Volume 3: Site Specific Sampling Plan
Ruetgers-Nease Salem, Ohio Site

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Submitted to

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1.0 INTRODUCTION

This Site Specific Sampling Plan (SSSP) Work Plan Volume 3 is one of four Work Plan volumes developed pursuant to and in accordance with the provisions of a Consent Order with an effective date of February 26, 1988 between Ruetgers-Nease Chemical Company (Ruetgers-Nease), the United States Environmental Protection Agency (U.S. EPA) and Ohio Environmental Protection Agency (OEPA). The Consent Order requires that a Remedial Investigation/Feasibility Study be conducted at the Ruetgers-Nease site in Salem, Ohio (the Site). This section of the SSSP describes the purpose of the SSSP and provides background information on Site conditions.

1.1 Plan Purpose

The project objectives described in this section define the purpose of the Remedial Investigation/Feasibility Study (RI/FS) being conducted. The objectives of the RI/FS are to gather data of adequate technical content, quality and quantity to:

- Determine fully the fact, nature, extent and magnitude of contamination on and off the Site.
- Determine if contaminants relating to the Ruetgers-Nease Site pose a threat to human health or the environment through the development of an Endangerment Assessment.
- Fully identify and characterize the source, migration pathways, routes of entry and receptors for contaminants.

- Support the identification, development and evaluation of remedial alternatives during an FS, an Endangerment Assessment, remedial technology screening, alternative development and screening, and detailed alternative evaluation.

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

The SSSP is organized into the following sections:

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 DECONTAMINATION

Appendix A - Sampling and Field Testing Procedures

Appendix B - Aquatic Biota Investigation

The overall sequence of RI/FS activities planned for the Site are discussed in the RI Work Plan (Volume 1). Analytical methods and Quality Control/Quality Assurance (QA/QC) procedures are provided in the Quality Assurance Project Plan (QAPP Volume 2). The remainder of this section of the SSSP provides a brief history and background data for the Site and study area.

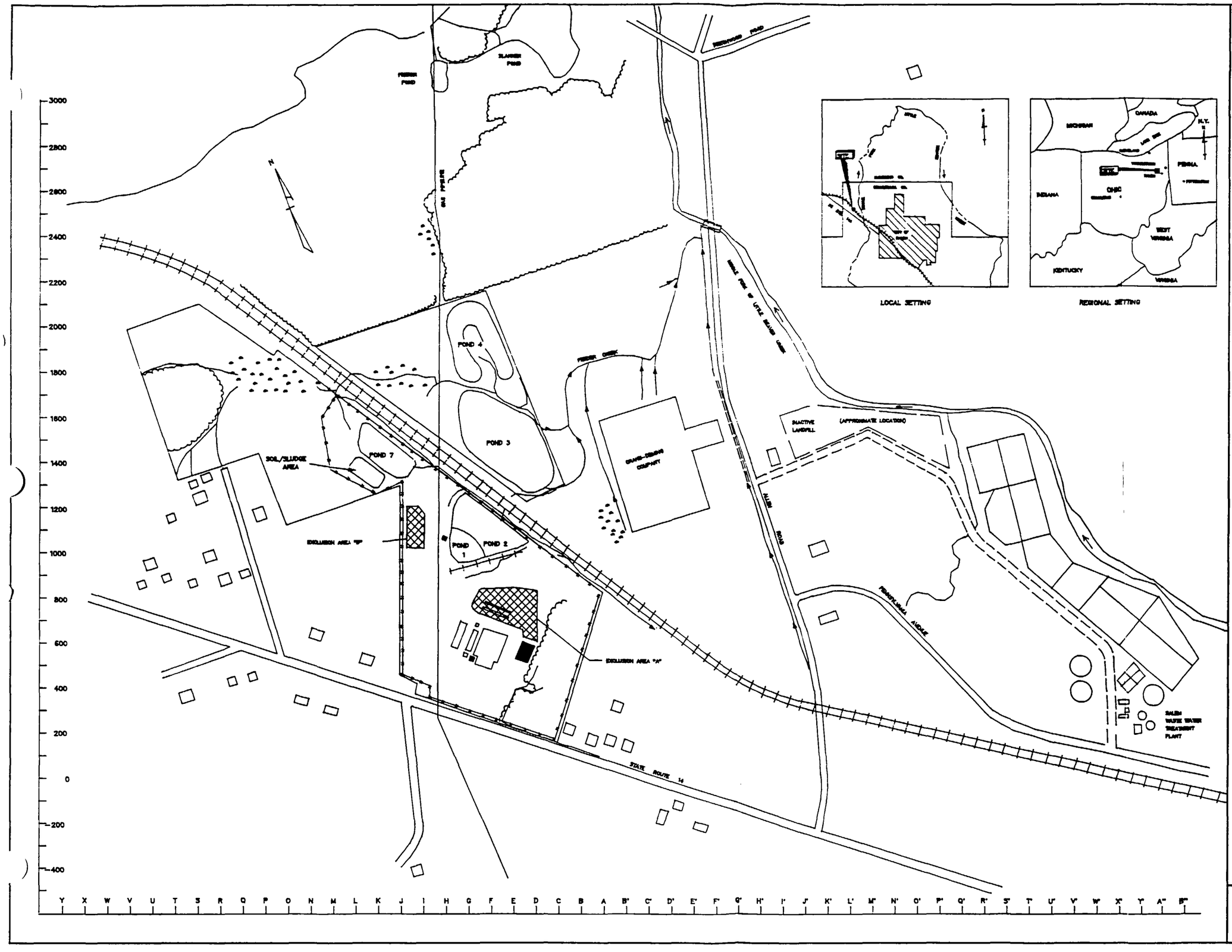
1.2 Background and History

Site background information presented in this section is necessary to understand the rationale behind the RI approach and the sample collection methods described in this SSSP.

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 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 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.



LOCAL SETTING

REGIONAL SETTING

LEGEND

- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- SITE BOUNDARY

FIGURE SSSP 1 – 1

SITE LOCATION MAP

RUETGERS-NEASE SALEM SITE RI/FS

SCALE

0 200 400 600 (FEET)

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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.

Prior to drift deposition, glacial forces eroded a valley into the sedimentary rocks east of the Site. This eroded valley was filled as 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 apparently is moving in the general direction of 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

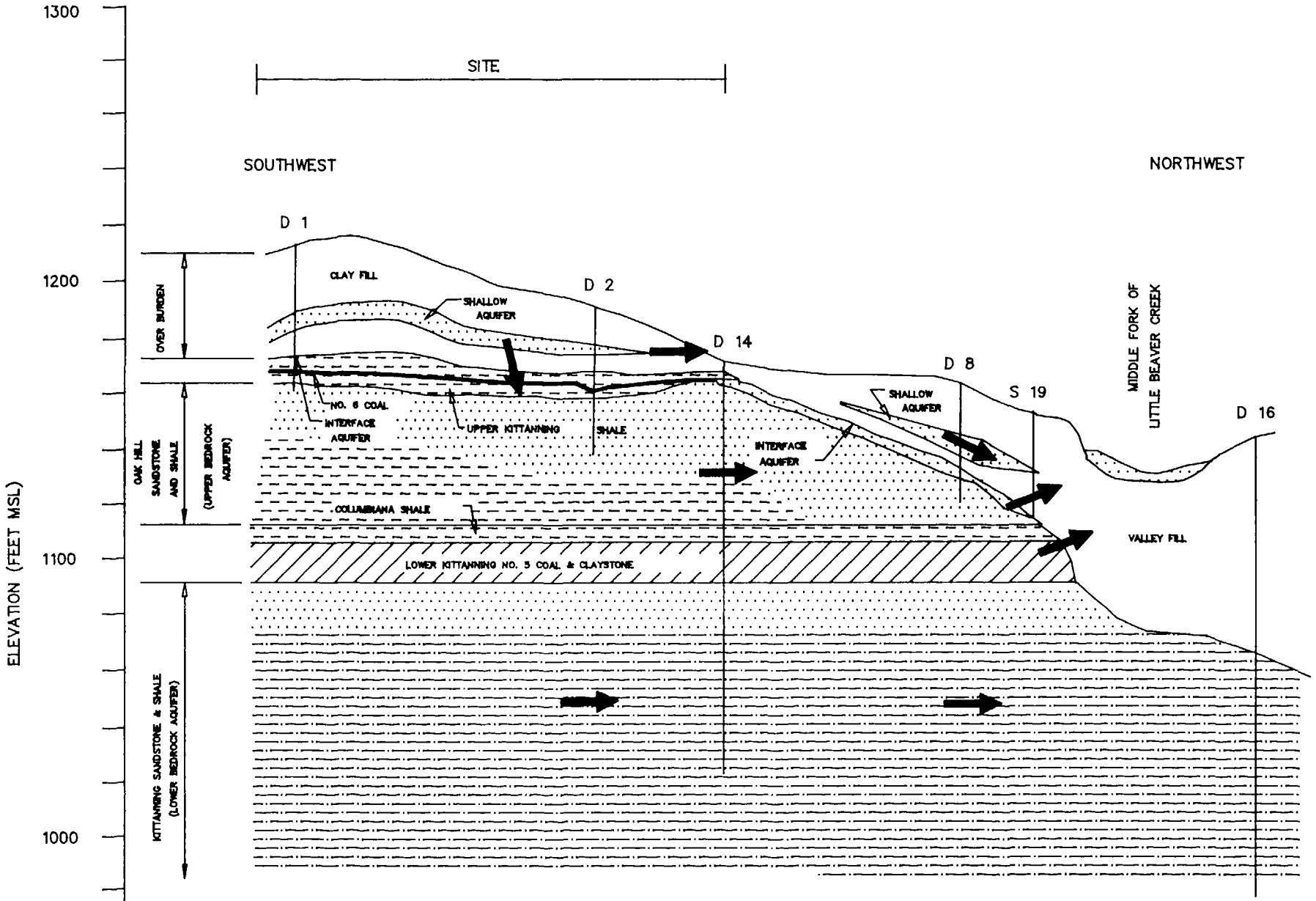

FIGURE SSSP 1 – 2

STRATIGRAPHIC AND HYDROGEOLOGIC

CROSS SECTION

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DIRECTION OF GROUND WATER FLOW

RUETGERS–NEASE SALEM SITE RI/FS

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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.

Since 1982, various environmental investigations and remedial actions have been conducted by Nease at the Site. The objectives of these studies were to define hydrogeological conditions, identify potential migration of contaminants, and evaluate remedies. In 1983, the Site was placed on the National Priorities List (NPL). Investigation activities conducted to date have included:

1. Soil borings and test pit excavations to characterize on-site contamination.

2. Soil borings through ponds to characterize contaminant concentrations and quantities.

3. Geophysical surveys at Exclusion Areas A and B to locate drum burial areas.

4. Sediment and surface water sampling and analysis to identify contaminant migration and extent.

Feeder Creek, the Crane-Deming Swamp, Slanker Pond, and MFLBC surface water and sediments have been sampled. In addition, the U.S. EPA and the OEPA have sampled fish from MFLBC and Slanker Pond.

5. Air monitoring to evaluate on-site and off-site atmospheric releases.

6. Installing and sampling monitoring wells and sampling nearby residential wells. At present, there are 38 monitoring wells at the Site, including:

   - o Fourteen Shallow Aquifer wells.
   - o Six Interface Aquifer wells.
   - o Fourteen Upper Bedrock wells.
   - o Four Lower Bedrock wells.

Three of these wells are installed into or through the Valley Fill.

7. Conducting a Risk Assessment in 1986.

8. Completing a site grid system based on two sets of perpendicular lines with adjacent lines 100 feet apart.

Table 1-1 lists compounds qualitatively identified at the Site. Most analyses were targeted to volatile and other organic compounds handled on-site. Several priority pollutant scans have been completed.

SSSP TABLE 1-1

COMPOUNDS THAT HAVE BEEN QUALITATIVELY IDENTIFIED
AT THE RUETGERS-NEASE SALEM SITE PRIOR TO THE RI/FS

Compounds	Range of MDLs Solid	Range of MDLs Aqueous
1,1-Dichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Chloroform	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,1,1-Trichloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloropropane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,3-Dichloropropene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Trichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Benzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Tetrachloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,1,2,2-Tetrachloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Toluene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Chlorobenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Ethylbenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
o,m,p-Xylene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,3+1,2-Dichlorobenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Methoxychlor	50 ug/kg[2,3]	0.005 ug/L[2]-0.05 ug/L[3]
Mirex	50 ug/kg[2,3]	0.005 ug/L[2]-0.05 ug/L[3]
3,4-Dichloronitrobenzene	500 ug/kg[3]-1000 ug/kg[1]	50 ug/L[3]-200 ug/L[1]
Diphenyl Sulfone	500 ug/kg[3]-1,000 ug/kg[1]	50 ug/L[3]-200 ug/L[1]

NOTES

MDL - Method Detection Limit

[1]By GC/FID

[2]By GC/ECD

[3]By GC/MS(SIM)

Actions taken to date include:

1. Removing 115 buried drums from Exclusion Area A.

2. Excavating and removing contaminated soils including approximately:

   - 5,400 $yd.^3$ from Exclusion Area A.
   - 684 $yd.^3$ from Exclusion Area B.
   - 2,790 $yd.^3$ from Pond 1.
   - 630 $yd.^3$ from the ditch paralleling the south side of the railroad tracks.

3. Seeding of Pond 2.

4. Installing geotextile fabric barriers and rock dams across drainage swales and ditches.

5. Installing hay-bale barriers around the exclusion areas.

6. Completing a leachate collection system between the railroad tracks and Exclusion Area A. Leachate is collected on a regular basis and disposed of at a permitted off-site wastewater treatment facility.

7. Installed a fence surrounding the western portion of the Site.

These actions were taken by Ruetgers-Nease.

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 Pond 3, 4 and 7 consist of weeds, grasses, small shrubs and some small trees. A soil/sludge pile west of Pond 1 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. This 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 1-2. The actual contribution of sources to affected media have not been qualified or quantified. The complete set of contaminants migrating from these sources has not been confirmed by samples meeting U.S. EPA 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 1-3.

1.3.3 Sampling Rationale

Sample locations and analysis have been identified that will provide data necessary to meet the objectives of the RI to complete EA activities. Sample type, locations, collection method, and sampling objectives are listed on Table 1-4. Scheduled analysis 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 development and validation study design and its completion will both be submitted for U.S. EPA and OEPA approval before investigation activities begin.

SSSP TABLE 1-2

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.

SSSP TABLE 1-3

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

SSSP TABLE 1-3 (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

SSSP TABLE 1-4

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

SSSP TABLE 1-4 (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	Soil borings, sampling and analysis of split spoon and shelby tube samples
		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.	
4.	Characteristics of Native Pond materials (soils under the pond bottom).	1.	Pond contaminant distribution - vertical horizonal	Soil borings, sampling and analysis of split spoon and shelby tube samples
		2.	Native material physical characterization	
		3.	Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment	

SSSP TABLE 1-4 (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	Soil borings, sampling and analysis of split spoon or auger samples
		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.	
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

SSSP TABLE 1-4 (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

SSSP TABLE 1-4 (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	Sampling and analysis of monitoring and residential wells
		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.	
9.	Air monitoring station upwind and downwind of the site	1.	Define areal extent of contaminant concentrations	Sampling and analysis of 6 stations
		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.	

SSSP TABLE 1-4 (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

SSSr Table 1-5

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 componsite (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.

2.0 FIELD ACTIVITIES SUMMARY

The RI will include field sample collection and subsequent physical and chemical analysis. This section of the SSSP summarizes work that will be conducted during the RI. Figure 2-1 shows the planned phasing of tasks that will be completed.

2.1 Preliminary Activities

Preliminary field activities will include:

1. Coordinating arrangements with RI subcontractors and investigation personnel.

2. Confirming access approvals (and permits if required).

3. Staging equipment to the Site.

4. Conducting an on-site orientation meeting with all subcontractor and project staff.

5. Completing a site reconnaissance and initial walk-through air monitoring survey.

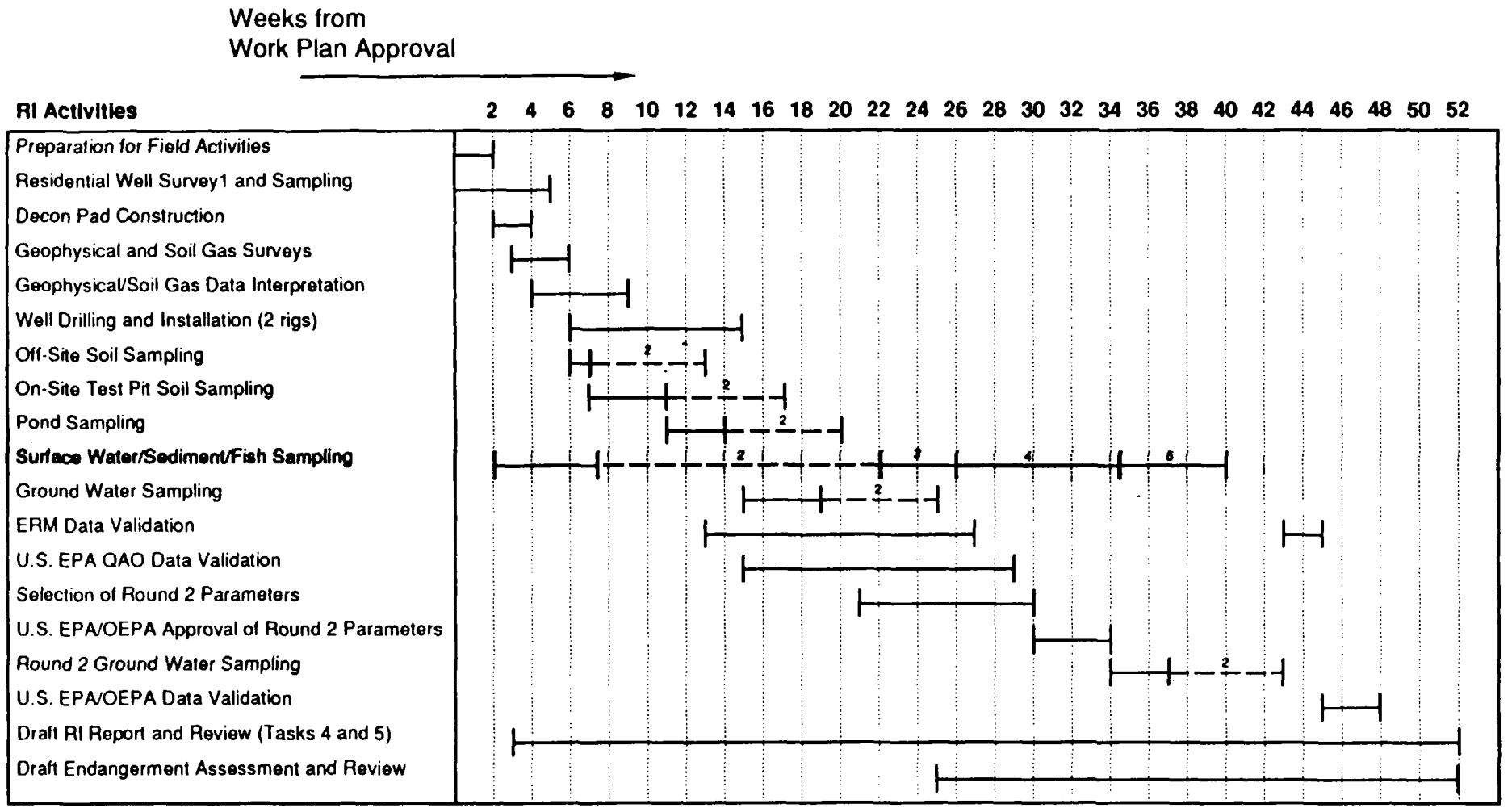
6. Establishing site exclusion zones, contaminant reduction zones, and the support zones previously identified in the Health and Safety Plan.

7. Constructing the decontamination pad and area.

8. Completing the survey of residential wells.

FIGURE SSSP 2-1

Schedule for Implementation of RI Activities at the Ruetgers-Nease Salem Site RI/FS



1. 1/2 mile radius confirmation of 1 mile radius search, residential well sampling
2. Laboratory analysis time
3. ERM data validation for surface water/sediment/fish sample analyses
4. U.S. EPA QAO data validation for surface water/sediment/fish sample analyses
5. Preparation of technical memorandum concerning surface water/sediment/fish sample analytical results

NOTE: Start-up and completion dates may change depending on field conditions

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9. Obtaining permits required for work to be completed during the investigation.

During the residential well survey, water user records within a radius of one mile from the Site will be obtained from the local water supply utility and Ohio DNR. The Township will be contacted and tax maps reviewed to obtain locations and addresses for all homes, residences, and businesses within that same area. Those homes, residences and businesses in the area which do not have active water utility accounts will be identified as possible ground water users. Ground water users within a one half mile radius from the Site will be confirmed by direct contact (e.g., by return of survey forms, phone calls, or by visits). Within the one half mile radius, five wells will be selected for sampling. In addition, the flowing well at the Salem Country Club will be sampled. The sampling of these wells will be conducted as soon as possible after the completion of the well inventory and Work Plan approval.

2.2 Field Investigation Activities

After completion of the preliminary activities (and Work Plan approval) field efforts will begin. Tasks to be completed include:

1. Air monitoring investigation.

2. Conducting geophysical surveys, including electromagnetic conductivity, seismic, and soil gas surveys.

3. Evaluating geophysical data.

4. Drilling and installing approximately 21 individual wells and an estimated 15 wells in four clusters.

5. Sampling 30 existing and all new monitoring wells, plus six off-site private wells.

6. Collecting soil samples from 30 test pits and from test pits along the railroad tracks.

7. Collecting samples of 3 foot cores from 14 soil borings for chemical analysis through five waste ponds and the soil/sludge area west of Pond 7.

8. Collecting a series of three foot core samples to bedrock for physical analysis at one location through Ponds 2, 3, 4 and 7.

9. Collecting sediment samples and surface water samples at Slanker Pond, Feeder Creek, and the MFLBC.

10. Collecting fish samples from Slanker Pond and the MFLBC.

11. Collecting surface and subsurface soil samples at 11 locations off-site.

12. Collecting sludge samples from the Salem wastewater treatment plant.

13. Aquifer testing of at least 16 monitoring wells, and collecting water level elevations from all monitoring wells.

As planned, these activities will be conducted according to the sequence shown in Figure 2-1.

2.3 Sampling and Analysis Program

Table 1-5 summarizes the samples that will be collected from each media and location, and identifies the analysis that will be completed for the samples. All TCL analysis will follow CLP procedures. Analysis for mirex, kepone, photomirex, DCNB and DPS will follow procedures developed and validated as documented in the QAPP (Volume 2) and approved by U.S. EPA Region 5 and OEPA. The analytical program is discussed in detail in the QAPP (Volume 2).

After analysis and quality assurance of all chemical results, and validation of data by ERM (and subsequently by U.S. EPA Region 5's Quality Assurance Office (QAO), if desired) parameters for the second round of ground water sampling may be reduced to those parameters found above background levels in the first sampling round, if no anomalies are observed in the sampling and analysis of the first round of sampling. This list of parameters for the second round of sampling will be submitted to the U.S. EPA and OEPA for approval.

The need, locations, and parameters for additional samples of other media to be collected will be reviewed pursuant to the Additional Work provisions of Paragraph XIII of the Consent Order. 3,4-DCNB, dioxins and furans, and CLP inorganics may be analyzed if they are found above background levels in samples from the selected sampling stations detailed in the CO-SOW. These will be submitted to the U.S. EPA and the OEPA for approval. In addition, U.S. EPA and OEPA may request additional sampling and analysis pursuant to the Additional Work provisions of paragraph XIII of the Consent Order.

2.4 Project Organization and Responsibility

Figure 2-2 is an organizational chart illustrating the structure for field activities. Specific responsibilities of personnel are described below.

2.4.1 Project Coordinator

Steven Foard, P.E. of Ruetgers-Nease is the Project Coordinator for this investigation. The alternate Project Coordinator is Brian Greene of Ruetgers-Nease. The responsibilities of the Project Coordinator include:

1. Providing an interface with the U.S. EPA RPM, the OEPA Project Coordinator, the Principal-in-Charge, and the Project Manager.

2. Approving on-site activities.

3. Initiating modification requests.

4. Ensuring that the terms of the Consent Order and SOW are met.

2.4.2 Principal-In-Charge

David E. Johe of ERM-Midwest is the Principal-In-Charge for this investigation. The responsibilities of the Principal-In-Charge include:

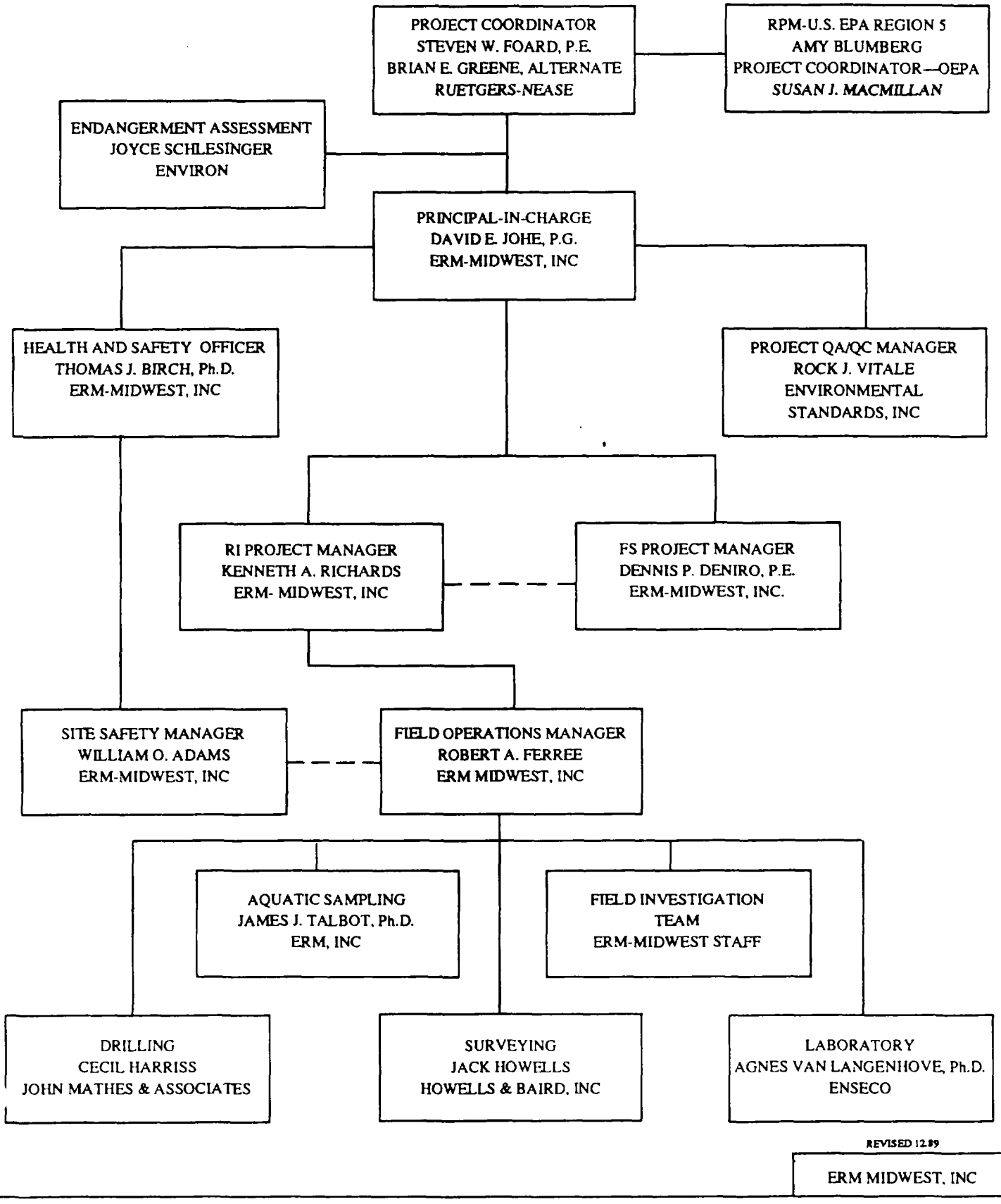
1. Providing an interface with the Project Coordinator and the Project Manager.

2. Committing ERM-Midwest resources to performing for the Site investigation.

3. Coordinating technical direction of the project.

FIGURE SSSP 2-2

FIELD INVESTIGATION ORGANIZATION
RUETGERS-NEASE SALEM SITE RI/FS



2.4.3 Project Manager

Kenneth A. Richards of ERM-Midwest is the Project Manager for the remedial investigation. The responsibilities of the Project Manager include:

1. Providing an interface between the Project Coordinator and the Site 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.

2.4.4 Field Operations Manager

The Field Operations Manager for this investigation will be Robert A. Ferree, an experienced member of the ERM-Midwest staff. The responsibilities of the Field Operations Manager include:

1. Managing field operations.
2. Reviewing and evaluating field data.
3. Implementing SSSP, HSP, and QAPP protocols.
4. Enforcing safety procedures.

2.4.5 Health and Safety Officer

Thomas J. Birch of ERM-Midwest will be the Health and Safety Officer. The responsibilities of the Health and Safety Officer include:

1. Selecting proper clothing and equipment to ensure the safety of on-site personnel.

2. Confirming each field team member's suitability for work based on a physician's recommendation.

3. Monitoring on-site hazards and conditions.

4. Monitoring the effectiveness of the Health and Safety Plan.

2.4.6 Project QA/QC Manager

Rock J. Vitale of Environmental Standards, Inc. is the Project QA/QC Manager for this investigation. He will be responsible for assuring that field, office, and laboratory activities and analyses are conducted in accordance with the QAPP. Specific responsibilities include the following:

1. Conduct performance and system audits.

2. Review all documents with respect to adherence to QA/QC procedures provided in the QAPP.

3. Review SAS data and RAS data including mass spectral library searches for tentative identifications.

4. Preparation of analytical data tables and quality assurance reviews.

5. Recommend and institute corrective actions based on reviews and audits.

2.4.7 Site Safety Manager

The Site Safety Manager will be William O. Adams of ERM-Midwest or his designee. 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:

1. Conducting ambient air monitoring.

2. Assuring all equipment and clothing availability at the Site.

3. Completing project safety briefings and reports.

4. Investigating accidents and implementing appropriate corrective actions.

5. Conducting daily safety briefing.

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

Amy Blumberg and Susan MacMillan are the Remedial Project Manager and Project Coordinator respectively for U.S. EPA Region 5 and the OEPA for this RI/FS. Their responsibilities include:

1. Technical review and approval of all plans and data submitted as part of this RI/FS.

2. Coordination of RI/FS 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 RI/FS Work Plans, 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.

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

Valerie Jones is the U.S. EPA/OEPA QA/QC Manager for this investigation. She is 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 EPA Region V CRL. Although neither EPA Region V or OEPA are responsible for data validation, both agencies reserve the right to perform data validation on any of the data generated of this project, within the time frame presented on the schedule in Figure 2-1.

3.0 FIELD INVESTIGATION ACTIVITIES

This section describes in detail the RI field investigation activities. Detailed instructions for sample collection and field testing are discussed in Appendix A and B.

3.1 Air Monitoring

In order to determine the nature, extent, and magnitude of potential contaminants present in the air pathways, an air monitoring program will be completed during the RI that will consist of a site reconnaissance survey, and the collection of ambient air samples across the study area. The first task to be performed during this survey will consist of the Site reconnaissance survey. Once this is completed, sampling for volatile and semivolatile compounds using Tenax, carbon molecular sieve, and XAD-2 adsorption tubes, respectively, will be conducted. Diphenyl sulfone and 3,4 dichloronitrobenzene (which may also be present at the Site) should also adsorb onto the XAD-2 resin. Organochlorine pesticides such as mirex, kepone, and photomirex, as well as PCBs, will be collected upon polyurethane foam (PUF) adsorbent utilizing modified hi-volume air particulate samplers.

3.1.1 Site Reconnaissance

A survey of the Site using flame ionization (FID) and photoionization (PID) detectors will be completed prior to the initiation of any field work. The survey will define specific work zone boundaries identified in the Health and Safety Plan, and identify areas with elevated levels of volatile organic compounds that may also require inclusion as an exclusion area. Measurements of VOCs at ground surface and three feet above ground surface using the PID

and FID will be made at 100-foot intervals along the Site boundaries, around each pond at stations every 50 feet, and at several locations within the proposed clean support zone. The areas between discrete sampling points will be walked with the instrument operating in order to detect any additional elevated levels of volatile compounds. The work and exclusion zones around each test pit excavation, boring or well drilling location will be screened during site work using a PID and/or FID to determine the proper health and safety protection. All measurements at discrete monitoring stations will be recorded in the field log book along with location, time and area weather conditions. If no elevated levels of volatiles are detected during the walk through between discrete monitoring stations using the organic vapor meter (FID) and/or photoionization instrument (PID), only the concentrations measured at discrete monitoring stations will be individually recorded in the field notebook.

3.1.1.1 Operation of the Century Organic Vapor Analyzer

In general, the procedures described in the owners manual will be utilized. Briefly they are as follows: The battery pack of the analyzer is charged for 16 hours before use. Prior to field utilization the instrument will be allowed to warm up for five minutes. The calibrate switch will be set to X1 (the most sensitive position) and the meter will be set to read zero. The pump is then turned on and the pump will be adjusted to between 1.5 to 2.5 units on the rotameter. The hydrogen tank value and the hydrogen supply value will be opened. The igniter button is then depressed after one minute. The OVA will be zeroed by pumping ambient air through activated charcoal and adjusting the reading instrument response to zero. The instrument will then be calibrated using a known standard such as benzene or methane in air, for example. This would be

accomplished on the X2 scale. For maximum sensitivity the calibrate switch is set to X1. To avoid false flame-out alarm indication, the meter will be set to 1 ppm and differential readings made from there. The instrument is now ready for field use. Once monitoring is complete the OVA will be shut down using the following procedure: Close the hydrogen supply valve and then the hydrogen tank valve. Move the instrument switch and pump switch to the off position.

3.1.1.2 Operation of the HNU Model P1-101 Photoionizer Instrument

In general the operation of the HNU instrument will be as described in the owners manual. Briefly these procedures are as follows. Charge the Instrument battery for 16 hours prior to field 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 approximately 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. The HNU is now ready for sampling. Simply turn the instrument to the off position once sampling is completed.

3.1.2 Air Sampling

Samples of ambient air will be collected to characterize ambient air quality, and identify contaminants potentially emanating from the Site and potentially impacting air quality in and around the study area. Results of the sampling will be used to establish potential hazards

to human health and welfare. Sample stations will be set up at the following locations:

1. Off-site upwind

2. Off-site downwind

3. Pond 1 downwind

4. Pond 2 downwind

5. Pond 7 downwind

6. Between the leachate collection system and railroad tracks.

Specific sample locations will be determined based on results of the reconnaissance survey and the prevailing wind directions at the time of sampling. All attempts will be made to collect the samples during warm, moderately calm conditions with a prevailing southwesterly wind.

Weather conditions (wind speed, wind direction, temperature, etc.) will also be obtained from a local weather bureau. Windrose data from the weather bureau will be used as a preliminary guideline to establish upwind and downwind sampling stations.

Portable sampling pumps, operated at low flow rates, will be used to pull air through Tenax adsorption tubes, carbon molecular sieve adsorption tubes, and XAD tubes equipped with particulate prefilters. These tubes will collect samples for volatile organics, highly volatile organics and semivolatile organics/pesticide analysis, respectively. The prefilters will collect particulates for pesticide analysis. Two sets of samples will be collected

at each location simultaneously to allow for backup analysis. Sampling intakes will be located no more than 3 feet and 5 to 6 feet above ground surface for on-site and off-site locations, respectively. Pumps and adsorption tubes will be shielded from wind and weather by a protective shelter, with the prefilter unit located outside the shelter. Upon removal, sample tubes/filters will be sealed and placed in cooled shipping containers for shipment to the laboratory. One replicate sample for analysis will be collected at the location identified as having the highest VOC level during the reconnaissance PID and FID survey. One trip blank comprised of an unopened Tenax and XAD tube will also be submitted for analysis.

3.1.2.1 Use of Tenax Traps for Collection of Volatile Organic Compounds

Ambient air will be drawn through a cartridge containing one to two grams of Tenax. Certain volatile organic compounds will be retained on the Tenax. Highly volatile organic compounds, such as vinyl chloride, will pass through the Tenax. The Tenax traps will be delivered to the laboratory (Enseco) for analysis.

The Tenax traps will be of either glass or metal construction (stainless steel). This is dependent upon the thermal desorption module which the analytical laboratory employs. Figure 1 in the TO1 Procedure in Appendix A depicts the two types of construction. EPA Method TO1 is entitled "Method for the Determination of Volatile Organic Compounds in Ambient Air Using Tenax Absorption and Gas Chromatography/Mass Spectrometry (GC/MS)." All methodology will be consistent with this procedure. All Tenax resin will be purified by the laboratory using a series of solvent extraction and thermal desorption steps as described in EPA Method TO1. All trap materials will be pre-cleaned by this

procedure. Approximately 0.5-1 cm glass wool plugs are placed in both ends of the trap with Tenax in between. The traps are then appropriately stored depending upon their construction; in a glass culture tube if glass is used and capped with stainless steel plugs if metal is used. Traps will be used within two weeks of preparation and analyzed within two weeks of use.

Each compound of interest has a characteristic retention volume (liters of air per gram of adsorbent) which must not be exceeded or breakthrough will occur. Since the retention volume is a function of temperature, and possibly other sampling variables, an adequate margin of safety to measure good collection efficiency must be maintained. Table 3-1 presents the estimated retention volume at 100°F in liters/gram for selected compounds. Data obtained from published literature values will be used to select the flow rate, the maximum flow rate, and the maximum total volume of air which may be sampled. Refer to page TO1-9 Method TO1 in Appendix A to this Site Specific Sampling Plan for the equations which will be utilized to determine the above parameters.

Collection of a known volume of air is critical to the accuracy of the results. Please refer to Figure 3, Method TO1, located in Appendix A, for two acceptable sampling systems. The sampling system using mass flow controllers (two parallel trains per pump instead of three as is in the figure) will be utilized since samples may be taken in parallel for additional quality assurance and simultaneously at different flow rates as an added insurance that the optimum sampling flow rate is being utilized. Two complete sampling systems (two pumps with four mass flow controllers) will be on-site; therefore, the sampling at all six monitoring stations (minimum) will not occur simultaneously. There are no significant advantages in sampling all six

SSSP TABLE 3-1

RETENTION VOLUME ESTIMATES FOR COMPOUNDS ON TENAX

COMPOUND	ESTIMATED RETENTION VOLUME AT 100°F (38°C)-LITERS/GRAM*
Benzene	19
Toluene	97
Ethyl Benzene	200
Xylene(s)	200
Cumene	440
n-Heptane	20
1-Heptene	40
Chloroform	8
Carbon Tetrachloride	8
1,2-Dichloroethane	10
1,1,1-Trichloroethane	6
Tetrachloroethylene	80
Trichloroethylene	20
1,2-Dichloropropane	30
1,3-Dichloropropane	90
Chlorobenzene	150
Bromoform	100
Ethylene Dibromide	60
Bromobenzene	300

* - Liters of air per grams of Tenax

sites at once as long as ambient conditions between sites are similar.

The system will be calibrated with respect to flow rate using a soap bubble flow meter; a single Tenax/CMS trap (which will not be analyzed) will be dedicated for flow rate calibration. During sampling the flow rate will be checked before and after sampling. The flow rate utilized will be approximately 50-200 ml/min per cartridge. The exact flow rate depends on the diameter of the trap because a specific flow velocity range must be maintained. (See TO1 methodology for flow rate determination in Appendix A, Section 10.1, "Flow Rate and Total Volume Selection.") An intermediate flow rate check of the reading on the monitor controller will be incorporated if the sampling time exceeds four hours. During sampling for volatiles, a particulate filter will not be placed ahead of the sampling tube since only the total concentrations of each volatile is desired.

Tenax/CMS traps and pumps will be protected from wind and weather by a shelter which will allow free transport of volatile and non-volatile organics from the soil. Tenax/CMS traps at on-site locations will be placed 3 feet above the ground. The two off-site sample locations (upwind and downwind) will have the Tenax/CMS traps 5 to 6 feet above the ground to better assess ambient breathing air quality. The pump will be started and the following parameters recorded on an appropriate data sheet: date, sampling location, time, ambient temperature, barometric pressure, relative humidity (obtained from the local weather station), flow rate, and Tenax trap number.

After completion of the sampling period, the above sampling variables will again be recorded on the data sheet. If the beginning and ending flow rates differ by more than

10% for any sample, the validity of this sample will be questioned. This may necessitate collecting these samples again.

The sampling traps will be removed, placed in labeled culture tubes (if glass) or capped (if stainless steel), and placed in a gallon friction top can containing a layer of charcoal. Appropriate chain of custody forms will be completed and accompany the shipment of samples. The sealed Tenax tubes will be placed in a cooled shipping container for shipment to the laboratory.

A trip blank and a field blank will be included for each day of sampling. The trip blank will be kept with all Tenax traps prior to sampling and will be placed in one of the shelters during sampling; however, the trip blank will not be opened during sampling. The trip blank and the field blank will be shipped to the laboratory with the day's samples. Two duplicate samples will be collected at one of the six stations and two samples in replicate will be collected at the other five stations, resulting in a total of 12 samples collected. Not all of the replicates will be analyzed. These replicates will verify QA/QC procedures and will provide additional samples for analysis if problems are encountered with thermal desorption of the Tenax traps. The following provides a further explanation of duplicate and replicate air samples.

A duplicate sample for volatiles in air will be collected on Tenax at one of the sampling locations and analyzed. The sampling apparatus which will be utilized is enumerated on pages SSSP-40, SSSP-42, and on page T01-35 in Appendix A. In short, two mass flow controllers in parallel will be connected to a pump which will be used to aspirate the air through the Tenax traps. Individual flow rates through the traps will be regulated by the mass flow meters.

For duplicate sampling, a Tenax trap will be connected to each mass flow controller of a sampling train. The times of sampling and the flow rates for the duplicate sampling will be identical; therefore, the volumes of air passed through the two traps will be the same. The traps will provide further quality control and verification of the laboratory analytical procedures.

To provide verification of the sample collection procedures, for the purposes of this discussion what will be called replicates will be obtained at the other five air sampling stations. The flow rates through each Tenax trap will be different for replicates; the time of sampling for each trap will, however, be identical. Collection of parallel samples at different flow rates (one sample flow rate should be lower than the selected flow rate for routine sampling) adds a measure of quality control. Agreement of results to within ± 25 percent verifies that the correct flow rate range has been selected and validates sampling procedures. If a trend of lower apparent concentration with increasing flow rate is observed for a series of replicate samples, it may be necessary to utilize a reduced flow rate and longer sampling interval. The replicates will be collected and shipped to the laboratory to verify correct selection of the flow rate. At the commencement of the sampling for each classification of compounds (volatiles, etc.), the replicates should be analyzed immediately for flow rate verification. Only those replicates necessary to verify correct flow rate will be analyzed; at this time we anticipate that only one replicate analysis will be necessary.

Both an organic vapor analyzer and a photoionization organic meter will be utilized to obtain appropriate organic chemical concentrations in ambient air. Based on this information, as well as the retention volumes on Tenax for

the volatiles to be collected, and the sampling flow rate to be used, a sample time will be selected to avoid sample breakthrough. Additionally, certain samples will have duplicate traps in tandem, that will be analyzed to provide proof that breakthrough did not occur. These samples will be collected at the commencement of the sampling project and will be analyzed as soon as possible to insure that the appropriate flow rates and sample times were selected.

Table 3-2 indicates which volatile compounds will be adsorbed onto the Tenax resins. The table also indicates which highly volatile compounds will be adsorbed onto carbon molecular sieves as described in the following section.

3.1.2.2 Use of Carbon Molecular Sieve Traps for Collection of Highly Volatile Organic Compounds in Ambient Air

Compounds such as vinyl chloride, benzene, toluene, and vinylidene chloride are not captured efficiently using Tenax. Therefore, EPA Method TO2 (see Appendix A for the complete methodology) is recommended for the sampling of these compounds. EPA Method TO2 is entitled "Method for the Determination of Volatile Organic Compounds in Ambient Air by Carbon Molecular Sieve Adsorption and Gas Chromatography/Mass Spectrometry (GC/MS)." The methodology utilized for adsorption of highly volatile compounds will be consistent with this methodology. Briefly, the methodology is as follows. In using this methodology, ambient air is drawn through a cartridge containing approximately 0.4 grams of a carbon molecular sieve (CMS) adsorbants such as 60/80 mesh-Sperocarb.

The cartridge design will be (unless otherwise specified) as presented in Figure 1 of EPA Method, TO2, in Appendix A of this document. The cartridge will be prepared

TABLE 3-2

Adsorption of Volatiles onto Tenax
and Carbon Molecular Sieves

	Adsorption Material	
Parameter	Tenax	CMS[a]
Chloromethane		X
Bromomethane		X
Vinyl chloride		X
Chloroethane		X
Methylene chloride		X
Acetone[b]	-[b]	-[b]
Carbon disulfide	X	
1,1-Dichloroethene	X	
1,1-Dichloroethane		X
1,2-Dichloroethene (total)	X	
Chloroform	X	
1,2-Dichloroethane		X
2-Butanone[b]	-[b]	-[b]
1,1,1-Trichloroethane		X
Carbon tetrachloride	X	
Vinyl acetate	X	
Bromodichloromethane	X	
1,2-Dichloropropane	X	
cis-1,3-Dichloropropene	X	
Trichloroethene	X	
Dibromochloromethane	X	
1,1,2-Trichloroethane	X	
Benzene		X
trans-1,3-Dichloropropene	X	
Bromoform	X	
4-Methyl-2-pentanone[b]	-[b]	-[b]
2-Hexanone[b]	-[b]	-[b]
Tetrachloroethene	X	X
Toluene		X
1,1,2,2-Tetrachloroethane	X	
Chlorobenzene	X	
Ethyl benzene	X	
Styrene	X	
Total xylenes	X	

[a] Carbon molecular sieve
[b] Compound is a ketone which will not require analysis by agreement with regulatory agencies because ketones do not readily adsorb on Tenax or CMS.

with approximately 0.4 grams of CMS with glass wool plugs at each end. The cartridges are conditioned for initial use by heating at 400°C for at least 16 hours with 100 ml/minute purge of ultra-pure nitrogen. Reused cartridges should be heated for four hours and a selected trap reanalyzed prior to use to ensure complete disorption of impurities. After heating, the cartridges are capped and placed in a metal friction top can containing charcoal. An unused cartridge from each set of conditioned cartridges will be analyzed prior to field sampling to document complete desorption.

Since vinyl chloride (which has a low retention volume) is one of the compounds of interest, the maximum allowable sampling volume is approximately 20 liters. The maximum allowable sampling flow rate will be determined using an appropriate equation referenced in Method TO2. It is estimated that a flow rate of between 50 ml/min and 200 ml/min will be dictated. The exact flow rate depends upon the retention volume of the least adsorbed compound of interest and upon the diameter of the sampling trap. See EPA method TO2, Section 10.1 "Flow Rate and Total Volume Selection," Appendix A for a discussion of this.

The same sampling apparatus will be used for the CMS sampling as was proposed for the Tenax trap sampling. Flow rate calibration will be the same for the CMS sampling as for the Tenax trap sampling. During sampling, no particulate filter will be utilized. The sampling procedure for the CMS will be the same as used for the Tenax trap as will be the sample preparation for shipment to the laboratory.

For quality assurance, at least one field blank and trip blank per day of sampling will be utilized. A duplicate sample for highly volatile compounds in air will be collected on CMS at one of the sampling locations and

analyzed. The sampling apparatus will be exactly the same as that used for Tenax sampling except CMS will be utilized as the adsorbent. In short, two mass flow controllers in parallel will be connected to a pump, which will be used to aspirate air through the CMS traps. Individual flow rates through the traps will be regulated by the mass flow meter. The times of sampling and the flow rates for the duplicate sampling will be identical; therefore, the volumes of air passed through the two traps will be the same. The traps will provide further quality control and verification of the laboratory analytical procedures.

To provide verification of the sample collection procedures, for the purposes of this discussion what will be called replicates will be obtained at the other five air sampling locations. For replicate sampling a CMS trap will be connected to each mass flow controller of a sampling train. The flow rates through each CMS trap will be different; the time of sampling for each trap will, however, be identical. Collection of parallel samples at different flow rates (one sample flow rate should be lower than the selected flow rate for routine sampling) adds a measure of quality control. Agreement of results to within ±25 percent verifies that the correct flow rate range has been selected and validates sampling procedures. If a trend of lower apparent concentration with increasing flow rate is observed for a series of replicate samples, it may be necessary to utilize a reduced flow rate and longer sampling interval. The replicates will be collected and shipped to the laboratory to verify correct selection of the flow rate. At the commencement of the sampling for each classification of compounds (volatiles, etc.), the replicates should be analyzed immediately for flow rate verification. Only those replicates necessary to verify correct flow rate will be analyzed; at this time we anticipate that only one replicate analysis will be necessary.

Two samples in duplicate will be collected at one of the stations and two samples in replicate will be collected at the other five stations, resulting in a total of 12 samples collected as described for the Tenax trap sampling. Both of the duplicates will be analyzed, but not all of the replicates will be analyzed. However, all replicates will be submitted to the laboratory as backup samples to be used in case a problem is encountered with the thermal desorption of a sample. At least one replicate sample will be analyzed by the laboratory as described in the preceding paragraph.

At the start of the carbon molecular sieve sampling, certain samples will also be collected with two sampling tubes in series. The back tubes will be analyzed to ensure that no breakthrough occurred. Also, certain of the parallel replicate samples will be run at lower flow rates in case breakthrough were to occur during the initial sampling. Coordinating the flow rate and sampling time, with a knowledge of retention volume of the various compounds of interest, should preclude breakthrough.

Table 3-2 indicates the highly volatile compounds which will be adsorbed onto the carbon molecular sieve. As the table indicates, certain compounds will be adsorbed both onto Tenax and onto carbon molecular sieves.

Dual tubes containing both Tenax (front half) and carbon molecular sieves (back half) will not be utilized in this program. Different flow rates and different sampling durations may have to be used due to differences in the retention volume of the compounds adsorbed by Tenax and by carbon molecular sieves.

3.1.2.3 Use of XAD-2 Traps for Collection of Semi-Volatile Organic Compounds in Ambient Air

XAD-2 resin is the preferred polymer for the sampling of higher boiling organic compounds such as semivolatile species. XAD-2 has better volumetric capacity and substantially greater (10X) weight capacity than Tenax-GC. Additionally, diphenyl sulfone and 3,4-dichloronitrobenzene should be adsorbed onto XAD-2.

Organic compound categories for which adsorption on XAD-2 is the recommended vapor sampling procedure are listed in Table 3-3. It should be noted that, while virtually all types of organic compounds are represented in this list, a number of these categories include some substances with boiling points below 100°C. The vapor sampling procedure is not expected to be effective for these substances whose high volatility places them in the categories of gases which are generally better adsorbed by Tenax. An evaluation of XAD-2 as an adsorbent for ambient air organic chemical sampling is detailed in the publication "EPA/IERL-RTP, Procedures for Level 2 Sampling and Analysis of Organic Materials, EPA-600-7-79-033, February, 1979".

The procedure utilized will be extremely similar to those detailed in the volatile organic adsorption section using Tenax; therefore, these procedures will not be repeated in this section. The principles will be the same with XAD-2 utilized in lieu of Tenax. Enseco will perform the method validation study for XAD-2 adsorption of the semivolatiles diphenyl sulfone and dichloronitrobenzene. Table 3-4 depicts the specific retention volumes of various compounds on XAD; data are also presented for Tenax.

The flow rate utilized will be between 50 and 200 ml/min. The exact flow rate will depend upon the retention

SSSP TABLE 3-3

COMPOUND CATEGORIES FOR WHICH ADSORPTION ON XAD-2 IS THE RECOMMENDED LEVEL 2 VAPOR SAMPLING METHOD

Category:
Aliphatic Hydrocarbons
Alkyl Halides
Ethers
Halogenated Ethers
Alcohols
Glycols, Epoxides
Aldehydes, Ketones
Amides, Esters
Nitriles
Amines
Nitrosamines
Sulfides, Disulfides
Sulfonic Acids, Sulfoxides
Benzenes
Halogenated Aromatics
Aromatic Nitro Compounds
Phenols
Halophenols
Nitrophenols
Polynuclear Aromatic Hydrocarbons
Heterocyclic N Compounds
Heterocyclic O Compounds
Heterocyclic S Compounds

SSSP TABLE 3-4

Specific Retention Volumes (Vg)* for Adsorbate Vapors on Sorbent Resins (20°C)

Adsorbate	Tenax-GC	XAD-2
n-Hexane	2.58×10^4	7.53×10^7
n-Octane	1.89×10^5	2.29×10^6
n-Decane	3.08×10^6	2.09×10^7
n-Dodecane	2.19×10^6	---
Benzene	6.09×10^4	5.24×10^4
Toluene	7.88×10^5	2.58×10^5
p-Xylene	3.81×10^5	9.05×10^5
Ethylbenzene	8.36×10^5	5.64×10^5
n-Propylbenzene	1.53×10^6	4.61×10^6
1,2-Dichloroethane	2.32×10^4	1.96×10^4
Fluorobenzene	8.82×10^4	3.13×10^4
1,1,2-trichloroethylene	8.82×10^4	3.06×10^4
Chlorobenzene	2.36×10^6	2.43×10^5
Bromobenzene	8.41×10^6	6.39×10^5
1,4-Dichlorobenzene	1.73×10^7	2.33×10^6
2-Butanone	2.21×10^4	4.39×10^3
2-Heptanone	5.55×10^6	1.49×10^6
4-Heptanone	3.22×10^6	1.52×10^6
Cyclohexanone	1.36×10^6	3.66×10^5
3-Methyl-2-butanone	6.46×10^4	2.53×10^4
3,3-Dimethyl-2-butanone	--	8.59×10^4
2,6-Dimethyl-4-heptanone	--	1.61×10^7
Acetophenone	1.23×10^7	7.70×10^6
n-Butylamine	2.67×10^4	1.80×10^4
n-Amylamine	1.96×10^5	1.29×10^5
n-Hexylamine	7.35×10^5	4.80×10^5
Benzylamine	1.58×10^6	7.87×10^6
Di-n-butylamine	1.91×10^6	6.90×10^6
Tri-n-butylamine	4.85×10^5	--

SSSP TABLE 3-4
(Cont'd)

Specific Retention Volumes (V_g)* for Adsorbate Vapors on Sorbent Resins (20°C)

Adsorbate	Tenax-GC	XAD-2
Ethanol	9.08×10^2	1.75×10^3
n-Propanol	5.71×10^3	9.31×10^3
n-Butanol	4.34×10^4	2.07×10^4
2-Butanol	1.86×10^4	2.04×10^4
2-Methyl-2-propanol	7.08×10^2	1.60×10^3
2-Methyl-1-propanol	2.88×10^4	1.28×10^4
Phenol	2.47×10^6	3.68×10^6
o-Cresol	1.00×10^7	1.33×10^7
p-Cresol	1.40×10^7	1.51×10^7
m-Cresol	1.18×10^7	1.55×10^7
Acetic Acid	3.20×10^3	7.07×10^3
Propionic Acid	1.73×10^4	4.00×10^4
n-Butanoic Acid	1.04×10^5	7.74×10^4
n-Pentanoic Acid	5.53×10^5	2.89×10^5

* In units of mL/g.

volume of the least adsorbed semivolatile compound, the total sampling time, and the diameter of the sampling tube containing the XAD-2 adsorbent.

A total of 12 samples (six samples, one duplicate, five replicates) will be collected; that is, two samples will be collected at each of the six stations. Not all of the five replicates will be analyzed, but all will be sent to the laboratory in case problems develop with the analysis of any particular sample. A duplicate sample for semivolatiles in air will be collected on XAD-2 at one of the sampling locations. The sampling apparatus will be exactly the same as that used for Tenax sampling except XAD-2 will be utilized as the adsorbent. In short, two mass flow controllers in parallel will be connected to a pump which will be used to aspirate air through the XAD-2 traps. Individual flow rates through the traps will be regulated by the mass flow meters. For duplicate sampling a XAD-2 trap will be connected to each mass flow controller of a sampling train. The times of sampling and the flow rates for the duplicate sampling will be identical; therefore, the volumes of air passed through the two traps will be the same. The traps will provide further quality control and verification of the laboratory analytical procedures.

To provide verification of the sample collection procedures, for the purposes of this discussion what will be called replicates will be obtained at the other five air sampling locations. The flow rates through each XAD-2 trap will be different for replicates; the time of sampling for each trap will, however, be identical. Collection of parallel samples at different flow rates (one sample flow rate should be lower than the selected flow rate for routine sampling) adds a measure of quality control. Agreement of results to within ±25 percent verifies that the correct flow rate range has been selected and validates sampling

procedures. If a trend of lower apparent concentration with increasing flow rate is observed for a series of replicate samples, it may be necessary to utilize a reduced flow rate and longer sampling interval. The replicates will be collected and shipped to the laboratory to verify correct selection of the flow rate. At the commencement of the sampling for each classification of compounds (volatiles, etc.), the replicates should be analyzed immediately for flow rate verification. Only those replicates necessary to verify correct flow rate will be analyzed; at this time we anticipate that only one replicate analysis will be necessary.

The possibility of breakthrough will be minimized by regulating the flow rate so as not to exceed the retention volume of the least adsorbed semivolatile compound. Additionally, tandem tube sampling (two XAD-2 tubes in series) will be utilized at the start of the sampling to ascertain if breakthrough is occurring. These will be shipped overnight to the laboratory for rush analysis.

Table 3-5 lists the semivolatile compounds of interest which will be adsorbed onto XAD-2. This is the target compound list for semivolatile organics.

3.1.2.4 Collection of Organochlorine Pesticides and PCBs in Ambient Air

Method TO4 will be utilized to collect particulate and vapor phase samples for organochlorine pesticides including mirex, kepone, and photomirex, as well as PCBs. Generally, detection limits in excess of 1 ng/m^3 are achievable using a 24-hour sampling period. A modified high volume sampler consisting of a glass fiber filter with a polyurethane foam (PUF) backup absorbent cartridge is used to sample ambient air at a rate of approximately 200-280 liters/minute. A

TABLE 3-5

Semi-Volatile Compounds
Which Will be Adsorbed Onto XAD-2

Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Benzyl alcohol
1,2-Dichlorobenzene
2-Methylphenol
bis(2-Chloroisopropyl)ether
4-Methylphenol
N-nitroso-di-n-dipropylamine
Hexachloroethane
Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Benzoic acid
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
Hexachlorobutadiene
4-Chloro-3-methylphenol
(para-chloro-meta-cresol)
2-Methylnaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2-Chloronaphthalene
2-Nitroaniline
Dimethylphthalate
Acenaphthylene
2,6-Dinitrotoluene
3-Nitroaniline
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran
2,4-Dinitrotoluene
Diethylphthalate
4-Chlorophenyl-phenyl ether
Fluorene
4-Nitroaniline
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylaminec
4-Bromophenyl-phenylether
Hexachlorobenzene
Pentachlorophenol
Phenanthrene
Anthracene
Di-n-butylphthalate
Fluoranthene
Pyrene
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Benzo(a)anthracene
Chrysene
bis(2-Ethylhexyl)phthalate
Di-n-octylphthalate
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene
Diphenyl sulfone
Dichloronitrobenzene

diagram of the sampling apparatus is provided in Figure 1, page TO4-17, Method TO4, located in Appendix A to this document. The procedures enumerated in this method will be adhered to in the sampling for organochlorine pesticides and PCBs.

A calibration of the venturi/magnehelic assembly will have already been conducted by the supplier of the PUF hi-volume air samplers. The sampler will be calibrated according to the following procedure:

1. Calibration of the PUF Sampler is performed without a foam slug or filter paper in the sampling module. However the empty glass cartridge must remain in the module to insure a good seal through the module.

2. Install the GMW-40 Calibrator on top of the 4" filter holder.

3. Connect an 8" water manometer to the Calibrator.

4. Open the ball valve fully.

5. Turn the system on by tripping the manual switch on the timer. Allow a few minutes for warm-up.

6. Adjust the voltage control screw to obtain a reading of 70 inches on the dial gage, (Magnehelic Gage).

7. With 70 inches on the dial gage as your first calibration point, record it and the manometer reading on the data sheet.

8. Close the ball valve slightly to readjust the dial gage down to 60 inches. Record this figure and manometer reading on the data sheet.

9. Using the above procedure, adjust the ball valve for readings at 50, 40, and 30 inches and record on the data sheet.

10. Using these two sets of readings, plot a curve on the data sheet. This curve will be used for determining the actual flow rate in the field.

11. Readjust the voltage control fully clockwise to its maximum setting. Open ball valve fully.

The PUF plugs will be obtained from a supplier such as General Metal works, Cleves, Ohio. Enseco will perform Soxhlet extraction upon these plugs prior to use. At least one assembled cartridge will be analyzed as a laboratory blank.

Two PUF hi-volume air samplers will be used for this project. The PUF hi-volume air samplers will be moved from one sampling location to another as is necessary. The on-site samples will be collected at a height of 3 feet above ground and off-site samples will be collected at a height of 5 to 6 feet. The samples will be located in an unobstructed area, at least two meters from any obstacle to air flow. The PUF plug and quartz fiber filter will be installed in the sampler, the hi-vol will be turned on, and the flow rate measured and adjusted if necessary. The ambient temperature, barometric pressure, sampler serial number, filter number and PUF cartridge number will be recorded. Temperature and pressure will be recorded. Temperature and pressure will also be recorded at the end of the 24-hour sampling period. The sampler will be monitored at six hour intervals during sampling. At the end of the sampling period, the filter and PUF cartridge will be wrapped in the original aluminum foil cleaned in the laboratory and placed in sealed, labeled containers. The container will be placed

in an ice chest at approximately 4°C and shipped to the laboratory. Appropriate chain of custody procedures will be utilized. Appropriate equations presented in Method T04 will be utilized to calculate the actual volume of air sampled and to convert to the volume sampled at standard conditions of temperature and pressure.

A total of eight samples using the PUF hi-volume air sampler will be obtained. There will be six samples (one from each of the six stations) and two duplicates. A duplicate sampler for organochlorine pesticides and PCBs in ambient air will be collocated in order to collect the duplicate samples. This sampling will occur at the two sites where the maximum concentrations (if any) would be expected to occur. The two samplers will be located approximately two meters apart to preclude air flow interference. One of the samplers will be identified as the sampler for the normal monitoring; the other will be identified as the duplicate sampler. The calibration, sampling, and analysis will be the same for the collocated sampler as for the other sampler. The exhaust hose for each sampler will point in a directions that will avoid biasing the results. The samples will operate simultaneously over a 24-hour period. The filter and foam will be sent back to the laboratory for analysis.

The sampling will be conducted according to EPA Method TO4 procedures using a recommended flow rate of 200-280 l/min. Because pesticide levels which may be present should be very minimal (if present at all), breakthrough is not anticipated.

3.1.2.5 Determination of Suspended Particulates in the Atmosphere (High Volume Method)

The objective of this sampling is to obtain particulate data which may be used in a health risk assessment study of

the site. The reference method for this sampling is presented in Appendix A and will be followed except that the intake to the hi-vol will be located at a height of 5 to 6 feet in order to assess particulates in the breathing zone. The method is entitled "Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)."

The following is a brief description of the methodology. Air is drawn into a covered housing and through a filter by means of a high flow rate blower at a flow rate of 40 to 60 ft^3/min that allows suspended particulate having a diameter of less than 100 microns to pass to the filter surface.

Particles within the size range of 100 to 0.1 micron diameter are ordinarily collected on the glass fiber filters. The mass concentration of suspended particulates in the ambient air (microgram/cubic meter) is calculated by measuring the mass of collected particulates and the volume of air sampled.

Each filter must be assigned a serial number. This serial number should be stamped on two diagonally opposite corners on opposite sides of the filters. Equilibrate the filters in a dessicator for a period of at least 24 hours prior to weighing. All filters must be weighed to the nearest tenth of a milligram. The filters should be weighed on a balance with a special filter tray, the clean filters must not be folded before being weighed. Before weighing the filter, perform a balance check by weighing a standard weight of 5 grams. Record the actual and measured weight, along with the data and operator's initials. If the actual and measured weight values differ by more than $\pm$.5 milligrams, do not proceed with weighing the filters. The balance must be checked before proceeding with filter

weighing. Record the tare weight and the serial number of each filter. Place the weighed filter in a folder to protect the filter from damage during transport to the sampling site.

Installation of a clean filter: Remove the face plate by loosening the four wing nuts and rotating the bolts outward. Place the filter rough-side up in the wire screen. EXTREME CARE WILL BE EXERCISED TO PREVENT DAMAGE OR DIRT SMUDGED ON THE CLEAN FILTER. Center the filter on the screen so that when the face plate is in position, the gasket will form an air-tight seal on the filter. Once the filter is aligned and the face plate is in place, the four wing nuts are tightened so that the gasket is air-tight against the filter. Also before the new filter is installed, the inside surface of the shelter should be cleaned of loose particles by wiping with a clean rag.

After the filter has been installed, make flow rate measurements while the sampler is at normal operating temperature. This requires a warm-up time of at least five minutes before a valid measurement can be obtained. Attach a rotameter to the sampler using the same tubing as was used to calibrate the sampler, place or hold the rotameter in vertical position at eye level. Read the widest part of the float. After connecting the rotameter to the sampler, observe the response for at least one minute before taking a reading. If a gradual change in flow rate is observed, do not take a reading until an equilibrium is reached. A gradual change will usually be observed when the rotameter is at a substantially different temperature from the sampler exhaust air, and may require two to three minutes to equilibrate. Set the timer for the correct time at each filter change. Record temperature, barometric pressure, filter number and initial flow rate. The hi-volume sampler then is allowed to operate from 12 noon to 12 noon (24-hour

period). After operation, before the filter is removed, make a flow rate measurement. Remove exposed filter from support screen, by grasping it at the ends (not at the corners) and lifting it from the screen. Fold the filter length-wise at the middle with the exposed sides in. Place the filter in the filter holder for transportation back to the laboratory. EXTREME CARE WILL AGAIN BE EXERCISED TO PREVENT DAMAGE OR DIRT SMUDGED ON THE FILTER. Then record the station number, the temperature and barometer pressure and the ending flow rate. Variation in flow rates during the sampling will be minimized by using Accu-vols which have flow controllers. A flow rate of approximately 40 CFM will be utilized.

The following briefly describes the sampling analysis procedure once the filters are returned to the laboratory. Exposed filters should be returned to the laboratory and placed in the dessicator the same day the samples are received by the laboratory. The filters should remain in the dessicators for 24 hours. The 24-hour equilibrium period should be adhered to for uniformity of results. EXTREME CARE WILL BE EXERCISED WHEN PLACING FILTER IN THE DESSICATOR TO MAKE SURE THAT THE FILTER DOES NOT COME IN CONTACT WITH LOOSE PARTICLES. Also, the filter should not be placed in the position such that some of the sample might fall or be knocked loose. The filter must be weighed immediately after removal from the dessicator. Weigh exposed filters to the nearest milligram. Record filter weights in the Laboratory Log book. At this point all documentation should be checked for completeness and accuracy. All data necessary for computing the concentrations must be recorded in the appropriate forms.

The following procedure briefly describes the rotameter calibration and the particulate concentration calculation. Assemble a high-volume sampler with a clean filter in place and run for at least five minutes. Attach a rotameter, read

the ball, adjust so that the ball reads 65, and seal the adjusting mechanism so that it cannot be changed easily. Shut off the motor, remove the filter, and attach the orifice calibration unit in its place. Operate the high-volume sampler at a series of different, but constant, airflows (usually six). Record the reading of the differential manometer on the orifice calibration unit, and record the readings of the rotameter at each flow. Measure atmospheric pressure and temperature. Convert the differential manometer reading to m^3/min., Q, then plot the rotameter reading versus Q. Calculate the air volume measured by the positive displacement primary standard.

$$Va = \frac{(Pa-Pm)}{Pa} (Vm)$$

Va = True air volume at atmospheric pressure, m^3
Pa = Barometric pressure, mm.Hg.
Pm = Pressure drop at inlet of primary standard, mm.Hg.
Vm = Volume measured by the primary standard, m^3

Conversion factors are as follows:

Inches Hg. x 25.4 = mm.Hg
Inches water x 73.48 x 10^{-3} = inches Hg.
Cubic feet air x 0.0284 = cubic meters air.

True air flow rate is as follows:

$$Q = \frac{Va}{T}$$

Q = flow rate, m^3/min.
T = time of flow, min.

Convert the initial and final rotameter readings to a true air flow rate, Q, using the calibration curve. Calculate the volume of air sampled using the following equation.

$$V = \frac{(Qi + Qt)}{2} \times T$$

V = Air volume sampled, m^3
Qi = Initial airflow rate, m^3/min.
Qt = Final airflow rate, m^3/min.
T = Sampling time, min.

Calculate mass concentration of suspended particulates by:

$$SP = \frac{(Wf - Wi) \times 10^6}{V}$$

SP = Mass concentration of suspended particulates ug/m^3
Wi = Initial weight of filter, g.
Wf = Final weight of filter, g.
V = Air volume sampled, m^3
10^6 = conversion of g to ug

Two hi-volume air samplers will be utilized to measure particulate at the site. The exact sampling locations will be determined prior to sampling. Representative areas of the site will be sampled; that is, the sampling will attempt to not be biased toward worst or best case scenarios. The primary goal of this portion of the sampling is to provide particulate data for the health risk assessment. Particulate matter in the atmosphere is much more area dependent than point source dependent as is the case with volatile compounds.

One upwind off-site and one downwind off-site sample will be collected. The upwind off-site sampling location will represent background. Two on-site samples will be collected, at least one of which will be near the downwind boundary line.

No duplicate samples will be collected. Because the sampling will be conducted according to EPA flow rates and using approved equipment, and because the particulate matter is collected on a glass fiber filter, no breakthrough will occur.

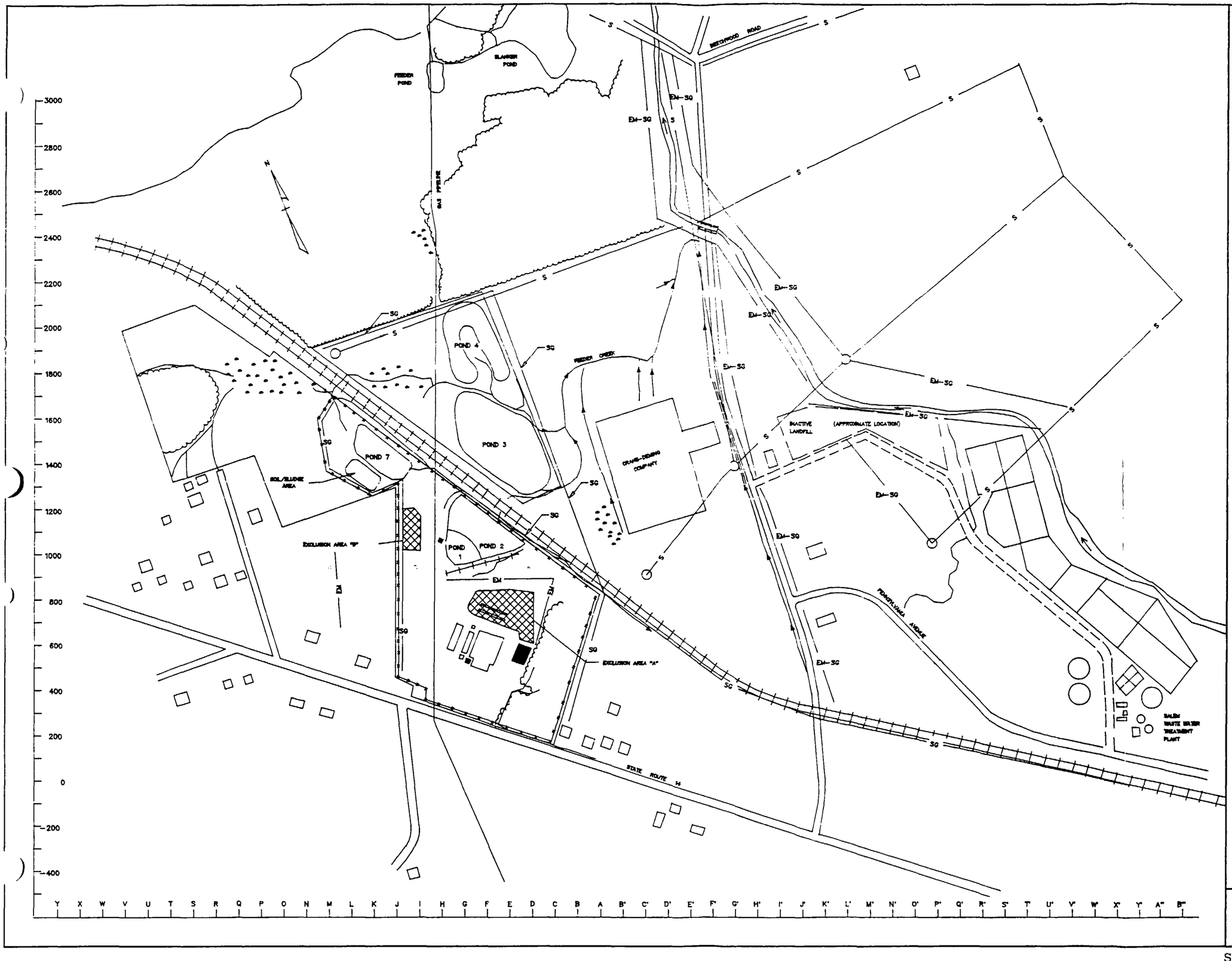
3.2 Geophysical Investigations and Soil Gas Survey

Combinations of conductivity and seismic geophysical methods will be used to investigate potential inorganic contamination, and to characterize the sub-surface geology of the Valley. Specific geologic objectives of the seismic survey include a delineation of the Valley Fill unit, and of the subsurface depth of the fill/bedrock interface.

Along with yielding sub-surface data, the results of the conductivity surveys may be useful in determining the occurrence of contaminants (if present) within groundwater. A soil gas survey will be performed concurrent with the geophysical surveys to delineate the presence or absence of organic vapors in the unsaturated soil pore spaces. Geophysical and soil gas surveys will be conducted along the transects shown in Figure 3-1.

3.2.1 Conductivity Surveys

Conductivity measurements will be taken using two types of equipment depending on the survey location:



LEGEND

- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- S — SEISMIC SURVEY LINE
- SG — SOIL GAS SURVEY LINE
- EM — ELECTROMAGNETIC CONDUCTIVITY SURVEY LINE
- SITE BOUNDARY

FIGURE SSSP 3 – 1

GEOPHYSICAL SURVEY LOCATIONS

RUETGERS-NEASE SALEM SITE RI/FS

SCALE

0 200 400 600 (FEET)

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1. A Geonics EM34-3 unit will be operated in the horizontal dipole mode at off-site transect lines east of the Crane-Deming facility. The horizontal dipole mode of the instrument is less sensitive to outside electromagnetic interferences than the vertical dipole mode and so should be a more efficient method of collecting representative conductivity data. Data will be collected at 100 foot intervals. The data will be compiled either in a field log book or on a magnetic tape polycorder. Either method will effectively store the raw field data for later reduction and compilation into a report. A printed copy of the original field data will be available with either method.

   The EM34-3 unit is equipped with three intercoil spacings. They are 10, 20, and 40 meters. Operated in the horizontal dipole mode, the signal generated at these intercoil spacings will detect conductivity contrasts to depths of approximately 25 feet (7.5m), 55 feet (16m), and 100 feet (30m), respectively.

   The 10 and 20 meter intercoil spacings will be used for those areas of the survey in which the overburden thickness (as historically defined for the site) is less than 55 feet. In those areas of the survey where the overburden thickness is greater than 55 feet or when the on-site geologist does not feel that the conductivity data adequately defines the overburden/bedrock interface, additional data will be collected using the 40 meter intercoil spacing.

2. A Geonics EM31-DL unit will be used on-site along the two lines located north and east of Exclusion Area "A". The unit will be used to detect a potential shallow conductivity contrast near that area. The EM31-DL unit uses a fixed intercoil spacing of 3.7 meters. The depth of investigation using this instrument will be approximately 10 feet in the horizontal dipole mode and 20 feet in the vertical dipole mode. Measurements will be taken continuously along the two transect lines. Data will be recorded using a magnetic tape polycorder or analog recorder. Data from both the horizontal and vertical dipole modes of the instrument can be stored simultaneously on either recording device.

   Background conductivity values for the EM-31 and EM-34 will be obtained in the open field west of Exclusion Area "B." The background survey will be completed before conducting surveys at other locations. Equipment operating procedures are described in Appendix A.

3.2.2 Seismic Surveys

The seismic survey is designed to investigate the subsurface overburden and bedrock stratigraphy across the study area. Seismic refraction data from six transect lines totaling approximately 12,000 linear feet will be collected and analyzed to determine the depth to bedrock and Valley Fill geometry in the vicinity of the Site (see Figure 3-1).

Seismic data will be collected using an ABEM 12-channel signal enhancement "Terraloc" seismograph (or equivalent). The line geometry will consist of 25 foot geophone spacings which will be variably displaced along a direct line from the shot source. The total proposed spread length of the

geophone array will be 575 feet. The proposed line geometry includes a 125 foot overlap (the equivalent of six geophones) at each end of each seismic line. Such a line geometry will duplicate 50 percent of the data on each line, thereby enhancing the accuracy of the data.

The seismic energy source will be a 12-gauge shot gun. Should the need to increase depth of penetration or data quality arise, a larger energy source, such as small explosive charges placed in shallow drill holes will be utilized. If explosives are necessary, a local blaster will be subcontracted to provide these services. The small charges used in this type of work do not create excessive noise and cause minimal surface damage. Signs will be posted and all shots will be detonated while under visual contact.

The survey will collect refraction data which will provide sufficient delineation of the overburden thickness, and definition of the bedrock/overburden interface up to a depth of 200 feet. Based on the available information, it appears that use of seismic reflection techniques will not be feasible for the purposes of this study due to the anticipated shallow depth of rock (estimated to be 30 to 100 feet deep), and the lack of a near-surface water table. Seismic reflection techniques are generally more successful for mapping reflectors at depths over 100 feet. However, a seismic reflection test will be run at the site to evaluate this method further. The identical equipment would be used for a reflection survey, but a revised geophone/shot geometry would utilized.

Reflection data may be collected during this survey under the following conditions:

1. A velocity inversion layer (i.e., a higher velocity layer immediately overlying a lower velocity layer)

2. An extremely deep occurrence of bedrock (below 250 feet)

3. The presence of a boulder field or other extremely unconsolidated layer in the overburden material

The seismic and monitor well pilot boring data will be used to produce cross sections parallel and perpendicular to the buried valley.

3.2.3 Soil Gas

A soil gas survey will be completed along the transects marked "SG" shown on Figure 3-1. Measurements of total organic concentrations will be recorded at 100 foot intervals using an FID to measure total volatile organic concentrations.

A KV Associates, Inc. (KV) soil gas system will be utilized to conduct the soil gas survey. At each measurement point, a KV hammerdrill will be used to drive a stainless steel sampling probe approximately three feet into the soil. The soil surrounding the probe should effectively "seal" the probe in the ground. Following probe installation, a section of tygon tubing will be connected to the top of the probe, and to a Foxboro Model 128 Organic Vapor Analyzer (OVA).

The self-contained pump within the OVA will be utilized to purge the system by evacuating approximately three volumes of gas. Upon completion of purging, a stabilized OVA reading will be recorded.

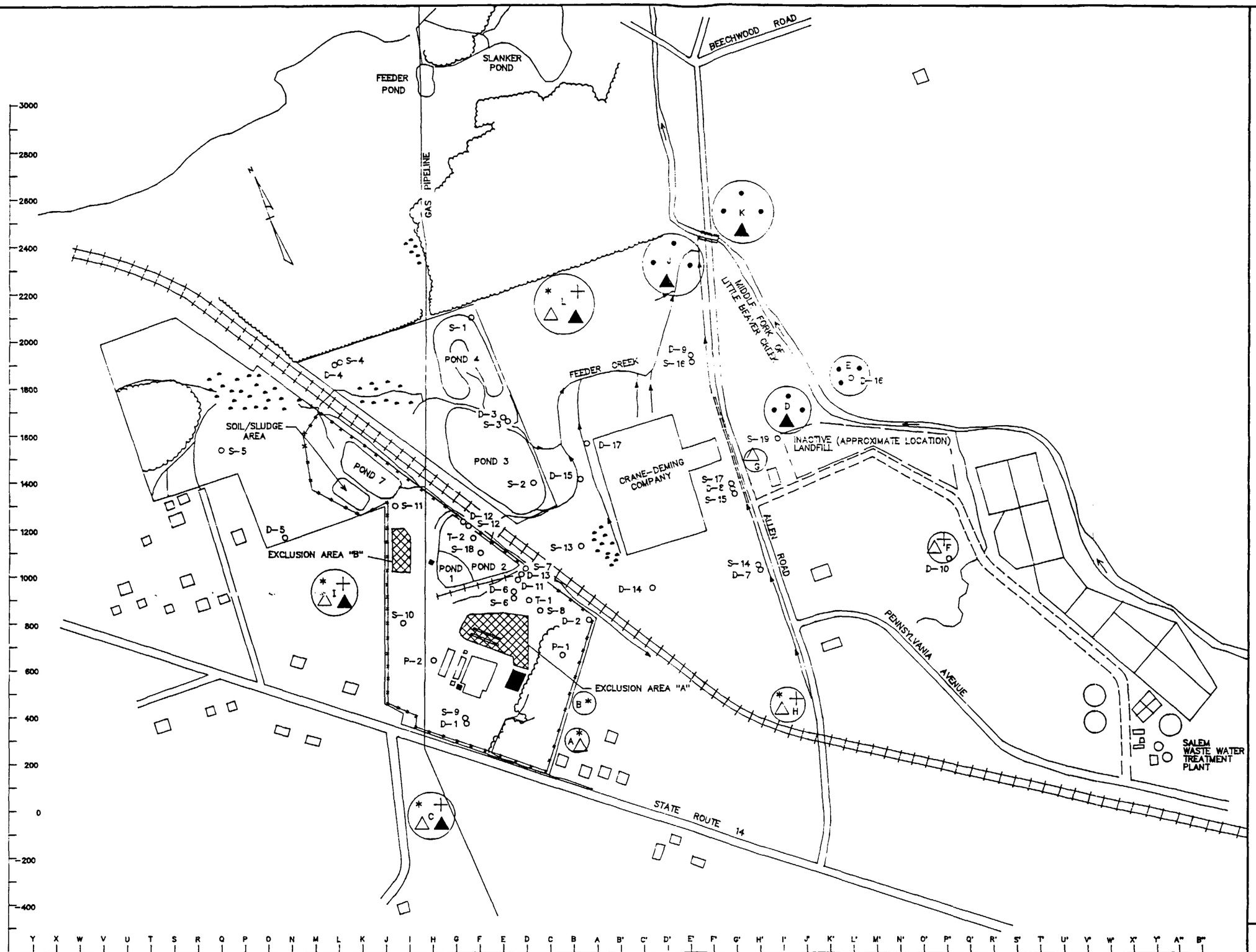
A GC attachment to the OVA will measure the concentrations of the organic constituents in the soil gas. In order for the volatile constituents to elute faster, a thermal attachment is used to heat the sample prior to running the GC. The results will be printed out on a strip recorder that provides a copy of the peaks from the GC. These peaks can be compared against fingerprints of known contaminants for identification.

The OVA will then be disconnected, and a PID will be connected to the tygon tubing. After re-purging the system, maximum and stabilized PID readings will be measured and recorded.

Prior to initial use and after each sampling taken, the probe will be decontaminated according to the procedures described in Section 7.3.3. The sample probe will be screened with the FID and PID to ensure complete decontamination and prevent cross contamination and false positive readings.

3.3 Well Drilling and Installation

An estimated 36 monitoring wells will be installed at the 12 locations shown on Figure 3-2. This network is designed to monitor five potential aquifer zones within the study area. These aquifers are the Shallow, Interface, Upper Bedrock, Lower Bedrock, and the Valley Fill in the area of the MFLBC. Target aquifers at each drilling location are identified on Table 3-6. The two bedrock aquifers may consist of interbedded sandstones, shales and coals, while the upper three aquifers may consist of unconsolidated sands and gravels. Information obtained from the wells will be used to evaluate site hydrogeology. All drilling and sampling equipment will be decontaminated according to the procedures described in Section 7.0.



LEGEND

- VALLEY FILL WELL
- SHALLOW AQUIFER WELL
- INTERFACE AQUIFER WELL
- UPPER BEDROCK WELL
- LOWER BEDROCK WELL

NOTE: ACTUAL LOCATIONS OF WELLS WITHIN DRILLING AREAS WILL BE DETERMINED AT THE TIME OF WELL CONSTRUCTION.

- (A) PROPOSED DRILLING AREA
- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- D-16 EXISTING MONITORING WELL NO.
- EXISTING MONITORING WELL LOCATION
- SITE BOUNDARY

FIGURE SSSP 3 - 2
MONITORING WELL
LOCATION AREAS

RUETGERS-NEASE SALEM SITE RI/FS

SCALE
0 200 400 600 (FEET)

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SSSP TABLE 3-6

MONITORING WELLS AND TARGET AQUIFERS BY DRILLING AREA
RUETGERS-NEASE SALEM SITE RI/FS

Proposed Drilling Area	Shallow Aquifer	Interface Aquifer	Upper Bedrock Aquifer	Lower Bedrock Aquifer	Note
A	1		1		
B	1				
C	1	1	1	1	1
D					2
E					3
F		1	1		1,4
G			1		
H	1	1	1		1
I	1	1	1	1	5
J					2
K					2
L	1	1	1	1	1
TOTAL	6	5	7	3	

NOTES:

1. If the Interface Aquifer is not encountered, the well will be completed in an overlying water bearing zone if one is encountered.
2. Well cluster, assume 4 water bearing zones.
3. Well cluster, assume 3 water bearing zones.
4. The Upper Bedrock well can only be installed if the Upper Bedrock Aquifer is encountered at this location.
5. Potential Background wells.

3.3.1 Drilling Procedures

Monitoring well pilot boreholes will be advanced through overburden materials with a drilling rig employing hollow stem auger, or rotary techniques using formation water or filtered air as a drilling fluid. Hollow stem augers will be used to advance the borehole through unconsolidated material. Rotary methods will be used to advance boreholes through bedrock.

To prevent downward migration of contaminants from shallower aquifer zones into deeper aquifer zones, telescoped, permanent outer PVC well casings will be installed as drilling proceeds. The procedure for installation of such casings will be as follows: the borehole will be advanced to at least ten feet below the base of the aquifer to be cased off, grout will be placed in the borehole using a tremie pipe, and then the casing (with bottom plug) will be inserted into the borehole thus displacing the grout. This will ensure continuous grout distribution outside of the casing. After the grout has hardened (minimum 24 hours), a rotary bit will be used to drill through the bottom plug, and advancement of the borehole will continue. It should be noted that temporary well casings may be installed in order to complete wells under artesian conditions.

At each location, the well proposed for the deepest aquifer will be drilled first, and all location specific soil and rock samples will be collected from this boring. In this boring only, continuous split spoon soil samples will be collected, and a wireline coring system will be utilized to collect rock core samples. Upon reaching the target depth, a rotary bit will be used to ream out the wireline cored borehole to a proper size for well installation.

Soil and rock core samples obtained will be used to delineate subsurface stratigraphy, and identify target depths to be screened in other wells at that drilling location. When drilling other pilot borings, split spoon or core samples will be collected at the projected target depths only, to confirm that the zone to be screened has been reached. In order to identify zones of contamination, all samples and drilling cuttings will be screened for volatile organic contamination using an FID and PID. U.S. EPA and OEPA have requested that a field screening method to detect mirex and its degradation products be utilized. However, field screening for these constituents is not believed to be technically feasible. Cuttings will be handled and disposed of according to the procedures outlined in Section 9.4 of the Health and Safety Plan (Volume 4).

Boreholes targeted for the Shallow, Interface, or Valley Fill Aquifers will be advanced using at least four inch I.D. hollow stem augers. Wells will be installed through the augers.

If the Interface Aquifer is not encountered at a target location, a well will be completed in a saturated zone if encountered at the expected depth of the Interface Aquifer.

Upper Bedrock pilot boreholes will be advanced with rotary methods to at least 15 feet into the Upper Bedrock Aquifer. A temporary casing will be installed if necessary to prevent the borehole from collapsing. The well will be installed through the temporary casing. As the well casing is grouted in place, the temporary casing will be removed from the borehole.

Lower Bedrock boreholes will be advanced with rotary methods to the Kittanning confining layer, if present.

Casing will only be temporarily installed into the borehole if the confining unit is not encountered. Drilling will proceed through casing until the target zone is reached. The well will then be installed through casing, and the outer casing will be withdrawn as the well casing is grouted in place.

All recordings, measurements, and split spoon descriptions taken during drilling will be recorded into a field notebook.

Upon completion of each borehole, a descriptive log with the following information will be completed:

1. If pumping pressure meters are installed on the equipment used, meter pressure readings during drilling or purging operations will be recorded.

2. Type and amount of drilling fluid used, depth at which its use started, and reason for starting. Drilling fluids other than formation water or filtered air will be used only with prior approval of the OEPA and the U.S. EPA.

3. Description of drill rig configuration, manufacturer, model, pump type, bit type, rod sizes, and specifications of tools used.

4. Evidence including VOC measurements of possible contamination zones, depth to these zones and thicknesses of the zones.

5. A record of the sequence of drilling operations used at each site.

6. All special problems encountered at a site (e.g., lost casing, screen, tools) along with hole heaving, bridging or cavern development.

7. Commencement and completion dates for each boring.

8. Sequential lithologic boundaries, and, if estimated, the degree of accuracy which applies to the boundary.

9. Blow counts, hammer weight, and length of fall.

10. The length of the sampled interval and the length of the sample recovered for that interval for all split spoon, thin wall, and cored samples.

11. Depth to the first and subsequent water bearing zones encountered, along with the method of determination.

12. Visual and any numerical estimates of secondary soil constituents.

13. Location, spacing, and nature of all core breaks (natural or coring induced), intervals of possible sample losses, and probable reasons for the loss.

3.3.2 Well Construction Specifications

The monitoring wells will be constructed according to the following specifications:

1. Well riser pipe located more than 10 feet above the anticipated maximum piezometric level elevation, and all permanent outer casings will be constructed of threaded Schedule 40 PVC material.

2. Well riser pipe located within ten feet of the anticipated maximum piezometric level, and all well screens will be constructed of threaded flush-joint, Schedule 304 stainless steel. All well risers and screen will be two inch inside diameter, and will be steam-cleaned prior to installation.

3. Screens will be 10 feet long, and will have a 2 foot sediment trap installed at the base, unless the target zone is less than 10 feet in thickness, in which case, a 5 foot screen will be installed with the prior approval of the OEPA and the U.S. EPA. Due to the nature of the fine grained material present in the subsurface, a screen with 0.010 inch openings will be used.

4. Washed sand filter packs will extend to approximately 2 feet above the top of screen.

5. Bentonite seals will extend approximately three feet above the top of the filter pack.

6. A cement-bentonite grout (of approximately 94 pounds cement to six pounds bentonite) will extend from the bentonite seal to approximately three feet below land surface.

7. A cement apron extending from the ground surface to below the frost line (approximately 3 feet) will be installed. A protective outer steel casing with locking cap will extend approximately three feet into this apron and will extend approximately two feet above the ground surface.

8. If wells are completed flush with land surface, the cement apron will be below land surface and a vault will replace the protective outer casing.

Wells may be single-cased, or have multiple casings (i.e., a telescoping system) depending on the target aquifer, and presence or absence of significant water bearing zones (which produce 1 GPM or more) overlying the target aquifer. Outer casings will be constructed of Schedule 40 PVC. Monitoring well installation procedures are described in the following text. A well construction summary log will be completed for each monitoring well installed, and will contain, at a minimum, the following information:

1. Borehole specifications (i.e., depth, diameter, drilling fluids used, etc.).

2. Amount of casing/screen used and depths at which it is installed.

3. Depth intervals for which filter material, grout, and bentonite seal are installed and the amount used.

4. Log detailing construction time for major tasks (i.e., drilling, casing installation, development, etc.).

Shallow, Interface, and Valley Fill Aquifer Wells

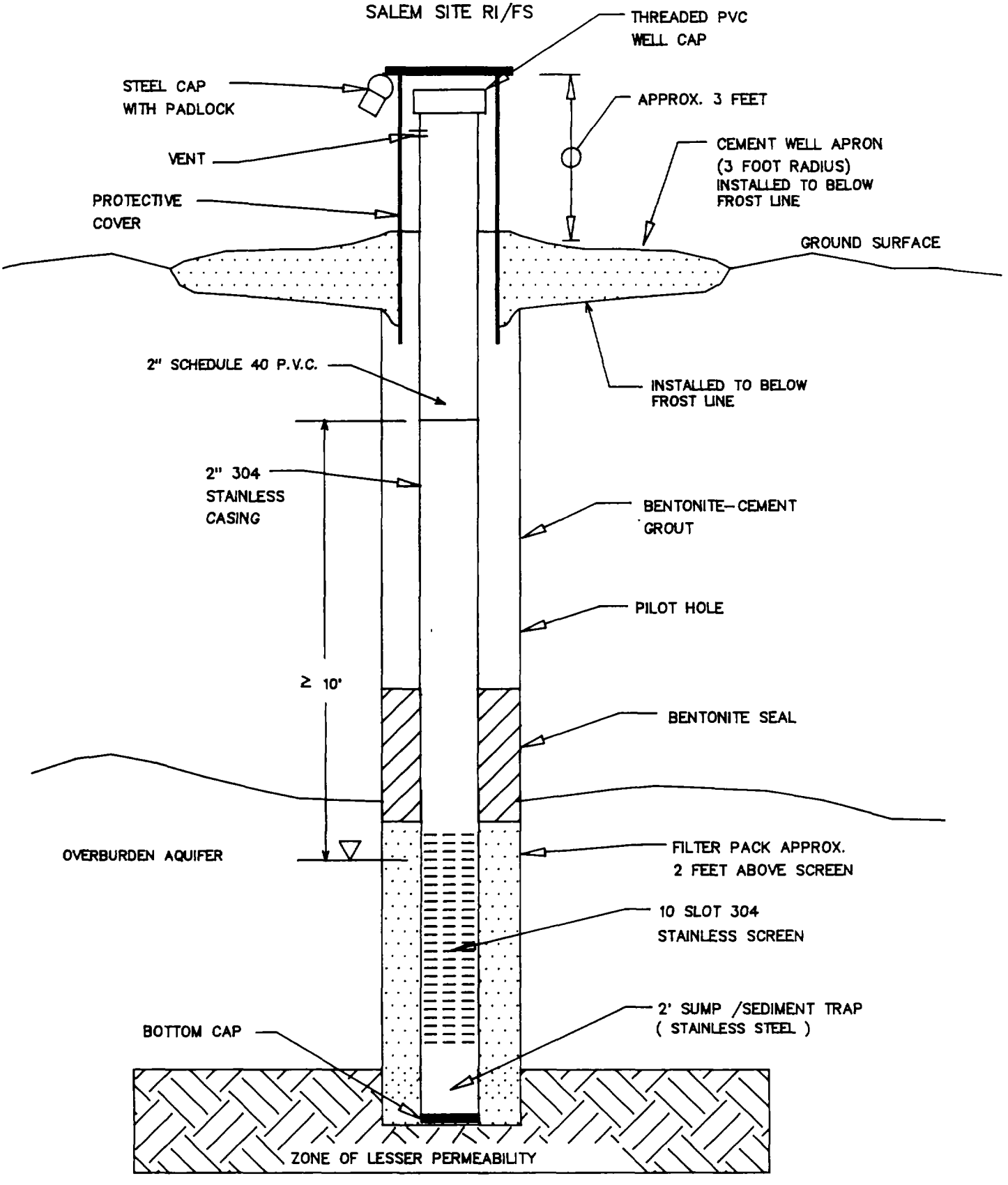
Wells in the Shallow Aquifer will be constructed as shown in Figure 3-3. After drilling has reached the target aquifer, a well screen, sediment trap, bottom cap and riser will be set into the borehole. Whenever hollow stem augers are used, the well screen and riser will be placed through

FIGURE SSSP 3-3

SCHEMATIC OVERBURDEN WELL

RUETGERS-NEASE

SALEM SITE RI/FS



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the hollow stem augers. A washed sand filter pack will be installed in the annulus around the well screen to a height 2 feet above the well screen followed by the bentonite seal. A cement and bentonite grout seal will be pumped into the annulus to ground surface using a small diameter tremie pipe or hose starting by extending it down to the top of the bentonite. The well will be completed with a cement apron placed around the well. A 6 inch protective steel casing with locking cap and cement apron will be installed over the riser pipe. The locking well cap will be appropriately labeled with a monitoring well identification number.

Double cased wells will be installed in the Interface Aquifer if the zone is encountered, and if a significant and apparently hydraulically separate overlying surface aquifer zone (defined as a zone which produces a minimum of 1 GPM) is present. Such an installation is shown in Figure 3-4. After the outer casing is installed in the borehole to a depth of five or more feet below the base of the surface aquifer, and is grouted in place (as described in Section 3.3.1), the borehole will then be advanced past this zone by drilling through the casing and completing the well in the same manner as a single-cased well.

Monitoring wells installed within the Valley Fill may be constructed as single-, or multiple-cased wells, The total number of wells per Valley Fill well cluster location will be determined by the number of water bearing zones (if any) present. A single-cased well will be installed in the shallowest significant water bearing zone (defined as a zone which will produce a minimum of 1 GPM) encountered. This well will be installed using the construction and protocol outlined for a surface aquifer well.

The next occurring significant, and hydraulically separate Valley Fill well will be double-cased, and will be

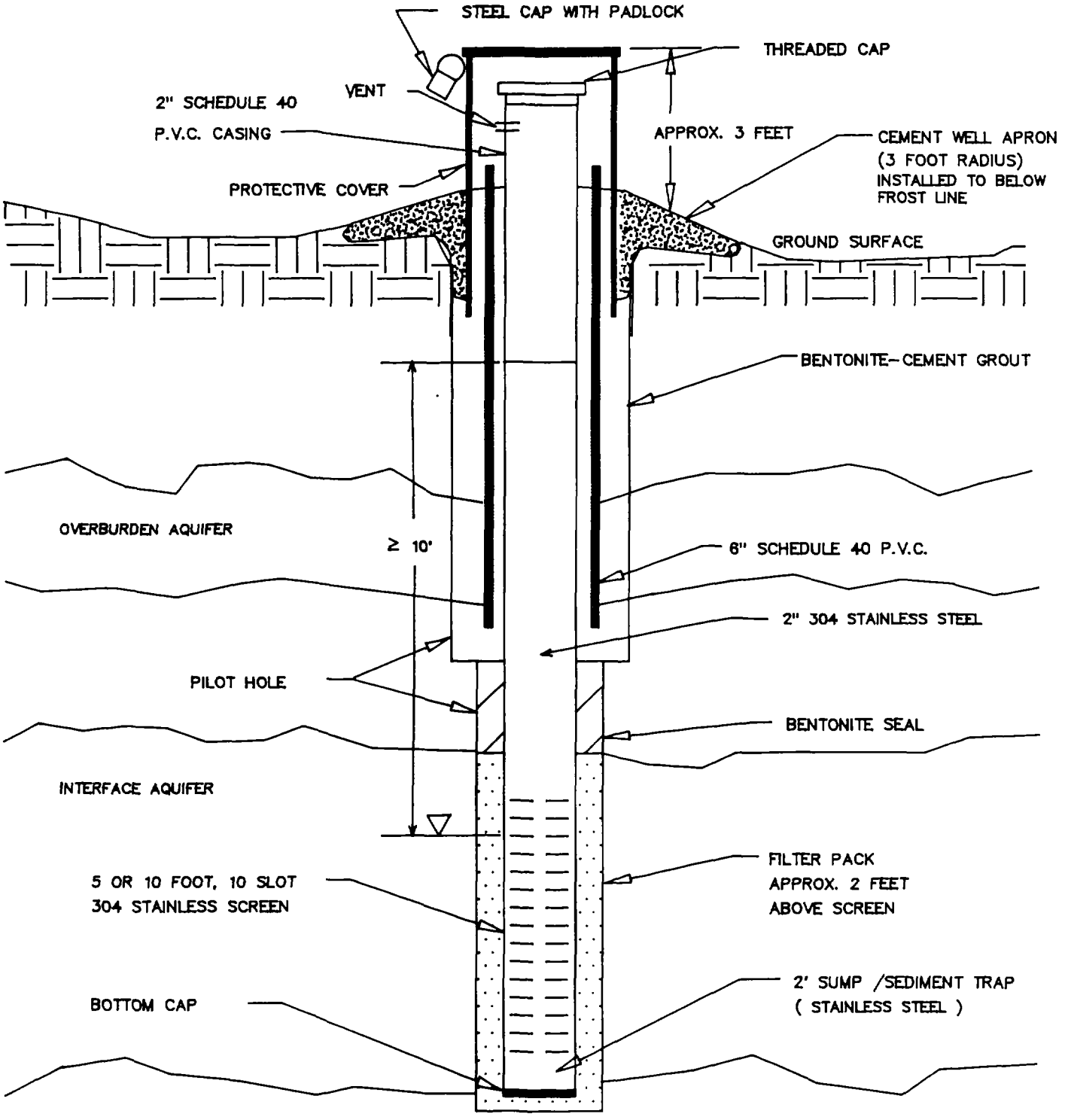
FIGURE SSSP 3-4

SCHEMATIC DOUBLE CASED

INTERFACE WELL

RUETGERS-NEASE

SALEM SITE RI/FS



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installed using the construction protocol outlined for an interface aquifer well. Wells installed in deeper significant, and hydraulically separate water-bearing Valley Fill zones will be multiple-cased (as described in Section 3.3.1), and the well components (screen and riser) will be installed through casing, in the same manner as a double-cased well. In the event the Valley Fill contains one continuous aquifer of significant thickness, a joint decision between the Field Operations Manager and the on-site agency representative will be made as to whether more than one well should be installed within the aquifer.

Upper Bedrock Wells

Double-, or multiple-cased wells will be installed in the Upper Bedrock Aquifer if the zone is encountered (i.e., has not been removed by erosion) and, if significant and apparently hydraulically separate overlying surface, and/or Interface Aquifer Zones are present. If the well is to be multiple-cased, it will be constructed as shown in Figure 3-5.

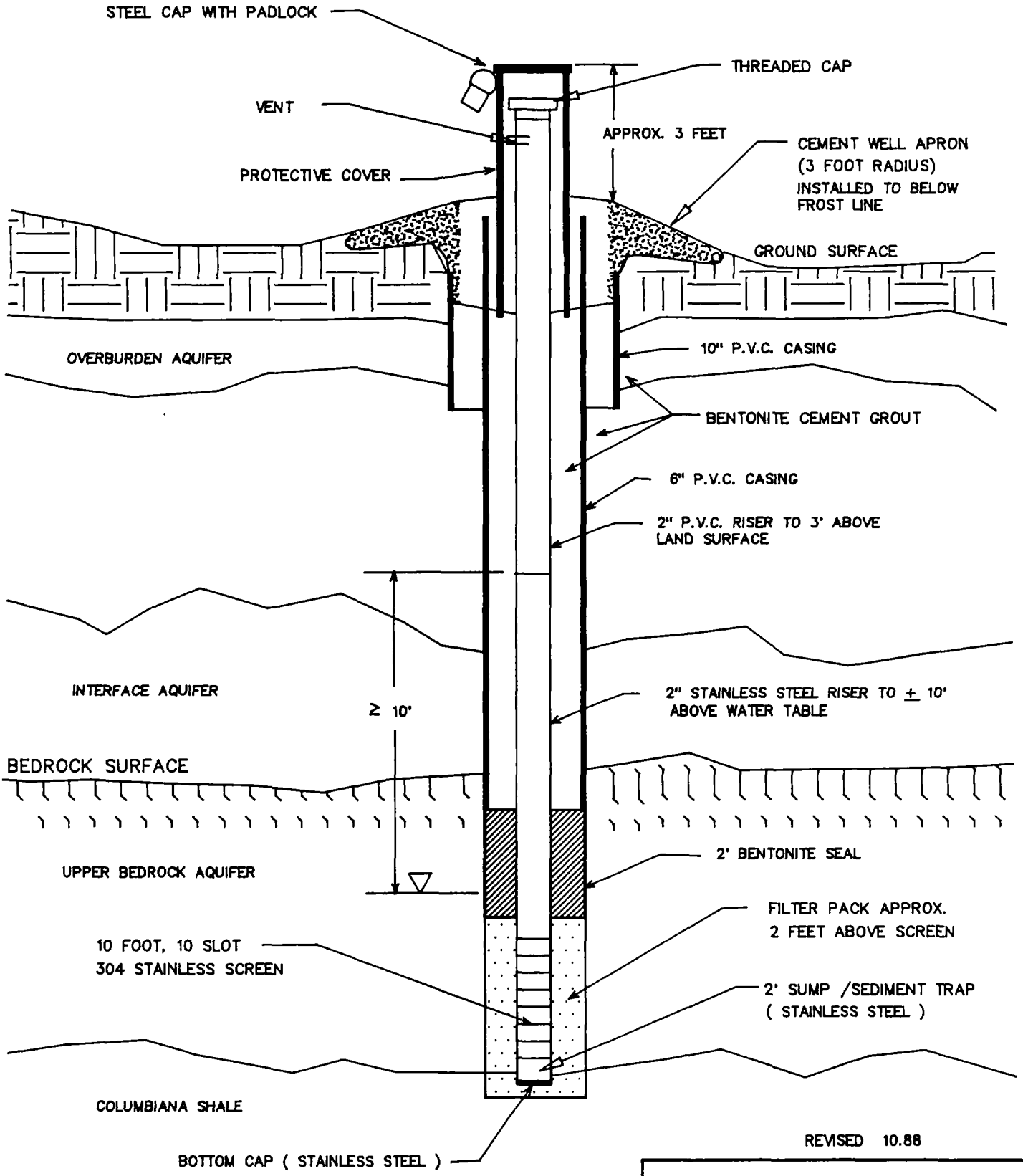
A pilot boring will be advanced to bedrock utilizing the drilling and casing installation methods discussed in Section 3.3.1 and as required by field conditions. Once bedrock is encountered, the boring will be advanced 15 feet into competent bedrock. Well casing will be installed through the augers or casing. The washed sand filter pack will be placed into the annulus using a small diameter tremie pipe or hose. A 2 foot bentonite seal will be placed above the sand pack and the remainder of the annular space will be filled with cement/bentonite grout to a height 3 feet below grade. A cement apron with a protective steel casing or flush mount well vault will complete the well. The locking well cap or vault will be labeled with the well identification number.

FIGURE SSSP 3-5

SCHEMATIC UPPER BEDROCK WELL

RUETGERS-NEASE

SALEM SITE RI/FS



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Lower Bedrock Wells

Double-, or multiple-cased wells will be installed in the Lower Bedrock Aquifer if the zone is encountered and if significant and apparently hydraulically separate overlying Surface, Interface, Upper Bedrock, or Valley Fill Aquifer zones are present. If the well is to be multiple-cased, it will be constructed as shown in Figure 3-6.

Unconsolidated material will be drilled and cased-off as appropriate using the methods discussed in Section 3.3.1, and as field conditions dictate. Rotary drilling methods will be used to advance the borehole from the top of bedrock into the confining layer (if present) over the Lower Bedrock Aquifer. An outer casing will be securely grouted in the borehole when the confining layer is reached. If the confining layer is not encountered, the upper bedrock zone will not be cased-off.

The borehole will be advanced approximately 15 feet into the Lower Bedrock Aquifer, and then the well components will be installed. The filter pack will be installed in the annulus, followed by a bentonite seal. A cement and bentonite grout seal will be placed into the annulus using a small diameter tremie pipe or hose.

The well will be completed with a cement apron installed to the frost line depth and either a protective steel casing or flush-mounted well vault. The locking cap or vault of each well will be labeled with the well identification number.

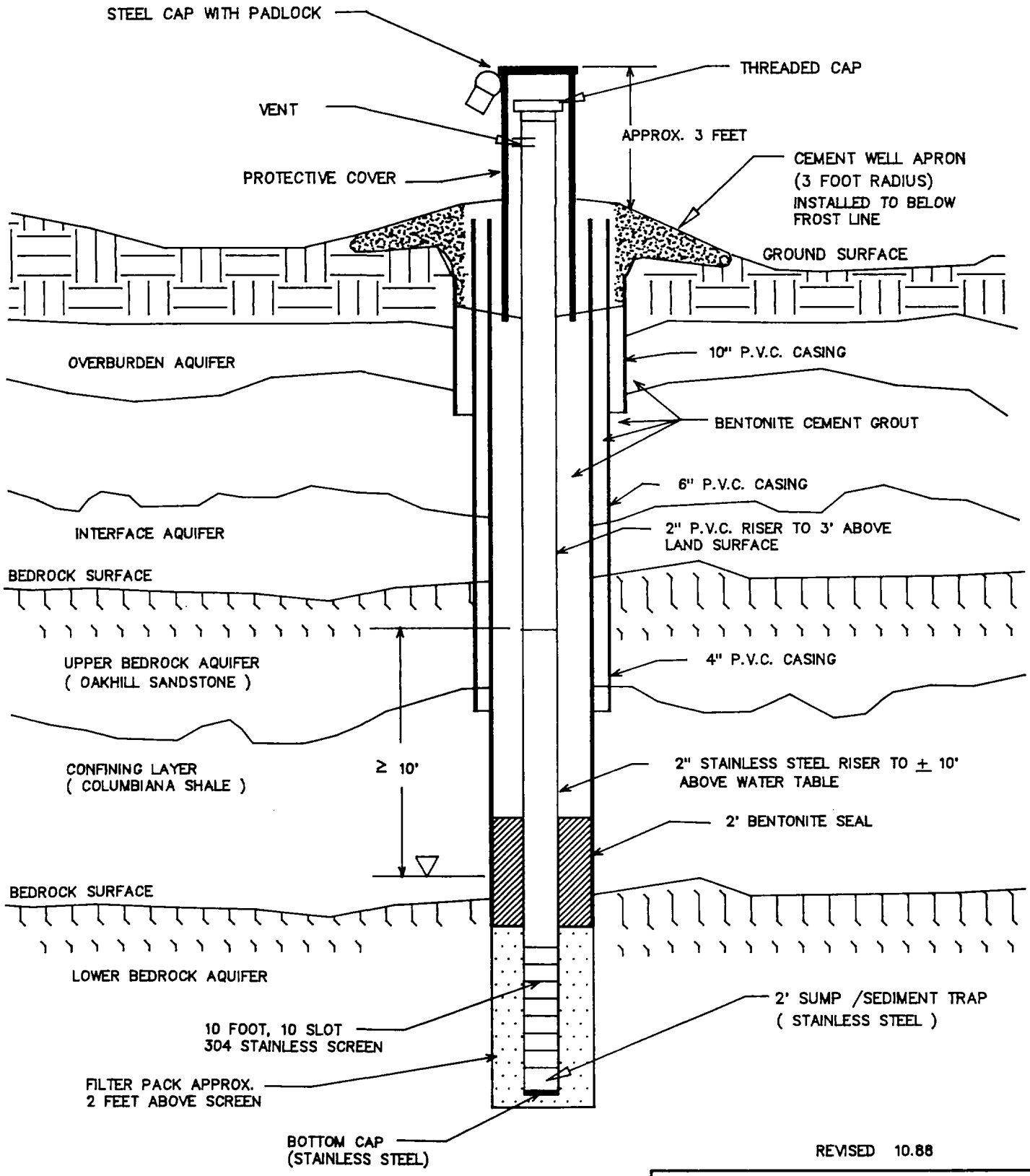
If no overlying water-bearing zones are encountered, which may occur near the Valley Fill, a temporary outer casing will be installed into the Lower Bedrock Aquifer, and the well will be installed through the casing. As the well

FIGURE SSSP 3-6

SCHEMATIC LOWER BEDROCK WELL

RUETGERS-NEASE

SALEM SITE RI/FS



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is being grouted, the temporary casing will be removed and the protective cover installed.

3.3.3 Artesian Well Installation

Zones where artesian conditions are present will be delineated while drilling the deep well at each monitoring location. If flows are encountered which prevent the normal installation of a monitoring well the procedures described below will be followed.

1. Advance the pilot borehole beyond the artesian zone and install a temporary casing in order to seal off the flow of ground water.

2. Backfill the overdrilled area to the projected bottom of the screened interval with clean gravel.

3. Slowly lower the well screen and riser into the outer casing.

4. Install the gravel pack and bentonite seal into the well annulus.

5. Slowly extract the outer casing from the pilot borehole while installing the grout seal into the well annulus. If the well is to be installed into the Lower Bedrock Aquifer, the outer casing shall be pulled back until it is approximately three feet into the bedrock or the confining layer (if present).

6. Complete the well by installing the grout seal to ground surface followed by the protective cover.

If the artesian zone cannot be adequately sealed, well installation procedures will be dependent upon the depth the

zone is encountered and the type of lithology the well is to be completed into. Wells to be installed into overburden material will consist of 2 inch (I.D.) stainless steel well points with wire wound screens. A pilot borehole will be advanced using hollow stem augers to within 5 feet of the target screen zone but not to the depth where the artesian conditions were encountered. The well point will be lowered inside the augers and driven down until the target interval is reached. The annulus will be filled with grout to ground surface and a protective cover installed.

Bedrock wells in which the artesian zone cannot be isolated will be completed as open borehole wells. In this case, the outer casing will be installed or pulled back (if possible) and sealed into bedrock or the confining layer (if present) approximately three feet using a cement/bentonite grout.

3.3.4 Development

Well development will begin not less than 48 hours after the annular space cement-bentonite seal has been completed. A combination of surge, pumping, or pressurized air methods will be used, with methods for each well to be determined based on field conditions. Pressurized air is proposed to be the preferred method for development. All materials placed into wells will be decontaminated before use. Development will continue until:

1. The well is free of sediment.

2. Water removed from the well is clear.

Development waters will be disposed of according to Hazardous Materials Handling Plan procedures included in Section 9.4 of the Health and Safety Plan (Volume 4).

3.4 Sampling

Ground water, surface and subsurface soils, surface waters, sediments, air, and fish tissue samples will be collected during this RI/FS. Media to be collected and analysis to be performed are listed in Table 1-5. Procedures for collecting these samples are described in this section, with step-by-step instructions provided in Appendix A and B.

3.4.1 Ground Water

Ground water samples will be collected from all new wells, 30 existing wells, (as shown in Table 3-7), and six residential wells. Sampling procedures generally will follow protocols described in the U.S. EPA TEGD document. Wells will be sampled beginning with those suspected of being least contaminated and progressing to those assumed most contaminated.

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 to within 0.01 feet using a decontaminated, Oil Recovery Systems (or equivalent) electric interface probe with attached permanent depth marked taped. The static and total depth will be used to calculate the volume of standing water. One bailer of ground water 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. Purge water will be managed according to procedures set forth in the Health and Safety Plan (Volume 4), Section 9.4.

SSSP TABLE 3-7

EXISTING GROUNDWATER SAMPLE LOCATIONS[1]
RUETGERS-NEASE SALEM SITE RI/FS

Shallow Wells	Interface Wells	Upper Bedrock Wells	Lower Bedrock Wells	Residential Wells
S1	S2	T2[2]	D10	5 wells in addition to the Salem Country Club
S4	S13	D1	D13	
S6[2]	S16	D2	D14	
S8	S17	D3	D16	
S9	S18[2]	D5		
S11	S19	D7		
S12[2]		D8		
S14		D9		
S15		D11		
		D12		
		D15		

[1]Proposed sample locations for all new monitoring wells are shown on Figure 3-2.

[2]Wells to be sampled/analyzed for the additional parameters DCNB, dioxins/furans and CLP inorganics

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. All bailers, hoses, and pumps will be decontaminated before being introduced into wells. Pumps will not allow lubricated surfaces to come into contact with ground water. 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 will take place after purging procedures are completed. Sampling, whenever possible, will occur the same day as purging, however, this may not be feasible for slowly recharging wells. Purging procedures are described in Appendix A, Section 2.1.

Samples will be collected from monitoring wells using Teflon^{R} bailers dedicated to each well, after removing 3 well volumes or purging the well dry.* The first sample withdrawn from the well will be checked for temperature, pH and specific conductance. Subsequent samples collected from within the screened interval will be used to 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. Detailed sampling protocols are specified in Appendix A, Section 2.0.

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*Footnote: Ruetgers-Nease intends to utilize temporary or permanent dedicated bladder-type pump installations in wells which will be sampled on a regular basis as part of any long-term groundwater monitoring activities.

Samples will be collected from five residential wells and the well at the country club. If possible, sampling points will be located at a water tap closest to the pump prior to any water softeners. Prior to sample collection, the faucet aerator, if any, will be removed and any in line water conditioning units turned off. If the well is active, an appropriate volume of water in the supply line preceding the tap will be removed. The sample will be collected after stabilization of temperature, pH, and conductivity; or after five minutes, which ever is greater. An estimated 3 well volumes will be removed.

The sample will be collected directly from the tap into the sample containers. Bottles will be filled in the same order as described in the previous paragraph.

If artesian flow conditions exist at any of the ground water sampling locations, the well will be allowed to flow until the appropriate amount of water is removed from the well prior to sample collection. Detailed sampling procedures are included in Section 2.3 of Appendix A.

3.4.2 Soil Borings Through Ponds

Eighteen soil borings will be completed through the five ponds and the soil/sludge area west of Pond 7, at locations shown on Figure 3-7. These borings will be completed to bedrock or to 9 feet into native soils below the ponds, whichever is less. Continuous core samples will be collected using split spoons (for chemical analysis borings) or Shelby tubes (for physical parameter borings), depending on field conditions. Each of the 3 foot core samples from fourteen borings will undergo chemical analysis, while each of the 3 foot core samples from latter four borings will undergo testing for physical parameters.

LEGEND

- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- SOIL BORING-CHEMICAL
- SOIL BORING-PHYSICAL
- SOIL TEST PIT-CHEMICAL
- SITE BOUNDARY
- TEST PIT FOR EXPANDED ANALYSIS

FIGURE SSSP 3 - 7

SOIL SAMPLING LOCATIONS ON-SITE AND ADJACENT AREAS

RUTGERS-NEASE SALEM SITE RI/FS

SCALE

0 200 400 600 (FEET)

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Within the ponds, and until the native soils under the ponds are reached, each 3 foot interval sampled will undergo scheduled analysis. Samples will be opened or the sample extruded, and screening will be performed on each three-foot interval using an OVA to select the sub-interval with the highest response. The sub-interval will be taken as a grab sample and will be analyzed for CLP volatile organics and library searches for up to 15 compounds. If there are no observed differences in OVA responses between sub-intervals, the middle of the interval will be samples as a grab and will be analyzed for CLP volatile organics and library searches for up to 15 compounds.

A composite of the entire 3-foot interval of soils/sludges (non-native soils) will then be homogenized and analyzed for CLP non-volatile* organics plus a library search for up to 25 additional compounds plus mirex, kepone, photomirex, and DPS. These composite samples will be prepared by thoroughly mixing the materials to be composited in a large stainless steel bowl using stainless steel utensils.

Samples of non-native soils/sludges from a series of 3 foot cores from one borehole in each pond and in the soil/ sludge area west of Pond 7 will be analyzed additionally for 3,4-DCNB, dioxins/furans and CLP inorganics.

Once native soils are reached, a portion of either the upper, middle or lower thirds of each 3 foot interval sampled will be selected for CLP volatile organic analysis plus library searches for up to 15 additional compounds. The remainder of the split spoon sample will be composited, and a sample will be collected and analyzed for mirex, photomirex, kepone, methoxychlor and DPS. The remainder of the composite will be placed on ice, and will be combined with samples from the same depth from the other boreholes within individual ponds for CLP non-volatile* organics plus

library searches for up to 25 additional compounds without deviating from the established holding times. In other words, samples from the same interval below the pond, from all the boreholes within a pond, will be combined to make one composite sample which will undergo analysis for all chemical parameters except the volatile organics. This sampling strategy is illustrated on Figure 3-8. If it appears that the holding times for the non-volatile samples will not be met then individual, not composite samples, will be analyzed.

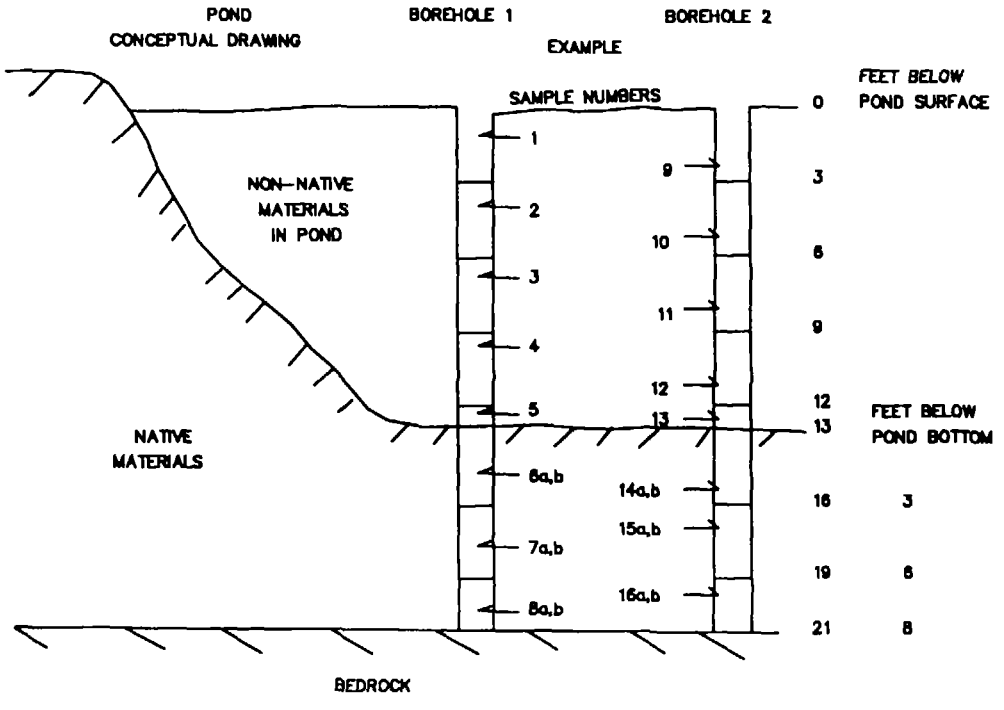
The bottom of the soil sludge pile west of Pond 7 is assumed to be at approximately the same elevation as found in the adjacent Pond 7. The bottom of Pond 1 is assumed to be approximately the same elevation as found in the adjacent Pond 2.

Samples for physical analysis will be collected using Shelby tubes, when possible, so that relatively undisturbed samples can be collected. Upon collection, the ends of the tube will be sealed, and the tube wrapped and taped for shipment to the physical testing laboratory.

The physical parameters that will be determined for each of the soil samples are: Unified Soil Classification (ASTM revised D2487), specific gravity (ASTM D854-83), permeability (Army Corps of Engineers EM1110-2-1906 Appendix 7, "Falling Permeability Test with Back Pressure"), effective porosity (a standard calculation without reference), sieve analysis (ASTM D422-63), moisture content (ASTM D2216-80), and Atterburg Limits (ASTM-D4318-84).

*Non-volatile compounds are defined as the TCL semivolatile organic compounds and the TCL pesticides/PCBs.

FIGURE SSSP 3 – 8
SCHEMATIC DIAGRAM, POND SLUDGE/SOIL SAMPLING STRATEGY
RUETGERS–NEASE SALEM SITE RI/FS



SCHEDULED ANALYSIS BY EXAMPLE SAMPLE NUMBERS

	A	B	C	D
	1	9	6a	6b+14b
	2	10	7a	7b+15b
	3	11	8a	8b+16b
	4	12	14a	
	5	13	15a	
			16a	
TOTAL SAMPLES	5	5	6	3

SCHEDULED ANALYSIS

A. CLP ORGANICS + 40 COMPOUNDS, MIREX, KEPONE, PHOTOMIREX, DPS, DCNB, DIOXINS, CLP INORGANICS

B. CLP ORGANICS + 40 COMPOUNDS, MIREX, KEPONE, PHOTOMIREX, DPS

C. CLP VOLATILE ORGANICS + 15 COMPOUNDS, MIREX, KEPONE, PHOTOMIREX, METHOXYCHLOR, DPS

D. CLP NON–VOLATILE ORGANICS + 25 COMPOUNDS

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3.4.3 Test Pit Soil Sampling

Test pits will be excavated on-site and in the Crane-Deming swamp at locations shown on Figure 3-7 and along the railroad tracks. Thirty pits are planned on-site and in the swamp, as well as an undetermined number to be located along the railroad tracks. The number and location of test pits along the railroad tracks will be selected in consultation with the OEPA/U.S. EPA based upon the results of the soil gas survey. At least one pit will be located along the tracks next to a southern corner of the site property. The number and location of these test pits will be subject to U.S. EPA and OEPA approval.

A backhoe will open the pits, and samples will be collected of undisturbed soils in the walls or from the base of the excavation. Samples will be collected from land surface to 0.5 feet below land surface (BLS), at 0.5 to 3.5 feet BLS, and 3.5 to 6.5 feet BLS. Successively deeper three foot samples will be collected if:

1. PID or FID measurements are above 10 ppm in the top 0.5 feet of remaining soils,

2. Bedrock has not been encountered, and

3. The water table has not been reached, if below 9.5 feet.

Samples from below approximately 6.5 feet will be collected from the backhoe bucket due to health and safety considerations. Under no circumstances will any personnel enter a pit deeper than 4 to 5 feet without protective sheeting or one that is of suspect stability (e.g., walls of pit have potential for collapse or depth of unsheeted pit is too excessive for personnel to enter safely).

Screening will be performed on each 3-foot interval using an OVA to select the 1-foot subinterval with the highest response. This subinterval will be taken as a grab sample and will be analyzed for CLP volatile organics and library searches for up to 15 compounds. If there are no observed differences in OVA responses between subintervals, the middle of the interval will be sampled as a grab and will be analyzed for CLP volatile organics and library searches for up to 15 compounds.

A composite of the entire 3-foot interval of soils/sludges (non-native soils) will then be homogenized and analyzed for CLP non-volatile* organics plus a library search for up to 25 additional compounds plus mirex, kepone, photomirex, and DPS.

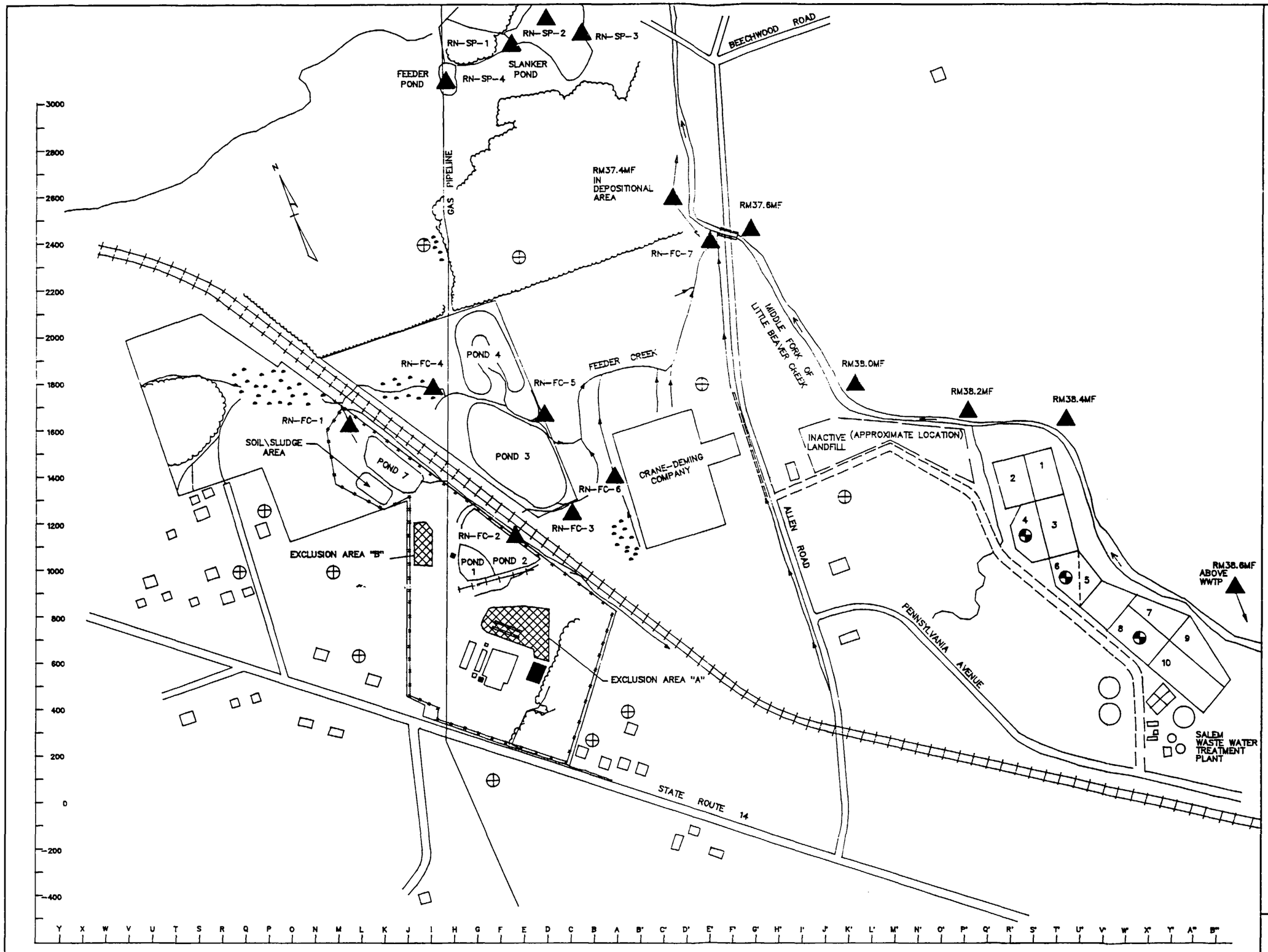
Samples from one test pit in each of Exclusion Areas A and B, and from four of the remaining potentially most contaminated on-site areas (see Figure 3-7) will be analyzed additionally for 3,4-DCNB, dioxins/furans and CLP inorganics.

Material for organic and inorganic analysis will be collected using decontaminated stainless steel utensils. All utensils will be decontaminated before each new sample is collected. Backhoe buckets will be decontaminated between test pit locations.

As each test pit is being opened, soils removed from the excavation will be placed on plastic on the ground in piles corresponding to sample intervals. After sampling, soils will be placed back into the pit in reverse order of removal.

3.4.4 Off-Site Soil Sampling

Subsurface soil samples will be collected from soil borings at 11 off-site locations (Figure 3-9) and submitted



NOTE:

MFLBC-1 THROUGH 6 DESCRIBED ON TABLE 5.

OTHER SAMPLES DESCRIBED ON TABLE 4.

LEGEND

- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- STREAM (DRAINAGE) POND SAMPLING STATION
- OFFSITE SOIL SAMPLE
- SITE BOUNDARY
- SALEM WWTP SLUDGE SAMPLE
- CELL NUMBERS

FIGURE SSSP 3 - 9

SURFACE WATER, SEDIMENT AND OFF-SITE SOIL SAMPLES

RUETGERS-NEASE SALEM SITE RI/FS

SCALE

0 200 400 600 (FEET)

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for laboratory analysis of TCL non-volatile organics plus library searches for up to 25 additional compounds in addition to mirex, kepone, photomirex and DPS. 3,4-DCNB, dioxins/furans, and CLP inorganics may be analyzed for if they are detected above background levels in on-site samples. Soil borings will be completed using a bucket auger or power auger. If auger refusal is encountered, a drilling rig will complete the soil borings. Samples will be collected from ground level to 0.5 feet BLS and from 0.5 feet to 3.5 feet below ground surface directly from the bucket auger or split- spoon samples if a drilling rig is used. Additional 3 foot cores will be collected from ground level to 0.5 feet BLS, from 0.5 to 3.5 BLS plus additional 3 foot cores until HNU and OVA measurements are less than 10 ppm in the top 6 inches of remaining subsurface soils. Samples will be collected below 9.5 feet BLS only if the water table has not been encountered.

Samples will be collected according to the procedures described in Appendix A, Section 3.1. Borings will be backfilled to ground surface with clean native soil: Drill cuttings will be containerized and disposed of according to the Hazardous Materials Handling procedures described in Section 9.4 of the Health and Safety Plan (Volume 4).

3.4.5 Surface Water and Sediment - Feeder Creek and Slanker Pond

Five surface water and 11 sediment samples will be collected from Feeder Creek and Slanker Pond at the approximate locations shown in Figure 3-9. Table 3-8 lists the media to be sampled at each location.

At each sample location along Feeder Creek the flow of the stream will be estimated using a flow meter (e.g., pygmy meter) prior to sample collection. Water samples will be

SSSP TABLE 3-8
Sampling Program for Survey of Feeder Creek, Slanker Pond, and Middle Fork of Little Beaver Creek

Description	Station	5/16/89 Agreed Location No.	Analysis: Media F	SW	S	FP	Analysis: Parameters CLP +40	CLP Non-Vol +25	M, P, K, DPS	M, P, K, DPS, ME
Upstream of the WWTP as stream crosses Rte. 45	RM 38.6 MF	#1	2	1	1		4		4	0
NE corner WWTP	RM 38.4 MF	#2		1	1		2		2	0
Golf course stream	RM 38.2 MF	#3		1	1		2		2	0
Discharge zone	RM 38.0 MF	#4		1	1		2		2	0
Upstream Allen Road	RM 37.6 MF	#5	2	1	1		4		4	0
Feeder/Slanker Pond	RN -SP- 4	#6A			1				1	0
Slanker Pond, inlet	RN - SP - 1	#6B			1				1	0
Slanker Pond, middle	RN - SP - 2	#6C	2	1	1		4		4	0
N. of Slanker Pond beach	RN - SP - 3	#6D			1				1	0
Allen Road downstream (Slanker Bridge, north)	RM 37.4 MF	#7	2	1	1		4		4	0
Pine Lake Road bridge	RM 36.7 MF	#8	2	1			3		3	0
Between Goshen Road and Rte. 165	RM 35.4 MF	#9	2					2	2	0
Miller Farm	RM 35.0 MF	#10			1	4		0	0	5
Swamp area 0.3 RM south of Middletown Road		#11			1			0	1	0
Ruthraff Farm	RM 33.3 MF	#12			1	4		0	0	5
Rte. 45 (0.7 mi. N of Middletown Road)	RM 32.0 MF	#13	2	1	1			4	4	0
Swamp area between Rte. 45 and Rte. 62		#14			1			0	1	0
Rte. 62		#15	2		1			3	3	0
Swamp area 0.45 RM south of Rte. 62		#16			1			0	1	0
Sherwood Farm		#17			1	4		0	0	5
Rte. 165		#18	2	1	1			4	4	0
Beaver dam 1.85 RM south of Rte. 165		#19			1			0	1	0
Large swamp are west of beaver dam		#19A			1	4		0	0	5
Large swamp are east of beaver dam (Shepherd dam)		#19B			1	4		0	0	5
Pine Lake Road bridge		#20	2	1	1			4	4	0
0.7 RM south of Pine Lake Road bridge		#21			1			0	1	0
Due east of intersection of E. 10th St. & Egypt Rd.		#22	2		1			3	3	0
Private bridge 0.45 RM south of Rte. 14 bridge		#23	2	1	1			4	4	0
N. Lisbon Rd-Rte. 14 at river bend	RM 24.5 MF	#24			1			0	1	0
Swamp area due west of EPA '89 station 24		#25			1			0	1	0
Swamp area 0.53 RM south of EPA '89 station 24		#26			1			0	1	0
Camp Farm		#27			1	4		0	0	5

KEY:
F = Fish
SW = Surface Water
S = Sediment
B = Benthos
FP = Floodplain Sediment
M = Mirex
P = Photomirex
K = Kepone
DPS = Diphenyl sulfone
ME = Methoxychlor

ASSUMPTIONS:
- 2 fish samples per station
- 4 floodplain samples per location

NOTES:
-No Station #36
-The analysis of CLP+40 and CLP non-volatile+25 includes the analysis of methoxychlor

SSSP TABLE 3-8
Sampling Program for Survey of Feeder Creek, Slanker Pond, and Middle Fork of Little Beaver Creek

Description	Station	5/16/89 Agreed Location No.	Analysis							
			Media				Parameters			
			F	SW	S	FP	CLP +40	CLP Non-Vol +25	M, P, K, DPS	M, P, K, DPS, ME
Railroad bridge over Lisbon-Canfield Road	RM 23.5 MF	#28	2	1	1			4	4	0
Cunningham Road bridge over Stone Mill Run	RM 2.0 SMR	#29	2	1	1			4	4	0
Erie-Lackawanna bridge over E. Branch Cherry Valley Run		#30	2	1	1			4	4	0
SE bank of confluence of MFLBC & Cherry Valley Cr.		#31			1			0	1	0
0.23 RM south of old Rte. 344 bridge		#32			1			0	1	0
Swamp area due west of EPA '89 station 32		#33			1			0	1	0
Swamp area 0.68 RM north of Rte. 45		#34			1			0	1	0
Teagarden bridge on Eagleton Road	RM 17.5 MF	#35	2	1	1			4	4	0
Coleman Road bridge	RM 15.1 MF	#37	2		1			3	3	0
0.37 RM south of Furnace Road bridge		#38			1			0	1	0
Above Lisbon dam	RM 12.5 MF	#39	2		1			3	3	0
Below Lisbon spillway	RM 12.5 MF	#40	2	1	1			4	4	0
0.6 RM west of EPA '89 station 42		#41			1			0	1	0
Elkton West Point Road bridge		#42	2	1	1			4	4	0
0.2 RM east of EPA '89 station 42		#43			1	4		0	0	5
Beaver Creek State Park canoe livery 2.25 mi. east of Elkton	RM 4.6 MF	#44	2		1			3	3	0
Beaver Hollow Road Bridge		#45	2		1			3	3	0
Swamp area by Rte. 7 north of Williamsport		#46			1			0	1	0
Y Camp Road bridge	RM 14.4 WB	#47	2	1	1			4	4	0
Bell School Road bridge	RM 14.4 LBC	#48	2	1	1			4	4	0
Sprucevale Bridge-Beaver Creek State Park	RM 11.0 LBC	#49	2		1			3	3	0
Fredricktown bridge	RM 0.2 LBC	#50	2	1	1			4	4	0
1 RM south of MFLBC/NFLBC confluence		#51	2		1			3	3	0
Grimms Road bridge gauging station	RM 4.5 LBC	#52	2	1	1			4	4	0
Feeder Creek NNW of Pond 7	RN-FC-1	#53			1		1		1	
Feeder Creek East of Pond 2	RN-FC-2	#54			1		1		1	
Feeder Creek S of Pond 3	RN-FC-3	#55		1	1		2		2	
Feeder Creek (Swamp) W of Pond 4	RN-FC-4	#56			1		1		1	
Feeder Creek S of Pond 4	RN-FC-5	#57		1	1		2		2	
Feeder Creek W of Crane-Deming	RN-FC-6	#58		1	1		2		2	
Feeder Creek Prior to entering MFLBC	RN-FC-7	#59		1	1		2		2	
Subtotal			56	26	61	28	36	82	136	35
Total			171				289			

KEY:
F = Fish
SW = Surface Water
S = Sediment
B = Benthos
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M = Mirex
P = Photomirex
K = Kepone
DPS = Diphenyl sulfone
ME = Methoxychlor

ASSUMPTIONS:
- 2 fish samples per station
- 4 floodplain samples per location

NOTES:
-No Station #36
-The analysis of CLP+40 and CLP non-volatile+25 includes the analysis of methoxychlor

collected in mid-stream and sediment samples will be collected in areas of deposition. The sampler will stand downstream of the actual sample location so as not to disturb bottom sediments when collecting water and sediment samples. Surface water sampling procedures are described in Appendix A, Section 2.4. Sediment will be collected using stainless steel utensils from the stream bottom to a depth of 6 to 8 inches. Samples for volatile organic analyses will be filled directly from the utensils. The remaining fractions will be collected from a stainless steel bowl after the sediment is thoroughly mixed together.

At Slanker Pond, one sediment sample will be collected from the deepest water depth and one surface water sample at the same location from mid-point in the water column. The sediment sample will be collected using a Ponar Dredge sampler according to the procedures described in Appendix A, Section 3.2. The water sample will be collected 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 container, filling the volatile organic bottles first. Mirex, kepone, photomirex and DPS containers will be filled next. Two additional sediment samples will be collected from the pond using a stainless steel trowel for the inlet/outlet locations and a stainless steel bucket auger for the beach area location. The beach area sample will be taken northeast of the beach away from the beach sand that was introduced into the pond and in water that is at least three feet deep. Sediment sampling procedures are described in Appendix A, Section 3.1. Slanker Pond and Feeder Creek samples will be analyzed for the parameters identified in Table 3-8. 3,4-DCNB, dioxins/furans and CLP inorganics may be analyzed for if they are detected above background levels in on-site samples.

All equipment used to collect samples will be decontaminated prior to use and between each sampling location according to the procedures described in Section 7.0.

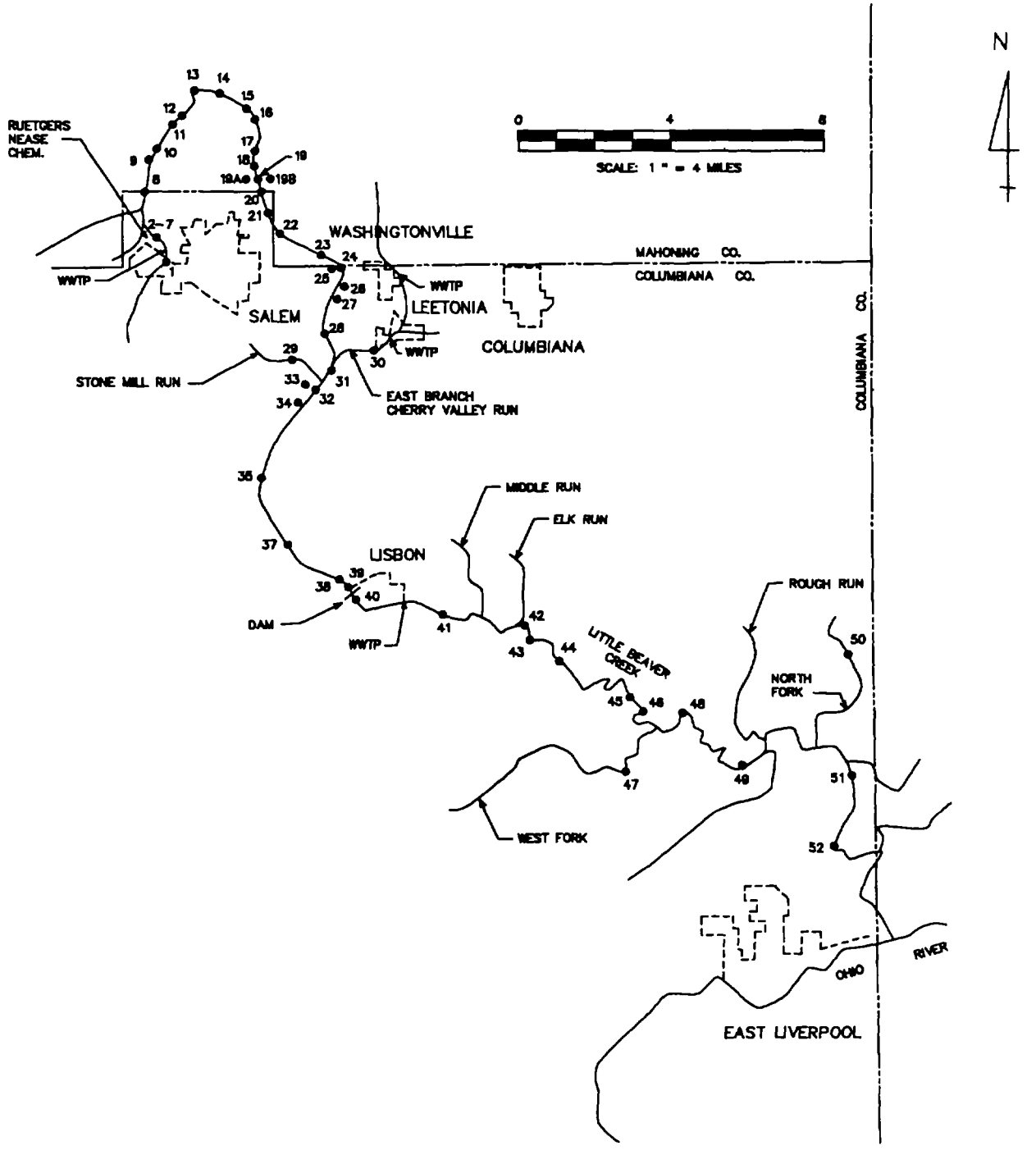
3.4.6 Surface Water and Sediment - Middle Fork of Little Beaver Creek (MFLBC)

Along the MFLBC, 50 sediment and 21 water and 28 floodplain soils samples will be collected. The locations of the sediment and water samples are shown on Figures 3-9 and 3-10. Each location and media to be sampled is listed on Table 3-8. Based upon the U.S. EPA/OEPA sampling programs conducted between August and November 1987 and the results of the 1985 OEPA survey, the surface water and sediment sampling program has been expanded beyond that described in the SOW.

Sediment and soil samples will be collected using stainless steel utensils. The steel utensils used for sediment samples will depend on water depth at the depositional location. Samples for volatile organic analysis will be filled directly from the sampler using a stainless steel spatula. All other samples will be filled after the sample has been mixed thoroughly in a stainless steel bowl.

Surface water samples will be collected near mid-stream, if possible, directly into the sample container. If the container has had preservatives added, then a clean glass beaker will be used to fill the sample container. The sampler will stand downstream of the sediment sample point so as not to stir bottom sediments. Measurements of pH, conductivity, DO and temperature will be performed at each surface location in-situ. Stream flow rates will be

FIGURE SSSP 3 – 10
SCHEMATIC MAP OF THE MIDDLE FORK LITTLE BEAVER CREEK
SHOWING GENERAL AREAS OF RI SAMPLING LOCATIONS
(REFER TO TABLE 3–8 FOR SPECIFIC LOCATION DESCRIPTIONS)
RUETGERS – NEASE SALEM SITE RI/FS



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estimated using a flow meter (e.g., pygmy meter) at each sampling location and recorded in the field notebook.

Analysis of the specified surface water and sediment samples from MFLBC will be for those parameters specified in Table 3-3. 3,4-DCNB, dioxins/furans, and CLP inorganics may be analyzed if they are detected in above-background levels in on-site samples.

All sampling equipment will be decontaminated prior to use and between each sampling location according to the procedures described in Section 7.0. Specific sampling procedures are described in Appendix A.

Sampling in MFLBC will be one of the initial tasks to be conducted following agency approval of the Work Plan. A technical memorandum reporting analytical results from this task will be submitted to the U.S. EPA/OEPA following data validation.

3.4.7 Aquatic Biota Investigation

To determine the levels in aquatic life of contaminants which possibly are leaving or have left the site, fish tissue samples will be collected at the 28 surface water locations shown on Figures 3-9 and 3-10 and described in Table 3-8. Specific sampling procedures are described in Appendix A.

Fish species representing two trophic levels, if present, will be collected from each sampling station described in Table 3-8. The representative fish species from the lower and upper trophic levels will be determined in the field to facilitate the collection of the same species at the sampling locations. The objective will be to collect coincident species for each trophic level at as

many of the 28 sampling stations as possible so that interstation differences in tissue contaminant levels will be discernible within a particular species and trophic level.

In their 1985 survey, the OEPA selected the white sucker, grasspike, yellow bullhead, and blue gill sunfish for tissue analysis. Examination of the OEPA report of the 1985 survey reveals that the number of resident species range from 17 in the headwaters of the MFLBC above the Salem Wastewater Treatment Plant and the Ruetgers-Nease site to 34 species near the mouth of the MFLBC. Four of the 17 species occurring in the upper reach of the creek also occur at all the selected downstream sampling locations. These four species include the white sucker, creek chub, yellow bullhead, and green sunfish. The U.S. EPA's referenced order of collection was: upper trophic - bass, bluegill, catfish; lower trophic - carp, sucker, bullhead.

Fish will be collected using electroshocking equipment, gill nets, and seines. One or more of each of these methods may be used, either separate, or in combination, depending on the physical characteristics of the water body at each sampling location. To augment the efficiency of the sample collection process, electroshocking will be the primary means of sampling in the MFLBC. Each station will be assessed prior to electrofishing and a reasonable area of stream, usually not exceeding 300 meters will be established as a sampling reach. The reach will be established so as not to overlap with either upstream or downstream stations. Once a reach is established, electrofishing will be conducted beginning at the downstream end of the reach and proceeding upstream. The sampling crew leader will bias the sampling towards optimal or preferred fish habitat in order to obtain sufficient numbers of the preferred (EPA recommended lower and upper trophic level) species. All

fish captured during electrofishing will be placed in clean stainless steel buckets filled with stream water collected at that station. Electrofishing will continue until a sufficient number of preferred fish are obtained (at least 5 of each trophic group), or until the entire reach of stream is shocked. If sufficient numbers of the preferred fish are not obtained during the first pass through of the reach, the team will move back to the starting point and shock the same reach again. This will continue until sufficient numbers of preferred fish species are obtained or until it is obvious that sufficient numbers of fish do not exist at the station. At the conclusion of electrofishing, all collected fish will be identified as to species and counted. If sufficient numbers of fish (at least 5 of each species) in both the upper and lower trophic levels have been obtained, the preferred fish will be weighed and measured to ascertain that at least 150 grams of preferred fish of similar size and age (based upon observation of age classes, a preference will be given to mature fish, e.g., three years of age and older) have been obtained for each sample. If 150 grams have not been obtained, sampling will continue. Excess preferred fish and unpreferred species will be released to the stream. Detailed field notes will be taken at each station. The preferred fish retained for the samples will be rinsed twice with fresh stream water, packaged and labeled and placed in a cooler filled with dry ice. This procedure will be conducted at each station.

Slanker Pond will be sampled using Fyke nets and gill nets placed in appropriate locations within the pond. Nets will be placed at dusk and will be checked at dawn of the following day. If insufficient amounts of fish are present, the nets will be re-set at dusk and checked at dawn until sufficient additional fish are captured. Seines may also be used to sample the near shore areas of the pond.

The expanse of the area sampled for each location will depend on the success of the sampling effort. The sampling effort at each location will continue until sufficient numbers (at least 5 fish of the same species weighing more than 150 grams) of representative fish species have been collected. The fish garnered at each sampling location will be sorted by species, counted, and recorded. After all samples have been collected at a station and prior to proceeding to the next station, the number of each species collected will be determined to allow for the selection of representative species. The U.S. EPA's 1985 referenced order of collection (upper trophic - bass, bluegill, catfish; lower trophic - carp, sucker, bullhead) will be followed in the selection process. The samples from the chosen representative species will be weighed, measured and submitted for laboratory analysis. To prepare the lower trophic samples for shipment to the laboratory, each fish will be washed in stream water, wrapped in aluminum foil (dull side down), tagged, sealed in a plastic bag, and frozen on dry ice. To prepare the upper trophic samples for shipment to the laboratory, each fish will be filleted (skin left on); fillets will be washed with stream water, wrapped in aluminum foil (dull side down), tagged, sealed in a plastic bag and frozen on dry ice. All samples will be shipped by overnight delivery to the laboratory except on occasions of late night or Sunday sampling. On these occasions, refrigeration will occur on-site for any samples requiring this preservation and the samples will be shipped at the next available delivery time. However, the next available delivery day shall not exceed more than one day after sample collection.

Analysis of the lower trophic level fish for contaminants of concern will be performed on whole specimens that will be homogenized by the analytical laboratory. Whole specimens are preferred for this analysis to ensure

that contaminant levels possibly affecting specific target organs within a specimen will not be omitted. Fillets are the primary exposure pathway for humans ingesting contaminated fish; therefore, upper trophic level fish will be filleted by the field crew and submitted for analysis. The detection of contaminants in fillets will provide an indication of the concentrations that are available for human consumption. Parameters to be analyzed are identified in Table 3-6. In addition, lipid content will be determined on both fillet and whole fish specimens. Dioxins/furans, 3,4-DCNB and CLP inorganics may be analyzed for, if they are detected above background levels in on-site samples.

A scientific collecting permit is required by the Ohio Department of Natural Resources Division of Wildlife. Application for the collecting permit will be submitted when a starting date for the project can be established. The application will require a statement of purpose for the collections, type of wildlife to be collected, the ultimate disposition of the samples and recommendations from two well- known scientific persons or teachers of science. The permit remains in effect for one year from the date of issue after which a written report must be filed with the Division of Wildlife of the operations conducted under the permit and the type and number of organisms collected.

Sampling in MFLBC will be one of the initial tasks to be conducted following agency approval of the Work Plan. A technical memorandum reporting analytical results from this task will be submitted to the U.S. EPA/OEPA following data validation.

3.5 Aquifer Testing

At each new and old well cluster, all monitoring wells will be tested by volume-displacement methods after the

first sampling event is completed. Equipment operating procedures and test protocols are described in Appendix A, Section 4.0. Results will be analyzed using the Hvorslev (1951) method or the Bouwer and Rice (1976) method for unconfined conditions and the Cooper, Bredehoft, and Papadopulus (1970) method for confined conditions.

The results of ground water monitoring and the slug tests will be evaluated to determine the need for and location of any long term pump tests that may be required to support the RI/FS and EA reports. If required, these pump tests will be conducted pursuant to the additional work provisions of Paragraph XIII of the Consent Order.

Wells which may be under artesian conditions, (i.e., possibly east of the Crane-Deming plant), and are not amenable to slug test or pump/recovery test may require measurement of the steady state flow from which to assess hydraulic conductivity.

3.6 Soil Hydraulic Conductivity Testing

Soil hydraulic conductivity in the unsaturated zone across the site will be assessed through the measurement of field saturated hydraulic conductivity (K_{fs}) using the constant head permeameter method (Guelph method). In-situ permeameter tests will be conducted at ten locations across the Site. One test will be completed at Pond 1, 3, 4 and 7 near the location where the physical soil samples are collected. Two tests in Exclusion Area "A" and one test in Exclusion Area "B" will be performed in the vicinity of the test pit excavations. Tests will also be completed at three (3) additional locations in and around the known waste management areas, where relatively undisturbed conditions exist. Equipment setup and specific test procedures are described in Section 4.2 of Appendix A. All equipment will

be decontaminated between each test according to the procedures set forth in Section 7.0.

3.7 Topographic Mapping and Surveying

3.7.1 Mapping

Topographic mapping and surveying activities will be completed to update and confirm the existing base maps. The on-Site map will have a horizontal scale of one inch equals 100 feet and contour interval of two feet. The off-Site map will have a horizontal scale of one inch equals 500 feet and a five foot contour interval. Both maps will display cultural features such as buildings, and fences, as well as the location of existing and proposed data-collection sites. Elevations will be referenced to the National Geodetic Vertical Datum (NGVD). These maps will be used to plot additional field data, including sampling locations, and existing and newly installed monitoring wells.

3.7.2 Site Grid

The existing site survey grid will be reestablished using a spacing of 100 feet with the center point at (A,0). Flags will be placed at end lines so that RI/FS activities can be plotted.

3.7.3 Well Location and Elevation Survey

All new monitoring wells will be surveyed to determine vertical elevation and horizontal locations. The base of the protective casing, and the top of the well casing without cap will be used as datum points. Accuracy will be to 0.1 foot horizontal and 0.01 foot for vertical. Only one horizontal measurement will be taken at each location.

3.7.4 Soil Boring and Test Pit Locations

A stake will mark each soil boring and test pit location. At each location the horizontal distances will be determined to the nearest foot and elevations to the nearest 0.1 foot. Horizontal measurements will be referenced to the site specific grid.

3.7.5 Surface Water Elevation Markers

Several surface water elevation markers installed in Feeder Creek and Slanker Pond will be surveyed to determine vertical elevation and horizontal locations. At each location horizontal and vertical occurrences will be 1 and 0.1 foot, respectively.

3.8 Quality Assurance/Quality Control Samples

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

1. 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 day for sediment, soil and biota samples analyzed for volatile organics. A trip blank (unopened CMS and Tenax tube) will also be submitted for air samples.

2. Field Blanks - Field 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 poured over the device, transferred to the appropriate sample bottles, preserved and returned to the laboratory for analysis. One field blank will be collected for each 10 or fewer surface water samples per day. Because dedicated Teflon bailers will be used for each groundwater monitoring well, which eliminates the possibility of cross-contamination between wells, one field blank will be collected per 20 groundwater samples. Solid matrix field blanks prepared by pouring deionized water through the sampling device directly into the appropriate sample bottles will be collected for every 20 soil/sediment samples. Field blanks for air samples will be prepared by removing the caps/covers from the traps and allowing the blanks to passively monitor the sampling event.

3. Field Replicate Samples - are samples from a single source, which are split into two distinct samples, labeled with unique sample numbers, and submitted to the laboratory without cross-referencing data and without identification as replicates on the parameter request sheet. At least one replicate will be prepared for every 10 samples per matrix.

The results of analyses of these QC samples are used as independent, external checks on laboratory and field contamination, and the accuracy and precision of analyses.

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 with the exception of geophysical instrumentation), maintained, and repaired in accordance with manufacturer's specifications. In addition, prior to and after use, each major piece of equipment will and cleaned, decontaminated, checked for damages, and repaired as needed. 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 in Table 4-1.

SSSP TABLE 4-1

EQUIPMENT MAINTENANCE AND CALIBRATION PROTOCOLS
RUETGERS-NEASE SALEM SITE RI/FS

Equipment	Maintenance/Calibration	Frequency
EM34 and 31 conductivity meters	Internal instrumentation is factory calibrated/ routinely maintained.	Every 5 yrs.
	A background conductivity survey will be performed to calibrate the equipment.	Prior to initiation of the geo-physical survey.
Resistivity meter	Internal instrumentation is factory calibrated/ routinely maintained.	Every 5 yrs.
	A background conductivity survey will be performed to calibrate the equipment.	Prior to initiation of the geo-physical survey.
Seismic	Internal instrumentation is factory calibrated/ routinely maintained.	As Required.
PID photoionization detector	Calibrate with isobutylene gas.	Start and end of each. day.
FID flame ionization detector	Calibrate with methane and/or benzene gas.	Start and end of each day.
Explosimeter	Calibrate with methane and carbon monoxide.	Once per month.
	Zero instrument in air.	Start of each day in clean area (e.g., support zone)
pH meters	Calibrate with three pH buffer solutions.	Before and after use, and after every 20 samples

SSSP TABLE 4-1 (cont'd)

EQUIPMENT MAINTENANCE AND CALIBRATION PROTOCOLS
RUETGERS-NEASE SALEM SITE RI/FS

Equipment	Maintenance/Calibration	Frequency
Temperature	Check against a mercury thermometer.	Every 10 samples.
Sp. conductance	Calibrate with one calibration solution.	Before and each use.
Dissolved oxygen meter	Calibration according to manufacturer's recommendations with ambient air.	At the beginning of each day, and every 30 minutes.
Rechargable equipment batteries	Charge	After use as, required.
Sampling accessories (tubing, submersible pumps)	Periodic maintenance performed and recorded in equipment maintenance log.	As required.

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:

1. Is in one's actual possession, (or)

2. Is in one's view, after being in one's physical possession, (or)

3. Is in one's physical possession and then locked up so that no one can tamper with it, (or)

4. Is kept in a secured area, restricted to authorized personnel only.

One member of each 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 ERM-Midwest and RNCC. 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 code, matrix type, sample number code, and depth code.

For the project "RNS" (Ruetgers-Nease, Salem) will designate the Site code. The matrix code will provide a general description of the sample type, examples are shown below. Matrix codes to be used for this investigation are as follows:

GW = Ground Water

SW = Surface Water

SS = Surface Soil

SD = Sediment

SL = Sludge/Waste

AO = Air Organics

AP = Air Particulates

AI = Air Inorganics

SB = Soil Boring

TP = Test Pit

FI = Fish

SG = Soil Gas

The sample number and depth codes will indicate the composite location within the study area and at what depth the sample was collected. For example, a sample on the chain-of-custody log and designated RNS-TP-30-05 would indicate that the sample was collected at the Ruetgers-Nease, Salem facility from test pit number 30 at a depth of five feet.

Soil gas samples will be both collected and analyzed in the field. Therefore, chain-of-custody logs will not be completed, but each sampling station will be identified using the previously described system. Field blanks and trip blanks will be identified by one hundred and two hundred series numbers respectively in place of a depth code. The sample identification numbers will be used in field log books, Chain-of-Custody forms, laboratory results and the final RI report. Each sample will be labeled using waterproof ink immediately after it is collected. 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 wrapped in individual plastic bags and 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:

1. 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.

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

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

4. Place all sample containers in the case.

5. "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.

6. 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.

7. Small containers such as 40 milliliter vials will be placed in small plastic 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.

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

9. The lid of the case will be closed and fastened. Duct tape will be used to seal the seam 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.

10. 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 Special Procedures - Soil Samples For Physical Parameters

In the five ponds on site, one core boring will be completed through each pond and soil samples collected for physical soil analysis. The soil samples may have residual amounts of various organics including mirex, kepone, DPS, 3,4-DCNB, and metals.

The following procedures will allow the safe handling of contaminated soils.

1. Shelby Tubes will be sealed on each end. Once sealed, the sample will be wrapped with aluminum foil, labeled, and placed in wooden boxes for shipment. Vermiculite packing or equivalent will be placed between each sample.

2. A project chain-of-custody and sample analysis request will be filled out and will accompany the samples in the shipping box.

5.5 Sample Shipping

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

1. Laboratory address and sample custodian identification.

2. Date shipped.

3. Return address and Site Manager's identification.

4. 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 RI/FS 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 Ruetgers-Nease 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:

1. Date
2. Time of each data entry
3. Description of work being performed that day
4. Names and affiliations of all personnel at location

5. Weather Conditions on site

6. Location and type of activity (monitor well, surface water sample, etc.)

7. Sample or Boring Methods in use

8. Visual Observations

9. Pertinent field data (pH, specific conductance, temperature, and any other field measurements such as from an FID or Explosimeter).

10. Serial numbers, if any, on seals and transportation cases.

11. Name of field custodian

12. 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.

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 Sample Log Book

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

1. Unique Sample number

2. Sample Date

3. Sampler's Initials

4. Sample Matrix (soil, water, etc.)

5. Number of samples

6. Analyses performed

7. Further analyses required

8. Date shipped to the lab

9. Method of shipment

6.4 Photo-Documentation

All photographers 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.

6.5 Correspondence/Communications

All documents including field notes will be copied at the field office once a week and will be checked for completeness and filed. Filing cabinets will hold files in the field office. 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 drilling and sampling equipment. Detailed personnel decontamination procedures are discussed in Section 9.0 of the Health and Safety Plan. Decontamination protocols will be strictly adhered to in order to minimize the potential for cross-contamination between sampling locations and contamination of areas off-site.

7.1 General Considerations

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

1. All decontamination and subsequent use of decontaminated equipment will be documented in a field book.

2. 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.

3. All spent wash and rinse waters will be collected, and transferred to and stored in an on-site tank pending proper disposal. Depending on the contamination found in the samples, the water may be discarded on-site or taken off-site for appropriate treatment and disposal.

4. Verification of the sampling equipment cleaning procedures will be documented by the collection of field blank samples.

5. 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 (Volume 4).

6. All properly decontaminated equipment will be stored in plastic bags (if possible) when not in use.

7. 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.

8. 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:

1. Remove all loose soil from equipment with a brush.

2. Steam or high pressure wash using non-phosphate soap.

3. Potable water rinse.

4. 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:

1. Remove all loose soil.

2. Steam or high pressure wash using non-phosphate soap.

3. Potable water rinse.

4. 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.

All cleaned equipment will be transported and stored on plastic sheets.

7.3 Sampling Equipment

Equipment used to collect environmental samples will be cleaned prior to its initial 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

Soil sampling equipment will be decontaminated at the sample locations. 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. Remove 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 Ground Water, Surface Water and Fish Sampling

Equipment used for ground water, surface water and fish 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, bailers, fillet knives, buckets and trays. The following procedures will be used for decontaminating equipment:

Inorganics	Organics
1. Remove loose soil/solid	1. Remove 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, eh, Temperature, Dissolved Oxygen and Depth to Water Probes

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

1. Wash with non-phosphate detergent solution.

2. Potable water rinse.

3. Deionized water rinse.

All equipment will be transported and stored in plastic sheeting.

7.3.4 Soil Gas Probe

The soil gas probe used during the survey will be decontaminated using the procedures specified below:

1. Remove loose soil.

2. Non-phosphate soap wash.

3. Potable water rinse.

4. Deionized water rinse.

5. Field scan with PID.

7.4 Monitor Well Materials

Prior to use, well screens, riser pipes, and outer casings will be steam cleaned at the decontamination area, wrapped in plastic sheeting, and stored in the warehouse.

7.5 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 each day. The procedure for decontaminating this equipment is a follows:

1. Remove particulate contamination.

2. Wipe down with clean damp cloth (deionized water).

3. Air dry.

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

Site Specific Sampling Plan Appendix A: Sampling and Field Testing Procedures

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1.0 INTRODUCTION

The RI objectives are to collect data of adequate technical content, quality and quantity to:

- Determine the characteristics, extent and magnitude of contamination on and off the Site.
- Determine if contaminants at the Ruetgers-Nease Site pose a threat to human health or the environment through the development of an endangerment assessment.
- Identify the pathways of contaminant migration from the Site, and characterize the contaminant flux across the Site boundaries.
- Quantify existing and potential future endangerment for each contaminant pathway.
- Evaluate the nature and magnitude of contamination, if any, in any nearby private water wells.
- Define the site's physical features and facilities that could affect contaminant migration, containment, or clean-up.
- Develop, screen and evaluate potential remedial action alternatives.
- Recommend the most cost-effective remedial action alternative(s) that adequately protect health, welfare and the environment.

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

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

2.0 GROUND WATER 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.

2.1 Monitoring Well Purging

All ground water 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:

1. Inspect well for effects of tampering.

2. Measure inside diameter of well casing.

3. Measure depth to water and total depth of well from the top of the well casing.

4. 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.

5. 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.

6. 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 SSSP.

2.2 Monitor Well Sample Collection

The following procedures will be used to collect samples from monitoring wells after the well has been purged.

1. Remove and inspect sample containers, sample forms, and chain-of-custody forms for consistency with sample location.

2. Attach 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.

3. Retrieve bailer and slowly transfer the sample to 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, pesticide/PCB, 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.

4. Label sample containers with time and date of collection.

5. Place sample in the appropriate shipment containers.

6. Wrap bailer in aluminum foil.

7. Replace well cap and secure the protective lid.

2.3 Potable Well Sampling

The following procedures will be used to collect samples from potable wells.

1. Locate faucet or tap closest to well prior to any water softeners.

2. Turn off or remove in line water conditioners (i.e., water softener, filters, etc.) and remove, if present, the aerator from the faucet.

3. If the well is active, an appropriate volume of water in the supply line preceding the tap will be removed until there is a stable temperature, pH, and conductivity or the faucet will be run for five minutes which ever is more. If the well has been inactive for more than one month prior to sampling, it will be purged until stable temperature, pH, and conductivity readings are obtained. At a minimum, an estimated three well volumes will be removed, or

the faucet will be run for 10 minutes, which ever is more.

4. Fill sample containers as described in Section 2.2 Step 3.

5. Follow Steps 3 through 5 as described in Section 2.2.

2.4 Surface Water Sampling

Surface water samples will be collected prior to sediment and fish samples according to the following procedures:

1. Commence sampling at the furthest downstream point and continue sampling moving upstream. Identification flags will mark each location for future sampling.

2. Remove sample containers, sample forms, and chain-of-custody forms and check for consistency with sample location.

3. 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.

4. Transfer sample into appropriate sample containers.

3.0 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.

3.1 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 test pit locations, and split spoon (for chemical analysis samples) 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 sampled; and field conditions. The following describes the sample collection procedures for each piece of equipment that may be utilized.

3.1.1 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 used 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 soils surficial sample collection, test pit investigations or collection of sediment samples. The following technique is used in obtaining a surface soil sample:

1. Using a decontaminated trowel, obtain soil from the required depth at the proper location.

2. 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.

3. Close container, label in indelible ink, place in a plastic bag, and seal.

4. Complete COC label for analysis desired.

5. Preserve and/or refrigerate to $4^{o}C$.

6. Record appropriate data and information into project log book.

7. Decontaminate equipment prior to next use or before storage.

3.1.2 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. A hand auger may be used during collection of off-site soil samples or when sediment samples are collected. The following technique is used when using a hand auger:

1. Clear the surface of any debris that may impede the auger's penetration.

2. Place stainless hand auger perpendicular to the ground and rotate the auger while exerting pressure on the hand bar until the desired depth.

3. 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.

4. Close the sample container, label in indelible ink, place in a plastic bag, and seal.

5. Complete COC, label and specify analysis desired.

6. Preserve and/or refrigerate to $4^{o}C$.

7. Record appropriate data and information in project log book.

8. Decontaminate equipment prior to next use or before storage.

3.1.3 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 trier may be used during the off-site soils investigation and during sediment sample collection. The procedure for sampling is similar to the hand auger sampling procedure:

1. Place decontaminated trier perpendicular to the sampling location.

2. Press down while rotating trier.

3. After reaching the desired depth, rotate the trier out in the opposite direction.

4. Inspect contents of trier and transfer sample to laboratory supplied containers, using a stainless steel spatula.

5. Attach labels and complete chain-of-custody protocols.

6. Seal, preserve and/or refrigerate sample at $4^{o}C$.

7. Record data and appropriate information in project log book.

8. Decontaminate trier equipment prior to next use or before storage.

3.1.4 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 soils for physical parameters such as moisture content and Atterburg Limits. Therefore, shelby tubes are usually only used once per sampling round or event. Shelby tubes, at a minimum, will be used for collection of on-site soils for physical parameter analysis. The following technique is used when employing a shelby tube to sample soils:

1. Position the shelby tube perpendicular to the surface of the soil to be sampled.

2. Push the tube into the soil without twisting or disrupting the soil.

3. 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.

4. After the desired depth has been obtained the tube will be rotated in order to shear the soil core off.

5. 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.

6. The shelby tube will be labeled and receive chain-of-custody protocols.

7. The shelby tube will be placed in a container for shipment.

8. Record all appropriate data and information per sample in the project log book.

3.1.5 Split Spoon Sampling

Split spoon sampling is a drilling technique employed to sample subsurface soils. The split spoon is similar to a shelby tube, but the driven end is attached to downhole rods and driven into the ground by a drill rig. Split spoon samples may be collected from on-site borings, off-site boring and during drilling of monitoring wells. The following procedures will be followed when collecting samples using a split spoon:

1. Lower the split spoon sampler to the bottom of a borehole.

2. Mark the drill rods in 6-inch increments above a fixed datum.

3. Drive the sampler downward with blows from a 140-pound hammer falling 30 inches onto the drill rod collar. (Verify the hammer weight and length of fall on each rig before the first test.)

4. Record the number of blows required to drive the sampler each 6-inch increment. The "blow count" is the total number of blows required to drive the sampler the last foot.

5. Retrieve samples from borehole and open it on a clean working surface.

6. Slice sample into thirds and transfer a portion of the third with the highest FID/PID finding into a container for analysis for CLP volatile organics and library searches for up to 15 compounds. If there are no observed differences in OVA responses between subintervals, the middle subinterval will be sampled.

7. Transfer the remaining sample to a clean stainless steel bowl, mix thoroughly and fill remaining sample containers in the same order as ground water (Section 2.2, Step 3) using a stainless steel spatula to fill containers for non-volatile organics plus a library search for up to 25 additional compounds plus mirex, photomirex, kepone, DPS (as required) and inorganic samples.

8. Label sample containers and follow chain-of-custody procedures.

9. Preserve accordingly and store in shipping containers.

10. Record all appropriate information on field book.

11. Decontaminate split spoon prior to next usage.

3.1.6 Test Pit Excavation and Sampling

Test pits are trenches that are dug using a backhoe with a decontaminated bucket. Test pits are useful in identifying soil strata, and retrieving samples from only the strata that is of interest or which indicates contamination via use of organic vapor meters. The following procedure is to be used when excavating test pits:

1. All excavated soils will be placed upon a sheet of heavy duty plastic.

2. Samples will be collected from the excavation wall by cutting out a block of soil using a stainless steel knife.

3. Sampling after 6.5 feet will be done solely from the backhoe bucket if conditions warrant and will continue until the desired depth has been reached.

4. Sampling will continue past 6.5 feet until bedrock or the water table is encountered, or the total organic vapor content of the soil six inches below the bottom of the pit is less than 10 ppm during soil screening with either an FID or PID organic vapor meter.

5. Samples will be collected with decontaminated tools such as a stainless steel trowel or stainless steel spoon.

6. Soil for volatile organic analysis will be collected from the test pit wall directly into the sample container using a stainless steel spatula. All other soil will be collected into a stainless steel bowl and mixed thoroughly. Soil will then

be transferred into the appropriate sample containers using stainless steel and plastic spatulas for organic and inorganic sample, respectively.

7. Containers will then be sealed, labeled, preserved and have all chain-of-custody protocols completed.

8. All data and appropriate information will be written in the project log book.

9. Excavated soils will be replaced in reverse order of removal as the test pit is backfilled.

3.2 Sediment Sampling

Sediment samples can be obtained using the same methods 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 depths. Sediment samples will be collected in areas of deposition in the vicinity of the surface water sample. General sampling procedures for sediment are as follows:

1. Identify the collection point location.

2. Using a decontaminated stainless steel sampling tool, retrieve sample from upper six inches of sediment at the location using procedures described in Section 3.1.

3. Transfer sample into a clean stainless steel bowl after filling the volatile organic sample containers.

4. Thoroughly mix the sample in the bowl and fill the remaining sample containers in the appropriate order.

5. Seal container, attach completed label and all chain-of-custody protocols, and specify analysis required.

6. Preserve and/or refrigerate at $4^{o}C$.

7. Record all data and appropriate information in the project log book.

8. Decontaminate all sampling equipment before next use and/or before storage.

3.2.1 Pond Bottom

Samples of pond bottom sediments will be collected using a ponar dredge from a small boat. 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:

1. Attach a clean sample line to the dredge (i.e., nylon).

2. Measure distance to bottom sediments with a weighted tape.

3. Open sampler until the jaws are latched.

4. 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.

5. Allow sample line to slack several inches.

6. Slowly raise sampler to surface and place in a clean stainless steel bowl.

7. Open dredge and transfer sediment to the appropriate sample containers using a clean stainless steel spatula filling the containers for volatile organic analysis first.

8. Seal and label container, and complete chain-of-custody procedures.

9. Record all data and appropriate information in the project log book.

4.0 FIELD TESTING

To ensure that data collected during field testing are representative and comparable, the procedures described in the following procedures will be implemented during the RI/FS.

4.1 Geophysical Surveys

When conducting geophysical surveys using the Geonics EM-34 or 31 conductivity meter for the procedures described in the following subsections will be implemented.

4.1.1 Conductivity

The EM-34-3 conductivity meter is operated according to the following procedures:

1. Using the appropriate wires, connect the transmitter and receiver units to their respective coils. Connect the intercoil wire of desired length (10, 20, 40 meters) from the transmitter coil to the receiver unit. All wires have unique connectors, so the units cannot be assembled improperly. Turn both units on.

2. Battery condition in the transmitter is continuously indicated by a gauge on the unit face. If needle deflection is not near full-scale, batteries must be replaced. On the receiver, batteries are checked by placing the range switch in the BATTERY position. If the needles of both gauges read inside the "BATT"

marks, batteries are in good condition, otherwise they need to be replaced.

3. Electronic calibration, if necessary, is done by disconnecting the receiver coil, and then adjusting the NULL control to obtain zero readings on the gauges.

4. Carrying the units by shoulder straps, center the intercoil wire at a measurement station. Turn the range switch so that the meter reads in the upper two thirds of the scale. Full-scale deflection is indicated by the range switch, and terrain conductivity can be read directly from the gauge in millimhos per meter. To center the "coil separation" gauge, move the receiver coil back and forth slightly until the needle centers. Holding the coils vertically will measure conductivity to a depth D = coil separation X 0.75. Laying the coils horizontally on the ground measures conductivity to a depth of D = separation X 1.5.

The EM31 conductivity meter is operated according to the following procedures:

1. Using the identifying labels on the tubes align the transmitter coil tube with respect to the main tube and fix it with the clamp.

2. Check battery condition, plus and minus, by setting the mode switch to the "OPER" position and the range switch to the "+B" and the "-B" positions respectively. If needle reads inside the "Batt" mark on the meter, batteries are in good condition, otherwise replace the batteries with a fresh set of "C" size alkaline batteries.

3. Electronic nulling of the instrument, if necessary, is done by setting the Mode switch to the "OPER" position, setting the range switch to the least sensitive position (1000 millimhos/meter), and then adjusting the "NULL" control to obtain zero reading. (See note Section 3.2)

4. Align and connect the receiver coil tube to the main tube. Ensure that the mode switch is set to the "OPER" position.

5. Wearing the instrument as shown in the data sheet with the shoulder strap adjusted so that the instrument rests comfortably on the hip, switch the Mode switch to the "OPER" position and rotate the range switch so that the meter reads in the upper two thirds of the scale. The full scale deflection is now indicated by the range switch and the instrument is reading the terrain conductivity directly in millimhos per meter.

6. In moving to the next measurement station the Mode switch may be left in the "OPER" position to provide a continuous reading of the terrain conductivity. The instrument has a time constant of approximately one second to which the operator should adjust his walking speed for the greatest accuracy.

7. Alternately, to extend battery life, the instrument can be switched on at each measurement station. The operator will notice that the type of integrator used results in a slight initial overshoot of the needle, which is normal, and that approximately two seconds after switch-on the measurement can be recorded.

4.2 In Situ Testing

To ensure the collection of accurate and representative data when conducting in situ tests on monitoring wells and in soil, the procedures described in the following text will be implemented during the investigation.

4.2.1 Hydraulic Conductivity Testing

The procedures below will be utilized in conducting a slug test with the suitcase cone recorder, the Transducer interface and the Druck Transducer.

A. Slug Test

1. On the back of the recorder, place the cone function switch in the off position (up) and the power switch in the "battery" position (up).

2. On the front of the recorder set the paper speed switch on cm/min and the selector above it on 30. Turn the power switch on. Remove the plastic cap from the pen tip. With the span switch "off", take a screwdriver and adjust the zero control until the pen is in the middle of the paper (50). Set the span select knob on 10. Now put the span switch on MV.

3. Plug the two pin banana plug from the interface (Black Box) into the recorder just below the zero control with the ground tap on the plug to the right. Plug the transducer into the interface socket. Turn the interface power switch to "Battery" and adjust the "Zero Adjust" knob on the interface until the pen is back on 50 or as close as possible.

4. Measure the static water level in the well with a cleaned steel tape or electronic measuring device and record.

5. Lower the transducer slowly into the well, watching the recorder pen as you do. When the transducer reaches the water surface the pen will move to the left. Make a note of this depth and now lower the transducer another 10 or 15 feet. The transducer can be damaged if it is lowered more than 20 feet below the water surface. Readjust the zero knob on the interface to bring the pen back to 50.

6. To calibrate the system, turn the chart switch on, make a final zero adjustment to get the pen on 50 and then lower the transducer one foot, return the transducer to its original position and then raise it one foot. Repeat this procedure with the span switch on 5 then 20. Turn charge switch off and write the span and paper speed settings on the test record. Now secure the transducer cable to keep the transducer at this level.

7. Slowly lower the slug to the water surface, watching for motion of the pen. Raise the slug an inch or two above water and hold it there with one hand. Turn the chart switch on and then with your free hand grab the slug rope a little more than one slug length above the other hand, and allow the slug to drop this distance. Care must be taken not to drop the slug to the level of the transducer as damage to the transducer could result.

8. Examine the resulting trace on the recorder chart. The trace should go to about 100 and return slowly

to 50. If not, readjust the span and try again. If the trace only goes to 60 or 70, set the span on 5. If the trace goes of scale, set the span on 20.

9. When the proper scale has been determined, run the test by dropping the slug with the chart switch on, as described above, wait for the trace to return to 50 (or very close) and then pull the slug up out of the water as quickly as possible. The pen will then go to the right. Let this chart run until the pen returns to 50. Be sure to note paper speed, span setting, and slug dimensions, on all records and in the bound project log book.

4.2.2 Guelph Permeameter

The following operating procedures for conducting a hydraulic conductivity test in soil using constant head well permeameter will be followed for this investigation:

1. Advance test hole to desired depth into the unsaturated zone.

2. Install the clean permeameter into the test hole and fill the reservoir with water. The air-inlet tube should be pushed down into the port to prevent flow out of the meter.

3. Pull air-inlet tube upward to produce the desired water level in the well (H) (usually 10-20 cm).

4. Measure and record rate of fall of water surface in meter reservoir until steady state is achieved.

5. Remove permeameter and backfill test hole.

6. Decontaminate equipment and move to next test hole.

4.3 Air Monitoring

To ensure that air monitoring data are collected properly, the following procedures will be implemented during the RI/FS.

4.3.1 Soil Gas Survey

The soil gas survey will employ three types of instruments capable of quantifying volatile organic compounds (VOC) present in the pore space of the near surface soils. The FID and PID organic vapor meters will be used to delineate areas where VOC levels are elevated above background. The gas chromatograph attachment to the FID will be used to identify whether the parameters of interest are present at locations of elevated VOC levels as well as the concentration of these parameters. Indicator parameters will be volatile organic compounds chosen from Table SSSP 1-1 that have high vapor pressures and mobility characteristics representative of those compounds listed on Table SSSP 1-1. The following procedures will be implemented to ensure the collection of the high quality data during the survey.

1. Advance the 1/2 inch diameter probe with sampling tip attached using a hammer drill.

2. Attach the FID to the sample port and purge the sample probe until a stabilized VOC level is measured. Both maximum and stabilized readings will be recorded. The FID portable GC attachment will then be used to record a sample spectrum.

3. Disconnect the FID and attach the PID to the sample port and measure the maximum and stabilized VOC levels.

4. Remove and decontaminate the sample probe, auger, sample port, PID and FID according to Section 7.0. of the SSSP. Discard the sample tubing attached to the sample port.

5. Screen the sample probe/port, and auger for residual contamination. If not fully clean, decontaminate again followed by an additional screen.

4.3.2 Explosimeter

An explosimeter is a personal air monitoring device. Three independent sensors simultaneously monitor the ambient air for the amount of toxic gas, combustible gas and oxygen (O_2) deficiency. The instrument may provide audio and visual alarms if concentrations of toxic or combustible gas becomes too high, and if the oxygen level is lower than the level necessary for normal breathing.

The following procedure will be implemented while operating the Enmet CGS-80 Tritector explosimeter:

The Enmet CGS-80 Tritector emits a high-pitched fluttering tone and red light when hazardous gas levels (toxic and combustible) exceeds the alarm points. When the oxygen level of the ambient air drops below the alarm level, the Tritector emits a steady high-pitched tone and red light.

During normal operation, in a non-alarm condition, the unit "chirps" softly and the red light blinks every eight seconds.

CHARGING THE UNIT

1. Make sure the instrument is off and connect the charger.

2. The green light on the charger goes out or slows to a pulse when unit battery is fully charged. The unit should operate 12 to 14 hours, continuously, after the battery is fully charged.

3. The unit will emit a distant-sounding tone, and steady amber light when the battery charge is low.

OPERATION OF THE UNIT IN A CLEAN AIR ZONE

1. Pull the locking toggle switch out and up into the TOXIC mode. Hold the PURGE/AUDIO OFF switch in the purge switch for one to five minutes.

2. When the TOXIC graph bars disappear, release the purge switch.

3. Allow 10 minutes for sensors to stabilize.

4. After the 10 minutes has expired, set the oxygen bar graph to 21 percent. This is achieved by pressing the oxygen calibration knob in while your turn it.

5. Move the function switch to whatever hazardous gas you want to be displayed (TOXIC or COMB). The unit alarms for both types of gases independent of what is selected with the function switch. Always allow the unit to adjust to temperature changes in

the ambient air. A change in temperature may cause an oxygen alarm. See the Tritector operation and maintenance manual for trouble-shooting procedures and further details on unit operation.

4.3.3 Flame Ionization Detector (FID) and Optional Gas Chromatograph (GC) Attachment

Note: The information in Section 4.3.3 is taken from the operational procedures manual, "Model OVA 128 Century Organic Vapor Analyzer," Foxboro Company, December 1985.

4.3.3.1 Introduction

GENERAL DESCRIPTION

The OVA 128 is a sensitive instrument designed to measure trace quantities of organic materials in air. It has broad application because it has a chemically resistant sampling system and can be calibrated to almost all organic vapors and gases found in most industries. The instrument has the sensitivity to measure organic compounds in the parts per million range (V/V) in the presence of atmospheric moisture, nitrogen oxides, carbon monoxide, and carbon dioxide.

The instrument has a single linearly scaled readout from 0 to 10 ppm with a X1, X10 and X100 range switch. This range expansion provides accurate reading across a wide concentration range with either 10, 100 or 1000 ppm full scale deflection.

In areas where mixtures of organic vapors are present, it often becomes necessary to determine the relative

concentration of the components and/or to make quantitative analysis of specific compounds.

To provide this capability, a gas chromatograph (GC) option is available. When the GC option is used, the capability of the OVA includes both qualitative and on-the-spot quantitative analysis of specific components present in the ambient environment.

The OVA 128 is certified by Factory Mutual Research Corporation for use in Class I, Groups A, B, C, & D, Division I hazardous locations. Instruments with this certification must be incapable, under normal or abnormal conditions, of causing ignition of hazardous mixtures in the air.

OPERATIONAL PRINCIPLE

The instrument utilizes the principle of hydrogen flame ionization for detection and measurement of organic vapors. The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated. During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system. The system stream is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber, the sample is exposed to a hydrogen flame which ionizes the organic vapors. When most organic vapors burn, they leave positively charged carbon-containing ions. An electric field drives the ions to a collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated. This current is measured with a linear electrometer preamplifier which has an output signal proportional to the

ionization current. A signal conditioning amplifier is used to amplify the signal from the pre-amp and to condition it for subsequent meter or external recorder display.

INSTRUMENT SENSITIVITY AND CALIBRATION

In general, the hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives repeatable results with all types of hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

Compounds containing oxygen, such as alcohols, ethers, aldehydes, carbolic acid and esters give a lower response than that observed for hydrocarbons. Nitrogen-containing compounds (i.e., amines, amides, and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Table 4-1 lists the responses and retention times of various compounds relative to methane which is typically used as a reference standard for calibration purposes.

There are two types of operation that are used for calibration. In type one, a non-regulatory (or non-target) compound such as methane is used for calibration. In this case, the instrument reading is reported in terms relative to the calibration compound used for calibration. For type two, the target compound or compounds are used for calibration. As a result, the instrument is calibrated to respond directly in ppm by volume of the target compound(s). For this

TABLE 4-1

RELATIVE RESPONSE CALIBRATED TO METHANE AND CHROMATOGRAPHIC RETENTION TIME FOR COMPOUNDS THAT HAVE BEEN QUALITATIVELY IDENTIFIED AT THE RUETGERS-NEASE SITE PRIOR TO THE RI/FS

COMPOUND	RELATIVE RESPONSE (%)	RETENTION TIME IN MINUTES AT 0°C WITH T-8 COLUMN
Benzene	150	1:43
Chlorobenzene	200	11:20
Chloroform	65	2:00
1,3+1,2-Dichlorobenzene	50	(1)
	113	(2)
1,2-Dichloroethane	80	3:50
1,1-Dichloroethene (vinylidene chloride)	40	22
1,2-Dichloroethene	50	31 (3)
1,2-Dichloropropane	90	2:56
1,3-Dichloropropene	(4)	(4)
Ethylbenzene	100	7:44
1,1,2,2-Tetrachloroethane	100	50:00 (3)
Tetrachloroethene	70	2:10
Toluene	110	4:30
1,1,1-Trichloroethane	105	:47
Trichloroethene	70	1:24
m-Xylene	111	8:31
o-Xylene	116	8:40
p-Xylene	116	8:23

(1) T-8 column will not identify this compound

(2) Retention time not available from manufacturer

(3) Due to field time constraints, analysis time in GC mode will be limited to 25 minutes; thus this compound will not be identified

(4) Relative response and retention time not available from manufacturer

(4) G-8 column at 40 °C retention time 2:37

investigation, the instrument will be calibrated using methane. Specific calibration instructions are presented in Section 4.3.3.2 of this operational procedure.

INSTRUMENT SPECIFICATIONS

Performance

Readout:	0 to 10, 0 to 100, 0 to 1000 ppm (linear)
Sample Flow Rate:	1 1/2 to 2 1/2 liter per minute at 22°C, 760 mm, using close area sampler
Response Time:	Approximately 2 seconds for 90% of final reading.
Hydrogen Flow Rate:	Factory set 12.5 ±0.5 mL/min (minus GC option); 11.0 ±0.5 mL/min (GC models).
Filters:	In-line sintered metal filters will remove particles larger than 10 microns.
Operating Temperature Range:	10°C to 40°C
Minimum Ambient Temperature:	15°C for Flame Ignition (cold start)
Accuracy:	Based on the use of calibration gas for each range:

Calibration Temp. °C	Operating Temp. °C	Accuracy in % of Individual Full Scale X1	X10	X100
20 to 25	20 to 25	±20	±10	±10
20 to 25	10 to 40	±20	±20	±20

Relative Humidity: 5% to 95%, Effect on accuracy: ±20% of individual scale.

Minimum Detectable Limit (Methane): 0.2 ppm.

Power Requirements and Operating Times

Primary Electrical Power: 12 volt (nominal) battery pack

Fuel Supply: Approximately 75 ml volume tank of pure hydrogen, maximum pressure 2400 psig, fillable in case.

Portable Operating Time: Minimum 8 hours with battery fully charged, hydrogen pressure at 1800 psig.

Battery Test: Battery charge condition indicated on readout meter. Upon activation of momentary contact switch, a meter reading above the indicator line means that there is four hours minimum service life remaining (at 22°C).

4.3.3.2 Operational Procedure

STARTUP PROCEDURE

Refer to Figure 4-1 for assembly, jack, switch and dial locations and nomenclature.

a) Connect the Probe/Readout Assembly to the Sidepack Assembly by attaching the sample line and electronic jack to the Sidepack.

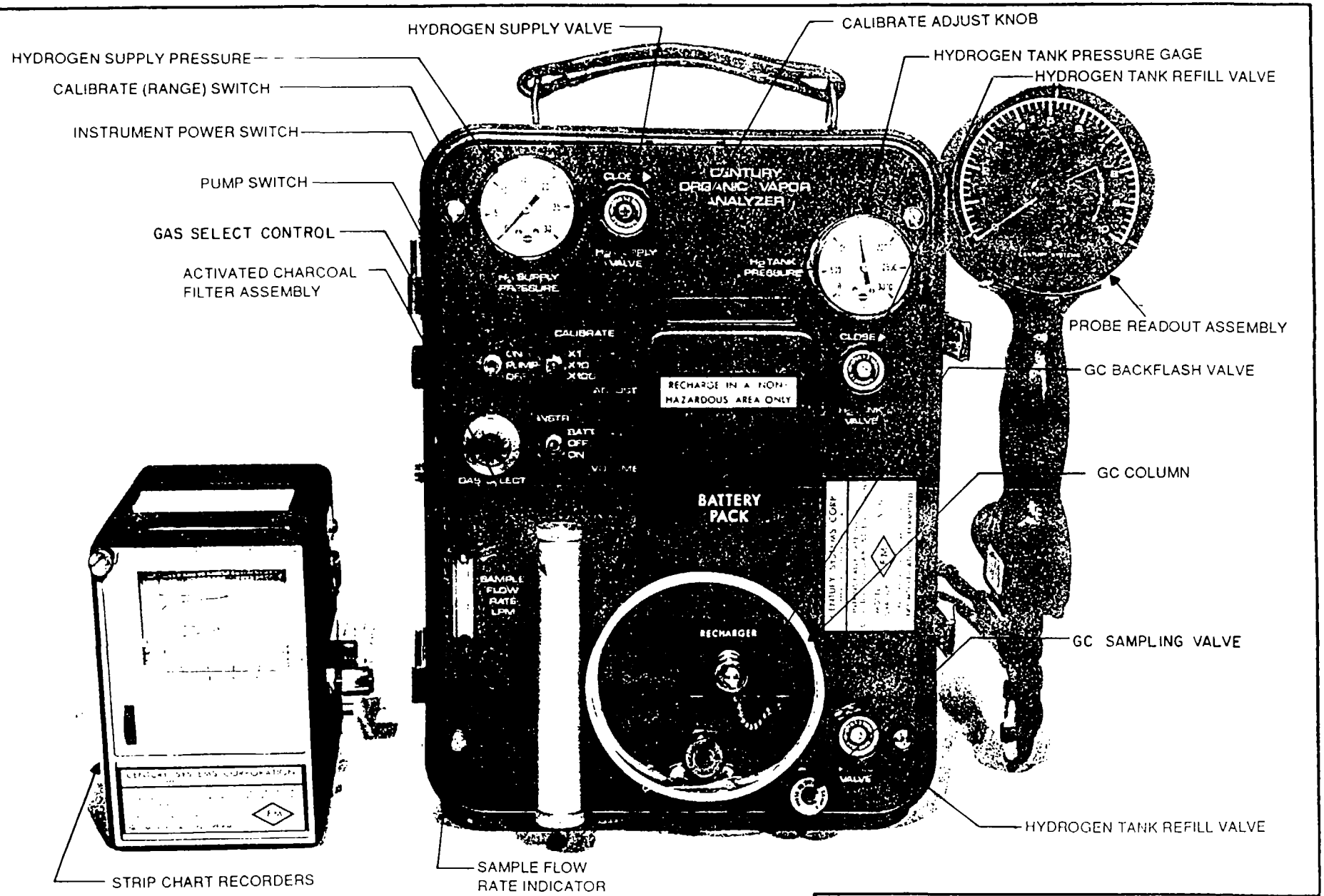
b) Select the desired sample probe (close area sampler or telescoping probe) and connect the probe handle. Before tightening the knurled nut, check that the probe accessory is firmly seated against the flat seals in the probe handle and in the tip of the telescoping probe.

c) Move the Instr/Batt Switch to the "test" position. The meter needle should move to a point beyond the white line, indicating that the integral battery has more than 4 hours of operating life before recharging is necessary.

d) Move the Instr/Batt Switch to the "ON" position and allow a 5 minute warm-up.

e) Turn the Pump Switch on.

f) Use the Calibrate Adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the Calibrate Switch must be set to the appropriate range.



NOTE TRIMPOTS R-31, R-32 AND R-33 USED FOR CALIBRATION ARE ACCESSED BY UNTIGHTENING THE FOUR KNURLED KNOBS ON THE FRONT PANEL AND REMOVING THE INSTRUMENT FROM THE PROTECTIVE CASE THE TRIMPOTS R-31, R-32 AND R-33 ARE LOCATED ON THE UNDERSIDE OF THE PANEL

ADDITIONAL CONTROLS AND COMPONENTS-GC OPTION
RUETGERS-NEASE CHEMICAL CO. INC
SALEM, OHIO

FIGURE 4-1

ERM–Midwest, inc.

g) Turn the Volume knob fully clockwise.

h) Using the Alarm Level Adjust knob, turn the knob

i) Move the Calibration Switch to X1 and adjust the meter reading to zero using the Calibration Adjust (zero knob).

j) Open the hydrogen Tank Valve 1 or 2 turns and observe the reading on the Hydrogen Tank Pressure Indicator. (Approximately 150 psi of pressure is required for each hour of operation).

k) Open the Hydrogen Supply Valve 1 or 2 turns and observe the reading on the Hydrogen Supply Pressure Indicator. The reading should be between 8 and 12 psi.

l) After approximately one minute, depress the Igniter Button until the hydrogen flame lights. The meter needle will travel upscale and begin to read "Total Organic Vapors." Caution: Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.

m) The instrument is ready for use. Note: If the ambient background organic vapors are "zeroed out" using the Calibrate Adjust knob, the meter needle may move off-scale in the negative direction when the OVA is moved to a location with lower background. If the OVA is to be used in the 0 to 10 ppm range, it should be "zeroed" in an area with very low background. A charcoal filter can be used to generate the clean background sample.

OPERATING PROCEDURE

The following procedure describes operation of the OVA in the "Survey Mode" to detect total organic vapors. Procedures for operation in the "Gas Chromatograph Mode" are explained in Section 4.3.3.4.

a) Set the calibrate switch to the desired range. Survey the areas of interest while observing the meter and/or listening for the audible alarm indication. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. For broad surveys outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative reading or pinpointing, the pickup fixture should be positioned at the point of interest.

b) When organic vapors are detected, the meter pointer will move up-scale and the audible alarm will sound when the setpoint is exceeded. The frequency of the alarm will increase as the detection level increases. If the flame-out alarm is actuated, check that the pump is running, then press the igniter button. Under normal conditions, flame-out results from sampling a gas mixture that is above the lower explosive level which causes the hydrogen flame to extinguish. If this is the case, re-ignition is all that is required to resume monitoring. Another possible cause for flame-out is restriction of the sample flow line which would not allow sufficient air into the chamber to support combustion. The normal cause for such restriction is a clogged particle filter.

It should be noted that the chamber exhaust port is on the bottom of the case and blocking this port with the hand will cause fluctuations and/or flame-out.

SHUT DOWN PROCEDURE

The following procedure should be followed for shut down of the equipment:

a) Close hydrogen tank valve

b) Close hydrogen tank supply valve

c) Move instr switch to off

d) Wait 5 seconds and move pump Switch to off. Instrument is now in a shut down configuration.

CALIBRATION

Primary Calibration for Methane

The accuracy stated in the instrument Specifications is obtained when the instrument is calibrated with known concentrations of methane for each concentration range. Prepare separate samples of methane-in-air in these concentration ranges: 7 to 10 ppm, 90 to 100 ppm, and 900 to 1000 ppm. Calibrate the instrument as follows:

a) Place the instrument in normal operation and allow a minimum of 15 minutes for warm-up and stabilization.

b) Set the gas select control to 300

c) Set the calibrate switch to X1

d) Set the calibrate adjust (zero) knob so that the meter reads zero.

e) Check that the meter reads zero on the X10 and X100 ranges.

f) Set the calibrate switch to X1 and introduce the sample with known concentration in the 7 to 10 ppm range.

g) Adjust electronic potentiometer R31 with a small screwdriver (inside of protective case, see Figure 4-1) so that the meter reading corresponds to the sample concentration.

h) Set the calibrate switch to X10 and introduce the sample with known concentration in the 90 to 100 ppm range.

i) Adjust R32 (see Figure 4-1) so that the meter reading corresponds to the sample concentration.

j) Set the calibrate switch to X100 and introduce the sample with known concentration in the 900 to 1000 ppm range.

k) Adjust R33 (see Figure 4-1) so that the meter reading corresponds to the sample concentration.

l) The instrument is now calibrated for methane and ready for service.

Using Empirical Data

Relative response data can be used to estimated the concentration of a vapor without the need to recalibrate the

analyzer. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The response factor (R) in percent, for the vapor is:

$$R = \frac{\text{Actual Concentration}}{\text{Measured Concentration}}$$

To determine the concentration of an unknown sample of that vapor, multiply the measured concentration by R.

4.3.3.3 Maintenance and Trouble-Shooting

GENERAL MAINTENANCE

Fuel Refilling

It is important to note that for proper operation and instrument accuracy, use of pre-purified or zero grade hydrogen (certified total hydrocarbons as methane <0.5 ppm) is recommended.

a) The instrument and the charger should be completely shut down during hydrogen tank refilling operations. Refilling should be done in a ventilated area. There should be no potential igniters or flame in the area.

b) If you are making the first filling on the instrument or if the filling hose has been allowed to fill with air, the filling hose should be purged with hydrogen prior to filling the instrument tank. This purging is not required for subsequent fillings.

c) The filling hose assembly should be left attached to the hydrogen supply tank when possible. Ensure

that the fill/bleed valve on the instrument end of the hose is in the off position. Connect the hose to the refill connection on the Side Pack Assembly.

d) Open the hydrogen supply bottle valve slightly. Open the refill valve and the hydrogen 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 be indicated on the hydrogen tank pressure indicator.

e) After the instrument fuel tank is filled, close the refill valve on the panel, the fill/bleed valve on the filling hose assembly and the hydrogen supply bottle valve.

f) 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 (g) below, because the hose will contain a significant amount of hydrogen at high pressure.

g) 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.

h) Close the hydrogen tank valve.

i) With the hydrogen tank valve and the hydrogen supply valve closed, a small amount of hydrogen at high pressure will be present in the regulators and plumbing. As a leak check, observe the hydrogen tank pressure indicator while the remainder of the system is shut down and ensure that the pressure reading does not decrease rapidly (more the 350 psi/h) which would indicate a significant leak in the supply system.

Battery Charging

WARNING: Never charge the battery in a hazardous environment.

a) Plug charger connector into mating connector on battery cover and insert AC plug into 115 V AC wall outlet.

b) Move the battery charger switch to the ON position. The lamp above the switch button should illuminate.

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

d) 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 from the Side Pack Assembly.

GENERAL TROUBLE-SHOOTING

Table 4-2 presents a summary of field troubleshooting procedures. If necessary, the instrument can be easily removed from the case by unlocking the four (4) ¼ turn fasteners on the panel face and removing the refill cap. The battery pack is removed by taking out the four (4) screws on the panel and disconnecting the power connector.

4.3.3.4 Gas Chromatograph (GC) Operation

The gas chromatograph (GC) option will be used to determine the relative concentration of organic components and/or to make quantitative analysis of specific compounds.

GENERAL DESCRIPTION

With the GC option, the OVA 128 functions as a portable gas chromatograph utilizing hydrogen as a carrier gas and a flame ionization detector as the sensor. In this mode, a fixed volume of sample air is injected (by means of an air injection valve) into the chromatographic column which contains a suitable packing material. At the same time that a sample is introduced into the column, the remaining sample air is directed through an integral charcoal filter to provide the detector with a supply of pure air.

TABLE 4-2

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator. Nominally 2 units on flow gauge. (See also 6 below)	a) Check 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, etc.	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, etc. NOTE: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when troubleshooting.	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.
2) Hydrogen flame will not light. (See also 6 below)	a) Check sample flow rate (see 1 above)	If sample flow rate is low, follow procedure 1 above.
	b) 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 plug. If igniter still does not light, check the battery and wiring.
	c) Check for rated Hydrogen 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.
	d) Check hydrogen flow rate by observing the psi decrease in pressure on the Hydrogen Tank Pressure gauge. The correct flow rate will cause about 130 psi decrease in pressure per hour. (Approximately 12 cm^3/min at detector).	The most likely cause for hydrogen flow restriction would be a blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the Hydrogen Supply Pressure by one-half or one psi. If flow rate cannot be compensated for, replace capillary tubing.
	e) Check all hydrogen plumbing joints for leaks using soap bubble solution. Also, shut off all valves and note pressure decay on hydrogen tank gauge. It should be less than 350 psi per hour.	Repair leaking joint.

TABLE 4-2 (Continued)

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	f) Check to see if hydrogen supply system is frozen up by taking unit into a warm area.	If there is moisture in the hydrogen supply system and the unit must be operated in subfreezing temperatures, purge the hydrogen system with dry nitrogen and ensure the hydrogen gas used is dry.
	g) Remove exhaust port and check for contamination.	If the chamber is dirty, clean with ethyl alcohol and dry by running pump for approximately 15 minutes. If hydrogen fuel jet is misaligned, ensure the porous metal flame arrestor is properly seated.
	h) Check spacing between collecting electrode and burner tip. Spacing should be 0.1 to 0.15 inches.	Adjust by screwing Mixer/Burner Assembly in or out. This spacing problem should only occur after assembling a Mixer/Burner Assembly to a Preamp Assembly.
3) Hydrogen flame lights but will not stay lighted.	a) Follow procedures 2(a), (c), (d), (e), (g) and (h) above. Also refer to 5 below.	
4) Flame-out alarm will not go on when hydrogen flame is out.	a) Check instrument calibration setting and GAS SELECT control setting.	Readjust as required to proper setting. Note that the flame-out alarm is actuated when the meter reading goes below zero.
	b) Remove exhaust port and check for leakage current path in chamber (probably moisture or dirt in chamber).	Clean contamination and/or moisture from the chamber using a swab and alcohol, dry chamber by running pump for approximately 15 minutes.
	c) If above procedures do not resolve the problem, the probable cause is a malfunction in the preamp or power board assemblies.	Return preamp chamber or power board assembly to the factory for repair.
	d) Check that volume control knob is turned up.	Adjust for desired volume.

TABLE 4-2 (Continued)

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
5) False flame-out alarm.	a) Flame-out alarm is actuated when signal goes below electronic zero (with flame on). This can be due to inaccurate initial setting, drift, or a decrease in *ambient concentration*. Verify if this is the problem by zeroing meter with flame out and reigniting.	When using the Xl range adjust meter to 1 ppm, rather than zero, be sure instrument has been zeroed to "lowest expected ambient background level".
6) Slow response, i.e., time to obtain response after sample is applied to input is too long.	a) Check to ensure that probe is firmly seated on the rubber seal in the readout assembly.	Reseat by holding the probe firmly against the rubber seat and then lock in position with the knurled locking nut.
	b) Check sample flow rate per procedure 1 above.	See 1 above.
7) Slow recovery time, i.e., too long a time for the reading to get back to ambient after exposure to a high concentration or organic vapor.	a) This problem is normally caused by contamination in the sample input line. This requires pumping for a long period to get the system clean of vapors. Charcoal in the lines would be the worst type of contamination. Isolate through the process of elimination. (See 1(b)).	Clean or replace contaminated sample line or assembly as required.
	b) Check flame chamber for contamination.	Clean as required.
8) Ambient background reading in clean environment is too high.	a) A false ambient background reading can be caused by hydrocarbons in the hydrogen fuel supply system. Place finger over sample probe tube restricting sample flow and if meter indication does not go down signficantly the contamination is probably in the hydrogen fuel.	Use a higher grade of hydrocarbon free hydrogen. Check for contaminated fittings on filling hose assembly.
	b) A false ambient background reading can also be caused by a residue of sample building up on the face of the sample inlet filter. If the test in 8(a) above produces a large drop in reading, this is usually the cause.	Remove the exhaust port (it is not necessary to remove instrument from case). Use the small wire brush from the tool kit or a knife blade and lightly scrub surface of sample inlet filter.

TABLE 4-2 (Continued)

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	c) A false ambient background reading can also be caused by hydrocarbon contamination in the sample input system. The most likely cause would be a contaminant absorbed or condensed in the sample line. NOTE: It should be emphasized that running the instrument tends to keep down the buildup of background vapors. Therefore, run the unit whenever possible and store it with the carrying case open in clean air.	Clean and/or replace the sample input lines. Normally the false reading will clear up with sufficient running.
9) Pump will not run.	a) Check that there is no short circuit in wiring.	If no short circuit, pump motor is defective.
10) No power to electronics but pump runs.	a) Short circuit in electronics.	There is a short in the electronics assembly. Return OVA to factory or authorized repair facility.
11) No power to pump or electronics	a) Place battery on charger and see if power is then available. Recharge in a non-hazardous area only.	If power is available, battery pack is dead or open. Recharge battery pack. If still defective, replace battery pack.

While moving through the chromatographic column, the sample constituents are separated based on their interaction with the column packing material. As the constituents leave the column, they are carried to the detector and register on logarithmic meter and the attached optional chart recorder. The time, measured from the moment of sample injection until the compound of interest exits the column, is known as the retention time and serves to identify the compound. The area under the chromatographic peak is proportional to the concentration of the compound in the air sample. The peak height can also be used to determine sample concentration because it closely correlates with peak area.

GC MODE OPERATION PROCEDURES

The gas chromatographic analysis mode (GC Mode) of operation can be initiated at any time during a survey by simply depressing the Sample Inject Valve. After completion of the analysis and backflush operations, the Sample Inject Valve is pulled out, and the survey is continued or another sample injected. Note that when the Sample Inject Valve is in the survey mode (out position), the OVA operates in the same manner as an OVA which does not incorporate the GC option.

Turn On Procedure

Place the Sample Inject Valve in the "out" position and put the OVA instrument in operation per "Operating Procedures" for the Survey Mode as explained in Section 4.3.3.2. NOTE: Leave the hydrogen fuel and pump "on" for three (3) to four (4) minutes before attempting ignition to allow time for hydrogen purging of the column.

Operation

A strip chart recorder is used to record the output concentration from the OVA as a function of time. This record, called a chromatogram, is utilized for interpretation of the GC data.

a) Turn on recorder and push Sample Inject Valve "in" with a fast, positive motion. This starts the GC analysis which is automatic up to the point of backflushing. NOTE: Rapid and positive motion should be used when moving either the Sample Inject or Backflush Valves. On occasion, the flame in the FID detector may go out, which would be indicated by a sharp and continued drop of the concentration level. If this occurs, reignite the flame and continue the analysis. NOTE: A negative "air" peak typically occurs shortly after sample injection and should not be confused with flame-out.

b) The negative air peak and various positive compound peaks indicated on the OVA readout meter and the strip chart recorder represent the chromatogram.

c) After the predetermined time for the analysis has elapsed (normally immediately after the peak of the last compound of concern), rapidly move the Backflush Valve to its alternate position (in or out). Leave the instrument in this condition until the backflush peak returns to baseline, then pull the Sample Inject Valve to the "out" position. If no backflush peak appears, pull the Sample Inject Valve out after being in the backflush condition for a period at least twice as

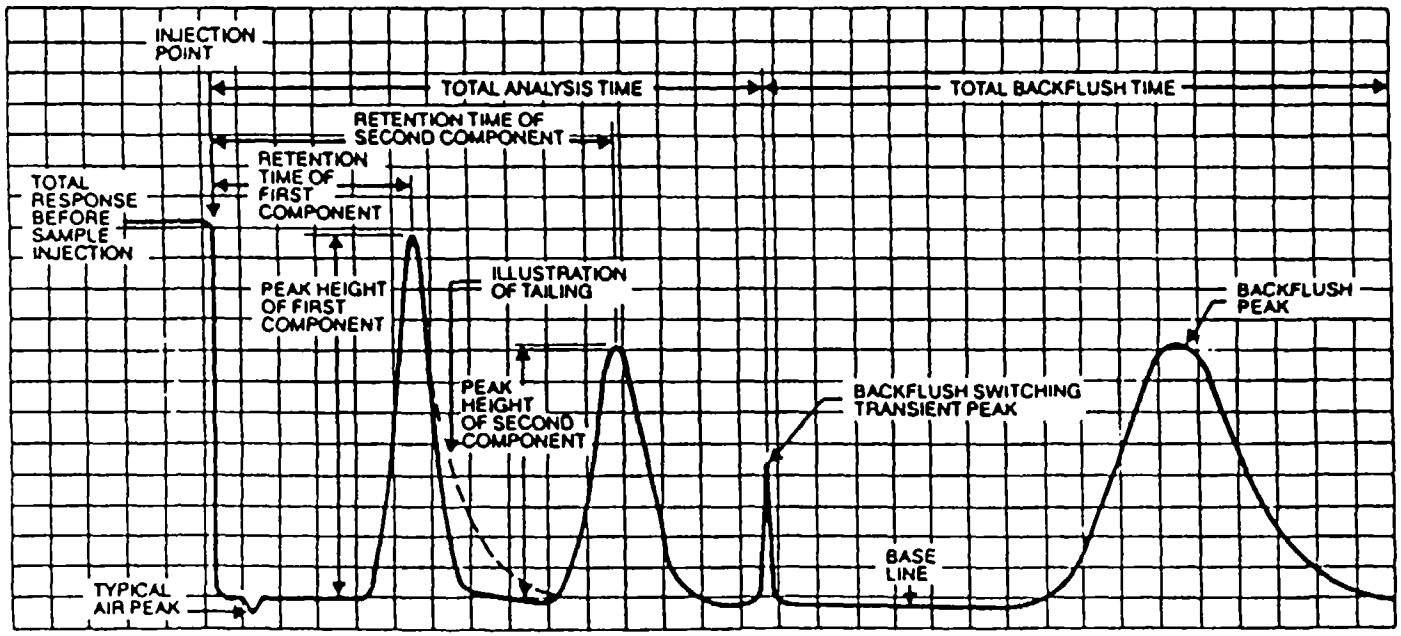
long as the analysis time. The OVA is now in the Survey Mode and is ready for survey or injection of another sample into the GC system.

INTERPRETATION OF RESULTS

The OVA 128 with GC option is intended for applications where there are a limited number of compounds of interest and the compounds are normally known. Under these conditions, the operator must know the retention time and peak height characteristics of the compounds under specific operation conditions. To calibrate the OVA in the GC Mode, the retention time and peak area (using peak height analysis) for the compounds of concern (Table 4-1) will be determined by test using available standards. For the purposes of the RI, these tests will be run and these standards will be prepared by the company from which the instrument will be obtained. These tests will be conducted on the column to be utilized and over the concentration and temperature range of concern. When representative characteristic data is available, a spot calibration check is normally all that is required.

Qualitative Analysis

Under a given set of operation conditions the retention time is characteristic of that particular substance and can be used to identify specific compounds. It will be necessary to calibrate retention times by making tests with the pure compounds of interest. The retention time (RT) is defined as that period of time from injection until the time of maximum detector response for each substance. Retention time is measured from the time of sample injection to the time the apex of the triangle shaped curve is obtained on the strip chart recorder. Refer to Figure 4-2. The strip



TYPICAL CHROMATOGRAM RUETGERS-NEASE SALEM, OHIO	FIGURE 4-2
ERM–Midwest, inc.	

chart recorder operates on a clock mechanism such that the distance along the baseline is proportional to time. While retention times are characteristic for each compound, it is possible that two materials could have the same retention times. Thus, if there is any question as to the identity of the vapor, it may be necessary to verify identification by retention times on different columns.

Use of a longer column will increase the retention times of those components it is capable of separating. The time between peaks will also be increased. This is especially useful if a component comes through too fast or if desired peaks are so close that they overlap.

An increase in carrier gas flow rate will decrease retention time. For reproducible data, the carrier gas (hydrogen) flow rate must be recorded in association with a chromatogram. Primary control of the hydrogen flow rate is accomplished in the OVA by regulating the hydrogen pressure across a capillary tube. The hydrogen flow rate is also affected by the restriction of the GC column but most columns have a limited effect. The hydrogen flow rate is set at by the manufacturer at 12cm/minute with a typical 24 inch column.

Quantitative Analysis

For the purpose of quantitative analysis, peak height calibration will be used. Using the peak height method, a known concentration of the compound is injected and the peak height is recorded. Peak height characteristics can be established for various columns and various temperatures. Normally, both retention time and peak height characteristics will be measured.

In general, the more triangularly symmetrical the peak, the better the peak height analysis capability. However, may GC peaks have "tailing" as illustrated in Figure 4-2. Peak height calibration is an acceptable method for quantitative analysis as long as the area under the tail is small compared with the total peak area. If severe tailing occurs, empirical calibration data generated through tests may be required to plot the peak height versus the concentration curve.

CALIBRATION DATA

When conducting tests to obtain GC calibration data, the following information will be recorded.

a) Column description and serial number as applicable.

b) Temperature: Column temperature, normally room ambient.

c) Chart speed: Distance/unit time

d) Carrier flow rate: Hydrogen flow rate through the column (cm/min).

e) Sample concentration: Ppm for each compound.

f) Sample volume: OVA by serial number or typically 0.25 cm for standard value.

g) Recorder scaling: Ppm per unit deflection.

h) Range: range of OVA being used, i.e. X1, X10, X100.

i) OVA serial number.

4.3.3.5 Maintenance and Trouble-Shooting for the GC Attachment

GENERAL MAINTENANCE

Column

Any column can be contaminated with compounds having long retention times. This will result in high background readings. This condition can be checked by installing a new column or a blank column (tubing only). If this reduces the background reading, the contaminated column should be baked at 100 C for three (3) to four (4) hours in a drying oven while passing nitrogen through the column. Higher temperatures may permanently damage the column packing.

When installing any column, avoid touching the ends, as this may cause contamination. Also, ensure that the fittings are tight to avoid hydrogen leakage.

IMPORTANT: The following simple test may be run to determine whether the GC column is contaminated. While in a clean ambient air background, place the Sample Inject Valve in the "in" (GC Mode) position. Observe the background reading on the meter or recorder. After one (1) to two (2) minutes, change the position of the Backflush Valve and again observe the background reading. If the background reading went down and then started to increase in one to two minutes, the column is probably contaminated and needs to be cleaned. To clean a column, the purge gas must be run through the column in one direction until all contamination is removed. Contaminated columns can be avoided by backflushing the column after every analysis.

Charcoal Filter Assembly

After repeated use, the Charcoal Filter Assembly will become saturated. Periodically, the operator should check the effectiveness of the activated charcoal.

This can easily be done by operating the unit with the Sample Injection Valve "in" and passing the probe near a concentrated sample of the compound being analyzed. The readout should remain nearly steady (should not rise more than 0 to 2 parts per million (ppm)). If rise is more than 2 ppm, replace the old charcoal with new activated charcoal. Care should be taken to completely fill the tube to prevent a path for sample to bypass the charcoal. The life of the charcoal depends on the time (length) of exposure and the concentration level during that exposure. When changing charcoal, be sure that any fine charcoal dust is removed from the assembly.

Another test of the charcoal filter is to note the background reading with the Sample Inject Valve "out" and then note the reading with the valve "in". The level should never be higher when the valve is in the "in" position and the charcoal filter is in the air line. If the reading with the valve in the "in" position is higher, the charcoal filter is probably contaminated and is acting like a contamination emitter.

GENERAL TROUBLE SHOOTING

Table 4-3 presents recommended field trouble-shooting procedures which are associated with the GC system. These procedures are in addition to those found in the basic OVA trouble-shooting section of this manual.

TABLE 4-3

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator.	a) Check Teflon tubing on valve assembly for kinks, etc.	Straighten or replace teflon tubing.
	b) Check flow rate with valve in down position.	Check for over restriction of charcoal filter.
2) Hydrogen flame will not light.	a) Check column connections on top of unit to make sure they are tight.	Tighten fittings.
	b) Check column for sharp bends or kinks. (Hydrogen flows through this column at all times and a sharp bend will compact packing too tightly for proper hydrogen flow).	Replace column.
	c) Check charcoal filter fittings to make sure they are tight.	Tighten fittings.
	d) Check hydrogen flow rate from the column.	Adjust hydrogen pressure to obtain 12 cm^3/min flow rate.
	e) Check that the Inject and Backflush Valves are both completely in or out. A partially activated valve will block the hydrogen and air flow paths.	Ensure both valves are either completely in or out.
	f) If a new column was installed prior to problem identification, check for proper hydrogen flow rate through the column (should be approximately 12 cm /min).	Increase hydrogen pressure to obtain proper hydrogen flow rate or if column is excessively restrictive, replace or repack the column.
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.
4) Flame-out when operating either valve.	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with a positive motion.

TABLE 4-3 (Continued)

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	b) Either hydrogen 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.	Replace quad rings and grease as above.
5) Excessive peak tailing	a) Change or clean GC; see if problem disappears.	Ensure columns are clean prior to use. If one of the same type of column tails are worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessive 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" ring. Do not get grease into the "O" ring grooves.

Recommended Spares

The following spare parts and supplies are recommended to support the GC system and recorder. These are an addition to the spare parts list for the basic OVA described in the "OVA MAINTENANCE" section.

ITEM DESCRIPTION	PART NO.
1) Quad Rings	510496-1 (10/pkg.)
2) Tubing, .148 in ID .020 wall	12942
3) Tubing, Teflon .120 in ID .030 wall	12941
4) Activated Charcoal	CSC-004
5) "O" Ring for Charcoal Scrubber	U0118CE
6) Chart Paper (linear)	CSC-008 (6/rls/pkg)

ACCESSORIES

Recorder Accessory

A portable Strip Chart Recorder is available for use with the OVA (reference Figure 11). The recorder is powered from the OVA battery pack and the output can be scaled to match the OVA readout meter, thereby providing a permanent record for subsequent analysis or reference. P/N 510445-4 is FM certified intrinsically safe. P/N 510445-6 is BASEEFA certified.

The recorder can be used with the OVA to provide a long term monitoring profile of total hydrocarbon or can be used with the Gas Chromatograph Option to provide a chromatogram.

Features

The recorder prints dry (no ink) on pressure sensitive chart paper. The recorder is equipped with two gain ranges and an electronic zero adjustment. The HIGH gain position is normally used to provide a means of scale expansion.

4.3.4 Photoionization Detector (PID)

Note: The information presented in Section 4.3.4 is excerpted in part from "Operational Procedures for HNu Model PI 101 Photoionization Analyzer," prepared by Cheng-Wen Tsai, Chemist, Quality Assurance Office, U.S. EPA, Region 5, a document provided to Ruetgers-Nease by U.S. EPA Region 5.

4.3.4.1 Introduction

GENERAL DESCRIPTION

OPERATION PRINCIPLE

The photoionization detector is a portable trace gas analyzer that can be used to measure a wide variety of organic vapors including chlorinated hydrocarbons, heterocyclics and aromatics, aldehydes and ketones as well as several inorganic gases including hydrogen sulfide and ammonia.

The photoionization detector is a simple analytical instrument to use because it has only three operating controls and unskilled personnel are easily and quickly trained to operate it. An easy to read 4½" linear scale provides a readout directly in units of concentration (ppm). Other features include an electronic zero that eliminates the use of a zero gas, and instrument calibrations that hold for weeks. The elimination of a flame, igniters and compressed hydrogen fuel make the photo-ionizer simpler to use than a flame ionization analyzer while providing an unusually safe instrument.

The HNu Model 101 photoionization detector has been designed to measure the concentration of trace gases in many industrial or plant atmospheres. The instrument has similar

capabilities outdoors. The analyzer employs the principle of photoionization for detection. This process is termed photoionization because the absorption of ultraviolet light (a photon) by a molecule leads to ionization via:

$$RH + hv \text{ ----------} > RH^+ + e^-$$

where RH = trace gas
hv = a photon with an energy greater than or equal to an ionization potential of RH.

The sensor consists of a sealed ultraviolet light source that emits photons which are energetic enough to ionize many trace species (particularly organics), but do not ionize the major components of air such as O_2, N_2, CO, CO_2 or H_2O. A chamber adjacent to the ultraviolet light source contains a pair of electrodes. When a positive potential is applied to one electrode, the field created drives any ions, formed by absorption of UV light, to the collector electrode where the current (proportional to concentration) is measured. The useful range of the instrument is from a one-tenth of a ppm to about 2,000 ppm.

INSTRUMENT SENSITIVITY AND CALIBRATION

The instrument responds to atmospheric compounds with ionization potentials equal to or less than the ionization energy of the UV light source. If a compound in air has an ionization potential greater than the energy source of the lamp, it will not be detected. Table 4-4 present compounds identified at Ruetgers-Nease and the light sources that should be used to detect each compound. The instrument is capable of using one of the three light sources - 9.5, 10.2, and 11.7 ev lamps. In addition, not all compounds respond equally to each light sources and thus they vary in their

TABLE 4-4

RELATIVE RESPONSE WITH DIFFERENT LAMP ENERGIES
CALIBRATED TO ISOBUTYLENE
FOR COMPOUNDS THAT HAVE BEEN
QUALITATIVELY IDENTIFIED AT THE RUETGERS-NEASE SITE
PRIOR TO THE RI/FS

COMPOUND	RELATIVE RESPONSE (%)		
	9.5 ev	10.2 ev	11.7 ev
Benzene	1.18	1.35	1.17
Chlorobenzene	1.49	1.65	1.78
Chloroform	0.003	0.01	0.75
1,2-Dichloroethane	0.02	0.02	1.46
1,1-Dichloroethene (vinylidene chloride)	0.27	0.55	1.40
1,2-Dichloroethene	1.25	1.97	1.84
Ethylbenzene	2.09	1.64	1.85
Tetrachloroethene	1.12	1.42	2.63
Toluene	1.26	1.24	1.45
1,1,1-Trichloroethane	0.04	0.03	1.13
Trichloroethane	0.73	1.48	2.00
m-Xylene	1.83	1.62	1.83
o-Xylene	1.42	1.26	1.66
p-Xylene	1.60	1.59	1.90

RELATIVE RESPONSES WITH DIFFERENT LAMP ENERGIES

Relative Responses for Volatile Organic Compounds (VOC)
Each at 100 PPM. TIP Spanned at 100 PPM

$$\text{Relative Response} = \frac{\text{Response 100 PPM VOC}}{\text{Response 100 PPM Isobutylene}}$$

sensitivity to ionization. As a result of varying sensitivities to photoionization, the response given by the instrument may or may not reflect the actual atmospheric concentration of the compound being detected. Table 4-4 represents the relative responses for various gases relative to the three light sources. Use this table to determine the approximate response of the instrument to a compound of interest, and to select the appropriate light (lamp) source. The 11.7 ev lamp will be utilized for HNu use at the site.

There are two types of operations that are used for calibration. For Type 1 Operation, a non-regulatory (or non-target) compounds such as isobutylene is used for calibration. In this case, the instrument reading is reported in terms relative to the calibration compound used for calibration. For the type 2 operation, the target compound or compounds are used for calibration. As a result, the instrument is calibrated to respond directly in ppm by volume of the target compound(s). At the Ruetgers Nease site the instrument will be calibrated using isobutylene. Specific calibration instructions are presented in Section 4.3.4.2 of this operational procedure.

INSTRUMENT SPECIFICATIONS

Performance

Range: 0.1 to 2000 ppm
Detection Limit: 0.1 ppm
Sensitivity (max.): 0 to 2 ppm FSD over 100 division meter scale
Repeatability: ± 1% of FSD
Linear Range: 0.1 to 600 ppm
Useful Range: 0.1 to 2000 ppm
Response Time: less than 3 seconds to reach 90% full scale

Ambient humidity: up to 95% relative humidity

Operating Temperature: Ambient to 40°C (instrument is temperature compensated to that a 20°C change in temperature corresponds to a change in reading of ± 2% full scale at maximum sensitivity.

Power Requirements and Operating Times

Continuous use on battery: approximately 10 hours

Continuous use with HNu recorder: reduces instrument battery operating time to approximately 5 hours

Recharge time: overnight
(A 3 hour charge will charge up to 90% full charge.)

4.3.4.2 Operational Procedures

INSTRUMENT CHECK-OUT

a. Remove instrument box cover by pulling up on fasteners.

b. On the instrument panel, there will be a label containing information on light source, calibration date, calibration gas, and span setting.

c. If the instrument has not been calibrated in the last 14 days or since its field use, it should be recalibrated. Check the instrument log, which should be maintained with the instrument, for the instrument status and its calibration history. For general use, the instrument should be calibrated to isobutylene at a span setting of 9.8.

d. Check the table for light source and refer to Table 4.4 for ionization potentials of various compounds. If the compound you wish to detect is not listed for the light sources provided with instrument, then the light source will have to be changed. Use the probe with the proper light source for the compounds to be detected.

e. Once it has been determined that the instrument has the correct lamp, the instrument may need to be recalibrated for the specific compound of interest. Use procedures in this section to calibrate the instrument.

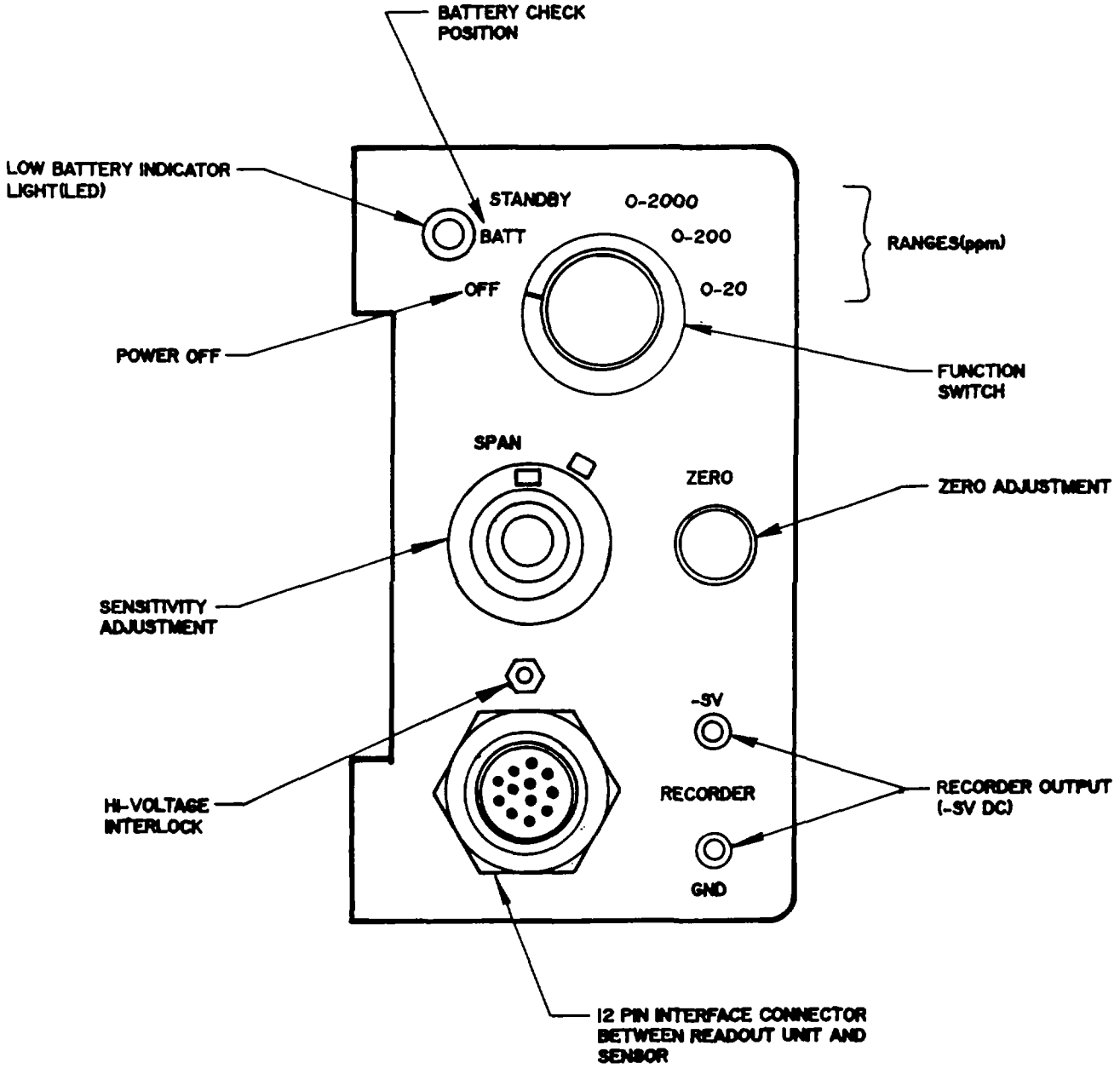
f. Check the battery supply by connecting the probe to the instrument box, and turning the function switch to the battery check position, refer to Figure 4-2. (Note: The battery check indicator will not function unless the probe is attached). The meter needle should deflect to the far right or above the green zone. If the needle is below or just within the green zone or the red LED indicator is on, the battery should be recharged. Follow the procedure described in Section 4.3.4.3 (Maintenance and Trouble Shooting) to recharge the battery.

g. Repack the instrument for shipment to the field.

STARTUP PROCEDURES

a. Remove instrument cover by pulling up on the side straps.

b. Prior to calibration, check the function switch (Figure 4-3) on the control panel to make sure it is in the OFF position. The probe nozzle is stored inside the



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INSTRUMENT CONTROL PANEL FEATURES	FIGURE 4-3
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instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.

c. Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.

d. Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.

e. Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements. Implement steps in Section 4.3.4.3 to recharge battery.

f. Turn the function switch to the ON position. In this position, the UV light source should be on. To verify, gaze at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to Maintenance, Section 4.3.4.3.

g. To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed because this is an electronic zero adjustment). If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If

necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

OPERATION PROCEDURE

a. Place function switch in 0-20 ppm range for field monitoring. This will allow for the most sensitive, quick response in detecting airborne contaminants.

b. Before entering a contaminated area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated areas. Under no circumstances should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.

c. Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measurements in notebook or on an appropriate form.

d. Keep in mind health and safety action guidelines for the level of protection you are wearing. Sustained readings above a certain level may force you to vacate an area or upgrade your level of protection.

Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected refer to Section 4.3.4.3 Maintenance and Trouble Shooting.

e. When finished, use the reverse Steps a thru e of the Startup Procedure to shut down the instrument.

CALIBRATION

This instrument should be calibrated after each field use and prior to each field use. Continuous calibration checks should be performed frequently during field operation (for example, check the instrument zero and calibration after every 10 measurements) and document the results properly. Caution: Do Not Change the Settings.

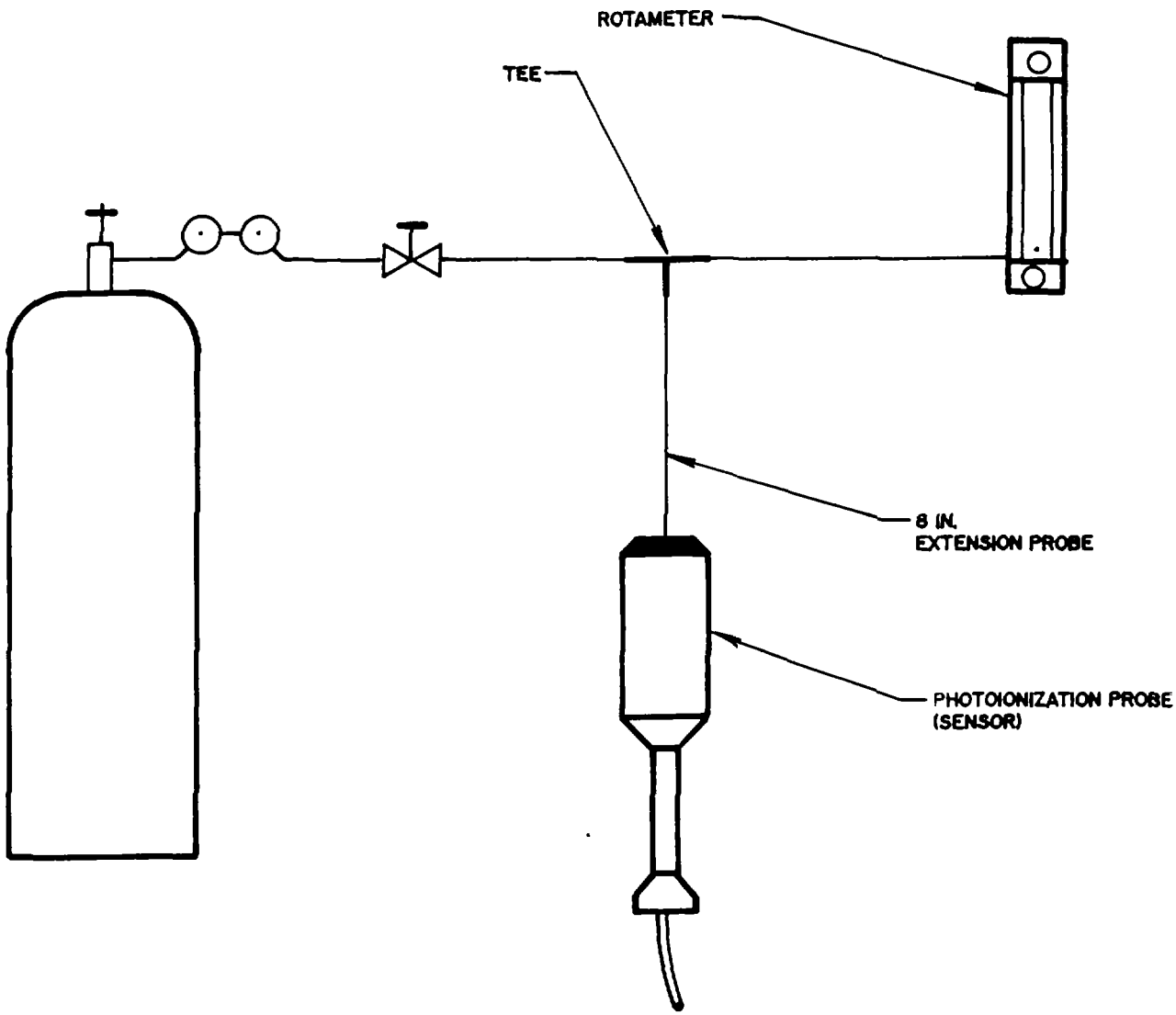
Primary Calibration for Isobutylene

Low range 0-20 ppm and mid-range 20-200 ppm concentration of isobutylene gas are used for standard field operation when contaminants are unknown or a mixture of gases may be present. The isobutylene gas is used for general calibration because of the instrument's relatively high sensitivity to this gas and because of the non-toxic nature of the gas.

Calibration Procedure

Use a three-points procedure to facilitate the proper instrument calibration over appropriate operating ranges. Distinct mixtures of calibration gas with known concentrations for selective operating range should be used for calibration. In this case isobutylene will be used. Each mixture should give a 3/4 scale deflection in its respective operating range.

a. Insert one end of T tube, as shown in Figure 4-4, into probe. Insert second end of probe into calibration gas



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RECOMMENDED CALIBRATION PROCEDURES FOR PHOTOIONIZATION ANALYZER	FIGURE 4-4
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in the 20-200 ppm range. The third end of probe should have a rotometer (bubble meter) attached.

b. Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection with the rotometer indicating excess flow.

c. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder).

d. Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following g) of Startup Procedure.

e. Record on form and field log all original and readjusted settings as specified in the form.

f. Next, set the function switch to the 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low range (0-20 ppm) calibration gas cylinder as described above.

g. Do not adjust the span potentiometer. The observed reading should be +3 ppm of the concentration specified for the low range calibration gas. If this is not the case, recalibrate the mid range scale repeating a thru f above. If the low range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window following Section 4.3.4.3 Maintenance and Trouble-Shooting. When the observed reading is within the required tolerances, the instrument is fully calibrated.

4.3.4.3 Maintenance and Trouble-Shooting

GENERAL MAINTENANCE

Battery Recharging

The instrument should be recharged 1 hour for each hour of use or overnight for a full day's use. (The battery will last 10 hours on a full charge).

To recharge the battery (or instrument):

a. Turn the function switch to the off position.

b. Remove the charger from the instrument top compartment.

c. Place the charger plug into the jack on the left side of the instrument box.

d. Connect the charger unit to a 120 V AC supply.

e. Check charger function by turning the instrument switch to the battery check position. The meter should go upscale if the charger is working and is correctly inserted into the jack.

f. Place instrument in instrument mode and charge for the appropriate time period.

g. Turn the instrument off following the recharge cycle. When disconnecting charger, remove 120 V AC supply before removing the mini phone plug.

GENERAL TROUBLE-SHOOTING

Battery level is low - Recharge if necessary implementing steps described under Battery Recharging. If the battery will not recharge, it will have to be replaced.

UV Lamp Function - Gaze at sample inlet when mode switch is on an instrument function position and observe for purple glow of lamp. If the lamp does not glow in any of the three instrument function positions, it may be burned out and will have to be replaced. To replace the lamp:

a. Turn the function switch to the off position and disconnect the probe connector from the readout unit.

b. Remove the exhaust screw found near the base of the probe.

c. Grasp the end cap in one hand and the probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.

d. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber does not fall out of the end cap and the lamp does not slide out of the lamp housing.

e. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out of it.

f. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

g. Replace lamp with one of same energy source as the one removed by sliding it into the housing. Note: the amplifier board and instrument circuitry are calibrated for one light energy.

h. Place the ion chamber on top of the lamp housing, checking to ensure that the contacts are aligned.

i. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enough to seal the "0" ring. Do not overtighten.

j. Line up the pins on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. Do not force the assembly as it only fits one way.

k. Replace and tighten the exhaust screw.

l. Reconnect the 12 pin connector and turn instrument mode switch to a function position. Check for glow of lamp. If lamp still does not function, the instrument has an electrical short or other problem that will have to be corrected at the factory.

Instrument appears to be functional, but responses are lower than expected or erratic - The window of the light source may be dirty and need to be cleaned. To clean the light source window:

a. Disassemble the probe assembly by repeating Steps a thru f.

b. Clean the window of the light source using compound provided with instrument and soft clean cloth. Important: Use cleaning compound on the window of the

10.2 eV lamp only. The cleaning compound may damage the windows of the 9.5 and 11.7 ev lamps.

c. Reassemble the probe assembly repeating Steps g thru i above.

4.3.5 Air Sampling Protocols, Procedures and Methods

The following separate section (blue pages) addresses the air sampling methods TO1, TO2, TO3, and TO4 that will be used for detecting VOCs on Tenax, VOCs on Carbon Molecular Sieves, and Organochloride pesticides and PCBs. Section 4.4 Water Monitoring continues after the blue pages.

4.4 Water Monitoring

To ensure that water monitoring data is collected properly the following procedures will be implemented during the RI/FS.

4.4.1 pH, eh, DO, and Temperature Meter

These meters are used to measure the pH, eh, and temperature of water samples. Often, temperature variations are automatically compensated for during the measurement.

The following procedure will be implemented while using the Cole-Parmer Model 5985-80 pH meter:

1. Connect pH electrode and the automatic temperature control (ATC) probe to the meter.

2. Push the ON/OFF switch to turn the unit on. Calibrate the unit with buffer solutions. The instrument can be calibrated with two buffers. The

calibration can use a pH 7.00 and either pH 4.01 or pH 10.00 standard buffers. If the pH of the sample to be measured is between 0 and 7 pH (acidic to neutral), pH 7.00 and pH 4.01 buffers will be used. If measurements will be between 7 and 14 pH (neutral to base), pH 7.00 and pH 10.00 buffers will be used.

After the two appropriate buffer solutions have been used to calibrate the instrument, a third buffer may be used with the meter in the standard pH mode. The use of this third buffer may be used to validate the effectiveness of the performed calibration.

3. Push RANGE button unit the display indicates the desired mode (pH, eh, or temperature).

   o For pH measurement: instrument is in pH mode when it is switched on. Dip the pH probe and ATC probe into the sample to be measured. Wait approximately 30 seconds and read pH value.

   o For temperature measurement: press RANGE button until "^{o}C" appears on display. Wait approximately 30 seconds for temperature probe to equilibrate with sample, and read pH value.

   o For eh measurement: press the RANGE button until "MV" appears on the display. After probe has equilibrated to sample for approximately 30 seconds read eh value. See the manufacturer's instructions for trouble shooting and details on meter operation.

4.4.2 Conductivity Meter

The following procedures will be implemented for the Cole-Parmer Model 1481-50 digital conductivity meters during the RI/FS.

OPERATION

1. After the unit has been calibrated with a standard solution, immerse the electrode in the liquid to be measured. Select the desire conductivity range by turning the function switch. The display will stop blinking when the proper range has been selected.

2. Turn the manual temperature adjustment knob to equal the temperature of the sample. Read the conductivity measure from the meter display. See the manufacturer's instructions for trouble shooting and details on meter operation.

4.4.3 Dissolved Oxygen Meter

The following procedure will be implemented during the RI/FS when the Cole-Parmer Model 5946-10 field oxygen meter is used.

OPERATION

1. Turn meter ON and to the O_2 mode. The meter will be on for 30 to 40 minutes before use. Calibrate meter.

2. Immerse probe at least one-inch into the sample solution. This will insure the correct temperature compensation by immersing the thermistor.

3. Slowly and gently move probe within the sample.

4. Wait two to three minutes then read the dissolved oxygen measurement.

5. Probe should be stored in 0.1 M sodium chloride solution for maximum probe performance.

METHOD TO1

Revision 1.0
April, 1984

ME ON OF VOLATILE ORGANIC COMPOUNDS NG TENAX® ADSORPTION AND MASS SPECTROMETRY (GC/MS)

1. Scope

1.1 The document describes a generalized protocol for collection and determination of certain volatile organic compounds which can be captured on Tenax® GC (poly(2,6-Diphenyl phenylene oxide)) and determined by thermal desorption GC/MS techniques. Specific approaches using these techniques are described in the literature (1-3).

1.2 This protocol is designed to allow some flexibility in order to accommodate procedures currently in use. However, such flexibility also results in placement of considerable responsibility with the user to document that such procedures give acceptable results (i.e. documentation of method performance within each laboratory situation is required). Types of documentation required are described elsewhere in this method.

1.3 Compounds which can be determined by this method are nonpolar organics having boiling points in the range of approximately 80° - 200°C. However, not all compounds falling into this category can be determined. Table 1 gives a listing of compounds for which the method has been used. Other compounds may yield satisfactory results but validation by the individual user is required.

2. Applicable Documents

2.1 ASTM Standards:

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis.

E355 Recommended Practice for Gas Chromatography Terms and Relationships.

2.3 Other documents:

Existing procedures (1-3).
U.S. EPA Technical Assistance Document (4).

3. Summary of Protocol

3.1 Ambient air is drawn through a cartridge containing ~1-2 grams of Tenax and certain volatile organic compounds are trapped on the resin while highly volatile organic compounds and most inorganic atmospheric constituents pass through the cartridge. The cartridge is then transferred to the laboratory and analyzed.

3.2 For analysis the cartridge is placed in a heated chamber and purged with an inert gas. The inert gas transfers the volatile organic compounds from the cartridge onto a cold trap and subsequently onto the front of the GC column which is held at low temperature (e.g. - 70°C). The GC column temperature is then increased (temperature programmed) and the components eluting from the column are identified and quantified by mass spectrometry. Component identification is normally accomplished, using a library search routine, on the basis of the GC retention time and mass spectral characteristics. Less sophistacated detectors (e.g. electron capture or flame ionization) may be used for certain applications but their suitability for a given application must be verified by the user.

3.3 Due to the complexity of ambient air samples only high resolution (i.e. capillary) GC techniques are considered to be acceptable in this protocol.

4. Significance

4.1 Volatile organic compounds are emitted into the atmosphere from a variety of sources including industrial and commercial facilities, hazardous waste storage facilities, etc. Many of these compounds are toxic; hence knowledge of the levels of

such materials in the ambient atmosphere is required in order to determine human health impacts.

4.2 Conventional air monitoring methods (e.g. for workspace monitoring) have relied on carbon adsorption approaches with subsequent solvent desorption. Such techniques allow subsequent injection of only a small portion, typically 1-5% of the sample onto the GC system. However, typical ambient air concentrations of these compounds require a more sensitive approach. The thermal desorption process, wherein the entire sample is introduced into the analytical (GC/MS) system fulfills this need for enhanced sensitivity.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356(6). All abbreviations and symbols are defined with this document at the point of use.

6. INTERFERENCES

6.1 Only compounds having a similar mass spectrum and GC retention time compared to the compound of interest will interfere in the method. The most commonly encountered interferences are structural isomers.

6.2 Contamination of the Tenax cartridge with the compound(s) of interest is a commonly encountered problem in the method. The user must be extremely careful in the preparation, storage, and handling of the cartridges throughout the entire sampling and analysis process to minimize this problem.

7. Apparatus

7.1 Gas Chromatograph/Mass Spectrometry system - should be capable of subambient temperature programming. Unit mass resolution or better up to 800 amu. Capable of scanning 30-440 amu region every 0.5-1 second. Equipped with data system for instrument control as well as data acquisition, processing and storage.

7.2 Thermal Desorption Unit - Designed to accommodate Tenax cartridges in use. See Figure 2a or b.

7.3 Sampling System - Capable of accurately and precisely drawing an air flow of 10-500 ml/minute through the Tenax cartridge. (See Figure 3a or b.)

7.4 Vacuum oven - connected to water aspirator vacuum supply.

7.5 Stopwatch

7.6 Pyrex disks - for drying Tenax.

7.7 Glass jar - Capped with Teflon-lined screw cap. For storage of purified Tenax.

7.8 Powder funnel - for delivery of Tenax into cartridges.

7.9 Culture tubes - to hold individual glass Tenax cartridges.

7.10 Friction top can (paint can) - to hold clean Tenax cartridges.

7.11 Filter holder - stainless steel or aluminum (to accommodate 1 inch diameter filter). Other sizes may be used if desired. (optional)

7.12 Thermometer - to record ambient temperature.

7.13 Barometer (optional).

7.14 Dilution bottle - Two-liter with septum cap for standards preparation.

7.15 Teflon stirbar - 1 inch long.

7.16 Gas-tight glass syringes with stainless steel needles - 10-500 µl for standard injection onto GC/MS system.

7.17 Liquid microliter syringes - 5,50 µL for injecting neat liquid standards into dilution bottle.

7.18 Oven - 60 $\pm$ 5°C for equilibrating dilution flasks.

7.19 Magnetic stirrer.

7.20 Heating mantel.

7.21 Variac

7.22 Soxhlet extraction apparatus and glass thimbles - for purifying Tenax.

7.23 Infrared lamp - for drying Tenax.

7.24 GC column - SE-30 or alternative coating, glass capillary or fused silica.

7.25 Psychrometer - to determine ambient relative humidity. (optional).

8. Reagents and Materials

8.1 Empty Tenax cartridges - glass or stainless steel (See Figure 1a or b).
8.2 Tenax 60/80 mesh (2,6-diphenylphenylene oxide polymer).
8.3 Glasswool - silanized.
8.4 Acetone - Pesticide quality or equivalent.
8.5 Methanol - Pesticide quality, or equivalent.
8.6 Pentane - Pesticide quality or equivalent.
8.7 Helium - Ultra pure, compressed gas. (99.9999%)
8.8 Nitrogen - Ultra pure, compressed gas. (99.9999%)
8.9 Liquid nitrogen.
8.10 Polyester gloves - for handling glass Tenax cartridges.
8.11 Glass Fiber Filter - one inch diameter, to fit in filter holder. (optional)
8.12 Perfluorotributylamine (FC-43).
8.13 Chemical Standards - Neat compounds of interest. Highest purity available.
8.14 Granular activated charcoal - for preventing contamination of Tenax cartridges during storage.

9. Cartridge Construction and Preparation

9.1 Cartridge Design

9.1.1 Several cartridge designs have been reported in the literature (1-3). The most common (1) is shown in Figure 1a. This design minimizes contact of the sample with metal surfaces, which can lead to decomposition in certain cases. However, a disadvantage of this design is the need to rigorously avoid contamination of the outside portion of the cartridge since the entire surface is subjected to the purge gas stream during the desorption porcess.

Clean polyester gloves must be worn at all times when handling such cartridges and exposure of the open cartridge to ambient air must be minimized.

9.1.2 A second common type of design (3) is shown in Figure 1b. While this design uses a metal (stainless steel) construction, it eliminates the need to avoid direct contact with the exterior surface since only the interior of the cartridge is purged.

9.1.3 The thermal desorption module and sampling system must be selected to be compatible with the particular cartridge design chosen. Typical module designs are shown in Figures 2a and b. These designs are suitable for the cartridge designs shown in Figures 1a and 1b, respectively.

9.2 Tenax Purification

9.2.1 Prior to use the Tenax resin is subjected to a series of solvent extraction and thermal treatment steps. The operation should be conducted in an area where levels of volatile organic compounds (other than the extraction solvents used) are minimized.

9.2.2 All glassware used in Tenax purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C.

9.2.3 Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartidge preparation should be cleaned in the same manner as Tenax.

9.2.4 The extracted Tenax is immediately placed in an open glass dish and heated under an infrared lamp for two

hours in a hood. Care must be exercised to avoid over heating of the Tenax by the infrared lamp. The Tenax is then placed in a vacuum oven (evacuated using a water aspirator) without heating for one hour. An inert gas (helium or nitrogen) purge of 2-3 ml/minute is used to aid in the removal of solvent vapors. The oven temperature is then increased to 110°C, maintaining inert gas flow and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature. Prior to opening the oven, the oven is slightly pressurized with nitrogen to prevent contamination with ambient air. The Tenax is removed from the oven and sieved through a 40/60 mesh sieve (acetone rinsed and oven dried) into a clean glass vessel. If the Tenax is not to be used immediately for cartridge preparation it should be stored in a clean glass jar having a Teflon-lined screw cap and placed in a desiccator.

9.3 Cartridge Preparation and Pretreatment

9.3.1 All cartridge materials are pre-cleaned as described in Section 9.2.2. If the glass cartridge design shown in Figure 1a is employed all handling should be conducted wearing polyester gloves.

9.3.2 The cartridge is packed by placing a 0.5-1cm glass-wool plug in the base of the cartridge and then filling the cartridge to within approximately 1 cm of the top. A 0.5-1cm glasswool plug is placed in the top of the cartridge.

9.3.3 The cartridges are then thermally conditioned by heating for four hours at 270°C under an inert gas (helium) purge (100 - 200 ml/min).

9.3.4 After the four hour heating period the cartridges are allowed to cool. Cartridges of the type shown in Figure 1a are immediately placed (without cooling) in clean culture tubes having Teflon-lined screw caps with a glasswool cushion at both the top and the bottom. Each tube should be shaken to ensure that the cartridge is held firmly in place. Cartridges of the type shown in Figure 1b are allowed to cool to room temperature under inert gas purge and are then closed with stainless steel plugs.

9.3.5 The cartridges are labeled and placed in a tightly sealed metal can (e.g. paint can or similar friction top container). For cartridges of the type shown in Figure 1a the culture tube, not the cartridge,is labeled.

9.3.6 Cartridges should be used for sampling within 2 weeks after preparation and analyzed within two weeks after sampling. If possible the cartridges should be stored at -20°C in a clean freezer (i.e. no solvent extracts or other sources of volatile organics contained in the freezer).

10. Sampling

10.1 Flow rate and Total Volume Selection

10.1.1 Each compound has a characteristic retention volume (liters of air per gram of adsorbent) which must not be exceeded. Since the retention volume is a function of temperature, and possibly other sampling variables, one must include an adequate margin of safety to ensure good collection efficiency. Some considerations and guidance in this regard are provided in a recent report (5). Approximate breakthrough volumes at 38°C (100°F) in liters/gram of Tenax are provided in Table 1. These retention volume data are supplied only as rough guidance and are subject to considerable variability, depending on cartridge design as well as sampling parameters and atmospheric conditions.

10.1.2 To calculate the maximum total volume of air which can be sampled use the following equation:

$$V_{MAX} = \frac{V_b \times W}{1.5}$$

where

V_{MAX} is the calculated maximum total volume in liters.

V_b is the breakthrough volume for the least retained compound of interest (Table 1) in liters per gram of Tenax.

W is the weight of Tenax in the cartridge, in grams.

1.5 is a dimensionless safety factor to allow for variability in atmospheric conditions. This factor is appropriate for temperatures in the range of 25-30°C. If higher temperatures are encountered the factor should be increased (i.e. maximum total volume decreased).

10.1.3 To calculate maximum flow rate use the following equation:

$$Q_{MAX} = \frac{V_{MAX}}{t} \times 1000$$

where

Q_{MAX} is the calculated maximum flow rate in milliliters per minute.

t is the desired sampling time in minutes. Times greater than 24 hours (1440 minutes) generally are unsuitable because the flow rate required is too low to be accurately maintained.

10.1.4 The maximum flow rate Q_{MAX} should yield a linear flow velocity of 50-500 cm/minute. Calculate the linear velocity corresponding to the maximum flow rate using the following equation:

$$B = \frac{Q_{MAX}}{\pi r^2}$$

where

B is the calculated linear flow velocity in centimeters per minute.

r is the internal radius of the cartridge in centimeters.

If B is greater than 500 centimeters per minute either the total sample volume (V_{MAX}) should be reduced or the sample flow rate (Q_{MAX}) should be reduced by increasing the collection time. If B is less than 50 centimeters per minute the sampling rate (Q_{MAX}) should be increased by reducing the sampling time. The total sample value (V_{MAX}) cannot be increased due to component breakthrough.

10.1.4 The flow rate calculated as described above defines the maximum flow rate allowed. In general, one should collect additional samples in parallel, for the same time period but at lower flow rates. This practice yields a measure of quality control and is further discussed in the literature (5). In general, flow rates 2 to 4 fold lower than the maximum flow rate should be employed for the parallel samples. In all cases a constant flow rate should be achieved for each cartridge since accurate integration of the analyte concentration requires that the flow be constant over the sampling period.

10.2 Sample Collection

10.2.1 Collection of an accurately known volume of air is critical to the accuracy of the results. For this reason the use of mass flow controllers, rather than conventional needle valves or orifices is highly recommended, especially at low flow velocities (e.g. less than 100 milliliters/minute). Figure 3a illustrates a sampling system utilizing mass flow controllers. This system readily allows for collection of parallel samples. Figures 3b shows a commercially available system based on needle valve flow controllers.

10.2.2 Prior to sample collection insure that the sampling flow rate has been calibrated over a range including the rate to be used for sampling, with a "dummy" Tenax cartridge in place. Generally calibration is accomplished using a soap bubble flow meter or calibrated wet test meter. The flow calibration device is connected to the flow exit, assuming the entire flow system is sealed. ASTM Method D3686 describes an appropriate calibration scheme, not requiring a sealed flow system downstream of the pump.

10.2.3 The flow rate should be checked before and after each sample collection. If the sampling interval exceeds four hours the flow rate should be checked at an intermediate point during sampling as well. In general, a rotameter should be included, as showed in Figure 3b, to allow observation of the sampling flow rate without disrupting the sampling process.

10.2.4 To collect an air sample the cartridges are removed from the sealed container just prior to initiation of the collection process. If glass cartridges (Figure 1a) are employed they must be handled only with polyester gloves and should not contact any other surfaces.

10.2.5 A particulate filter and holder are placed on the inlet to the cartridges and the exit end of the cartridge is connected to the sampling apparatus. In many sampling situations the use of a filter is not necessary if only the total concentration of a component is desired. Glass cartridges of the type shown in Figure 1a are connected using teflon ferrules and Swagelok (stainless steel or teflon) fittings. Start the pump and record the following parameters on an appropriate data sheet (Figure 4): data, sampling location, time, ambient temperature, barometric

pressure, relative humidity, dry gas meter reading (if applicable) flow rate, rotameter reading (if applicable), cartridge number and dry gas meter serial number.

10.2.6 Allow the sampler to operate for the desired time, periodically recording the variables listed above. Check flow rate at the midpoint of the sampling interval if longer than four hours.
At the end of the sampling period record the parameters listed in 10.2.5 and check the flow rate and record the value. If the flows at the beginning and end of the sampling period differ by more than 10% the cartridge should be marked as suspect.

10.2.7 Remove the cartridges (one at a time) and place in the original container (use gloves for glass cartridges). Seal the cartridges or culture tubes in the friction-top can containing a layer of charcoal and package for immediate shipment to the laboratory for analysis. Store cartridges at reduced temperature (e.g. - 20°C) before analysis if possible to maximize storage stability.

10.2.8 Calculate and record the average sample rate for each cartridge according to the following equation:

$$Q_A = \frac{Q_1 + Q_2 + \ldots Q_N}{N}$$

where

Q_A = Average flow rate in ml/minute.
$Q_1, Q_2, \ldots . Q_N$ = Flow rates determined at beginning, end, and immediate points during sampling.

N = Number of points averaged.

10.2.9 Calculate and record the total volumetric flow for each cartridge using the following equation:

$$V_m = \frac{T \times Q_A}{1000}$$

where

V_m = Total volume sampled in liters at measured temperature and pressure.
T_2 = Stop time.
T_1 = Start time.
T = Sampling time = $T_2 - T_1$, minutes

10.2.10 The total volume (V_s) at standard conditions, 25°C and 760 mmHg, is calculated from the following equation:

$$V_s = V_m \times \frac{P_A}{760} \times \frac{298}{273 + t_A}$$

where

P_A = Average barometric pressure, mmHg
t_A = Average ambient temperature, °C.

11. GC/MS Analysis

11.1 Instrument Set-up

11.1.1 Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 14 discusses specific performance criteria which should be met.

11.1.2 A block diagram of the typical GC/MS system required for analysis of Tenax cartridges is depicted in Figure 5. The operation of such devices is described in 11.2.4. The thermal desorption module must be designed to accommodate the particular cartridge configuration. Exposure of the sample to metal surfaces should be minimized and only stainless steel, or nickel metal surfaces should be employed.

The volume of tubing and fittings leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

11.1.3 The GC column inlet should be capable of being cooled to -70°C and subsequently increased rapidly to approximately 30°C. This can be most readily accomplished using a GC equipped with subambient cooling capability (liquid nitrogen) although other approaches such as manually cooling the inlet of the column in liquid nitrogen may be acceptable.

11.1.4 The specific GC column and temperature program employed will be dependent on the specific compounds of interest. Appropriate conditions are described in the literature (1-3). In general a nonpolar stationary phase (e.g. SE-30, OV-1) temperature programmed from 30°C to 200°C at 8°/minute will be suitable. Fused silica bonded phase columns are preferable to glass columns since they are more rugged and can be inserted directly into the MS ion source, thereby eliminating the need for a GC/MS transfer line.

11.1.5 Capillary column dimensions of 0.3 mm ID and 50 meters long are generally appropriate although shorter lengths may be sufficient in many cases.

11.1.6 Prior to instrument calibration or sample analysis the GC/MS system is assembled as shown in Figure 5. Helium purge flows (through the cartridge) and carrier flow are set at approximately 10 ml/minute and 1-2 ml/minute respectively. If applicable, the injector sweep flow is set at 2-4 ml/minute.

11.1.7 Once the column and other system components are assembled and the various flows established the column temperature is increased to 250°C for approximately four hours (or overnight if desired) to condition the column.

11.1.8 The MS and data system are set according to the manufacturer's instructions. Electron impact ionization (70eV) and an electron multiplier gain of approximately 5×10^4 should be employed. Once the entire GC/MS system has been setup the system is calibrated as described in Section 11.2. The user should prepare a detailed standard operating procedure (SOP) describing this process for the particular instrument being used.

11.2 Instrument Calibration

11.2.1 Tuning and mass standarization of the MS system is performed according to manufacturer's instructions and relevant information from the user prepared SOP. Bromofluorobenzene (BFB) will be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g. lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2 as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria.

However, these alternate values must be repeatable on a day-to-day basis.

11.2.2 After the mass standarization and tuning process has been completed and the appropriate values entered into the data system the user should then calibrate the entire system by introducing known quantities of the standard components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct syringe injection of dilute vapor phase standards, prepared in a dilution bottle, onto the GC column, 2) Injection of dilute vapor phase standards into a carrier gas stream directed through the Tenax cartridge, and 3) introduction of permeation or diffusion tube standards onto a Tenax cartridge. The standards preparation procedures for each of these approaches are described in Section 13. The following paragraphs describe the instrument calibration process for each of these approaches.

11.2.3 If the instrument is to be calibrated by direct injection of a gaseous standard, a standard is prepared in a dilution bottle as described in Section 13.1. The GC column is cooled to -70°C (or, alternately, a portion of the column inlet is manually cooled with liquid nitrogen). The MS and data system is set up for acquisition as described in the relevant user SOP. The ionization filament should be turned off during the initial 2-3 minutes of the run to allow oxygen and other highly volatile components to elute. An appropriate volume (less than 1 ml) of the gaseous standard is injected onto the GC system using an accurately calibrated gas tight syringe.

The system clock is started and the column is maintained at -70°C (or liquid nitrogen inlet cooling) for 2 minutes. The column temperature is rapidly increased to the desired initial temperature (e.g. 30°C). The temperature program is started at a consistent time (e.g. four minutes) after injection. Simultaneously the ionization filament is turned on and data acquisition is initiated. After the last component of interest has eluted acquisiton is terminated and the data is processed as described in Section 11.2.5. The standard injection process is repeated using different standard volumes as desired.

11.2.4 If the system is to be calibrated by analysis of spiked Tenax cartridges a set of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored as described in Section 9.3. If glass cartridges (Figure 1a) are employed care must be taken to avoid direct contact, as described earlier. The GC column is cooled to -70°C, the collection loop is immersed in liquid nitrogen and the desorption module is maintained at 250°C. The inlet valve is placed in the desorb mode and the standard cartridge is placed in the desorption module, making certain that no leakage of purge gas occurs. The cartridge is purged for 10 minutes and then the inlet valve is placed in the inject mode and the liquid nitrogen source removed from the collection trap. The GC column is maintained at -70°C for two minutes and subsequent steps are as described in 11.2.3. After the process is complete the cartridge is removed from the desorption module and stored for subsequent use as described in Section 9.3.

11.2.5 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

11.3 Sample Analysis

11.3.1 The sample analysis process is identical to that described in Section 11.2.4 for the analysis of standard Tenax cartridges.

11.3.2 Data processing for sample data generally involves 1) qualitatively determining the presence or absence of each component of interest on the basis of a set of characteristic ions and the retention time using a reverse-search software routine, 2) quantification of each identified component by integrating the intensity of a characteristic ion and comparing the value to that of the calibration standard, and 3) tentative identification of other components observed using a forward (library) search software routine. As for other user specific processes, a SOP should be prepared describing the specific operations for each individual laboratory.

12. Calculations

12.1 Calibration Response Factors

12.1.1 Data from calibration standards is used to calculate a response factor for each component of interest. Ideally the process involves analysis of at least three calibration levels of each component during a given day and determination of the response factor (area/nanogram injected) from the linear least squares fit of a plot of nanograms injected versus area (for the characteristic ion). In general quantities of component greater than 1000 nanograms should not be injected because of column overloading and/or MS response nonlinearity.

12.1.2 In practice the daily routine may not always allow analysis of three such calibration standards. In this situation calibration data from consecutive days may be pooled to yield a response factor, provided that analysis of replicate standards of the same concentration are shown to agree within 20% on the consecutive days. One standard concentration, near the midpoint of the analytical range of interest, should be chosen for injection every day to determine day-to-day response reproducibility.

12.1.3 If substantial nonlinearity is present in the calibration curve a nonlinear least squares fit (e.g. quadratic) should be employed. This process involves fitting the data to the following equation:

$$Y = A + BX + CX^2$$

where

Y = peak area

X = quantity of component, nanograms

A,B, and C are coefficients in the equation

12.2 Analyte Concentrations

12.2.1 Analyte quantities on a sample cartridge are calculated from the following equation:

$$Y_A = A + BX_A + CX_A$$

where

Y_A is the area of the analyte characteristic ion for the sample cartridge.

X_A is the calculated quantity of analyte on the sample cartridge, in nanograms.

A,B, and C are the coefficients calculated from the calibration curve described in Section 12.1.3.

12.2.2 If instrumental response is essentially linear over the concentration range of interest a linear equation (C=0 in the equation above) can be employed.

12.2.3 Concentration of analyte in the original air sample is calculated from the following equation:

$$C_A = \frac{X_A}{V_S}$$

where

C_A is the calculated concentration of analyte in nanograms per liter.

V_S and X_A are as previously defined in Section 10.2.10 and 12.2.1, respectively.

13. Standard Preparation

13.1 Direct Injection

13.1.1 This process involves preparation of a dilution bottle containing the desired concentrations of compounds of interest for direct injection onto the GC/MS system.

13.1.2 Fifteen three-millimeter diameter glass beads and a one-inch Teflon stirbar are placed in a clean two-liter glass septum capped bottle and the exact volume is determined by weighing the bottle before and after filling with deionized water. The bottle is then rinsed with acetone and dried at 200°C.

13.1.3 The amount of each standard to be injected into the vessel is calculated from the desired injection quantity and volume using the following equation:

$$W_T = \frac{W_I}{V_I} \times V_B$$

where

W_T is the total quantity of analyte to be injected into the bottle in milligrams

W_I is the desired weight of analyte to be injected onto the GC/MS system or spiked cartridge in nanograms

V_I is the desired GC/MS or cartridge injection volume (should not exceed 500) in microliters.

V_B is total volume of dilution bottle determined in 13.1.1, in liters.

13.1.4 The volume of the neat standard to be injected into the dilution bottle is determined using the following equation:

$$V_T = \frac{W_T}{d}$$

where

V_T is the total volume of neat liquid to be injected in microliters.

d is the density of the neat standard in grams per milliliter.

13.1.6 The bottle is placed in a 60°C oven for at least 30 minutes prior to removal of a vapor phase standard.

13.1.7 To withdraw a standard for GC/MS injection the bottle is removed from the oven and stirred for 10-15 seconds. A suitable gas-tight microber syring warmed to 60°C, is inserted through the septum cap and pumped three times slowly. The appropriate volume of sample (approximately 25% larger than the desired injection volume) is drawn into the syringe and the volume is adjusted to the exact value desired and then immediately injected over a 5-10 seconds period onto the GC/MS system as described in Section 11.2.3.

13.2 Preparation of Spiked Cartridges by Vapor Phase Injection

13.2.1 This process involves preparation of a dilution bottle containing the desired concentrations of the compound(s) of interest as described in 13.1 and injecting the desired volume of vapor into a flowing inert gas stream directed through a clean Tenax cartridge.

13.2.2 A helium purge system is assembled wherein the helium flow 20-30 mL/minute is passed through a stainless steel Tee fitted with a septum injector. The clean Tenax cartridge is connected downstream of the tee using appropriate Swagelok fittings. Once the cartridge is placed in the flowing gas stream the appropriate volume vapor standard, in the dilution bottle, is injected through the septum as described in 13.1.6. The syringe is flushed several times by alternately filling the syringe with carrier gas and displacing the contents into the flow stream, without removing the syringe from the septum. Carrier flow is maintain through the cartridge for approximately 5 minutes after injection.

13.3 Preparation of Spiked Traps Using Permeation or Diffusion tubes

13.3.1 A flowing stream of inert gas containing known amounts of each compound of interest is generated according to ASTM Method D3609(6). Note that a method of accuracy maintaining temperature within ± 0.1°C is required and the system generally must be equilibrated for at least 48 hours before use.

13.3.2 An accurately known volume of the standard gas stream (usually 0.1-1 liter) is drawn through a clean Tenax cartridge using the sampling system described in Section 10.2.1, or a similar system. However, if mass flow controllers are employed they must be calibrated for the carrier gas used in Section 13.3.1 (usually nitrogen). Use of air as the carrier gas for permeation systems is not recommended, unless the compounds of interest are known to be highly stable in air.

13.3.3 The spiked cartridges are then stored or immediately analyzed as in Section 11.2.4.

14. Performance Criteria and Quality Assurance

This section summarizes quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory. In many cases the specific QA procedures have been described within the appropriate section describing the particular activity (e.g. parallel sampling).

14.1 Standard Opreating Procedures (SOPs)

14.1.1 Each user should generate SOPs describing the following activities as they are performed in their laboratory:

1) assembly, calibration, and operation of the sampling system,
2) preparation, handling and storage of Tenax cartridges,
3) assembly and operation of GC/MS system including the thermal desorption apparatus and data system, and
4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by the laboratory personnel conducting the work.

14.2 Tenax Cartridge Preparation

14.2.1 Each batch of Tenax cartridges prepared (as described in Section 9) should be checked for contamination by analyzing one cartridge immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria the entire lot should be rejected.

14.3 Sample Collection

14.3.1 During each sampling event at least one clean cartridge will accompany the samples to the field and back to the laboratory, without being used for sampling, to serve as a field blank. The average amount of material found on the field blank cartridge may be subtracted from the amount found on the actual samples. However, if the blank level is greater than 25% of the sample amount, data for that component must be identified as suspect.

14.3.2 During each sampling event at least one set of parallel samples (two or more samples collected simultaneously) will be collected, preferably at different flow rates as described in Section 10.1. If agreement between parallel samples is not generally within $\pm$ 25% the user should collect parallel samples on a much more frequent basis (perhaps for all sampling points). If a trend of lower apparent concentrations with increasing flow rate is observed for a set

of parallel samples one should consider using a reduced flow rate and longer sampling interval if possible. If this practice does not improve the reproducibility further evaluation of the method performance for the compound of interest may be required.

14.3.3 Backup cartridges (two cartridges in series) should be collected with each sampling event. Backup cartridges should contain less than 20% of the amount of components of interest found in the front cartridges, or be equivalent to the blank cartridge level, whichever is greater. The frequency of use of backup cartridges should be increased if increased flow rate is shown to yield reduced component levels for parallel sampling. This practice will help to identify problems arising from breakthrough of the component of interest during sampling.

14.4 GC/MS Analysis

14.4.1 Performance criteria for MS tuning and mass calibration have been discussed in Section 11.2 and Table 2. Additional criteria may be used by the laboratory if desired. The following sections provide performance guidance and suggested criteria for determining the acceptability of the GC/MS system.

14.4.2 Chromatographic efficiency should be evaluated using spiked Tenax cartridges since this practice tests the entire system. In general a reference compound such as perfluorotoluene should be spiked onto a cartridge at the 100 nanogram level as described in Section 13.2 or 13.3. The cartridge is then analyzed by GC/MS as

described in Section 11.4. The perfluorotoluene (or other reference compound) peak is then plotted on an expanded time scale so that its width at 10% of the peak can be calculated, as shown in Figure 6. The width of the peak at 10% height should not exceed 10 seconds. More stringent criteria may be required for certain applications. The assymmetry factor (See Figure 6) should be between 0.8 and 2.0. The assymmetry factor for any polar or reactive compounds should be determined using the process described above. If peaks are observed that exceed the peak width or assymmetry factor criteria above, one should inspect the entire system to determine if unswept zones or cold spots are present in any of the fittings and is necessary. Some laboratories may chose to evaluate column performance separately by direct injection of a test mixture onto the GC column. Suitable schemes for column evaluation have been reported in the literature (7). Such schemes cannot be conducted by placing the substances onto Tenax because many of the compounds (e.g. acids, bases, alcohols) contained in the test mix are not retained, or degrade, on Tenax.

14.4.3 The system detection limit for each component is calculated from the data obtained for calibration standards. The detection limit is defined as

$$DL = A + 3.3S$$

where

DL is the calculated detection limit in nanograms injected.

A is the intercept calculated in Section 12.1.1 or 12.1.3.

S is the standard deviation of replicate determinations of the lowest level standard (at least three such determinations are required.

In general the detection limit should be 20 nanograms or less and for many applications detection limits of 1-5 nanograms may be required. The lowest level standard should yield a signal to noise ratio, from the total ion current response, of approximately 5.

14.4.4 The relative standard deviation for replicate analyses of cartridges spiked at approximately 10 times the detection limit should be 20% or less. Day to day relative standard deviation should be 25% or less.

14.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present in ambient air (e.g. perfluorotoluene). The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than $\pm$ 2 standard deviations from the mean (calculated

excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

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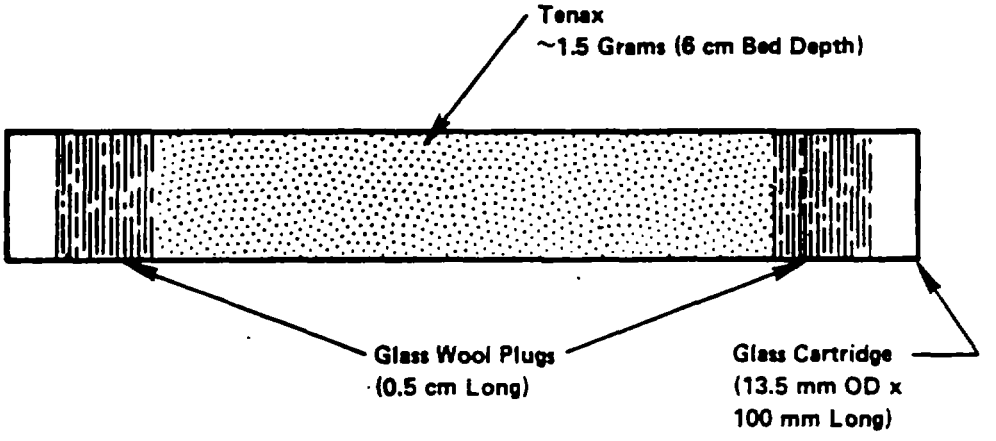
TABLE 1. RETENTION VOLUME ESTIMATES FOR COMPOUNDS ON TENAX

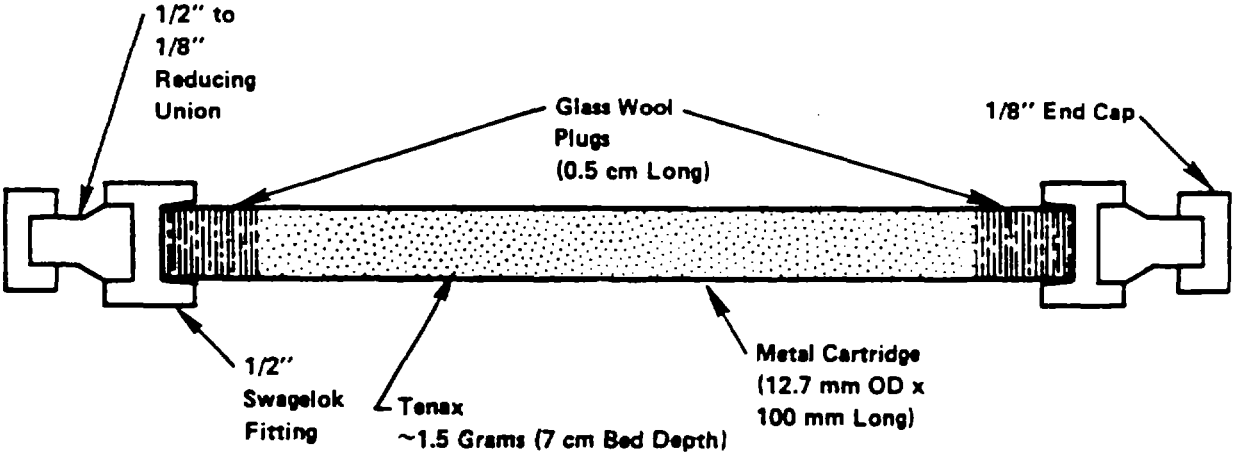
COMPOUND	ESTIMATED RETENTION VOLUME AT 100°F (38°C)-LITERS/GRAM
Benzene	19
Toluene	97
Ethyl Benzene	200
Xylene(s)	~ 200
Cumene	440
n-Heptane	20
1-Heptene	40
Chloroform	8
Carbon Tetrachloride	8
1,2-Dichloroethane	10
1,1,1-Trichloroethane	6
Tetrachloroethylene	80
Trichloroethylene	20
1,2-Dichloropropane	30
1,3-Dichloropropane	90
Chlorobenzene	150
Bromoform	100
Ethylene Dibromide	60
Bromobenzene	300

TABLE 2. SUGGESTED PERFORMANCE CRITERIA FOR RELATIVE ION ABUNDANCES FROM FC-43 MASS CALIBRATION

M/E	% RELATIVE ABUNDANCE
51	1.8 ± 0.5
69	100
100	12.0 ± 1.5
119	12.0 ± 1.5
131	35.0 ± 3.5
169	3.0 ± 0.4
219	24.0 ± 2.5
264	3.7 ± 0.4
314	0.25 ± 0.1

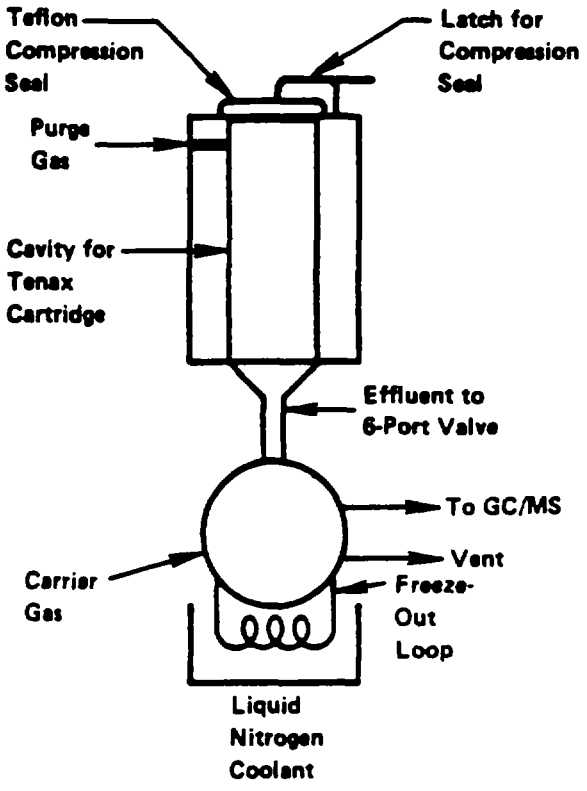


(a) Glass Cartridge

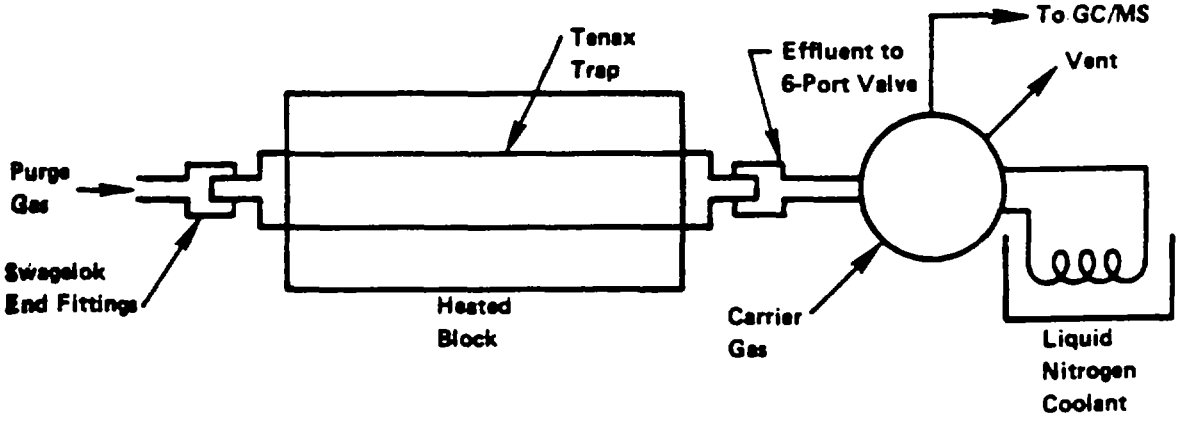


(b) Metal Cartridge

FIGURE 1. TENAX CARTRIDGE DESIGNS

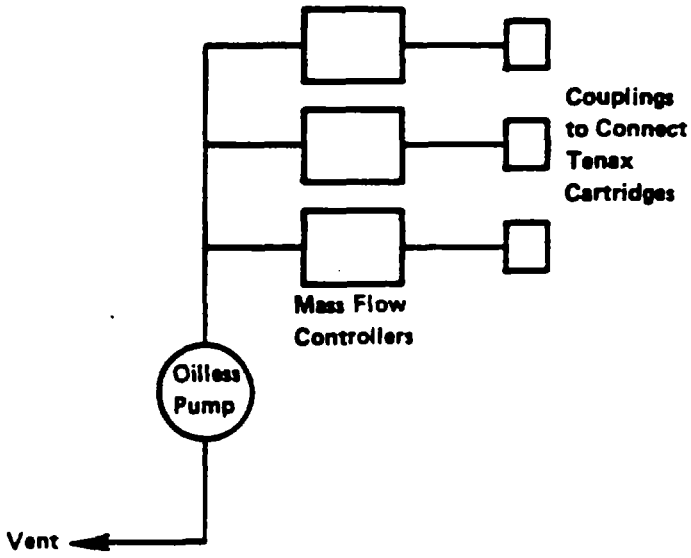


(a) Glass Cartridges (Compression Fit)

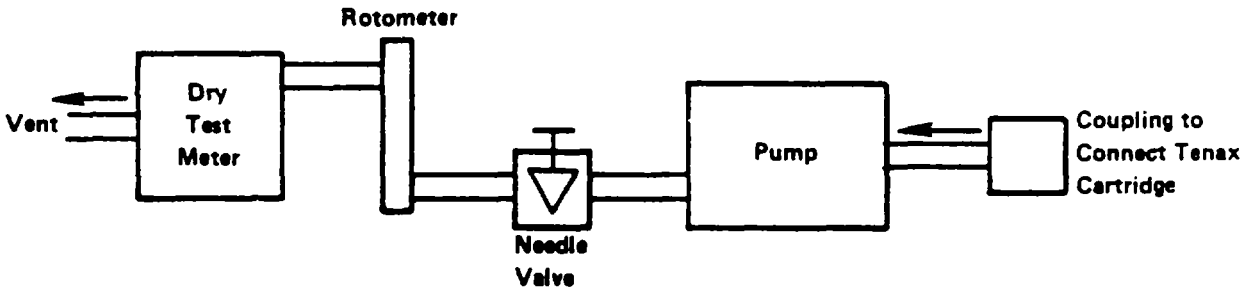


(b) Metal Cartridges (Swagelok Fittings)

FIGURE 2. TENAX CARTRIDGE DESORPTION MODULES



(a) Mass Flow Control



(b) Needle Valve Control

FIGURE 3. TYPICAL SAMPLING SYSTEM CONFIGURATIONS

SAMPLING DATA SHEET
(One Sample Per Data Sheet)

PROJECT: _____ DATE(S) SAMPLED: _____

SITE: _____ TIME PERIOD SAMPLED: _____

LOCATION: _____ OPERATOR: _____

INSTRUMENT MODEL NO: _____ CALIBRATED BY: _____

PUMP SERIAL NO: _____

SAMPLING DATA

Sample Number: _____

Start Time: _____ Stop Time: _____

Time	Dry Gas Meter Reading	Rotameter Reading	Flow Rate,*Q ml/Min	Ambient Temperature °C	Barometric Pressure, mmHg	Relative Humidity, %	Comments
1.							
3.							
4.							
N.							

Total Volume Data**

V_m = (Final - Initial) Dry Gas Meter Reading, or = _____ Liters

$$= \frac{Q_1 + Q_2 + Q_3 \ldots Q_N}{N} \times \frac{1}{1000 \times (\text{Sampling Time in Minutes})} = \text{_____ Liters}$$

* Flowrate from rotameter or soap bubble calibrator (specify which).
** Use data from dry gas meter if available.

FIGURE 4. EXAMPLE SAMPLING DATA SHEET

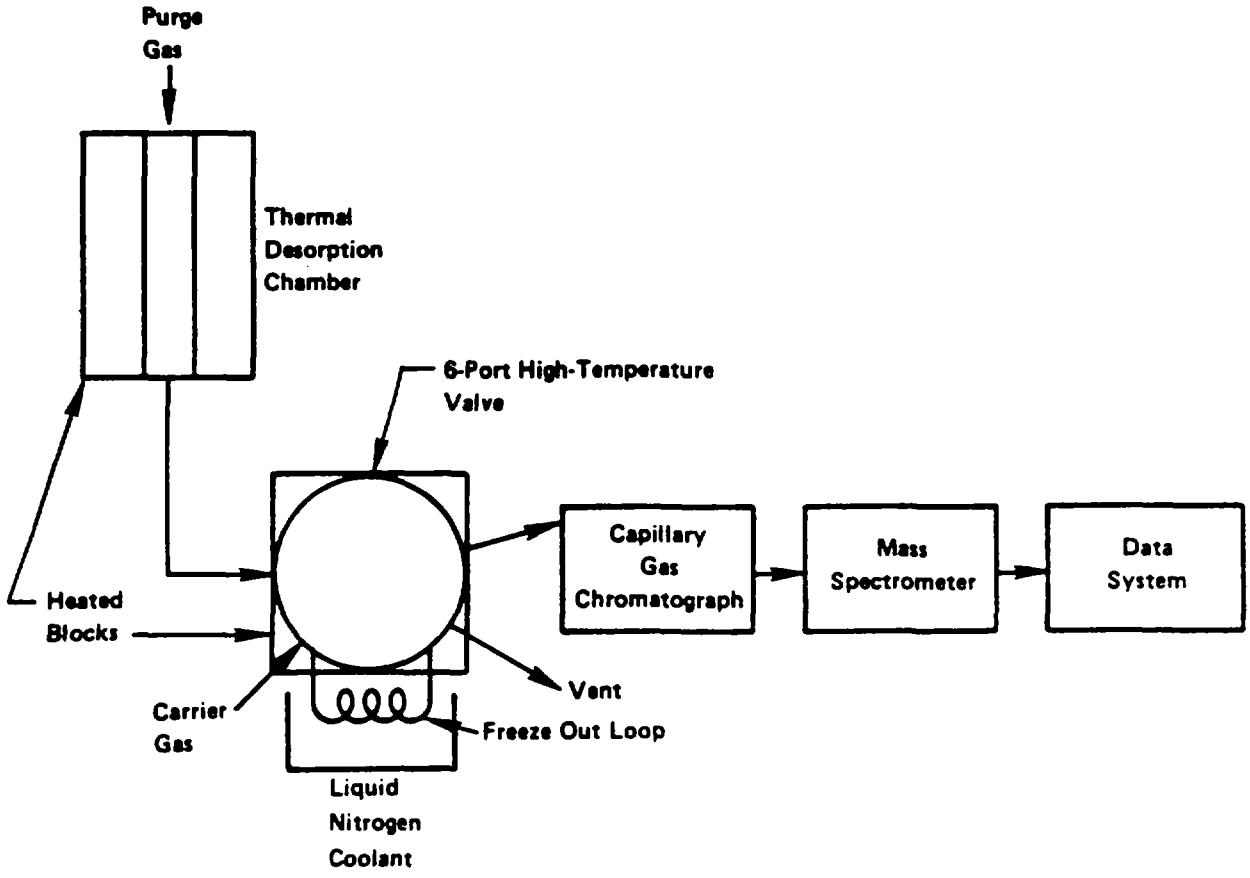
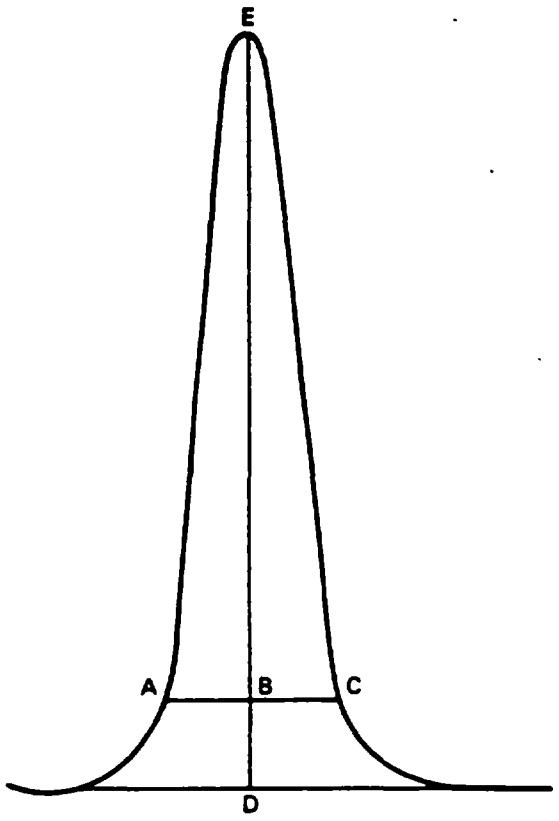


FIGURE 5. BLOCK DIAGRAM OF ANALYTICAL SYSTEM



$$\text{Asymmetry Factor} = \frac{BC}{AB}$$

Example Calculation:

Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Asymmetry Factor = $\frac{12}{11}$ = 1.1

FIGURE 6. PEAK ASYMMETRY CALCULATION

METHOD TO2

Revision 1.0
April, 1984

METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR BY CARBON MOLECULAR SIEVE ADSORPTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1. Scope

1.1 This document describes a procedure for collection and determination of selected volatile organic compounds which can be captured on carbon molecular sieve (CMS) adsorbents and determined by thermal desorption GC/MS techniques.

1.2 Compounds which can be determined by this method are nonpolar and nonreactive organics having boiling points in the range -15 to +120°C. However, not all compounds meeting these criteria can be determined. Compounds for which the performance of the method has been documented are listed in Table 1. The method may be extended to other compounds but additional validation by the user is required. This method has been extensively used in a single laboratory. Consequently, its general applicability has not been thoroughly documented.

2. Applicable Documents

2.1 ASTM Standards

D 1356 Definitions of Terms Related to Atmospheric Sampling and Analysis.

E 355 Recommended Practice for Gas Chromatography Terms and Relationships.

2.2 Other Documents

Ambient Air Studies (1,2).

U.S. EPA Technical Assistance Document (3).

3. Summary of Method

3.1 Ambient air is drawn through a cartridge containing ~0.4 of a carbon molecular sieve (CMS) adsorbent. Volatile organic compounds are captured on the adsorbent while major inorganic atmospheric constituents pass through (or are only partially retained). After sampling, the cartridge is returned to the laboratory for analysis.

3.2 Prior to analysis the cartridge is purged with 2-3 liters of pure, dry air (in the same direction as sample flow) to remove adsorbed moisture.

3.3 For analysis the cartridge is heated to 350°-400°C, under helium purge and the desorbed organic compounds are collected in a specially designed cryogenic trap. The collected organics are then flash evaporated onto a capillary column GC/MS system (held at -70°C). The individual components are identified and quantified during a temperature programmed chromatographic run.

3.4 Due to the complexity of ambient air samples, only high resolution (capillary column) GC techniques are acceptable for most applications of the method.

4. Significance

4.1 Volatile organic compounds are emitted into the atmosphere from a variety of sources including industrial and commercial facilities, hazardous waste storage and treatment facilities, etc. Many of these compounds are toxic; hence knowledge of the concentration of such materials in the ambient atmosphere is required in order to determine human health impacts.

4.2 Traditionally air monitoring methods for volatile organic compounds have relied on carbon adsorption followed by solvent desorption and GC analysis. Unfortunately, such methods are not sufficiently sensitive for ambient air monitoring, in most cases, because only a small portion of

the sample is injected onto the GC system. Recently on-line thermal desorption methods, using organic polymeric adsorbents such as Tenax® GC, have been used for ambient air monitoring. The current method uses CMS adsorbents (e.g. Spherocarb®) to capture highly volatile organics (e.g. vinyl chloride) which are not collected on Tenax®. The use of on-line thermal desorption GC/MS yields a sensitive, specific analysis procedure.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356 (4). All abbreviations and symbols are defined with this document at the point of use.

6. Interferences

6.1 Only compounds having a mass spectrum and GC retention time similar to the compound of interest will interfere in the method. The most commonly encountered interferences are structural isomers.

6.2 Contamination of the CMS cartridge with the compound(s) of interest can be a problem in the method. The user must be careful in the preparation, storage, and handling of the cartridges through the entire process to minimize contamination.

7. Apparatus

7.1 Gas Chromatograph/Mass Spectrometry system - must be capable of subambient temperature programming. Unit mass resolution to 800 amu. Capable of scanning 30-300 amu region every 0.5-0.8 seconds. Equipped with data system for instrument control as well as data acquisition, processing and storage.

7.2 Thermal Desorption Injection Unit - Designed to accommodate CMS cartridges in use (See Figure 3) and including cryogenic trap (Figure 5) and injection valve (Carle Model 5621 or equivalent).

7.3 Sampling System - Capable of accurately and precisely drawing an air flow of 10-500 ml/minute through the CMS cartridge. (See Figure 2a or b.)

7.4 Dewar flasks - 500 mL and 5 liter.

7.5 Stopwatches.

7.6 Various pressure regulators and valves - for connecting compressed gas cylinders to GC/MS system.

7.7 Calibration gas - In aluminum cylinder. Prepared by user or vendor. For GC/MS calibration.

7.8 High pressure apparatus for preparing calibration gas cylinders (if conducted by user). Alternatively, custom prepared gas mixtures can be purchased from gas supply vendors.

7.9 Friction top can (e.g. one-gallon paint can) - With layer of activated charcoal to hold clean CMS cartridges.

7..10 Thermometer - to record ambient temperature.

7.11 Barometer (optional).

7.12 Dilution bottle - Two-liter with septum cap for standard preparation.

7.13 Teflon stirbar - 1 inch long

7.14 Gas tight syringes - 10-500 µl for standard injection onto GC/MS system and CMS cartridges.

7.15 Liquid microliter syringes - 5-50 µL for injecting neat liquid standards into dilution bottle.

7.16 Oven - 60 $\pm$ 5°C for equilibrating dilution bottle.

7.17 Magnetic stirrer.

7.18 Variable voltage transformers - (120 V and 1000 VA) and electrical connectors (or temperature controllers) to heat cartridge and cryogenic loop.

7.19 Digital pyrometer - 30 to 500°C range.

7.20 Soap bubble flow meter - 1, 10 and 100 mL calibration points.

7.21 Copper tubing (1/8 inch) and fittings for gas inlet lines.

7.22 GC column - SE-30 or alternative coating, glass capillary or fused silica.

7.23 Psychrometer (optional).

7.24 Filter holder - stainless steel or aluminum (to accommodate 1 inch diameter filter). Other sizes may be used if desired. (optional)

8. Reagents and Materials

8.1 Empty CMS cartridges - Nickel or stainless steel (See Figure 1).

8.2 CMS Adsorbent, 60/80 mesh- Spherocarb® from Analabs Inc., or equivalent.

8.3 Glasswool - silanized.

8.4 Methylene chloride - pesticide quality, or equivalent.

8.5 Gas purifier cartridge for purge and GC carrier gas containing charcoal, molecular sieves, and a drying agent. Available from various chromatography supply houses.

8.6 Helium - Ultra pure, (99.9999%) compressed gas.

8.7 Nitrogen - Ultra pure, (99.9999%) compressed gas.

8.8 Liquid nitrogen or argon (50 liter dewar).

8.9 Compressed air, if required - for operation of GC oven door.

8.10 Perfluorotributylamine (FC-43) for GC/MS calibration.

8.11 Chemical Standards - Neat compounds of interest. Highest purity available.

9. Cartridge Construction and Preparation

9.1 A suitable cartridge design in shown in Figure 1. Alternate designs have been reported (1) and are acceptable, provided the user documents their performance. The design shown in Figure 1 has a built-in heater assembly. Many users may choose to replace this heater design with a suitable separate heating block or oven to simplify the cartridge design.

9.2 The cartridge is assembled as shown in Figure 1 using standard 0.25 inch O.D. tubing (stainless steel or nickel), 1/4 inch to 1/8 inch reducing unions, 1/8 inch nuts, ferrules, and endcaps. These parts are rinsed with methylene chloride and heated at 250°C for 1 hour prior to assembly.

9.3 The thermocouple bead is fixed to the cartridge body, and insulated with a layer of Teflon tape. The heater wire (constructed from a length of thermocouple wire) is wound around the length of the cartridge and wrapped with Teflon tape to secure the wire in place. The cartridge is then wrapped with woven silica fiber insulation (Zetex or equivalent). Finally the entire assembly is wrapped with fiber glass tape.

9.4 After assembly one end of the cartridge is marked with a serial number to designate the cartridge inlet during sample collection.

9.5 The cartridges are then packed with ~0.4 grams of CMS adsorbent. Glasswool plugs (~0.5 inches long) are placed at each end of the cartridge to hold the adsorbent firmly in place. Care must be taken to insure that no strands of glasswool extend outside the tubing, thus causing leakage in the compression endfittings. After loading the endfittings (reducing unions and end caps) are tightened onto the cartridge.

9.6 The cartridges are conditioned for initial use by heating at 400°C overnight (at least 16 hours) with a 100 mL/minute purge of pure nitrogen. Reused cartridges need only to be heated for 4 hours and should be reanalyzed before use to ensure complete desorption of impurities.

9.7 For cartridge conditioning ultra-pure nitrogen gas is passed through a gas purifier to remove oxygen,moisture and organic contaminants. The nitrogen supply is connected to the unmarked end of the cartridge and the flow adjusted to ~50 mL/minute using a needle valve. The gas flow from the inlet (marked) end of the cartridge is vented to the atmosphere.

9.8 The cartridge thermocouple lead is connected to a pyrometer and the heater lead is connected to a variable voltage transformer (Variac) set at 0 V. The voltage on the Variac is increased to ~15 V and adjusted over a 3-4 minute period to stabilize the cartridge temperature at 380-400°C.

9.9 After 10-16 hours of heating (for new cartridges) the Variac is turned off and the cartridge is allowed to cool to ~30°C, under continuing nitrogen flow.

9.10 The exit end of the cartridge is capped and then the entire cartridge is removed from the flow line and the other endcap immediately installed. The cartridges are then placed in a metal friction top (paint) can containing ~2 inches of granulated activated charcoal (to prevent contamination of the cartridges during storage) in the bottom, beneath a retaining screen. Clean paper tissues (e.g. Kimwipes) are placed in can to avoid damage to the cartridges during shipment.

9.11 Cartridges are stored in the metal can at all times except when in use. Adhesives initially present in the cartridge insulating materials are "burnt off" during initial conditioning. Therefore, unconditioned cartridges should not be placed in the metal can since they may contaminate the other cartridges.

9.12 Cartridges are conditioned within two weeks of use. A blank from each set of cartridges is analyzed prior to use in field

sampling. If an acceptable blank level is achieved, that batch of cartridges (including the cartridge serving as the blank) can be used for field sampling.

10. Sampling

10.1 Flow Rate and Total Volume Selection

10.1.1 Each compound has a characteristic retention volume (liters of air per unit weight of adsorbent). However, all of the compounds listed in Table 1 have retention volumes (at 37°C) in excess of 100 liters/cartridge (0.4 gram CMS cartridge) except vinyl chloride for which the value is ~30 liters/cartridge. Consequently, if vinyl chloride or similarly volatile compounds are of concern the maximum allowable sampling volume is approximately 20 liters. If such highly volatile compounds are not of concern, samples as large as 100 liters can be collected.

10.1.2 To calculate the maximum allowable sampling flow rate the following equation can be used:

$$Q_{Max} = \frac{V_{Max}}{t} \times 1000$$

where

Q_{Max} is the calculated maximum sampling rate in mL/minute.

t is the desired sampling time in minutes.

V_{Max} is the maximum allowable total volume based on the discussion in 10.1.1.

10.1.3 For the cartridge design shown in Figure 1 Q_{Max} should be between 20 and 500 mL/minute. If Q_{Max} lies outside this range the sampling time or total sampling volume must be adjusted so that this criterion is achieved.

10.1.4 The flow rate calculated in 10.1.3 defines the maximum allowable flow rate. In general, the user should collect additional samples in parallel, at successive 2- to 4-fold lower flow rates. This practice serves as a quality control procedure to check on component breakthrough and related sampling and adsorption problems, and is further discussed in the literature (5).

10.2 Sample Collection

10.2.1 Collection of an accurately known volume of air is critical to the accuracy of the results. For this reason the use of mass flow controllers, rather than conventional needle valves or orifices is highly recommended, especially at low flow rates (e.g. less than 100 milliliters/minute). Figure 2a illustrates a sampling system based on mass flow controllers which readily allows for collection of parallel samples. Figure 2b shows a commercially available sampling system based on needle valve flow controllers.

10.2.2 Prior to sample collection the sampling flow rate is calibrated near the value used for sampling, with a "dummy" CMS cartridge in place. Generally calibration is accomplished using a soap bubble flow meter or calibrated wet test meter connected to the flow exit, assuming the entire flow system is sealed. ASTM Method D 3686 (4) describes an appropriate calibration scheme, not requiring a sealed flow system downstream of the pump.

10.2.3 The flow rate should be checked before and after each sample collection. Ideally, a rotameter or mass flow meter should be included in the sampling system to allow periodic observation of the flow rate without disrupting the sampling process.

10.2.4 To collect an air sample the cartridges are removed from the sealed container just prior to initiation of the collection process.

10.2.5 The exit (unmarked) end of the cartridge is connected to the sampling apparatus. The endcap is left on the sample inlet and the entire system is leak checked by activating the sampling pump and observing that no flow is obtained over a 1 minute period. The sampling pump is then shut off.

10.2.6 The endcap is removed from the cartridge, a particulate filter and holder are placed on the inlet end of the cartridge, and the sampling pump is started. In many situations a particulate filter is not necessary since the compounds of interest are in the vapor state. However, if, large amounts of particulate matter are encountered, the filter may be useful to prevent contamination of the cartridge. The following parameters are recorded on an appropriate data sheet (Figure 4): date, sampling location, time, ambient temperature, barometric pressure, relative humidity, dry gas meter reading (if applicable), flow rate, rotometer reading (if applicable), cartridge number, pump, and dry gas meter serial number.

10.2.7 The samples are collected for the desired time, periodically recording the variables listed above. At the end of the sampling period the parameters listed in 10.2.6 are recorded and the flow rate is checked. If the flows at the beginning and end of the sampling period differ by more than 10%, the cartridge should be marked as suspect.

10.2.8 The cartridges are removed (one at a time), the endcaps are replaced, and the cartridges are placed into the original container. The friction top can is sealed and packaged for immediate shipment to the analytical laboratory.

10.2.9 The average sample rate is calculated and recorded for each cartridge according to the following equation:

$$Q_A = \frac{Q_1 + Q_2 + \ldots Q_N}{N}$$

where

Q_A = Average flow rate in ml/minute.
Q_1, Q_2,...Q_N = Flow rates determined at beginning, end, and immediate points during sampling.

N = Number of points averaged.

10.2.10 The total volumetric flow is obtained directly from the dry gas meter or calculated and recorded for each cartridge using the following equation:

$$V_m = \frac{T \times Q_A}{1000}$$

where

V_m = Total volume sampled in liters at measured temperature and pressure.
T = Sampling time = T_2-T_1, minutes.

10.2.11 The total volume sampled (V_s) at standard conditions, 760 mm Hg and 25°C, is calculated from the following equation:

$$V_s = V_m \times \frac{Pa}{760} \times \frac{298}{273 + ta}$$

where

Pa = Average barometric pressure, mm Hg
ta = Average ambient temperature, °C.

11. Sample Analysis

11.1 Sample Purging

11.1.1 Prior to analysis all samples are purged at room temperature with pure, dry air or nitrogen to remove water vapor. Purging is accomplished as described in 9.7 except that the gas flow is in the same direction as sample flow (i.e. marked end of cartridge is connected to the flow system).

11.1.2 The sample is purged at 500 mL/minute for 5 minutes. After purging the endcaps are immediately replaced. The cartridges are returned to the metal can or analyzed immediately.

11.1.3 If very humid air is being sampled the purge time may be increased to more efficiently remove water vapor. However, the sum of sample volume and purge volume must be less than 75% of the retention volume for the most volatile component of interest.

11.2 GC/MS Setup

11.2.1 Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore, each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 14 discusses specific performance criteria which should be met.

11.2.2 A block diagram of the analytical system required for analysis of CMS cartridges is depicted in Figure 3. The thermal desorption system must be designed to accommodate the particular cartridge configuration. For the CMS cartridge design shown in Figure 1, the cartridge heating is accomplished as described in 9.8. The use of a desorption oven, in conjunction with a

simplier cartridge design is also acceptable. Exposure of the sample to metal surfaces should be minimized and only stainless steel or nickel should be employed. The volume of tubing leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

11.2.3 The GC column oven must be capable of being cooled to -70°C and subsequently temperature programmed to 150°C.

11.2.4 The specific GC column and temperature program employed will be dependent on the compounds of interest. Appropriate conditions are described in the literature (2). In general, a nonpolar stationary phase (e.g. SE-30, OV-1) temperature programmed from -70 to 150°C at 8°/minute will be suitable. Fused silica, bonded-phase columns are preferable to glass columns since they are more rugged and can be inserted directly into the MS ion source, thereby eliminating the need for a GC/MS transfer line. Fused silica columns are also more readily connected to the GC injection valve (Figure 3). A drawback of fused silica, bonded-phase columns is the lower capacity compared to coated, glass capillary columns. In most cases the column capacity will be less than 1 microgram injected for fused silica columns.

11.2.5 Capillary column dimensions of 0.3 mm ID and 50 meters long are generally appropriate although shorter lengths may be sufficient in many cases.

11.2.6 Prior to instrument calibration or sample analysis the GC/MS system is assembled as shown in Figure 3. Helium purge flow (through the cartridge) and carrier flow are set at approximately 50 mL/minute and 2-3 mL/minute respectively. When a cartridge is not in place a union is placed in the helium purge line to ensure a continuous inert gas flow through the injection loop.

11.2.7 Once the column and other system components are assembled and the various flows established the column temperature is increased to 250°C for approximately four hours (or overnight if desired) to condition the column.

11.2.8 The MS and data system are set up according to the manufacturer's instructions. Electron impact ionization (70eV) and an electron multiplier gain of approximately 5×10^4 should be employed. Once the entire GC/MS system has been setup the system is calibrated as described in Section 11.3. The user should prepare a detailed standard operating procedure (SOP) describing this process for the particular instrument being used.

11.3 GC/MS Calibration

11.3.1 Tuning and mass standardization of the MS system is performed according to manufacturer's instructions and relevant user prepared SOPs. Bromofluorobenzene (BFB) will be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g., lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2, as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria. However, these alternate values must be repeatable on a day-to-day basis.

11.3.2 After the mass standardization and tuning process has been completed and the appropriate values entered into the data system, the user should then calibrate the entire GC/MS system by introducing known quantities of the components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct injection of dilute vapor phase standards, prepared in a dilution bottle or compressed gas cylinder, onto the GC column, 2) injection of dilute vapor phase standards into a flowing inert gas stream directed onto a CMS cartridge, and 3) introduction of permeation or diffusion tube standards onto a CMS cartridge. Direct injection of a compressed gas cylinder (aluminum) standard containing trace levels of the compounds of interest has been found to be the most convenient practice since such standards are stable over a several month period. The standards preparation processes for the various approaches are described in Section 13. The following paragraphs describe the instrument calibration process for these approaches.

11.3.3 If the system is to be calibrated by direct injection of a vapor phase standard, the standard, in either a compressed gas cylinder or dilution flask, is obtained as described in Section 13. The MS and data system are setup for acquisition, but the ionizer filament is shut off. The GC column oven is cooled to -70°C, the injection valve is placed in the load mode, and the cryogenic loop is immersed in liquid nitrogen or liquid argon. Liquid argon is required for standards prepared in nitrogen or air, but not for standards prepared in helium. A known volume of the standard (10-1000 mL) is injected through the cryogenic loop at a rate of 10-100 mL/minute.

11.3.4 Immediately after loading the vapor phase standard, the injection valve is placed in the inject mode, the GC program and system clock are started, and the cryogenic loop is heated to 60°C by applying voltage (15-20 volts) to the thermocouple wire heater surrounding the loop. The voltage is adjusted to maintain a loop temperature of 60°C. An automatic temperature controller can be used in place of the manual control system. After elution of unretained components (~3 minutes after injection) the ionizer filament is turned on and data acquisition is initiated. The helium purge line (set at 50 mL/minute) is connected to the injection valve and the valve is returned to the load mode. The loop temperature is increased to 150 °C, with helium purge, and held at this temperature until the next sample is to be loaded.

11.3.5 After the last component of interest has eluted, acquisition is terminated and the data is processed as described in Section 11.3.8. The standard injection process is repeated using different standard concentrations and/or volumes to cover the analytical range of interest.

11.3.6 If the system is to be calibrated by analysis of standard CMS cartridges, a series of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored (no longer than 48 hours) as described in Section 9.10. For analysis the injection valve is placed in the load mode and the cryogenic loop is immersed in liquid nitrogen (or liquid argon if desired). The CMS cartridge is installed in the helium purge line (set at 50 mL/minute) so that the helium flow through the cartridge is opposite to the direction of sample flow and the purge gas is directed through the cryogenic loop and vented to the

atmosphere. The CMS cartridge is heated to 370-400°C and maintained at this temperature for 10 minutes (using the temperature control process described in Section 9.8). During the desorption period, the GC column oven is cooled to -70°C and the MS and data system are setup for acquisition, but the ionizer filament is turned off.

11.3.7 At the end of the 10 minute desorption period, the analytical process described in Sections 11.3.4 and 11.3.5 is conducted. During the GC/MS analysis heating of the CMS cartridge is discontinued. Helium flow is maintained through the CMS cartridge and cryogenic loop until the cartridge has cooled to room temperature. At that time, the cryogenic loop is allowed to cool to room temperature and the system is ready for further cartridge analysis. Helium flow is maintained through the cryogenic loop at all times, except during the installation or removal of a CMS cartridge, to minimize contamination of the loop.

11.3.8 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved, the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

11.4 Sample Analysis

11.4.1 The sample analysis is identical to that described in Sections 11.3.6 and 11.3.7 for the analysis of standard CMS cartridges.

11.4.2 Data processing for sample data generally involves 1) qualitatively determining the presence or absence of each component of interest on the basis of a set of characteristic ions and the retention time using a reversed-search software routine, 2) quantification of each identified component by integrating the intensity of a characteristic ion and comparing the value to that of the calibration standard, and 3) tentative identification of other components observed using a forward (library) search software routine. As for other user specific processes, a SOP should be prepared describing the specific operations for each individual laboratory.

12. Calculations

12.1 Calibration Response Factors

12.1.1 Data from calibration standards is used to calculate a response factor for each component of interest. Ideally the process involves analysis of at least three calibration levels of each component during a given day and determination of the response factor (area/nanogram injected) from the linear least squares fit of a plot of nanograms injected versus area (for the characteristic ion). In general, quantities of components greater than 1,000 nanograms should not be injected because of column overloading and/or MS response nonlinearity.

12.1.2 In practice the daily routine may not always allow analysis of three such calibration standards. In this situation calibration data from consecutive days may be pooled to yield a response factor, provided that analysis of replicate standards of the same concentration are shown to agree within 20% on the consecutive days. In all cases one given standard

concentration, near the midpoint of the analytical range of interest, should be injected at least once each day to determine day-to-day precision of response factors.

12.1.3 Since substantial nonlinearity may be present in the calibration curve, a nonlinear least squares fit (e.g. quadratic) should be employed. This process involves fitting the data to the following equation:

$$Y = A + BX + CX^2$$

where

Y = peak area
X = quantity of component injected nanograms
A, B, and C are coefficients in the equation.

12.2 Analyte Concentrations

12.2.1 Analyte quantities on a sample cartridge are calculated from the following equation:

$$Y_A = A + BX_A + CX^2$$

where Y_A is the area of the analyte characteristic ion for the sample cartridge.
X_A is the calculated quantity of analyte on the sample cartridge, in nanograms.
A, B, and C are the coefficients calculated from the calibration curve described in Section 12.1.3.

12.2.2 If instrumental response is essentially linear over the concentration range of interest, a linear equation (C=0 in the equation above) can be employed.

12.2.3 Concentration of analyte in the original air sample is calculated from the following equation:

$$C_A = \frac{X_A}{V_s}$$

where

C_A is the calculated concentration of analyte in ng/L.

V_s and X_A are as previously defined in Section 10.2.11 and 12.2.1, respectively.

13. Standard Preparation

13.1 Standards for Direct Injection

13.1.1 Standards for direct injection can be prepared in compressed gas cylinders or in dilution vessels. The dilution flask protocol has been described in detail in another method and is not repeated here (6). For the CMS method where only volatile compounds (boiling point <120°C) are of concern, the preparation of dilute standards in 15 liter aluminum compressed gas cylinders has been found to be most convenient. These standards are generally stable over at least a 3-4 month period and in some cases can be purchased from commercial suppliers on a custom prepared basis.

13.1.2 Preparation of compressed gas cylinders requires working with high pressure tubing and fittings, thus requiring a user prepared SOP which ensures that adequate safety precautions are taken. Basically, the preparation process involves injecting a predetermined amount of neat liquid or gas into an empty high pressure cylinder of known volume, using gas flow into the cylinder to complete the transfer.

The cylinder is then pressurized to a given value (500-1000 psi). The final cylinder pressure must be determined using a high precision gauge after the cylinder has thermally equilibrated for a 1-2 hour period after filling.

13.1.2 The concentration of components in the cylinder standard should be determined by comparison with National Bureau of Standards reference standards (e.g. SRM 1805-benzene in nitrogen) when available.

13.1.3 The theoretical concentration (at 25°C and 760 mm pressure) for preparation of cylinder standards can be calculated using the following equation:

$$C_T = \frac{V_I \times d}{V_c} \times \frac{14.7}{P_c + 14.7} \times 24.4 \times 1000$$

where C_T is the component concentration, in ng/mL at 25°C and 760 mm Hg pressure.

V_I is the volume of neat liquid component injected, in µL.

V_c is the internal volume of the cylinder, in L.

d is the density of the neat liquid component, in g/mL.

P_c is the final pressure of the cylinder standards, in pounds per square inch gauge (psig).

13.2 Preparation of Spiked Traps by Vapor Phase Injection

This process involves preparation of a dilution flask or compressed gas cylinder containing the desired concentrations of the compound(s) of interest and injecting the desired volume of vapor into a flowing gas stream which is directed onto a clean CMS cartridge. The procedure is described in detail in another method within the Compendium (6) and will not be repeated here.

13.3 Preparation of Spiked Traps Using Permeation or Diffusion Tubes

13.3.1 A flowing stream of inert gas containing known amounts of each compound of interest is generated according to ASTM Method D3609 (4). Note that a method of accurately maintaining temperature within ± 0.1°C is required and the system generally must be equilibrated for at least 48 hours before use.

13.3.2 An accurately known volume of the standard gas stream (usually 0.1-1 liter) is drawn through a clean CMS cartridge using the sampling system described in Section 10.2.1, or a similar system. However, if mass flow controllers are employed, they must be calibrated for the carrier gas used in Section 13.3.1 (usually nitrogen). Use of air as the carrier gas for permeation systems is not recommended, unless the compounds of interest are known to be highly stable in air.

13.3.3 The spiked traps are then stored or immediately analyzed as in Sections 11.3.6 and 11.3.7.

14. Performance Criteria and Quality Assurance

This section summarizes the quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory. In many cases the specific QA procedures have been described within the appropriate section describing the particular activity (e.g. parallel sampling).

14.1 Standard Operating Procedures (SOPs)

14.1.1 Each user should generate SOPs describing the following activities as accomplished in their laboratory: 1) assembly, calibration and operation of the sampling system, 2) preparation, handling and storage of CMS cartridges, 3) assembly and operation of GC/MS system including the thermal desorption apparatus and data system, and 4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by the laboratory personnel conducting the work.

14.2 CMS Cartridge Preparation

14.2.1 Each batch of CMS cartridges, prepared as described in Section 9, should be checked for contamination by analyzing one cartridge, immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one-fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria, the entire lot should be rejected.

14.3 Sample Collection

14.3.1 During each sampling event at least one clean cartridge will accompany the samples to the field and back to the laboratory, having been placed in the sampler but without sampling air, to serve as a field blank. The average amount of material found on the field blank cartridges may be subtracted from the amount found on the actual samples. However, if the blank level is greater than

25% of the sample amount, data for that component must be identified as suspect.

14.3.2 During each sampling event at least one set of parallel samples (two or more samples collected simultaneously) should be collected, preferably at different flow rates as described in Section 10.1.4. If agreement between parallel samples is not generally within ±25% the user should collect parallel samples on a much more frequent basis (perhaps for all sampling points). If a trend of lower apparent concentrations with increasing flow rate is observed for a set of parallel samples one should consider using a reduced sampling rate and longer sampling interval, if possible. If this practice does not improve the reproducibility further evaluation of the method performance for the compound of interest might be required.

14.3.3 Backup cartridges (two cartridges in series) should be collected with each sampling event. Backup cartridges should contain less than 10% of the amount of components of interest found in the front cartridges, or be equivalent to the blank cartridge level, whichever is greater.

14.4 GC/MS Analysis

14.4.1 Performance criteria for MS tuning and mass standardization have been discussed in Section 11.2 and Table 2. Additional criteria can be used by the laboratory, if desired. The following sections provide performance guidance and suggested criteria for determining the acceptability of the GC/MS system.

14.4.2 Chromatographic efficiency should be evaluated daily by the injection of calibration standards. A reference compound(s) should be chosen from the calibration standard and plotted on an expanded time scale so that its width at 10% of the peak height can be calculated, as shown in Figure 6. The width of the peak at 10% height should not exceed 10 seconds. More stringent criteria may be required for certain applications. The asymmetry factor (see Figure 6) should be between 0.8 and 2.0. The user should also evaluate chromatographic performance for any polar or reactive compounds of interest, using the process described above. If peaks are observed that exceed the peak width or asymmetry factor criteria above, one should inspect the entire system to determine if unswept zones or cold spots are present in any of the fittings or tubing and/or if replacement of the GC column is required. Some laboratories may chose to evaluate column performance separately by direct injection of a test mixture onto the GC column. Suitable schemes for column evaluation have been reported in the literature (7).

14.4.3 The detection limit for each component is calculated from the data obtained for calibration standards. The detection limit is defined as

$$DL = A + 3.3S$$

where

DL is the calculated detection limit in nanograms injected.

A is the intercept calculated in Section 12.1.3.

S is the standard deviation of replicate determinations of the lowest level standard (at least three such determinations are required). The lowest

level standard should yield a signal to noise ratio (from the total ion current response) of approximately 5.

14.4.4 The relative standard deviation for replicate analyses of cartridges spiked at approximately 10 times the detection limit should be 20% or less. Day to day relative standard deviation for replicate cartridges should be 25% or less.

14.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present is ambient air (e.g. perfluorotoluene). Spiking is readily accomplished using the procedure outlined in Section 13.2, using a compressed gas standard. The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than $\pm$ 2 standard deviations from the mean (calculated excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

14.5 Method Precision and Recovery

14.5.1 Recovery and precision data for selected volatile organic compounds are presented in Table 1. These data were obtained using ambient air, spiked with known amounts of the compounds in a dynamic mixing system (2).

14.5.2 The data in Table 1 indicate that in general recoveries better than 75% and precision (relative standard deviations) of 15-20% can be obtained. However, selected compounds (e.g. carbon tetrachloride and

benzene) will have poorer precision and/or recovery. The user must check recovery and precision for any compounds for which quantitative data are needed.

References

1. Kebbekus, B. B. and J. W. Bozzelli. Collection and Analysis of Selected Volatile Organic Compounds in Ambient Air. Proceedings of Air Pollution Control Association, Paper No. 82-65.2, Air Pollution Control Association, Pittsburgh, Pennsylvania, 1982.

2. Riggin R. M. and L. E. Slivon. Determination of Volatile Organic Compounds in Ambient Air Using Carbon Molecular Sieve Adsorbants, Special Report on Contract 68-02-3745 (WA-7), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, September, 1983.

3. Riggin, R. M., "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air", EPA-600/4-83-027, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1983.

4. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis: Occupational Health and Safety", American Society for Testing and Materials, 1983.

5. Walling, J. F., Berkley, R. E., Swanson, D. H., and Toth, F. J. "Sampling Air for Gaseous Organic Chemical-Applications to Tenax", EPA-600/7-54-82-059, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1982.

6. This Methods Compendium - Tenax Method (TO 1).

7. Grob, K., Jr., Grob, G., and Grob, K., "Comprehensive Standardized Quality Test for Glass Capillary Columns", J. Chromatog., 156 1-20, 1978.

TABLE 1. VOLATILE ORGANIC COMPOUNDS FOR WHICH THE CMS ADSORPTION METHOD HAS BEEN EVALUATED

Compound	Retention Time, Minutes[a]	Characteristic Mass Fragment Used For Quantification	Method Performance Data[b]		
			Concentration, ng/L	Percent Recovery	Standard Deviation
Vinyl Chloride	6.3	62	17	74	19
Acrylonitrile	10.8	53	20	85	18
Vinylidene Chloride	10.9	96	36	94	19
Methylene Chloride	11.3	84	28	93	16
Allyl Chloride	11.4	76	32	72	19
Chloroform	13.8	83	89	91	12
1,2-Dichloroethane	14.5	62	37	85	11
1,1,1-Trichloroethane	14.7	97	100	75	9.1
Benzene	15.4	78	15	140	37
Carbon Tetrachloride	15.5	117	86	55	2.9
Toluene	18.0	91	4.1	98	5.4

a) GC conditions as follows:

Column - Hewlett Packard, crosslinked methyl silicone, 0.32 mm ID x 50 mm long, thick film, fused silica.

Temperature Program - 70°C for 2 minutes then increased at 8°C/minute to 120°C.

b) From Reference 2. For spiked ambient air.

TABLE 2. SUGGESTED PERFORMANCE CRITERIA FOR RELATIVE ION ABUNDANCES FROM FC-43 MASS CALIBRATION

M/E	% Relative Abundance
51	1.8 ± 0.5
69	100
100	12.0 ± 1.5
119	12.0 ± 1.5
131	35.0 ± 3.5
169	3.0 ± 0.4
219	24.0 ± 2.5
264	3.7 ± 0.4
314	0.25 ± 0.1

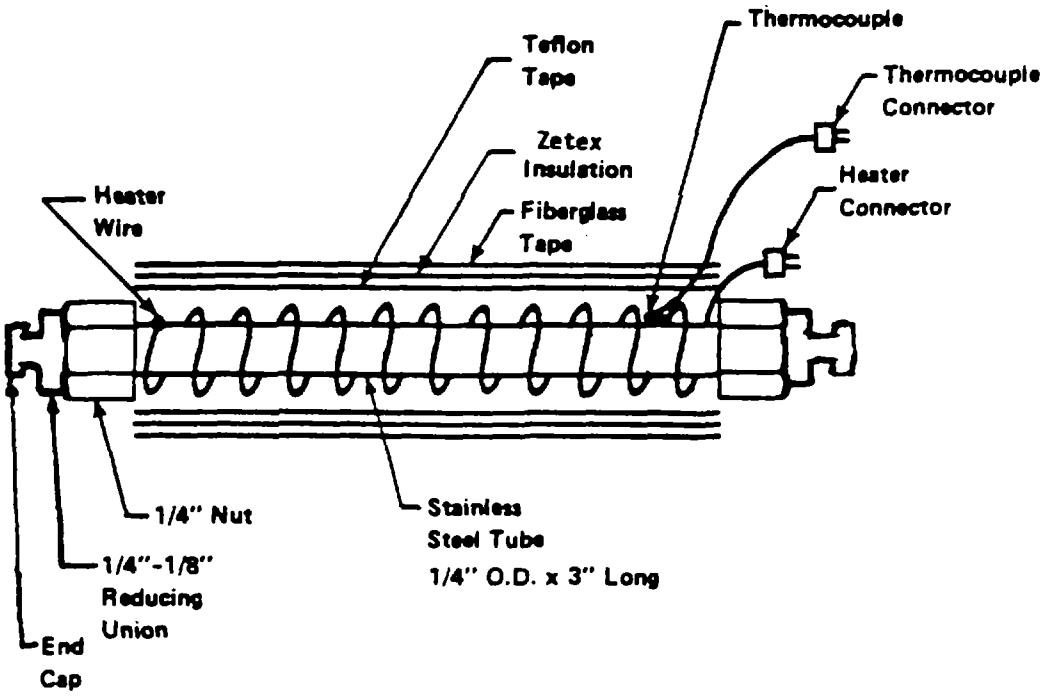
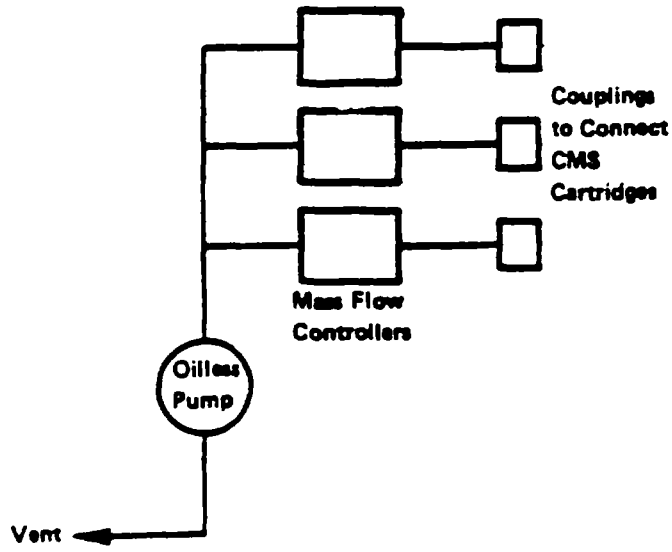
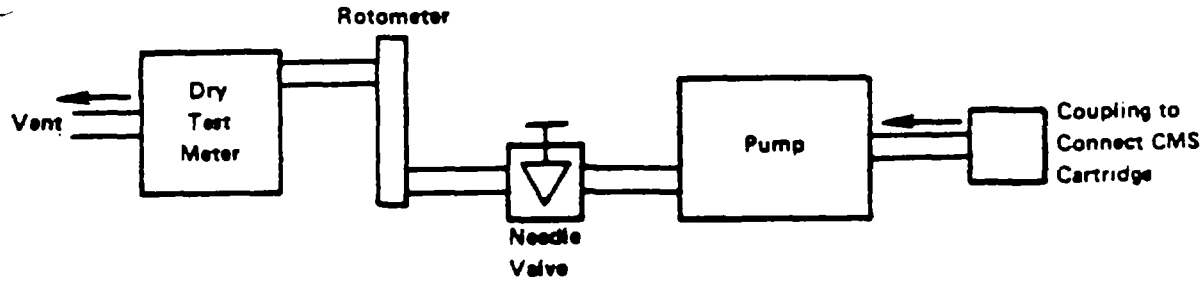


FIGURE 1. DIAGRAM SHOWING CARBON MOLECULAR SIEVE TRAP (CMS) CONSTRUCTION

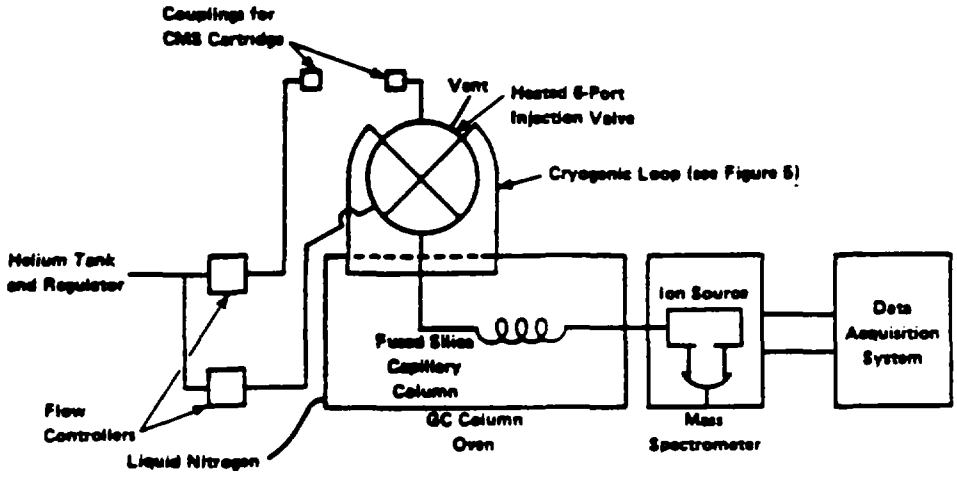


(a) Mass Flow Control

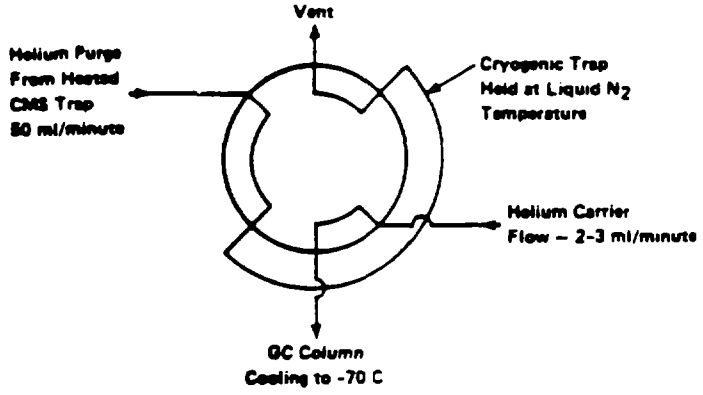


(b) Needle Valve Control

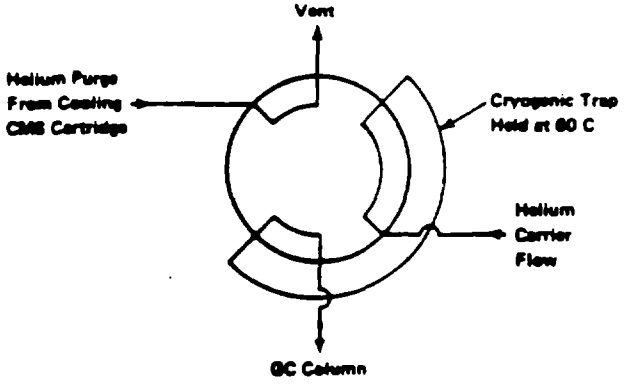
FIGURE 2. TYPICAL SAMPLING SYSTEM CONFIGURATIONS



(a) Overall System



(b) Valve – Load Mode



(a) Valve – Inject Mode

FIGURE 3. GC/MS ANALYSIS SYSTEM FOR CMS CARTRIDGES

SAMPLING DATA SHEET
(One Sample Per Data Sheet)

PROJECT:_____ DATE(S) SAMPLED:_____

SITE:_____ TIME PERIOD SAMPLED:_____

LOCATION:_____ OPERATOR:_____

INSTRUMENT MODEL NO:_____ CALIBRATED BY:_____

PUMP SERIAL NO:_____

SAMPLING DATA

Sample Number:_____

Start Time: _____ Stop Time: _____

Time	Dry Gas Meter Reading	Rotameter Reading	Flow Rate,*Q ml/Min	Ambient Temperature °C	Barometric Pressure, mmHg	Relative Humidity, %	Comments
2.							
3.							
4.							
n.							

Total Volume Data**

V_m = (Final - Initial) Dry Gas Meter Reading, or = _____ Liters

$$\frac{Q_1 + Q_2 + Q_3 \ldots Q_N}{N} \times \frac{1}{1000 \times (\text{Sampling Time in Minutes})} = \text{_____ Liters}$$

* Flowrate from rotameter or soap bubble calibrator (specify which).
** Use data from dry gas meter if available.

FIGURE 4. EXAMPLE SAMPLING DATA SHEET

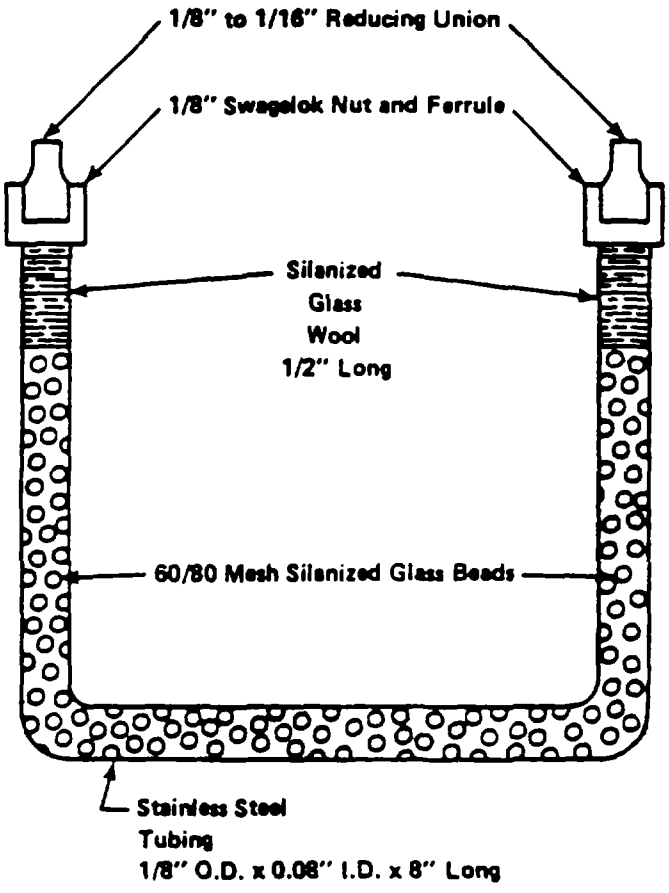
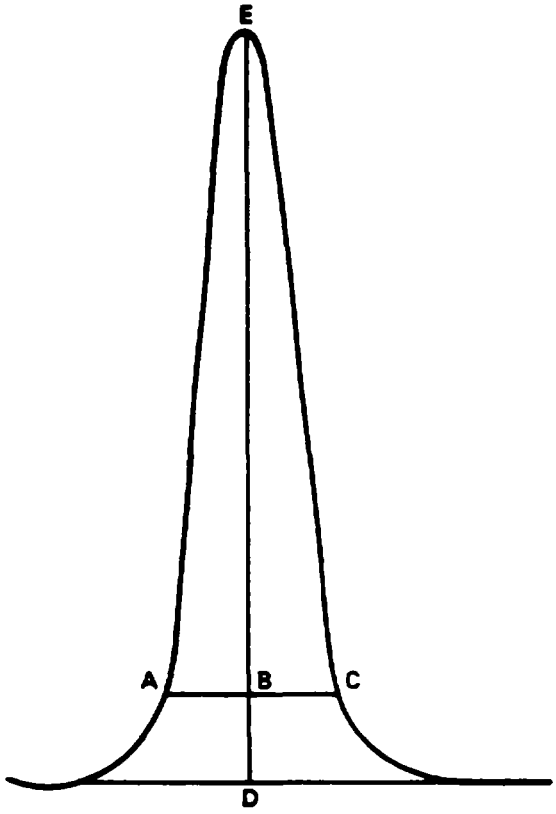


FIGURE 5. CRYOGENIC TRAP DESIGN



$$\text{Asymmetry Factor} = \frac{BC}{AB}$$

Example Calculation:

Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Asymmetry Factor = $\frac{12}{11}$ = 1.1

FIGURE 6. PEAK ASYMMETRY CALCULATION

METHOD FOR THE DETERMINATION OF ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS IN AMBIENT AIR

1. Scope

1.1 This document describes a method for determination of a variety of organochlorine pesticides and polychlorinated biphenyls (PCBs) in ambient air. Generally, detection limits of >1 ng/m^3 are achievable using a 24-hour sampling period.

1.2 Specific compounds for which the method has been employed are listed in Table 1. Several references are available which provide further details on the development and application of the method. The sample cleanup and analysis methods are identical to those described in U. S. EPA Method 608. That method is included as Appendix A of this methods compendium.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definition of Terms Related to Atmospheric Sampling and Analysis (7).

2.2 Other Documents

Ambient Air Studies (1-3)

U. S. EPA Technical Assistance Document (4).

U. S. EPA Method 608 (5). See Appendix A of methods compendium.

3. Summary of Method

3.1 A modified high volume sampler consisting of a glass fiber filter with a polyurethane foam (PUF) backup absorbent cartridge is used to sample ambient air at a rate of ~200-280 L/minute.

3.2 The filter and PUF cartridge are placed in clean, sealed containers and returned to the laboratory for analysis. The PCBs and pesticides are recovered by Soxhlet extraction with 5% ether in hexane.

3.3 The extracts are reduced in volume using Kuderna-Danish (K-D) concentration techniques and subjected to column chromatographic cleanup.

3.4 The extracts are analyzed for pesticides and PCBs using gas chromatography with electron capture detection (GC-ECD), as described in U. S. EPA Method 608 (5).

4. Significance

4.1 Pesticides, particularly organochlorine pesticides, are widely used in both rural and urban areas for a variety of applications. PCBs are less widely used, due to extensive restrictions placed on their manufacture. However, human exposure to PCBs continues to be a problem because of their presence in various electrical products.

4.2 Many pesticides and PCBs exhibit bioaccumulative, chronic health effects and hence monitoring ambient air for such compounds is of great importance.

4.3 The relatively low levels of such compounds in the environment requires the use of high volume sampling techniques to acquire sufficient sample for analysis. However, the volatility of these compounds prevents efficient collection on filter media. Consequently, this method utilizes both a filter and a PUF backup cartridge which provides for efficient collection of most organochlorine pesticides, PCBs, and many other organics within the same volatility range.

5. Definitions

Definitions used in this document and any user-prepared SOPs should be consistent with ASTM D1356 (7). All abbreviations

and symbols are defined within this document at the point of use.

6. Interferences

6.1 The use of column chromatographic cleanup and selective GC detection (GC-ECD) minimizes the risk of interference from extraneous organic compounds. However, the fact that PCBs as well as certain organochlorine pesticides (e.g. toxaphene and chlordane) are complex mixtures of individual compounds can cause difficulty in accurately quantifying a particular formulation in a multiple component mixture.

6.2 Contamination of glassware and sampling apparatus with traces of pesticides or PCBs can be a major source of error in the method, particularly when sampling near high level sources (e.g. dumpsites, waste processing plants, etc.) careful attention to cleaning and handling procedures is required in all steps of the sampling and analysis to minimize this source of error.

7. Apparatus

7.1 Hi-Vol Sampler with PUF cartridge - available from General Metal Works (Model PS-1). See Figure 1.

7.2 Sampling Head to contain glass cartridge with PUF plug - available from General Metal Works. See Figure 2.

7.3 Calibration orifice - available from General Metal Works.

7.4 Manometer - to use with calibration orifice.

7.5 Soxhlet extraction system - including Soxhlet extractors (500 and 250 mL), heating mantels, variable voltage transformers, and cooling water source - for extraction of PUF cartridges before and after sampling. Also for extraction of filter samples.

7.6 Vacuum oven connected to water aspirator - for drying extracted PUF cartridges.

7.7 Gas chromatograph with electron capture detector - (consult U. S. EPA Method 608 for specifications).

7.8 Forceps - to handle quartz fiber filter samples.

7.9 Die - to cut PUF plugs.

7.10 Various items for extract preparation, cleanup, and analysis - consult U. S. EPA Method 608 for detailed listing.

7.11 Chromatography column - 2 mm I.D. x 15 cm long - for alumina cleanup.

8. Reagent and Materials

8.1 Polyurethane foam - 3 inch thick sheet stock, polyether type used in furniture upholstering. Density 0.022 g/cm^3.

8.2 Polyester gloves - for handling PUF cartridges and filters

8.3 Filters, quartz fiber - Pallflex 2500 QAST , or equivalent.

8.4 Wool felt filter - 4.9 mg/cm^2 and 0.6 mm thick. To fit sample head for collection efficiency studies. Pre-extracted with 5% diethyl ether in hexane.

8.5 Hexane - Pesticide or distilled in glass grade.

8.6 Diethyl ether - preserved with 2% ethanol - distilled in glass grade, or equivalent.

8.7 Acetone - Pesticide or distilled in glass grade.

8.8 Glass container for PUF cartridges.

8.9 Glass petri dish - for shipment of filters to and from the laboratory.

8.10 Ice chest - to store samples at ∿0°C after collection.

8.11 Various materials needed for extract preparation, cleanup, and analysis - consult U. S. EPA Method 608 for details (Appendix A of this compendium).

8.12 Alumina - activity grade IV. 100/200 mesh

9. Assembly and Calibration of Sampling Apparatus

9.1 Description of Sampling Apparatus

9.1.1 The entire sampling system is diagrammed in Figure 1. This sampler was developed by Syracuse University

Research Corporation (SURC) under a U. S. EPA contract (6) and further modified by Southwest Research Institute and the U. S. EPA. A unit specifically designed for this method is now commercially available (Model PS-1 - General Metal Works, Inc., Village of Cleves, Ohio). The method writeup assumes the use of the commercial device, although the earlier modified device is also considered acceptable.

9.1.2 The sampling module (Figure 2) consists of a glass sampling cartridge and an air-tight metal cartridge holder. The PUF plug is retained in the glass sampling cartridge.

9.2 Calibration of Sampling System

9.2.1 The airflow through the sampling system is monitored by a venturi/Manehelic assembly, as shown in Figure 1. A multipoint calibration of the venturi/magnehelic assembly must be conducted every six months using an audit calibration orifice, as described in the U. S. EPA High Volume Sampling Method (8). A single point calibration must be performed before and after each sample collection, using the procedure described below.

9.2.2 Prior to calibration a "dummy" PUF cartridge and filter are placed in the sampling head and the sampling motor is activated. The flow control valve is fully opened and the voltage variator is adjusted so that a sample flow rate corresponding to ~110% of the desired flow rate is indicated on the magnehelic (based on the previously obtained multipoint calibration curve). The motor is allowed to warmup for ~10 minutes and then the flow control valve is adjusted to achieve the desired flow rate. The ambient temperature and barometric pressure should

be recorded on an appropriate data sheet (e.g. Figure 3).

9.2.3 The calibration orifice is then placed on the sampling head and a manometer is attached to the tap on the calibration orifice. The sampler is momentarily turned off to set the zero level of the manometer. The sampler is then switched on and the manometer reading is recorded, once a stable reading is achieved. The sampler is then shut off.

9.2.4 The calibration curve for the orifice is used to calculate sample flow from the data obtained in 9.2.3, and the calibration curve for the venturi/ magnehelic assembly is used to calculate sample flow from the data obtained in 9.2.2. The calibration data should be recorded on an appropriate data sheet (e.g. Figure 3). If the two values do not agree within 10% the sampler should be inspected for damage, flow blockage, etc. If no obvious problems are found the sampler should be recalibrated (multipoint) according to the U. S. EPA High Volume Sampling procedure (8).

9.2.5 A multipoint calibration of the calibration orifice, against a primary standard, should be obtained annually.

10. Preparation of Sampling (PUF) Cartridges

10.1 The PUF adsorbent is a polyether-type polyurethane foam (density No. 3014 or 0.0225 g/cm^3). This type of foam is used for furniture upholstery. It is white and yellows on exposure to light.

10.2 The PUF inserts are 6.0 cm diameter cylindrical plugs cut from 3 inch sheet stock and should fit with slight compression in the glass cartridge, supported by the wire

screen. See Figure 2. During cutting the die is rotated at high speed (e.g. in a drill press) and continuously lubricated with water.

10.3 For initial cleanup the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14-24 hours at approximately 4 cycles per hour. When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.

10.4 The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

10.5 The PUF is placed into the glass sampling cartridge using polyester gloves. The module is wrapped with hexane rinsed aluminum foil, placed in a labeled container and tightly sealed.

10.6 Other adsorbents may be suitable for this method as indicated in the various references (1-3). If such materials are employed the user must define appropriate preparation procedures based on the information contained in these references.

10.7 At least one assembled cartridge from each batch must be analyzed, as a laboratory blank, using the procedures described in Section 12, before the batch is considered acceptable for field use. A blank level of <10 ng/plug for single compounds is considered to be acceptable. For multiple component mixtures (e.g. Arochlors) the blank level should be <100 ng/plug.

11. Sampling

11.1 After the sampling system has been assembled and calibrated as described in Section 9 it can be used to collect air samples as described below.

11.2 The samples should be located in an unobstructed area, at least two meters from any obstacle to air flow. The exhaust hose should be stretched out in the downwind

direction to prevent recycling of air.

11.3 A clean sampling cartridge and quartz fiber filter are removed from sealed transport containers and placed in the sampling head using forceps and gloved hands. The head is tightly sealed into the sampling system. The aluminum foil wrapping is placed back in the sealed container for later use.

11.4 The zero reading of the Magnehelic is checked. Ambient temperature, barometric pressure, elapsed time meter setting, sampler serial number, filter number and PUF cartridge number are recorded. A suitable data sheet is shown in Figure 4.

11.5 The voltage variator and flow control valve are placed at the settings used in 9.2.3 and the power switch is turned on. The elapsed time meter is activated and the start time recorded. The flow (Magnehelic setting) is adjusted, if necessary using the flow control valve.

11.6 The Magnehelic reading is recorded every six hours during the sampling period. The calibration curve (Section 9.2.7) is used to calculate the flow rate. Ambient temperature and barometric pressure are recorded at the beginning and end of the sampling period.

11.7 At the end of the desired sampling period the power is turned off and the filter and PUF cartridges are wrapped with the original aluminum foil and placed in sealed, labeled containers for transport back to the laboratory.

11.8 The Magnehelic calibration is checked using the calibration orifice as described in Section 9.2.4. If the calibration deviates by more than 10% from the initial reading the flow data for that sample must be marked as suspect and the sampler should be inspected and/or removed from service.

11.9 At least one field blank will be returned to the laboratory with each group of samples. A field blank is treated exactly as a sample except that no air is drawn through the cartridge.

11.10 Samples are stored at ∿20°C in an ice chest until receipt at the analytical laboratory, at which time they are stored refrigerated at 4°C.

12. Sample Preparation and Analysis

12.1 Sample Preparation

12.1.1 All samples should be extracted within 1 week after collection.

12.1.2 PUF cartridges are removed from the sealed container using gloved hands, the aluminum foil wrapping is removed, and the cartridges are placed into a 500-mL Soxhlet extraction. The cartridges are extracted for 14-24 hours at ∿4 cycles/hour with 5% diethyl ether in hexane. Extracted cartridges can be dried and reused following the handling procedures in Section 10. The quartz filter can be placed in the extractor with the PUF cartridges. However, if separate analysis is desired then one can proceed with 12.1.3.

12.1.3 If separate analysis is desired, quartz filters are placed in a 250-mL Soxhlet extractor and extracted for 14-24 hours with 5% diethyl ether in hexane.

12.1.4 The extracts are concentrated to 10 mL final volume using 500-mL Kuderna-Danish concentrators as described in EPA Method 608 (5), using a hot water bath. The concentrated extracts are stored refrigerated in sealed 4-dram vials having teflon-lined screw-caps until analyzed or subjected to cleanup.

12.2 Sample Cleanup

12.2.1 If only organochlorine pesticides and PCBs are sought, an alumina cleanup procedure reported in the literature is appropriate (1). Prior to cleanup the sample

extract is carefully reduced to 1 mL using a gentle steam of clean nitrogen.

12.2.2 A glass chromatographic column (2 mm ID x 15 cm long) is packed with alumina, activity grade IV and rinsed with ~20 mL of n-hexane. The concentrated sample extract (from 12.2.1) is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as described in 12.3.

12.2.3 If other pesticides are sought, alternate cleanup procedures (e.g. Florisil) may be required. Method 608 (5) identifies appropriate cleanup procedures.

12.3 Sample Analysis

12.3.1 Sample analysis is performed using GC/ECD as described in EPA Method 608 (5). The user must consult this method for detailed analytical procedures.

12.3.2 GC retention times and conditions are identified in Table 1 for the compounds of interest.

13. GC Calibration

Appropriate calibration procedures are identified in EPA Method 608 (5).

14. Calculations

14.1 The total sample volume (V_m) is calculated from the periodic flow readings (Magnehelic) taken in Section 11.6 using the following equation.

$$V_m = \frac{Q_1 + Q_2 \ldots Q_N}{N} X \frac{T}{1000}$$

where

V_m = Total sample volume (m^3).

Q_1, $Q_2 \ldots Q_N$ = Flow rates determined at the beginning, end, and intermediate points during sampling (L/minute).

N = Number of data points averaged.

T = Elapsed sampling time (minutes).

14.2 The volume of air sampled can be converted to standard conditions (760 mm Hg pressure and 25°C) using the following equation:

$$V_s = V_m \times \frac{P_A}{760} \times \frac{298}{273+t_A}$$

where

V_s = Total sample volume at 25°C and 760 mm Hg pressure (m^3)

V_m = Total sample flow under ambient conditions (m^3)

P_A = Ambient pressure (mm Hg)

t_A = Ambient temperature (°C)

14.3 The concentration of compound in the sample is calculated using the following equation:

$$C_A = \frac{A \times V_E}{V_i \times V_s}$$

where

C_A = Concentration of analyte in the sample, $\mu g/m^3$

A = Calculated amount of material injected onto the chromatograph based on calibration curve for injected standards (*nanograms*)

V_i = Volume of extract injected (µL).

V_E = Final volume of extract (mL).
V_s = Total volume of air samples corrected to standard conditions (m^3).

14. Performance Criteria and Quality Assurance

This section summarizes the quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory.

14.1 Standard Operating Procedures (SOPs)

14.1.1 Users should generate SOPs describing the following activities as accomplished in their laboratory: 1) assembly, calibration and operation of the sampling system, 2) preparation, purification, storage and handling of sampling cartridges, 3) assembly, calibration and operation of the GC/ECD system, and 4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

14.2 Process, Field, and Solvent Blanks

14.2.1 One PUF cartridge and filter from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

14.2.2 During each sampling episode at least one PUF cartridge and filter should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

14.2.3 During the analysis of each batch of samples at least one solvent process blank (all steps conducted but no PUF cartridge or filter included) should be

carried through the procedure and analyzed.

14.2.4 Blank levels should not exceed ~10 ng/sample for single components or ~100 ng/sample for multiple component mixtures (e.g. PCBs).

14.3 Collection Efficiency and Spike Recovery

14.3.1 Before using the method for sample analysis each laboratory must determine their collection efficiency for the components of interest.

14.3.2 The glass fiber filter in the sampler is replaced with a hexane-extracted wool felt filter (weight 14.9 mg/cm^2, 0.6 mm thick). The filter is spiked with microgram amounts of the compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate and filter is placed into the sampling system for immediate use.

14.3.3 The sampling system, including a clean PUF cartridge, is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

14.3.4 The filter and PUF cartridge are then removed and analyzed as described in Section 12.

14.3.5 A second sample, unspiked is collected over the same time period to account for any background levels of components in the ambient air matrix.

14.3.6 A third PUF cartridge is spiked with the same amounts of the compounds used in 14.3.2 and extracted to determine analytical recovery.

14.3.7 In general analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

14.3.8 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of ± 15% or less is considered acceptable performance.

14.3.9 Blind spiked samples should be included with sample sets periodically, as a check on analytical performance.

14.4 Method Precision and Accuracy

Typical method recovery data are shown in Table 1. Recoveries for the various chlorobiphenyls illustrate the fact that all components of an Arochlor mixture will not be retained to the same extent. Recoveries for tetrachlorobiphenyls and above are generally greater than 85% but di- and trichloro homologs may not be recovered quantitatively.

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6. Bjorkland, J., Compton, B., and Zweig, G., "Development of Methods for Collection and Analysis of Airborne Pesticides." Report for Contract No. CPA 70-15, National Air Pollution Control Association, Durham, NC, 1970..

7. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis", American Society for Testing and Materials, Philadelphia, PA, 1983.

8. Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method). Federal Register, Sept. 14, 1972 or 40CFR50 Appendix B.

TABLE 1. SELECTED COMPONENTS DETERMINED USING HI-VOL/PUF SAMPLING PROCEDURE

Compound	GC Retention Time, Minutes[a]	24-Hour Sampling Efficiency[b]	
		Air Concentration ng/m^3	% Recovery
Aldrin	2.4	0.3-3.0	28
4,4'-DDE	5.1	0.6-6.0	89
4,4'-DDT	9.4	1.8-18	83
Chlordane	(c)	15-150	73
Chlorobiphenyls			
4,4' Di-	--	2.0-20	62
2,4,5 Tri-	---	0.2-2.0	36
2,4',5 Tri-	--	0.2-2.0	86
2,2',5,5' Tetra-	--	0.2-2.0	94
2,2',4,5,5' Penta-	--	0.2-2.0	92
2,2',4,4',5,5' Hexa	--	0.2-2.0	86

(a) Data from U.S. EPA Method 608. Conditions are as follows:

Stationary Phase - 1.5% SP2250/1.95% SP-2401 on Supelcoport (100/120 mesh) packed in 1.8 mm long x 4 mm ID glass column.

Carrier - 5/95 methane/Argon at 60 mL/Minute

Column Temperature - 160°C except for PCBs which are determined at 200°C.

(b) From Reference 2.

(c) Multiple component formulation. See U.S. EPA Method 608.

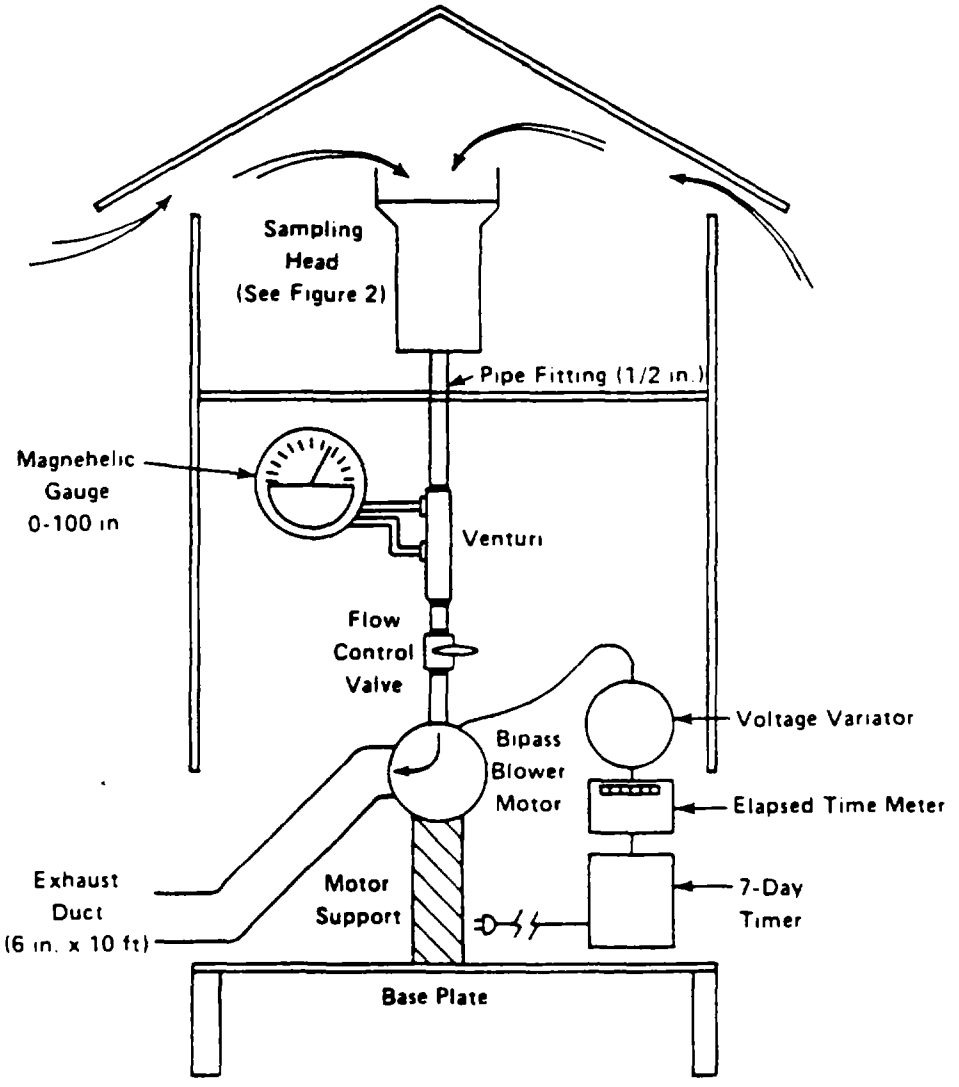

FIGURE 1. HIGH VOLUME AIR SAMPLER. AVAILABLE FROM GENERAL METAL WORKS (MODEL PS-1)

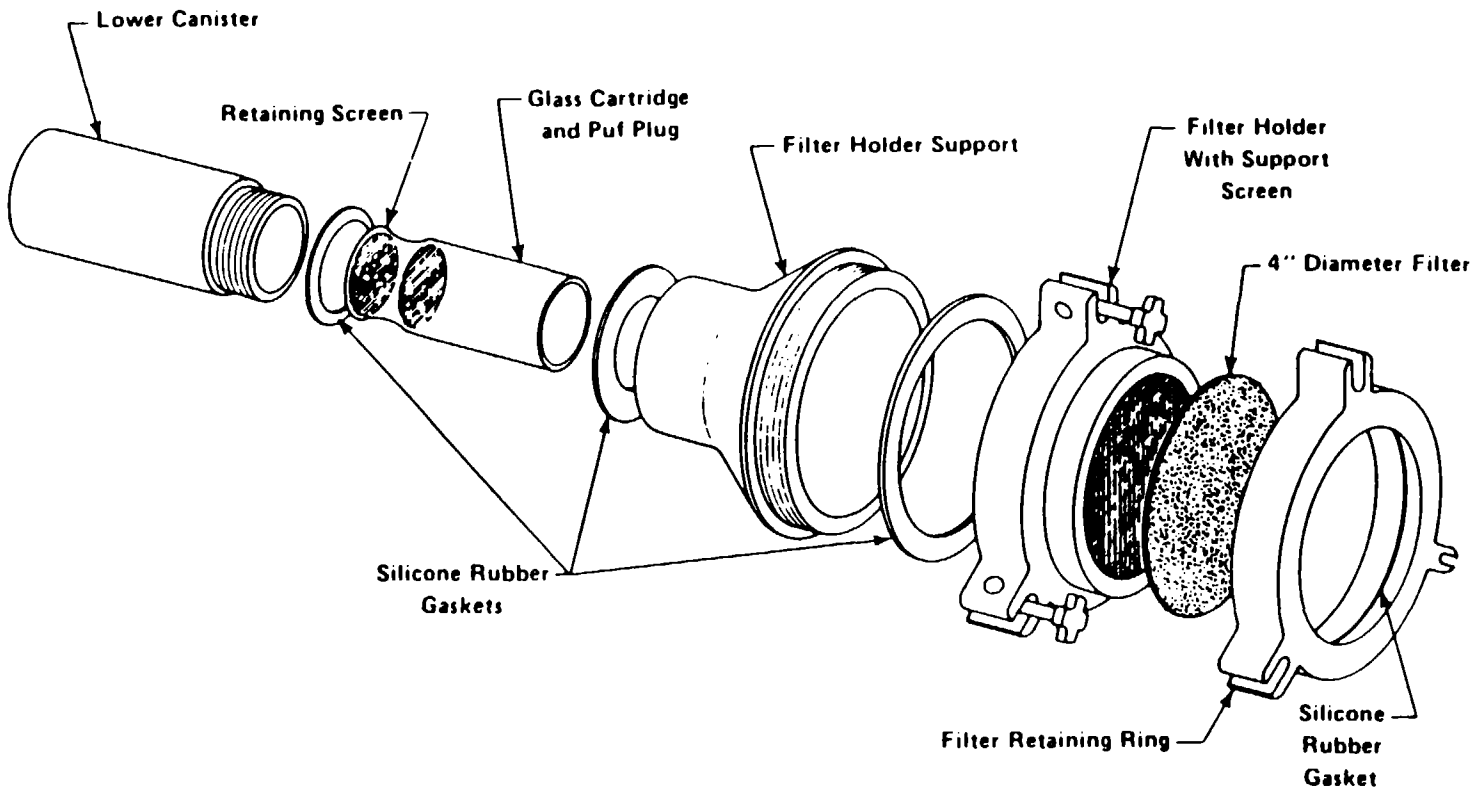

FIGURE 2. SAMPLING HEAD

Performed by_____ Calibration Orifice S/N_____ Ambient Temperature_____°C

Date/Time_____ Manometer S/N _____ Bar.Press. _____ mm Hg

Sampler S/N	Variac Setting V	Timer OK? Yes/No	Calibration Orifice Data		Sampler Venturi Data		% Difference Between Calibration and Sample Venturi Flow Rates	Comments
			Manometer, in. H_2O	Flow Rate, scm /min(a)	Magnehelic, in. H_2O	Flow Rate scm/min (b)		

(a) From Calibration Tables for Calibration Orifice or Venturi Tube

(b) From Calibration Tables for Venturi Tube in each Hi-Vol unit. Date check by_____ Date_____

FIGURE 3. TYPICAL CALIBRATION SHEET FOR HIGH VOLUME SAMPLER

Site ______________________ Date ______________________ Performed by ______________________

Sampler S/N	Sampling Location I.D.	New Filter (√)	PUF Cart No.	Variac Setting	Clock Time			Sampler Timer			Venturi Readings Time/Magnehelic in H_2O				Ambient Temperature °C		Barometric Pressure mm Hg		Comments
					Start hr CDT	Stop hr CDT	Min Elapsed	Start min	Stop min	Min Elapsed	1	2	3	4	Start	Stop	Start	Stop	

(a) Record any evidence of tampering with sampler and/or abnormalities in sampler operation, PUF cartridge condition or handling, etc.

Data Checked By ______________________ Date ______________

FIGURE 4. TYPICAL SAMPLING DATA FORM FOR HIGH VOLUME PESTICIDE/PCB SAMPLER

11.0 REFERENCE METHOD*

APPENDIX B—REFERENCE METHOD FOR THE DETERMINATION OF SUSPENDED PARTICULATES IN THE ATMOSPHERE (HIGH VOLUME METHOD)

1. *Principle and Applicability.*

1.1 Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 $m.^3$/min.: 40 to 60 $ft.^3$/min.) that allows suspended particles having diameters of less than 100 μm. (Stokes equivalent diameter) to pass to the filter surface. (*1*) Particles within the size range of 100 to 0.1μm. diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (μg./$m.^3$) is computed by measuring the mass of collected particulates and the volume of air sampled.

1.2 This method is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses.

2 *Range and Sensitivity.*

2.1 When the sampler is operated at an average flow rate of 1.70 $m.^3$/min. (60 $ft.^3$/min.) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 μg./$m.^3$. If particulate levels are unusually high, a satisfactory sample may be obtained in 6 to 8 hours or less. For determination of average concentrations of suspended particulates in ambient air, a standard sampling period of 24 hours is recommended.

2.2 Weights are determined to the nearest milligram, airflow rates are determined to the nearest 0.03 $m.^3$/min. (1.0 $ft.^3$/min.), times are determined to the nearest 2 minutes, and mass concentrations are reported to the nearest microgram per cubic meter.

3. *Interferences.*

3.1 Particulate matter that is oily, such as photochemical smog or wood smoke, may block the filter and cause a rapid drop in airflow at a nonuniform rate. Dense fog or high humidity can cause the filter to become too wet and severely reduce the airflow through the filter.

3.2 Glass-fiber filters are comparatively insensitive to changes in relative humidity, but collected particulates can be hygroscopic. (*2*)

4. *Precision, Accuracy, and Stability.*

4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst variation (repeatability of the method) is 3.0 percent. The corresponding value for multilaboratory variation (reproducibility of the method) is 3.7 percent. (*3*)

4.2 The accuracy with which the sampler measures the true average concentration depends upon the constancy of the airflow rate through the sampler. The airflow rate is affected by the concentration and the nature of the dust in the atmosphere. Under these conditions the error in the measured average concentration may be in excess of ±50 percent of the true average concentration, depending on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-hour sampling period. (*4*)

5. *Apparatus.*

5.1 *Sampling.*

5.1.1 *Sampler.* The sampler consists of three units: (1) the faceplate and gasket, (2) the filter adapter assembly, and (3) the motor unit. Figure B1 shows an exploded view of these parts, their relationship to each other, and how they are assembled. The sampler must be capable of passing environmental air through a 406.5 $cm.^2$ (63 $in.^2$) portion of a clean 20.3 by 25.4 cm. (8- by 10-in.) glass-fiber filter at a rate of at least 1.70 $m.^3$/min. (60 $ft.^3$/min.). The motor must be capable of continuous operation for 24-hour periods with input voltages ranging from 110 to 120 volts, 50–60 cycles alternating current and must have third-wire safety ground. The housing for the motor unit may be of any convenient construction so long as the unit remains airtight and leak-free. The life of the sampler motor can be extended by lowering the voltage by about 10 percent with a small "buck or boost" transformer between the sampler and power outlet.

5.1.2 *Sampler Shelter.* It is important that the sampler be properly installed in a suitable shelter. The shelter is subjected to extremes of temperature, humidity, and all types of air pollutants. For these reasons the materials of the shelter must be chosen carefully. Properly painted exterior plywood or heavy gauge aluminum serve well. The sampler must be mounted vertically in the shelter so that the glass-fiber filter is parallel with the ground. The shelter must be provided with a roof so that the filter is protected from precipitation and debris. The internal arrangement and configuration of a suitable shelter with a gable roof are shown in Figure B2. The clearance area between the main housing and the roof at its closest point should be 580.5±193.5 $cm.^2$ (90±30 $in.^2$). The main housing should be rectangular, with dimensions of about 29 by 36 cm. (11½ by 14 in.).

5.1.3 *Rotameter.* Marked in arbitrary units, frequently 0 to 70, and capable of being calibrated. Other devices of at least comparable accuracy may be used.

5.1.4 *Orifice Calibration Unit.* Consisting of a metal tube 7.6 cm. (3 in.) ID and 15.9 cm. (6¼ in.) long with a static pressure tap 5.1 cm. (2 in.) from one end. See Figure B3. The tube end nearest the pressure tap is flanged to about 10.8 cm. (4¼ in.) OD with a male thread of the same size as the inlet end of the high-volume air sampler. A single metal plate 9.2 cm. (3⅝ in.) in diameter and 0.24 cm. (3/32 in.) thick with a central orifice 2.9 cm. (1⅛ in.) in diameter is held in place at the air inlet end with a female threaded ring. The other end of the tube is flanged to

*Reproduced from Code of Federal Regulation 40, Part 50.11, Appendix B, July 1, 1975, Pages 12-16.

hold a loose female threaded coupling, which screws onto the inlet of the sampler. An 18-hole metal plate, an integral part of the unit, is positioned between the orifice and sampler to simulate the resistance of a clean glass-fiber filter. An orifice calibration unit is shown in Figure B3.

5.1.5 *Differential Manometer.* Capable of measuring to at least 40 cm. (16 in.) of water.

5.1.6 *Positive Displacement Meter.* Calibrated in cubic meters or cubic feet, to be used as a primary standard.

5.1.7 *Barometer.* Capable of measuring atmospheric pressure to the nearest mm.

5.2 *Analysis.*

5.2.1 *Filter Conditioning Environment.* Balance room or desiccator maintained at 15° to 35°C. and less than 50 percent relative humidity.

5.2.2 *Analytical Balance.* Equipped with a weighing chamber designed to handle unfolded 20.3 by 25.4 cm. (8- by 10-in.) filters and having a sensitivity of 0.1 mg.

5.2.3 *Light Source.* Frequently a table of the type used to view X-ray films.

5.2.4 *Numbering Device.* Capable of printing identification numbers on the filters.

6. *Reagents.*

6.1 *Filter Media.* Glass-fiber filters having a collection efficiency of at least 99 percent for particles of 0.3 μm. diameter, as measured by the DOP test, are suitable for the quantitative measurement of concentrations of suspended particulates, (5) although some other medium, such as paper, may be desirable for some analyses. If a more detailed analysis is contemplated, care must be exercised to use filters that contain low background concentrations of the pollutant being investigated. Careful quality control is required to determine background values of these pollutants.

7. *Procedure.*

7.1 *Sampling.*

7.1.1 *Filter Preparation.* Expose each filter to the light source and inspect for pinholes, particles, or other imperfections. Filters with visible imperfections should not be used. A small brush is useful for removing particles. Equilibrate the filters in the filter conditioning environment for 24 hours. Weigh the filters to the nearest milligram; record tare weight and filter identification number. Do not bend or fold the filter before collection of the sample.

7.1.2 *Sample Collection.* Open the shelter, loosen the wing nuts, and remove the faceplate from the filter holder. Install a numbered, preweighed, glass-fiber filter in position (rough side up), replace the faceplate without disturbing the filter, and fasten securely. Undertightening will allow air leakage, overtightening will damage the sponge-rubber faceplate gasket. A very light application of talcum powder may be used on the sponge-rubber faceplate gasket to prevent the filter from sticking. During inclement weather the sampler may be removed to a protected area for filter change. Close the roof of the shelter, run the sampler for about 5 minutes, connect the rotameter to the nipple on the back of the sampler, and read the rotameter ball with rotameter in a vertical position. Estimate to the nearest whole number. If the ball is fluctuating rapidly, tip the rotameter and slowly straighten it until the ball gives a constant reading. Disconnect the rotameter from the nipple; record the initial rotameter reading and the starting time and date on the filter folder. (The rotameter should never be connected to the sampler except when the flow is being measured.) Sample for 24 hours from midnight to midnight and take a final rotameter reading. Record the final rotameter reading and ending time and date on the filter folder. Remove the faceplate as described above and carefully remove the filter from the holder, touching only the outer edges. Fold the filter lengthwise so that only surfaces with collected particulates are in contact, and place in a manila folder. Record on the folder the filter number, location, and any other factors, such as meteorological conditions or razing of nearby buildings, that might affect the results. If the sample is defective, void it at this time. In order to obtain a valid sample, the high-volume sampler must be operated with the same rotameter and tubing that were used during its calibration.

7.2 *Analysis.* Equilibrate the exposed filters for 24 hours in the filter conditioning environment, then reweigh. After they are weighed, the filters may be saved for detailed chemical analysis.

7.3 *Maintenance.*

7.3.1 *Sampler Motor.* Replace brushes before they are worn to the point where motor damage can occur.

7.3.2 *Faceplate Gasket.* Replace when the margins of samples are no longer sharp. The gasket may be sealed to the faceplate with rubber cement or double-sided adhesive tape.

7.3.3 *Rotameter.* Clean as required, using alcohol.

8. *Calibration.*

8.1 *Purpose.* Since only a small portion of the total air sampled passes through the rotameter during measurement, the rotameter must be calibrated against actual airflow with the orifice calibration unit. Before the orifice calibration unit can be used to calibrate the rotameter, the orifice calibration unit *itself must be calibrated against* the positive displacement primary standard.

8.1.1 *Orifice Calibration Unit.* Attach the orifice calibration unit to the intake end of the positive displacement primary standard and attach a high-volume motor blower unit to the exhaust end of the primary standard. Connect one end of a differential manometer to the differential pressure tap of the orifice calibration unit and leave the other end open to the atmosphere. Operate the high-volume motor blower unit so that a series of different, but constant, airflows (usually six) are obtained for definite time periods. Record the reading on the differential manometer at each airflow. The different constant airflows are obtained by placing a

series of loadplates, one at a time, between the calibration unit and the primary standard. Placing the orifice before the inlet reduces the pressure at the inlet of the primary standard below atmospheric; therefore, a correction must be made for the increase in volume caused by this decreased inlet pressure. Attach one end of a second differential manometer to an inlet pressure tap of the primary standard and leave the other open to the atmosphere. During each of the constant airflow measurements made above, measure the true inlet pressure of the primary standard with this second differential manometer. Measure atmospheric pressure and temperature. Correct the measured air volume to true air volume as directed in 9.1.1, then obtain true airflow rate, Q, as directed in 9.1.3. Plot the differential manometer readings of the orifice unit versus Q.

8.1.2 *High-Volume Sampler.* Assemble a high-volume sampler with a clean filter in place and run for at least 5 minutes. Attach a rotameter, read the ball, adjust so that the ball reads 65, and seal the adjusting mechanism so that it cannot be changed easily. Shut off motor, remove the filter, and attach the orifice calibration unit in its place. Operate the high-volume sampler at a series of different, but constant, airflows (usually six). Record the reading of the differential manometer on the orifice calibration unit, and record the readings of the rotameter at each flow. Measure atmospheric pressure and temperature. Convert the differential manometer reading to m.³/min., Q, then plot rotameter reading versus Q.

8.1.3 *Correction for Differences in Pressure or Temperature.* See Addendum B.

9. *Calculations.*

9.1 *Calibration of Orifice.*

9.1.1 *True Air Volume.* Calculate the air volume measured by the positive displacement primary standard.

$$V_a = \frac{(P_a - P_m)}{P_a}(V_M)$$

V_a = True air volume at atmospheric pressure, m.³
P_a = Barometric pressure, mm. Hg.
P_m = Pressure drop at inlet of primary standard, mm. Hg.
V_M = Volume measured by primary standard, m.³

9.1.2 *Conversion Factors.*
Inches Hg. × 25.4 = mm. Hg.
Inches water × 73.48 × 10^{-4} = inches Hg.
Cubic feet air × 0.0284 = cubic meters air.

9.1.3 *True Airflow Rate.*

$$Q = \frac{V_a}{T}$$

Q = Flow rate, m.³/min.
T = Time of flow, min.

9.2 *Sample Volume.*

9.2.1 *Volume Conversion.* Convert the initial and final rotameter readings to true airflow rate, Q, using calibration curve of 8.1.2.

9.2.2 *Calculate volume of air sampled*

$$* \quad V = \frac{Q_i Q_f}{2} \times T$$

V = Air volume sampled, m.³
Q_i = Initial airflow rate, m.³/min.
Q_f = Final airflow rate, m.³/min.
T = Sampling time, min.

9.3 *Calculate mass concentration of suspended particulates*

$$S.P. = \frac{(W_f - W_i) \times 10^6}{V}$$

S.P. = Mass concentration of suspended particulates, μg/m.³
W_i = Initial weight of filter, g.
W_f = Final weight of filter, g.
V = Air volume sampled, m.³
10^6 = Conversion of g. to μg.

10. *References.*

(1) Robson, C. D., and Foster, K. E., "Evaluation of Air Particulate Sampling Equipment", *Am. Ind. Hyg. Assoc. J. 24*, 404 (1962).

(2) Tierney, G. P., and Conner, W. D., "Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters", *Am. Ind. Hyg. Assoc. J. 28*, 363 (1967).

(3) Unpublished data based on a collaborative test involving 12 participants, conducted under the direction of the Methods Standardization Services Section of the National Air Pollution Control Administration, October, 1970.

(4) Harrison, W. K., Nader, J. S., and Fugman, P. S., "Constant Flow Regulators for High-Volume Air Sampler", *Am. Ind. Hyg. Assoc. J. 21*, 114–120 (1960).

(5) Pate, J. B., and Tabor, E. C., "Analytical Aspects of the Use of Glass-Fiber Filters for the Collection and Analysis of Atmospheric Particulate Matter", *Am. Ind. Hyg. Assoc. J. 23*, 144–150 (1962).

ADDENDA

A. *Alternative Equipment.*

A modification of the high-volume sampler incorporating a method for recording the actual airflow over the entire sampling period has been described, and is acceptable for measuring the concentration of suspended particulates (Henderson, J. S., Eighth Conference on Methods in Air Pollution and Industrial Hygiene Studies, 1967, Oakland,

*This equation should read $V = \frac{(Q_i + Q_f)}{2} \times T$

Calif.). This modification consists of an exhaust orifice meter assembly connected through a transducer to a system for continuously recording airflow on a circular chart. The volume of air sampled is calculated by the following equation:

$$V = Q \times T.$$

Q = Average sampling rate, m.³/min.
T = Sampling time, minutes.

The average sampling rate, Q, is determined from the recorder chart by estimation if the flow rate does not vary more than 0.11 m.³/min. (4 ft.³/min.) during the sampling period. If the flow rate does vary more than 0.11 m.³ (4 ft.³/min.) during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average.

B. *Pressure and Temperature Corrections.*

If the pressure or temperature during high-volume sampler calibration is substantially different from the pressure or temperature during orifice calibration, a correction of the flow rate, Q, may be required. If the pressures differ by no more than 15 percent and the temperatures differ by no more than 100 percent (°C), the error in the uncorrected flow rate will be no more than 15 percent. If necessary, obtain the corrected flow rate as directed below. This correction applies only to orifice meters having a constant orifice coefficient. The coefficient for the calibrating orifice described in 5.1.4 has been shown experimentally to be constant over the normal operating range of the high-volume sampler (0.6 to 2.2 m.³/min.; 20 to 78 ft.³/min.). Calculate corrected flow rate:

$$Q_2 = Q_1 \left[\frac{T_2 P_1}{T_1 P_2} \right]^{1/2}$$

Q_2 = Corrected flow rate, m.³/min.
Q_1 = Flow rate during high-volume sampler calibration (Section 8.1.2), m.³/min.
T_1 = Absolute temperature during orifice unit calibration (Section 8.1.1), °K or °R.
P_1 = Barometric pressure during orifice unit calibration (Section 8.1.1), mm. Hg.
T_2 = Absolute temperature during high-volume sampler calibration (Section 8.1.2), °K or °R.
P_2 = Barometric pressure during high-volume sampler calibration (Section 8.1.2), mm. Hg.

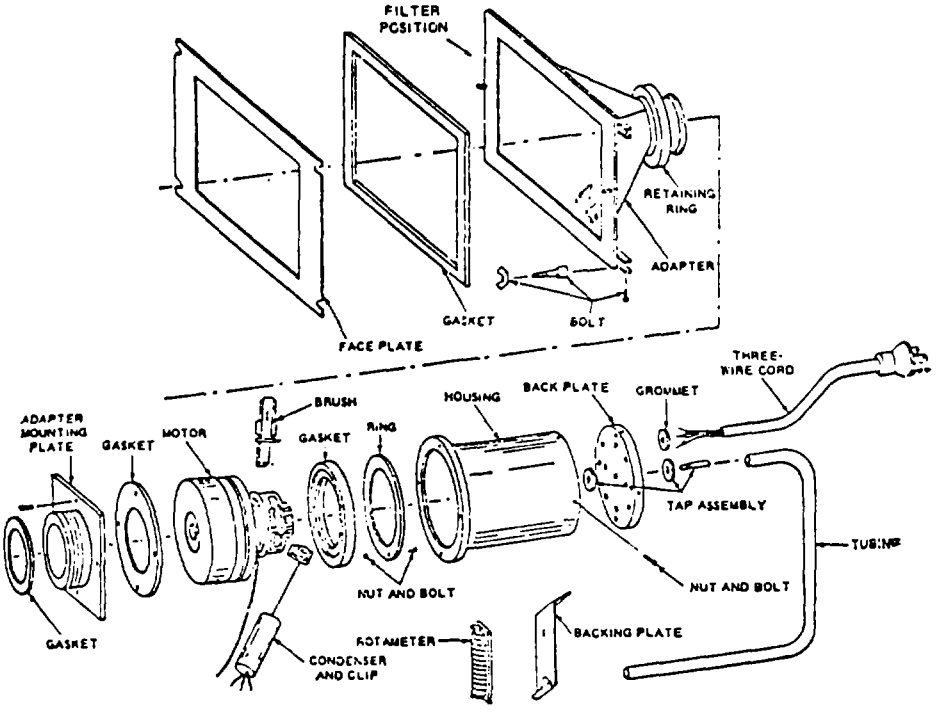

Figure B1. Exploded view of typical high-volume air sampler parts.

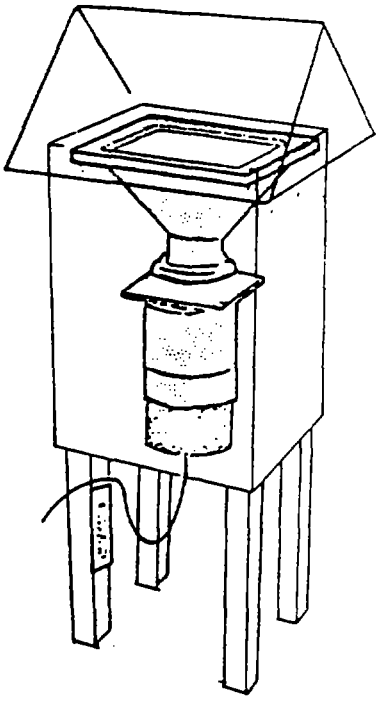

Figure B2. Assembled sampler and shelter.

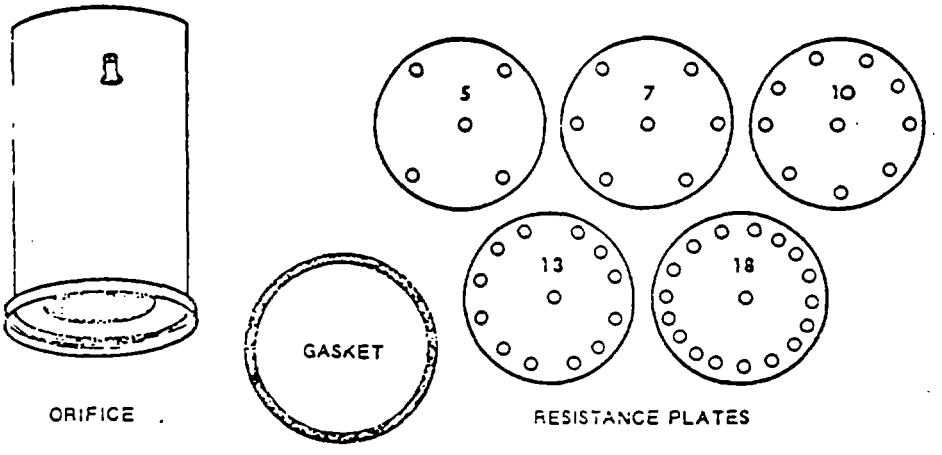

Figure B3. Orifice calibration unit.

Site Specific Sampling Plan Appendix B: Aquatic Biota Investigation

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APPENDIX B

Aquatic Biota Investigation

Fish samples will be collected using one or more of the following types of equipment: electroshocker, fyke net, seine net, and gill net. The specific sampling device will be determined in the field based upon the station location, accessibility, and station physical characteristics (stream width and water depth). The following describes the sample collection procedures for each piece of equipment.

Electroshocker

This method provides a fast and efficient means of collecting fish samples from water bodies with depths of less than four feet.

A Smith-Root Type VII backpack fish shocker and two fiberglass wrapped aluminum handle dip nets with 1/8 inch mesh netting will be the primary sampling devices used in the MFLBC. The accompanying technique is followed when using the electroshocker.

1. The backpack operator and the assistants will wear chest waders to protect against electric shock.

2. Sample collection will begin at the furthest downstream station and finish at the last upstream station.

3. Set the output switch of the backpack shocker to 200 volts and the pulse width and frequency switches to minimum. Turn the power switch on and

observe that the voltmeter indicates 12 volts or more.

4. Depress the anode push-button switch and observe the amount of amps generated. If the amps are below or above 0.5 amps then adjust the frequency and pulse width switches so that the ammeter indicates 0.5 amps.

5. Start electrofishing by slowly sweeping the probe back and forth while walking upstream.

6. As fish are stunned, net the representative upper and lower trophic level species and place then in a food grade 5 gallon stainless steel bucket.

7. Turn off the electroshocker after at least 5 fish of the same species totaling more than 150 grams have been collected from each trophic level at each station.

8. Fish will be sorted according to species, counted, weighed, measured and recorded immediately after sampling. One representative species from each trophic level will be selected for submission to the laboratory for the analyses identified in Table 3-3.

9. The selected lower trophic level fish species will be wrapped whole in aluminum foil, labeled and placed in zip-lock bags.

10. The selected fish species from the upper trophic level will be filleted in the field using a stainless steel knife in a stainless steel pan. Special care will be taken while filleting so that

muscle tissue will not come into contact with sediment or other sources of contamination. The fillets will then be washed, wrapped in aluminum foil, labeled and placed in zip-lock bags.

11. Decontaminate the stainless steel knife and pan after filleting each fish.

12. The fish will be placed in a cooler of dry ice and shipped frozen to the laboratory.

13. Complete chain of custody sheets for each sample.

Fyke Net or Hoop Net

This sampling device will be utilized to collect fish from Slanker Pond. The fyke net has a series of hoops that support mesh funnels so when fish enter the net they become trapped and cannot escape. The following technique is used to set a fyke net.

1. Depending on the depth of the water, the net will be set either by boat or by wading.

2. The net is set so that the mouth of the net is facing against the water current or against the direction of fish movement.

3. The seine net type wings that are attached to the opening hoop are stretched out and anchored to the bottom while the cod end (back end) is stretched and anchored downstream of the mouth opening.

4. The net will be baited at the cod end to facilitate fish collection.

5. The net will be placed at dusk and checked at dawn of the following day.

6. After completion of collection each day the net will either be retrieved or re-set at dusk and the fish will be processed in the same manner as previously discussed.

Seine Net

The seine net is a good sampling device for small fish when it is used as a haul seine or for any size fish when it is used to prevent fish from escaping the sampling station during electrofishing. The following technique is used when the net is used as a haul seine.

1. Attach two seven foot poles to the ends of the seine net.

2. Wade out from the shore with the net out of the water.

3. Place net in the water and stretch it out.

4. Walk swiftly toward shore keeping the bottom of the seine at the stream or lake bottom.

5. Walk the net onto shore and collect the fish according to the procedures outlined in the electroshocking section.

The following procedures are used when the seine net is used in conjunction with the electroshocker.

1. Position and set the seine net across the stream at the downstream portion of the station.

2. Place rocks on the bottom of the net so that fish cannot escape under the net.

3. Tie off the end poles so that the net will remain upright when unattended.

4. Begin shocking upstream of the net, moving slowly downstream toward the net.

5. Collect the representative fish as they are shocked and after they have been gathered into the net.

6. Samples are then processed in the same manner as stated in the previous sections.

Gill Net

Gill nets are designed to capture larger fish in ponds, lakes, reservoirs, or rivers where fish movement is expected. This technique may be used in Slanker Pond along with the Fyke net. The following technique is used to set the gill net.

1. Depending on the depth of the pond, the gill net will be set either by boat or by wading.

2. Stretch the gill net out as far across the pond as possible.

3. Anchor the ends with stakes or weights so that the net sits in the water perpendicular to the bottom.

4. The net will be placed at dusk and checked at dawn of the following day. After completion of collection each day, the net will either be retrieved or re-set at dusk.

5. Process the fish in the same manner as previously discussed.