

OVERSIZE PAGES

LOCATED AT

END OF

DOCUMENT

25-02 -02 -03 -02 -9408010001

Environmental Restoration Program

**PROPOSAL FOR ADDITIONAL WORK
SOIL GAS SURVEY AND GEOPHYSICAL
INVESTIGATIONS, MOUND PLANT
MAIN HILL AND SM/PP HILL AREAS**

**MOUND PLANT
MIAMISBURG, OHIO**

February 1992

FINAL

(Revision 2)

**Department of Energy
Albuquerque Field Office**

Environmental Restoration Program
Technical Support Office
Los Alamos National Laboratory



ENVIRONMENTAL RESTORATION PROGRAM

**PROPOSAL FOR ADDITIONAL WORK
SOIL GAS SURVEY AND GEOPHYSICAL INVESTIGATIONS
MOUND PLANT MAIN HILL AND SM/PP HILL AREAS**

**MOUND PLANT
MIAMISBURG, OHIO**

February 1992

**DEPARTMENT OF ENERGY
ALBUQUERQUE FIELD OFFICE**

**ENVIRONMENTAL RESTORATION PROGRAM
TECHNICAL SUPPORT OFFICE
LOS ALAMOS NATIONAL LABORATORY**

**FINAL
(REVISION 2)**

APPROVAL

This document was prepared under University of California, Los Alamos National Laboratory, Subcontract 9-XS8-0146R-1, by Roy F. Weston, Inc.

APPROVED--ROY F. WESTON, INC.

John B. Price (Date)
Installation Manager

Michael P. Mauzy, P.E. (Date)
Program Manager

APPROVED--LOS ALAMOS NATIONAL LABORATORY ER TSO

G. Thomas Farmer, Ph.D. (Date)
Installation Coordinator

Kenneth H. Rea, Ph.D. (Date)
Group Leader

CONTENTS

1. INTRODUCTION	1-1
2. BACKGROUND	2-1
2.1. MAIN HILL	2-1
2.2. SM/PP HILL	2-2
2.3. OLD FIRING RANGE DRUM SITE	2-2a
3. PROPOSED ADDITIONAL WORK	3-1
3.1. SOIL GAS SURVEY	3-1
3.2. GEOPHYSICAL SURVEYS	3-3
3.2.1. Geophysical Survey in Area J	3-3
3.2.2. Geophysical Survey for the Buried Boiler in Operable Unit 4	3-3a
4. REFERENCES	4-1

APPENDIX A - DESCRIPTION OF SOIL GAS SAMPLING AND ANALYSIS

LIST OF FIGURES

2.1. Old Firing Range showing drum removal activity	2-3
2.2. Old Firing Range drum removal	2-4
2.3. Old Firing Range showing drums in ponded water	2-5
2.4. Location map of Old Firing Range and Drum Storage Area, April 17, 1983	2-7
3.1. Proposed locations for soil gas samples.	3-2
3.2. Approximate location of Area J on the SM/PP Hill	3-4
3.3. Approximate location of the proposed geophysical survey area in Operable Unit 4.	3-6

ACRONYMS

DOE U.S. Department of Energy
EPA U.S. Environmental Protection Agency
FFA Federal Facility Agreement
RI/FS remedial investigation/feasibility study
VOCs volatile organic compounds

ACKNOWLEDGEMENTS

This report was prepared by Roy F. Weston, Inc., Albuquerque, New Mexico, under subcontract number 9-X58-0146R-1, to the University of California, Los Alamos National Laboratory. The manuscript was prepared by John Price, Bimal Mukhopadhyay, Bill Criswell, and Steve Gorin. The graphics and report were assembled, edited, and produced by WESTON's Albuquerque Office publications staff.

1. INTRODUCTION

Under Section XIII of the Federal Facility Agreement (FFA) between the U.S. Department of Energy (DOE) and the U.S. Environmental Protection Agency (EPA), Region V, for the DOE Mound Plant, "all additional work or modification to work determined to be necessary by U.S. DOE shall be subject to approval by U.S. EPA prior to initiating any work or modification of work." The DOE has determined the necessity for soil gas sampling and analysis and geophysical surveys, as described in the following sections. Therefore, this proposal for additional work has been prepared in order to comply with this guidance.

The additional work specified in this plan is mostly of a reconnaissance nature, i.e., it is a scoping task. The objective of the additional work is to provide sufficient data to enhance the planning of site characterization data. These data will be used during the preparation of work plans to identify areas needing further investigation.

2. BACKGROUND

The DOE is currently preparing reports specified by the FFA, including a background report (FFA Attachment I, Section 1.1) and remedial investigation/feasibility study (RI/FS) work plans for several operable units, including

- Operable Unit 2, Main Hill,
- Operable Unit 3, Miscellaneous Sites,
- Operable Unit 4, Miami-Erie Canal, and
- Operable Unit 5, Radioactively Contaminated Soil (SM/PP Hill).

Information developed during the research for the background report (DOE 1991d) indicates that additional work or modification to work is necessary for work planned or being planned in the respective work plans for Operable Units 2, 3, 4, and 5. Additional background on the Mound Plant RI/FS is contained in the Site-wide work plan (DOE 1991f).

2.1. MAIN HILL

Research for the background report included the investigation (records searches, employee interviews) of possible sources of volatile organic chemicals (VOCs) in seeps around the periphery of the Main Hill. The results of the investigation are that the source of the VOCs is possibly the result of waste solvents being disposed of into sinks connected to the sanitary sewer or radioactive waste lines, and subsequent leakage from these lines. Also, there was no indication that any solvents were disposed of in mass quantities by discharge or leaking onto the ground. The conceptual model of the sewers and waste lines as a source is supported by television surveys of storm and sanitary sewers that located breaks and leaks along these lines. Another important part of the conceptual model is that other buried utilities, such as water, electrical, and communication lines, may serve as preferential pathways for contaminant migration because of the permeable backfill surrounding them. The location of underground utilities is required by the FFA (Attachment I, Section 1.1.1) and has previously been documented (DOE 1991a).

Because the general source of the VOCs is not located, a soil gas survey may provide information necessary to focus further investigation. The proposed soil gas survey is described in section 3.

Solvents previously observed in the groundwater from the Main Hill seeps are predominately tetrachloroethane, trichloroethene, 1,2-dichloroethene, 1,1,1-trichloroethane, and trihalomethanes. Accordingly, trichloroethene, 1,2-dichloroethene, 1,1,1-trichloroethane and trichlorofluoromethane (Freon, a trihalomethane) will be used as target compounds for the soil gas survey. Tetrachloroethane was eliminated because its concentration in the seeps is usually lower than, and correlated with, those of trichloroethane.

In addition, toluene will be added as a target compound because

- it is representative of petroleum product contamination and the solvents are not, adding another class of compound, and
- it has been observed at Mound Plant in a previous soil gas survey (DOE 1989).

The compounds predominantly observed in groundwater would not necessarily be the same contaminants found in soil gas. The selected compounds are indicators and do not address all possible contaminants.

2.2. SM/PP HILL

Research for the background report indicated the possibility of waste disposal in Area J of Operable Unit 5. Area J is an area where construction debris (including excess soil) was pushed over the side of SM/PP Hill. However, there was no identification of any specific incident of solvent disposal.

In order to confirm the possibility of solvent disposal in Area J, and to focus any further investigation, a soil gas survey is proposed. The soil gas survey is described in section 3.

In addition, because of the bulk quantities of soil and debris (including concrete) disposed at Area J, a geophysical survey would provide the following benefits:

- identify areas of difficult drilling, e.g., dense concentrations of concrete, and
- confirm or exclude the possibility of ferro-magnetic wastes (rebar, wire, or drums).

Because there is no indication of disposal of liquid waste at Area J, the possibility of large quantities of chemical waste is not considered.

Area 7, in the valley between the Main Hill and the SM/PP Hill, has been the site of considerable infilling (DOE 1991c) with soil, debris and radioactive waste. A rumor that trash from the Historic Landfill (Area B, Operable Unit 1) was removed to the ravine could not be confirmed (DOE 1991d); if true, it would suggest the possibility that some hazardous chemicals could have been relocated to Area 7.

A geophysical survey has already been completed for Area 7 (DOE 1991e). Because of the disposal of bulk quantities of soil and debris, and the possibility of the relocation of hazardous chemicals to Area 7, a soil gas survey would help to scope further investigations.

Building 51, down-valley from Area 7, was the location of a tank used to store oil and solvents that were destroyed by an incinerator in the building. The incinerator was built and used in the early 1970s and was removed in 1990. Sampling of the tank contents and adjacent soil indicated the presence of trichloroethene. Therefore, the area around the tank will be included in the soil gas survey.

2.3. OLD FIRING RANGE DRUM SITE

The Old Firing Range Drum Site (Figures 2.1, 2.2, and 2.3) is a potential release site assigned to Operable Unit 3, Miscellaneous sites. In the Operable Unit 3 Work Plan (DOE 1991b), Figure 14.1 shows the sampling grid at the site. In preparation for the field work, the area was staked by WESTON and Ohio EPA personnel. Since the staking, research conducted for the Operable Unit 9 Site Scoping Report (DOE 1991d) indicated that the area was not properly located.

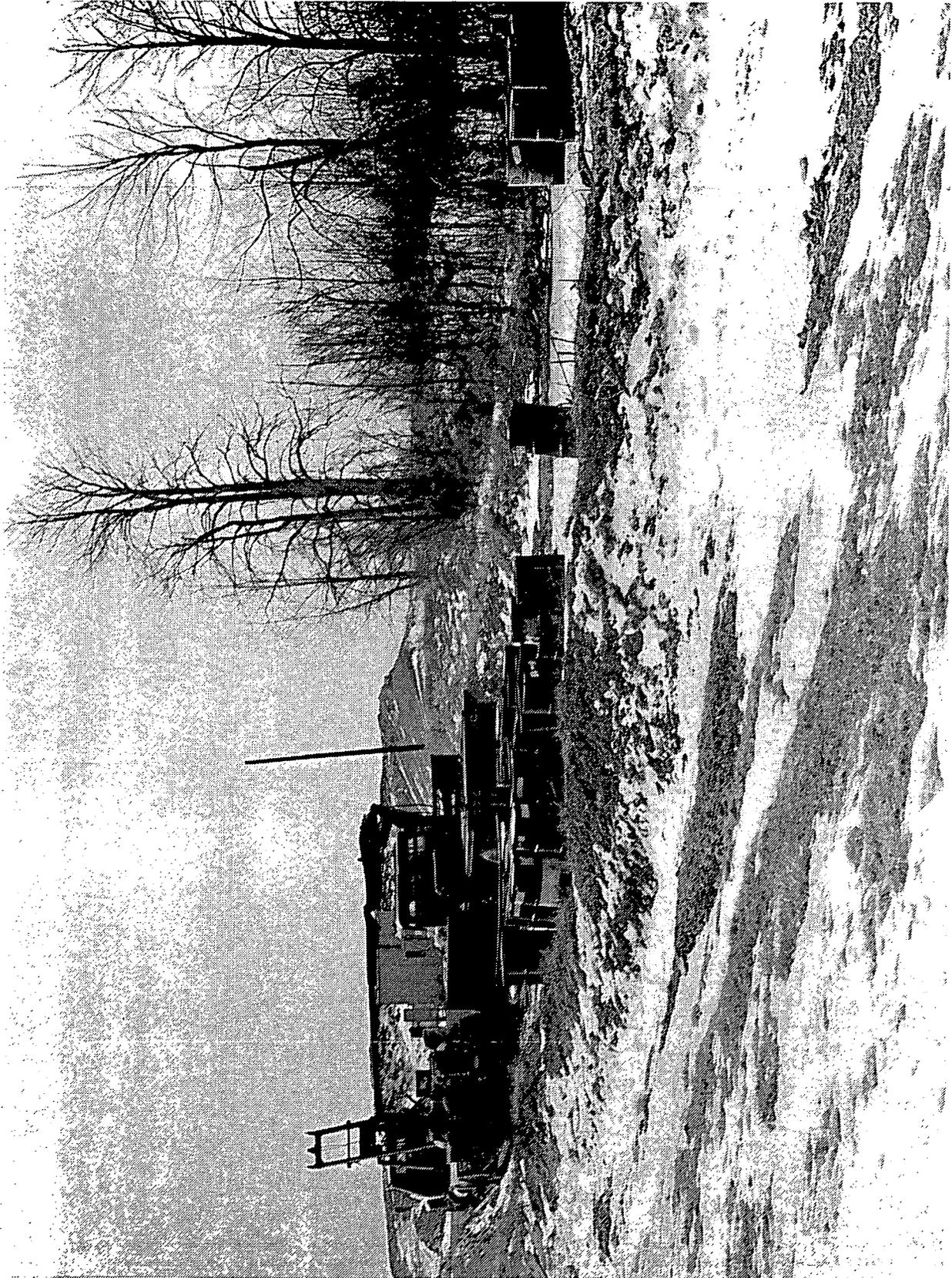


Figure 2.1. Old Firing Range showing drum removal activity (Mound Plant photo reference no. 740359).



Figure 2.2. Old Firing Range drum removal (Mound Plant photo reference no. 740360).

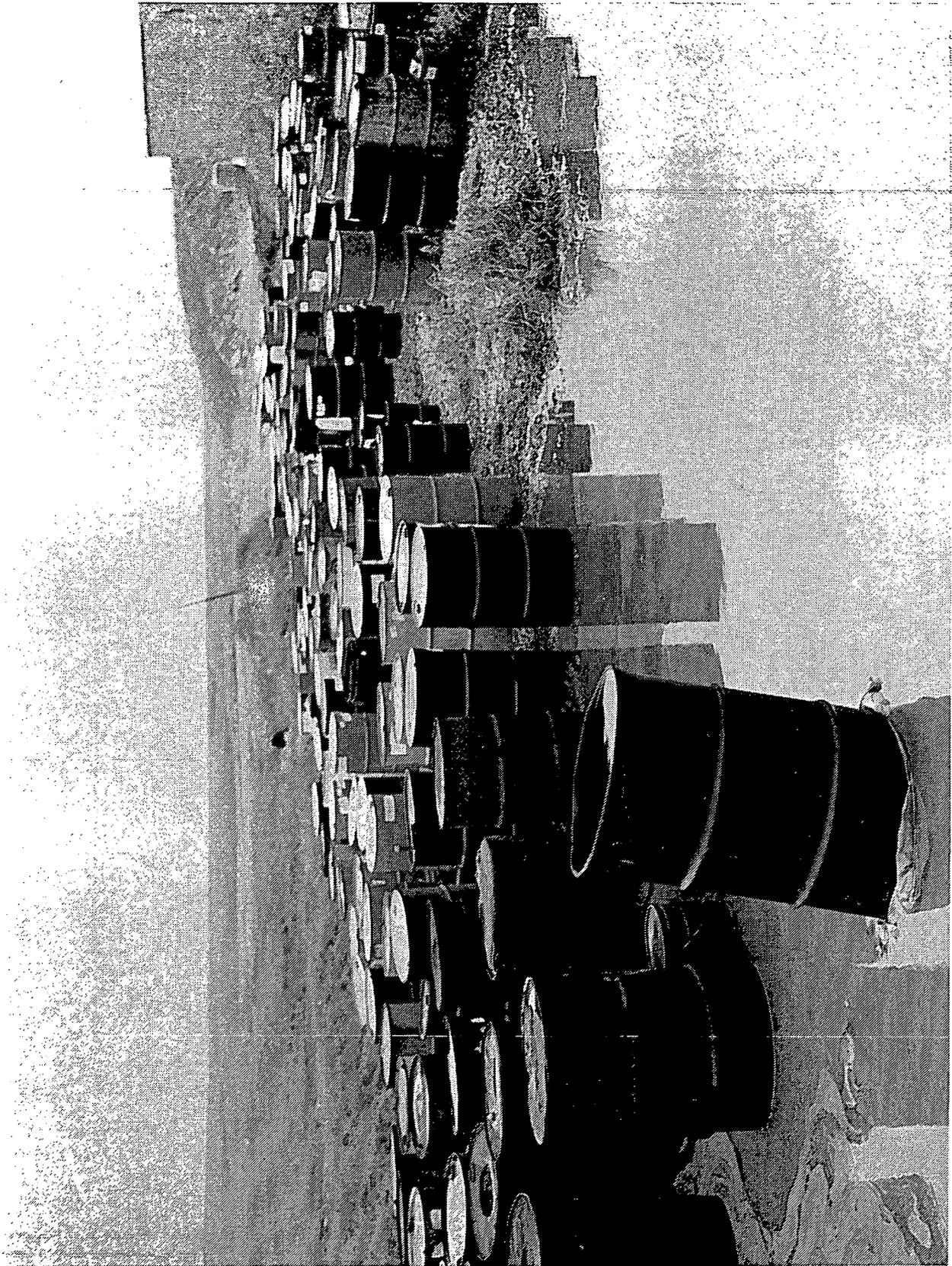


Figure 2.3. Old Firing Range showing drums in ponded water (Mound Plant photo reference no. 740123).

In an effort to resolve this conflict, a photo interpretation map was compiled from vertical aerial photographs taken by the Ohio Department of Transportation on April 14, 1973, and March 3, 1975. These photographs exactly locate the firing range and the drum storage area without the distortions common to oblique photographs, such as those used to locate the site originally (DOE 1991b). The location map (Figure 2.4) was compiled as an overlay onto the recent topographic base map (scale 1 inch = 200 ft) that currently serves as the standard for nearly all of the graphic illustrations used by the program over the past year.

Although the Old Firing Range Drum Site is now accurately located, the previous misidentification indicates that it would be prudent to do additional work to confirm the location. Because the contaminants of concern at the area include solvents, specifically trichloroethane and trichlorofluoromethane (TCF, or Freon, also a refrigerant and a product of chlorinating water), those will be included as target compounds in the soil gas survey. Acetone was also present at the Old Firing Range Drum Site; however, two characteristics constrain its use as a target compound:

- its lack of persistence in the environment due to a tendency to biodegrade rapidly, and
- its high water solubility that causes preferential partitioning to water and absence in soil gas.

Thus, the same target compounds will be used as on the Main Hill. The proposed soil gas survey is described in section 3.

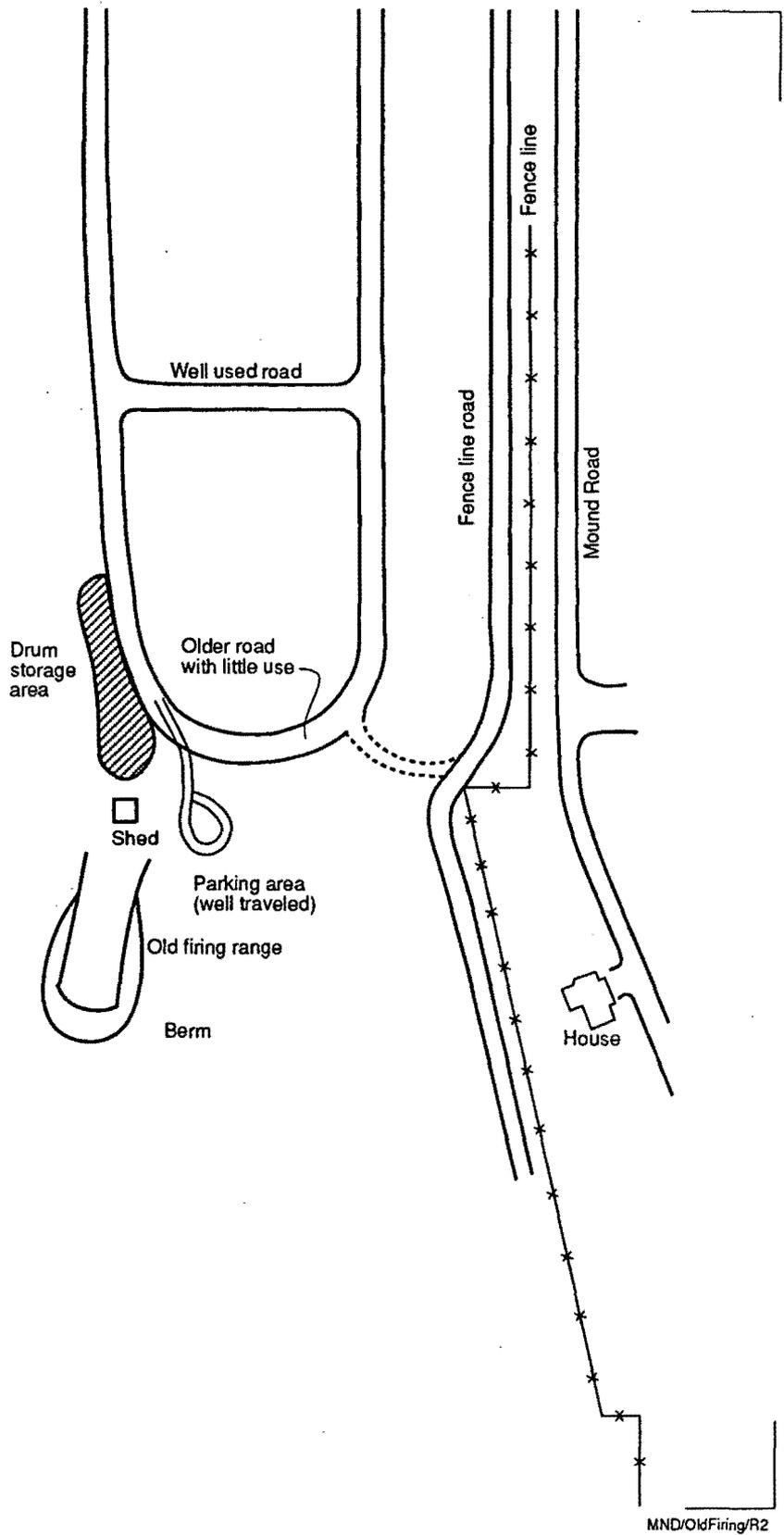
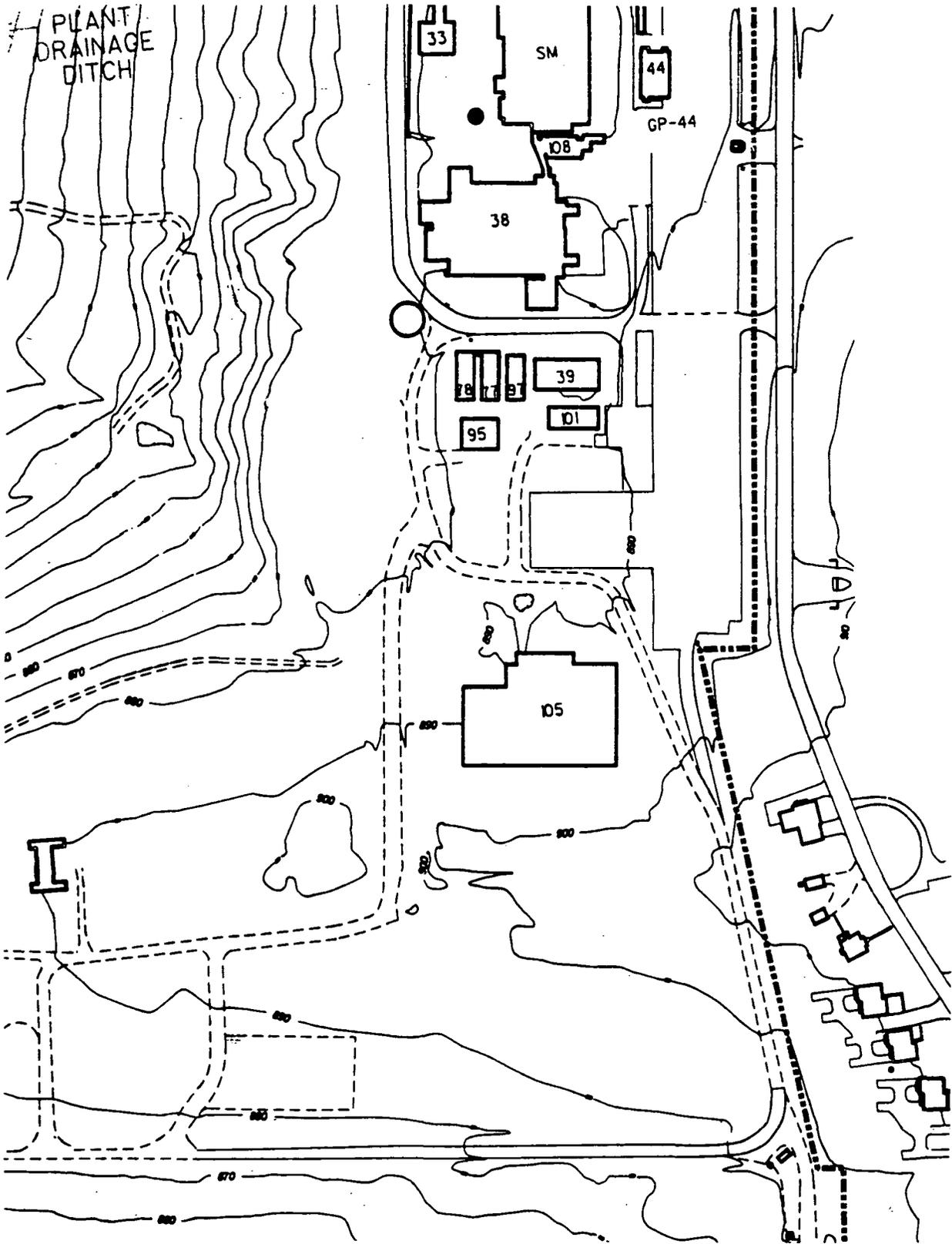


Figure 2.4. Location map of Old Firing Range and Drum Storage Area, April 17, 1983.



3. PROPOSED ADDITIONAL WORK

3.1. SOIL GAS SURVEY

Sampling and analysis for the soil gas survey will be similar to that described in Appendix A. Target compounds, based on the rationale described in section 2, will be

- trichloroethene,
- 1,2-dichloroethene,
- 1,1,1-trichloroethane,
- toluene, and
- trichlorofluoromethane.

Target compounds will be limited to these five in order to expedite and economize the analyses.

Proposed locations for soil gas samples on the Main Hill are shown in Figure 3.1. The rationale for these locations is based on the

- accessibility from roadways,
- locations of sanitary sewers and radioactive waste lines where they cross roadways,
- proximity of buildings where solvents have been used, and
- objective of sampling along roadways systematically at a set interval.

Proposed locations for soil gas sampling at Area J, the Old Firing Range Drum Site, and Area 7 are shown in Figure 3.1. These locations were chosen by a systematic grid covering the areas at an economical grid spacing. Samples will be collected at a constant depth of 5 feet.

Proposed sample locations for the vicinity of Building 51 are also shown in Figure 3.1. Samples for the latter site, only, will be collected at depths of 15 and 25 feet below ground, to be below the bottom elevation of the tank that was removed. Samples will be collected between the former tank location and the Plant drainage ditch, and in a down-valley direction, with the presumption that any contaminant migration would be in the hydraulically downgradient direction.

In addition to the proposed locations, optional or discretionary samples may number up to 45 (approximately four days of work). The samples will be collected at the discretion of the field manager, in consultation with other project personnel, to follow any contamination that is identified by the previously specified grids.

Soil gas results may be influenced by several factors that include the absorption properties of the soil and the soil's air permeability. The results will be used as an indicator of contamination and may lead to additional drilling, sampling, and laboratory analyses.

Soil gas samples will not be collected from saturated soils. Appendix A includes a procedure for collecting water samples from the soil gas probe. At the discretion of the field manager, a water sample may be collected in lieu of a soil gas sample when saturated soil is encountered. Appendix A addresses analysis of water samples collected in this way.

3.2. GEOPHYSICAL SURVEYS

3.2.1. Geophysical Survey in Area J

A geophysical survey will be performed in Area J to locate concentrations of buried metal debris. The survey will be performed over an area of approximately 4 acres. The survey area is steep and overgrown with vegetation, so field work will probably be performed in late autumn or in winter.

Initially, a grid will be established with labeled wooden stakes at 20-ft intervals in the east-west direction and at 10-ft intervals in the north-south direction. The grid will be established based on magnetic north, and will later be converted to the State Plane coordinate system. Figure 3.2 shows the location of Area J and the approximate areal coverage of the survey.

The geophysical surveys will include terrain conductivity and magnetics. Data will be gathered simultaneously by both methods. The terrain conductivity survey will be performed with a Geonics Model EM 31 instrument on north-south traverse lines spaced 10 ft apart, with measurements every 5 ft. The survey will measure apparent conductivity of materials (quadrature component) and proximity to metals (in-phase component) simultaneously. In-phase measurements with the EM 31 are particularly useful because they can detect non-ferrous metals, while magnetic measurements cannot. Concentrations of concrete should produce relatively low conductivity values, but it may be difficult to differentiate concrete among clean fill or natural soils. In addition, concrete containing rebar cannot be differentiated from concentrations of buried metal debris.

The magnetic survey will be performed on north-south traverse lines spaced 10 ft apart, with measurements every 5 ft. Two Gem GSM-19 portable Overhauser magnetometer/gradiometer instruments will be used in the survey: one for use as a field magnetometer/gradiometer, and the other to be used as a fixed base station. Base station readings will be synchronized with field magnetometer readings, and field magnetometer values will be subtracted from base station values to remove the effects

of diurnal (daily) variations in the earth's magnetic field and magnetic storms. Magnetic vertical gradient values will be acquired at the same time, and do not require base station corrections, as they are unaffected by changes in the earth's ambient magnetic field.

Upon the completion of the surveys, both terrain conductivity and magnetic survey results will be plotted in map view to identify anomalies. In addition, data will be plotted as contours in the field to allow review of the data and modifications to the survey.

3.2.2. Geophysical Survey for the Buried Boiler in Operable Unit 4

A magnetic survey will be performed as part of the Operable Unit 4 Investigation (Miami-Erie Canal) to locate the old City of Miamisburg power plant boiler reportedly buried in the area. This survey and the survey in Area J will be performed as part of the same field effort in order to minimize shipping and

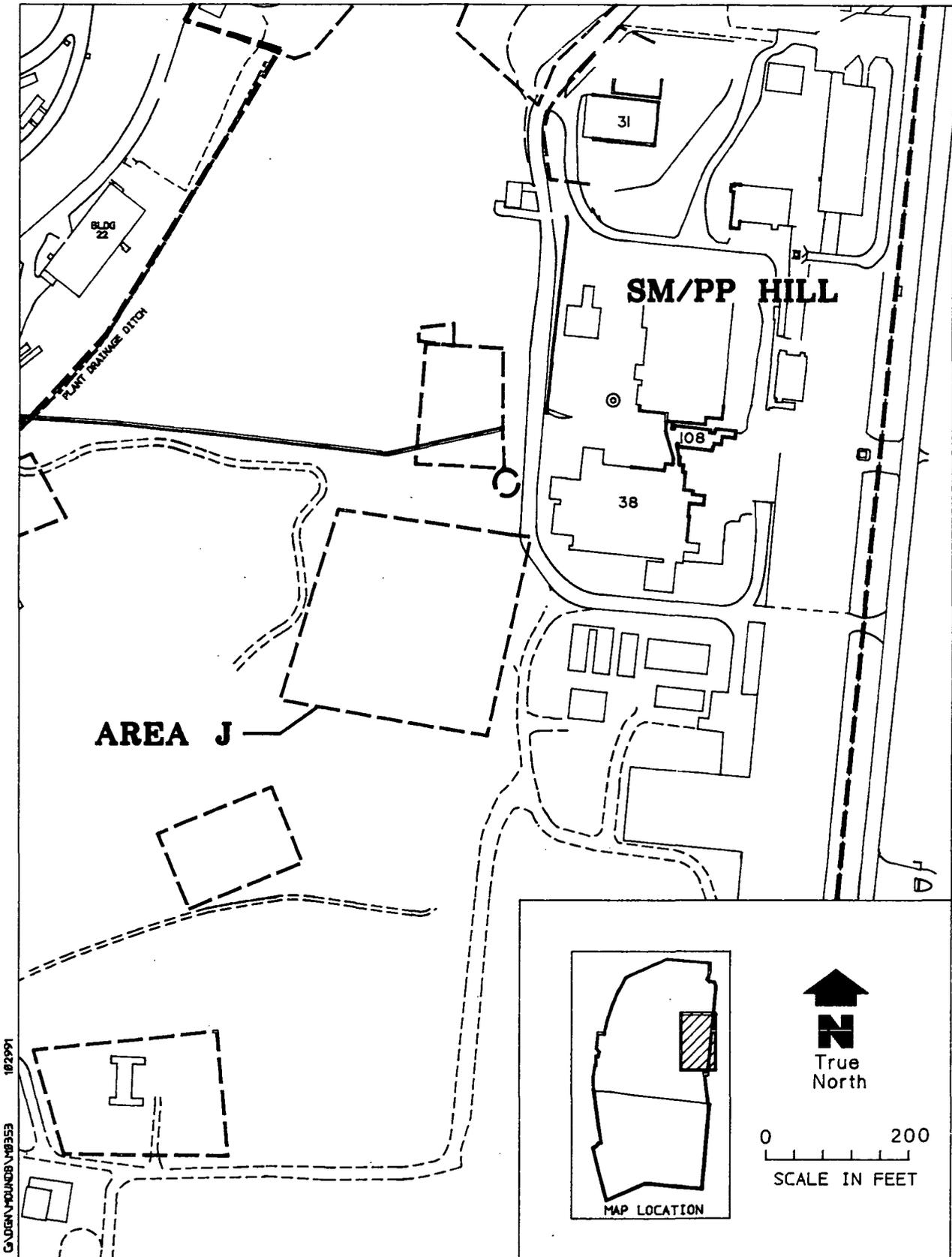


Figure 3.2. Approximate location of Area J on the SM/PP Hill.

mobilization costs. Figure 3.3. shows the Operable Unit 4 survey area with the approximate location of the survey grid. The survey grid will cover less than 90,000 ft². The grid will be established, based on magnetic north, with labeled wooden stakes every 20 ft. Magnetic vertical gradient and magnetic total field measurements will be taken every 10 ft on north-south traverse lines spaced 10 ft apart. Total field readings will be compared with simultaneous readings from a fixed base station. The two Gem GSM-19 magnetometer/gradiometer instruments will be used for this survey.

Upon completion of the magnetic survey, readings will be plotted in map view to locate anomalous zones that could be related to the buried boiler. When these zones have been identified, they will be investigated with the in-phase component of the EM 31 in continuous mode. Readings will be monitored on a real-time basis, but will not be recorded. The methodology for the EM 31 continuous mode, in-phase survey involves rotating the instrument boom over an anomaly that was identified in the magnetic survey and noting deflections in instrument readings. Linear features, such as buried pipes, can be differentiated from discrete buried objects, such as the buried boiler, using this technique. The EM 31 instrument will be used to confirm the identification and location of the buried boiler, and the boiler location will be marked in the field.

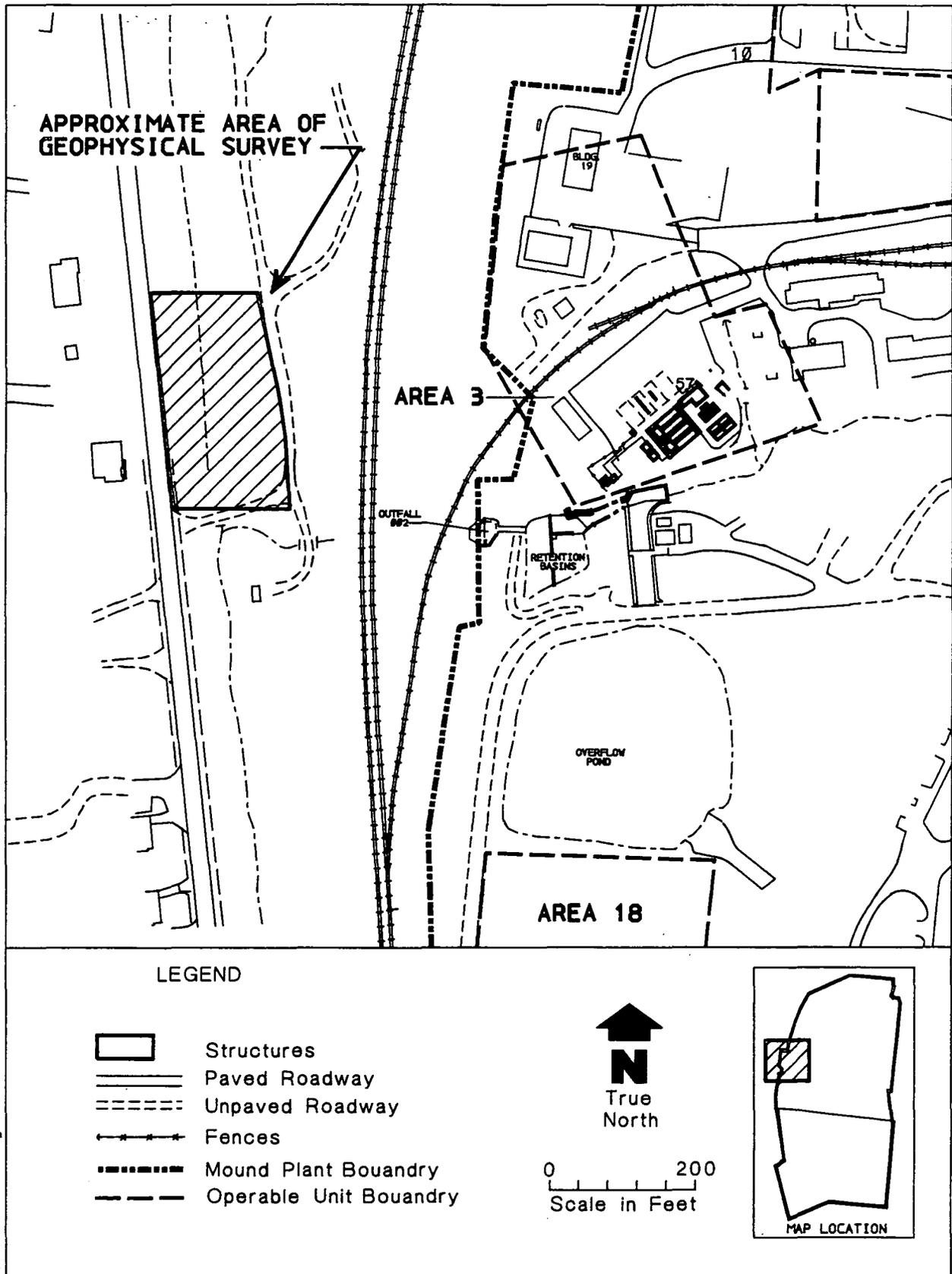


Figure 3.3. Approximate location of the proposed geophysical survey area in Operable Unit 4.

4. REFERENCES

- DOE 1989. "Operable Unit 1, Stage 3 RI/FS Work Plan." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. September 1991.
- DOE 1991a. "Site Scoping Report: Volume IV - Engineering Map Series." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. June 1991.
- DOE 1991b. "Limited Field Investigation Work Plan, Mound Plant Miscellaneous Sites, Operable Unit 3." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. June 1991.
- DOE 1991c. "Site Scoping Report: Volume VI - Photohistory Report." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. May 1991.
- DOE 1991d. "Site Scoping Report: Volume VII - Waste Management Report." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. November 1991.
- DOE 1991e. "Letter Report: Preliminary Results of Reconnaissance Magnetic Survey, Mound Plant Areas 2, 6, 7, and C." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. November 1990.
- DOE 1991f. "Site-wide RI/FS Work Plan, Mound Plant, Operable Unit 9." U.S. Department of Energy, Albuquerque Operations Office, Albuquerque, New Mexico. October 1991.

APPENDIX A

DESCRIPTION OF SOIL GAS SAMPLING AND ANALYSIS

**VOLATILE ORGANIC SAMPLING AND ANALYSES
IN SOIL GAS, SOILS, WATER, AND ATMOSPHERE
BY MODIFIED EPA 8021 HYDRO GEO CHEM, INC.**

**Hydro Geo Chem, Inc.
1430 North Sixth Avenue
Tucson, Arizona 85705
(602)623-6981**

TABLE OF CONTENTS

	Page
INTRODUCTION	1
Summary of Method	2
1. SAMPLING	5
1.1 Scope and Application	5
1.2 Sampling Equipment	5
1.3 Sample Collection	10
1.4 Decontamination of Equipment	13
2. ANALYSIS	15
2.1 Scope	15
2.2 Detection Limits	15
2.3 Apparatus and Equipment	15
2.4 Calibration	22
2.5 Quality Control	25
2.6 Procedures	32
2.7 Calculations	35

List of Figures

<u>Figure</u>		<u>Page</u>
1	Soil Gas Sampling Probe	6
2	Soil Gas Sample Housing	8
3	Soil Gas Sample Collection	11
4	Mobile Laboratory Analytical Schematic	17
5	Purge Apparatus for Shallow Water Samples	20
6	Field Data Sheet	33

TABLE OF CONTENTS (CONTINUED)

List of Tables

<u>Table</u>		<u>Page</u>
1	Compounds EPA Method 8021 (Hall/PID Analysis)	3
2	Breakthrough Volumes for Selected Hydrocarbons on the Carbosieve S-III/Carbopack B/Carbotrap C Thermal Desorption Tube	9

**VOLATILE ORGANIC SAMPLING AND ANALYSES IN SOIL GAS,
SOILS, WATER, AND ATMOSPHERE BY MODIFIED EPA 8021
HYDRO GEO CHEM, INC.**

INTRODUCTION

On site analysis of volatile organic compounds (VOC) is increasingly important to environmental assessments. The ability to perform real-time chemical analysis during investigations of potentially contaminated soils, water, and air allows field decisions to be made regarding the depth and areal extent of the investigation.

The applications of on-site VOC analysis include: soil analysis for evaluation of subsurface spills and leaking tanks; atmospheric analysis for evaluation of VOC emissions from landfills, contaminated soils, and industrial facilities; water analysis for identification and definition of the vertical and areal extent of groundwater VOC plumes; and soil gas surveys in which VOC's in the soil atmosphere are sampled and analysed in order to determine the vertical and areal distribution of VOC's in site soils.

Because VOC's are readily transported in soils by diffusive and advective processes, soil gas surveys have proved to be a powerful technique for determining whether spills have taken place on the site, for locating these spills, and, if subsurface conditions are favorable, for finding and delineating groundwater VOC plumes by the presence of VOC's in overlying soils.

The analytical requirements for real-time mobile-laboratory analysis are different than the conventional VOC analyses prescribed by EPA protocols. The laboratory productivity, that is, the number of analyses required per unit time, must be much greater for the mobile facility. Otherwise, the value of using the data to modify the investigation is diminished.

Despite the need for greater productivity, the other analytical requirements for detection limits, variety of analytes, and freedom from laboratory contamination are, if anything, more stringent than those of fixed laboratory facilities.

The following sampling and analytical protocols have been adopted by Hydro Geo Chem to meet these stringent requirements of on-site VOC analysis.

Summary of Method

In summary, the analytical method consists of the recently approved EPA 8021 protocol, modified to allow greater throughput and to minimize the potential for laboratory contamination. These modifications include temperature programming and flow changes to reduce analytical time, the use of gas rather than water-solution standards, purging of VOA bottles (40 or 250 ml bottles sealed with a teflon septum) directly rather than using a conventional water purging apparatus (a technique recently independently developed and used in EPA Region 5 RI/FS studies), methanol solvent extraction of soils and subsequent stripping of a methanol-water solution; and splitting of the sample injection stream to allow simultaneous analysis on a separate column and detector of other compounds not analyzed by the 8021 protocol. Table 1 lists the compounds that can be analyzed using EPA 8021 protocol.

Table 1. Compounds EPA Method 8021 (Hall/PID Analysis)

Benzene	1,2-Dichloropropane
Bromobenzene	1,3-Dichloropropane
Bromochloromethane	2,2-Dichloropropane
Bromodichloromethane	1,1-Dichloropropane
Bromoform	Ethylbenzene
Bromomethane	Hexachlorobutadiene
n-Butylbenzene	Isopropylbenzene
sec-Butylbenzene	p-Isopropyltoluene
tert-Butylbenzene	Methylene chloride
Carbon tetrachloride	Naphthalene
Chlorobenzene	n-Propylbenzene
Chloroethane	Styrene
Chloroform	1,1,1,2-Tetrachloroethane
Chloromethane	1,1,2,2-Tetrachloroethane
2-Chlorotoluene	Tetrachloroethene
4-Chlorotoluene	Toluene
Dibromochloromethane	1,2,3-Trichlorobenzene
1,2-Dibromo-3-chloropropane	1,2,4-Trichlorobenzene
1,2-Dibromoethane	1,3,5-Trichlorobenzene
Dibromomethane	1,1,2-Trichloroethane
1,2-Dichlorobenzene	1,1,1-Trichloroethane
1,3-Dichlorobenzene	Trichlorofluoromethane
1,4-Dichlorobenzene	1,2,3-Trichloropropane
Dichlorodifluoromethane	1,2,4-Trimethylbenzene
1,1-Dichloroethane	1,3,5-Trimethylbenzene
1,2-Dichloroethane	Vinyl chloride
1,1-Dichloroethene	o-Xylene
1,2-cis-Dichloroethene	m-Xylene
1,2-trans-Dichloroethene	p-Xylene
	Trichloroethene

The sampling methods included in the protocol have been designed to allow accurate, contamination-free sampling of soils, water, atmosphere, and soil gas. These methods offer a detection limit of at least 0.1 $\mu\text{g}/\text{kg}$ (soil), 0.01 $\mu\text{g}/\text{l}$ (soil gas or water), and 0.001 $\mu\text{g}/\text{l}$ (atmosphere) for any compounds listed in Table 1. Additional, simultaneous analysis is provided for total petroleum hydrocarbons, methane, and total chlorinated hydrocarbons. The following sections document the materials, apparatus, and procedures used.

1. SAMPLING

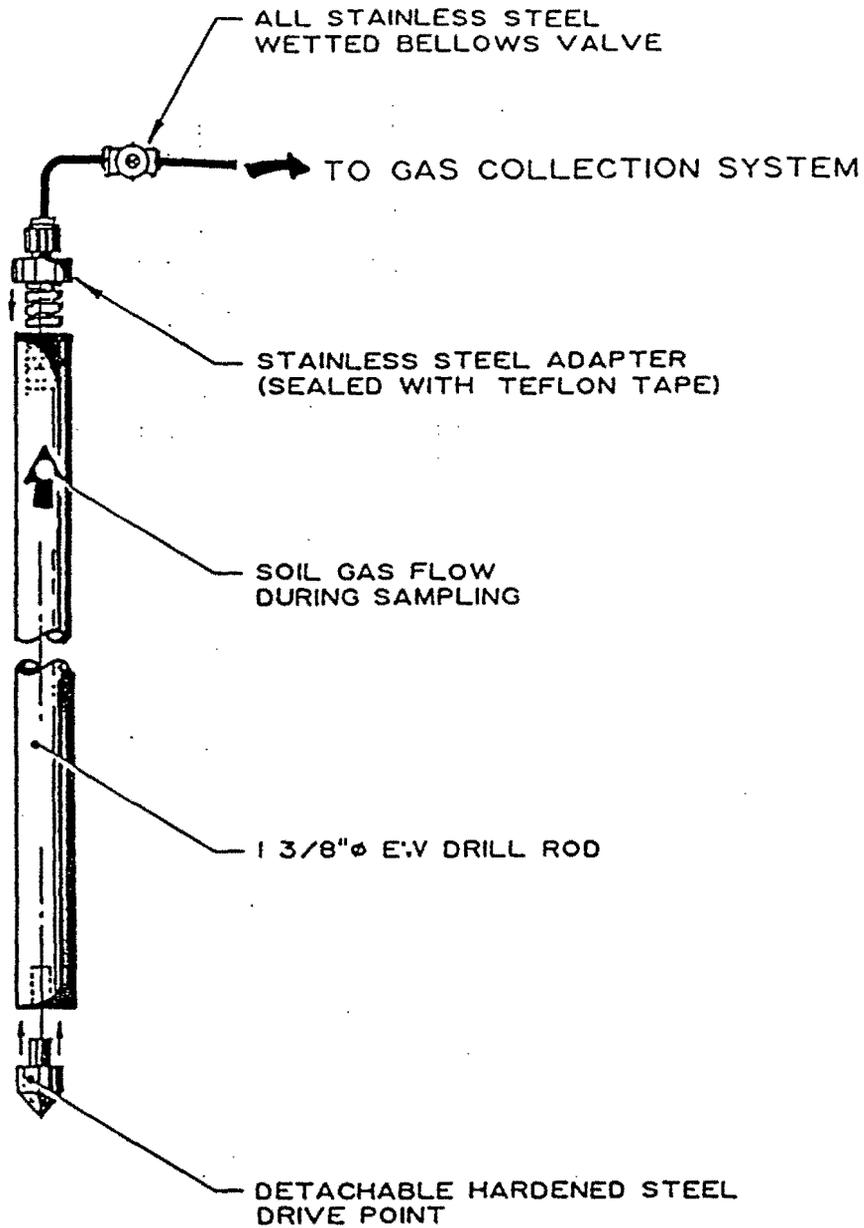
1.1 Scope and Application

This section covers the materials, equipment and procedures utilized by Hydro Geo Chem, Inc. for collecting soil gas, atmospheric, soil, and shallow groundwater samples in the field.

1.2 Sampling Equipment

1.2.1 Sampling Probes and Drive Point Rigs

Sampling probes consist of 5 foot sections of nickel plated $1\frac{3}{8}$ " hardened EW drill rod with Acme threads. The high carbon steel points are left behind when the pipe is hydraulically pulled back to expose the formation to pumping. Figure 1 shows our probe design. The probes are driven to the sampling depth using a heavy duty hydraulic hammer mounted on Ford F-450 trucks. These rigs are capable of driving sampling pipe to a depth in excess of 50 feet under normal driving conditions. The drive point rigs are equipped with hydraulic outriggers, pipe racks and a steam cleaner. The probes are removed by the drive point rig using a hydraulically activated pulling dog. Latex gloves are worn during handling and assembling of the sampling apparatus.



NO SCALE

SOIL GAS SAMPLING PROBE

FIGURE 1

1.2.2 Sampling Adaptors

Soil gas samples are collected from the probes via adaptors constructed of stainless steel pipe caps welded to stainless steel tubing connected to an inline stainless steel bellows valve.

1.2.3 Soil Gas Cartridges

Atmospheric or soil gas samples are collected in stainless steel cartridges housing a glass tube (Supelco) filled with a three layer packing of various types of adsorptive hydrophobic carbon (see Figure 2). The soil gas is passed through these layers, the first, Carbotrap, absorbing "heavy" volatiles such as dichlorobenzene, the second, Carbopack B, the lighter volatiles such as TCE and DCE, and the third, Carbosieve III, the ultralights such methylene chloride or vinyl chloride. The most mobile constituent, vinyl chloride, has a breakthrough volume of 158 liters (vinyl chloride detected at the tube outlet after 158 liters of 25 ppb vinyl chloride are passed through the cartridge). These cartridges are therefore rated to absorb at least 158 liters of soil gas or atmospheric gas before breakthrough of any of the priority pollutants listed in EPA method 8021. Table 2 shows some breakthrough volumes for the types of carbon sorbents making up the adsorption cartridge. Thus the sampling capacity of this technique far exceeds that of syringe collection. The high capacity is necessary to meet the wide range of specified detection limits encountered in site investigations.

FLOW DIRECTION
DURING SAMPLING

STAINLESS STEEL
CARTRIDGE HOUSING

ACTIVATED CARBON PACKED
GLASS CONCENTRATING
CARTRIDGE

TEFLON O-RING SEAL

SOIL GAS SAMPLE HOUSING

Figure 2

Table 2

Breakthrough Volumes (in Liters) for Selected Hydrocarbons
on the Carbosieve™ S-III/Carbopack B/Carbotrap C Thermal Desorption Tube

Hydrocarbon	Carbosieve S-III (125 mg)	Carbopack B (200 mg)	Carbotrap C (300 mg)
Vinyl Chloride	158		
Chloroform		1.1	
1,2-Dichloroethane		0.4	
1,1,1-Trichloroethane		2.7	
Carbon tetrachloride		4.7	
1,2-Dichloropropane		6.8	
Trichloroethylene		2.5	
Bromoform		1.7	
Tetrachloroethylene		2.2	
Chlorobenzene		316	
n-Heptane		262	
1-Heptene		284	
Benzene		2.3	
Toluene		130	
Ethylbenzene		4060	12.9
p-Xylene			11.2
m-xylene			11.0
o-xylene			11.0
Cumene			27.8

1.2.4 Groundwater Samples

Groundwater is sampled through the 1³/₈ inch EW drill rod using ³/₈" stainless steel bailers.

1.2.5 Soil Samplers

Soil is sampled at specified intervals using a 1 inch diameter by 12 inch length ring-barrel sampler containing multiple stainless steel sleeves.

1.3 Sample Collection

1.3.1 Soil Gas Sampling

After purging 3 probe volumes from the sampling train, the bellows valve on the adaptor is shut off and the stainless steel sample cartridge housing is attached in line using Swagelok compression fittings. The cartridge inlet leads to the adaptor and the outlet to a programmable mass flow controller equipped with a vacuum regulated oilless diaphragm vacuum pump (see Figure 3). The flow controller is typically programmed to pump 200 ml of soil gas at a flow rate of 100 ml/min. When the specified flow volume has been obtained, a

fig 3

SAMPLE TYPE

- SOIL GAS
- ATMOSPHERE

SAMPLE TRAPPING

WEAK, ($-C_6$)

MEDIUM, (C_3-C_6)

STRONG, (C_1-C_2)

3-LAYER CARBON SORPTION TUBE

OILESS DIAPHRAM VACUUM PUMP

VACUUM REGULATOR

INLINE FILTER

LED DISPLAY

FLOW 60

FLOW INDICATOR

VACUUM GAGE

.7	8	9	-
4	5	6	-
1	2	3	=
FR	S	R	O

KEYPAD

MASS FLOW CONTROLLER/METER

PROGRAMMABLE GAS SAMPLER

SOIL GAS SAMPLE COLLECTION

Figure 3

solenoid valve is automatically closed and the sample collection is complete. The mass flow meter delivers sample volumes between 20 and 5000 standard ml with less than 2% error independent of temperature and vacuum conditions.

1.3.2 Atmospheric Sampling

Atmospheric samples are collected by positioning the probe in the area of interest, and programming the mass flow controller to the appropriate pumping rate and total sample volume. If desired, the probe can be moved through a sampling volume at a specified rate to collect an integrated sample. No purging is necessary for atmospheric samples.

1.3.3 Soil Sampling

Soil samples are collected using 1" ring samplers equipped with three 4" stainless steel sleeves. The 1³/₈" sampling probe is first equipped at its end with a retrievable point and emplaced to depth. The sampling probe is then fully extracted and the retrievable point is replaced with the 1" ring sampler. The sampling probe is then run back into the original hole and the sampler is driven 12" beyond the hole bottom. The sampling probe is extracted again and the 1.0" ring sampler detached. No liquids (i.e., drilling mud, water, foam) are used

during the probe placement or sampling procedure. All soil samples to be analyzed or sent out for analysis are retained in the stainless steel tubes (1.0 inch diameter, 4 inches long). Immediately following removal of the stainless steel sleeves from the sampler, the center tube is capped with aluminum foil and plastic slip caps. The slip caps are then duct taped to the stainless steel sleeves to maintain a proper seal. Samples are labeled and placed in a zip lock plastic bag and stored in a cooler.

1.3.4 Water Sampling

Groundwater samples are collected using $\frac{3}{8}$ " stainless steel bailers. The bailers are lowered down the inside of the probe using a nylon cord that is replaced before each sampling. The water sample is carefully poured in 40 ml VOA vials in a manner not to allow air bubbles to pass through the water sample. The liquid full vials are then immediately capped with a teflon-lined septum cap and delivered to the mobile lab for analysis.

1.4 Decontamination of Equipment

1.4.1 Prior to each use and reuse, each soil sampler, stainless steel sleeve, sampling probe, point and bailer are steam cleaned and stored in clean storage areas on the drive point rigs. Care is taken with this equipment to eliminate both soil-surface and cross-hole contamination.

Vinyl or latex surgical gloves are worn during handling and assembly of the sampling apparatus.

1.4.2 Adaptors, stainless steel bottles, and stainless steel cartridge holders are heated to 120°C using a convection oven and held for 1 hour at that temperature. Carbon packed desorption cartridges are purged at 400°C with helium for 8 minutes.

1.4.3 Separate storage areas are provided for used and cleaned equipment. No equipment is reused without cleaning.

2. ANALYSIS

2.1 Scope

This section covers the equipment, materials, and procedures used to determine the concentrations of various volatile organic compounds in the soil gas, atmospheric, soil, and shallow groundwater samples.

2.2 Detection Limits

Method detection limits (MDL's) are matrix dependent. The MDL for soil gas samples is 0.01 $\mu\text{g}/\text{l}$, 0.01 $\mu\text{g}/\text{l}$ for water and 0.1 $\mu\text{g}/\text{kg}$ for soils. The MDL for atmospheric samples is 0.001 $\mu\text{g}/\text{L}$. The applicable concentration range of this method is influenced by sample size and instrument limitations.

2.3 Apparatus and Equipment

2.3.1 Gas Chromatographs

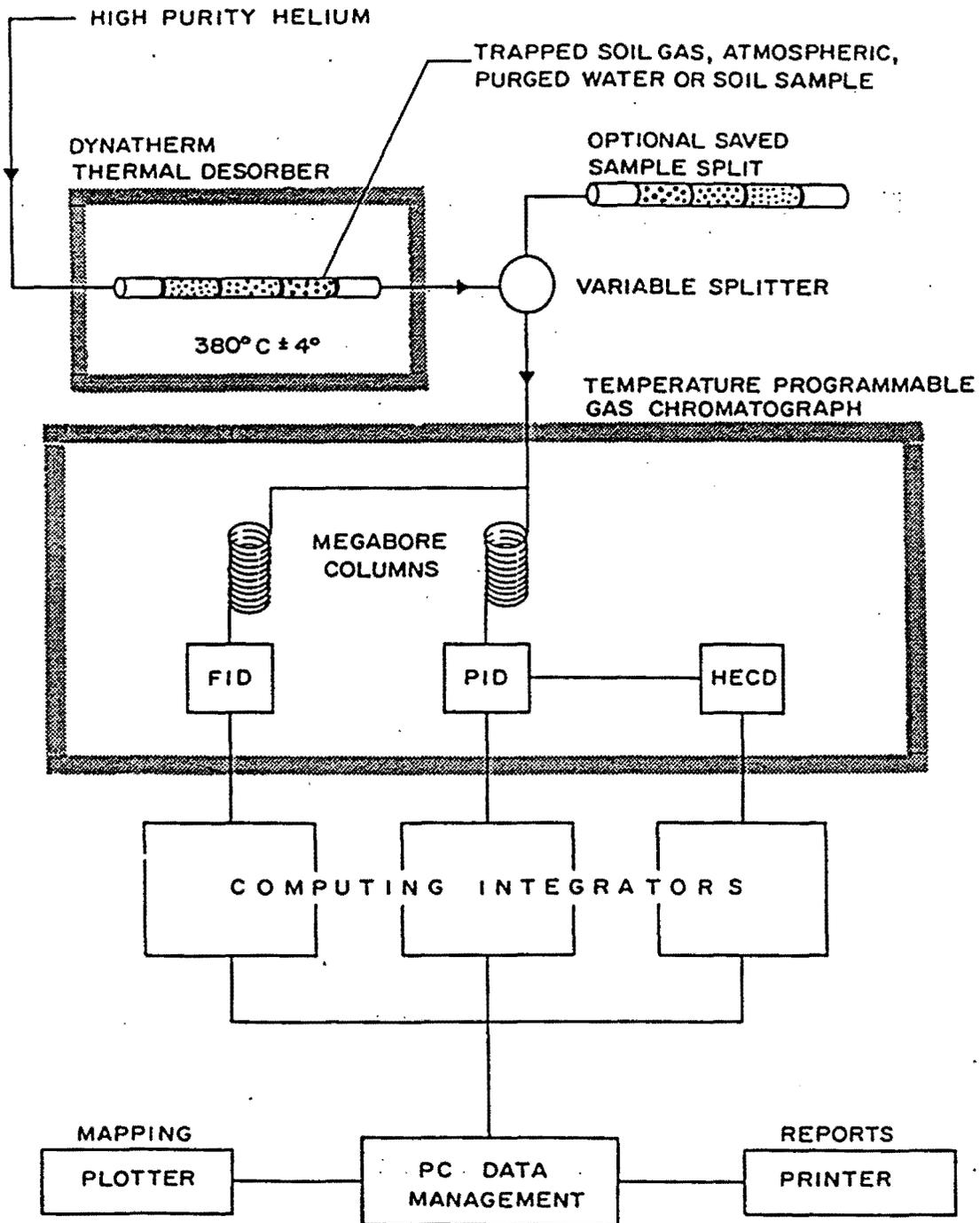
Hydro Geo Chem's mobile laboratories, used to provide on-site analyses, are housed in 18 foot custom built non-motorized trailers. The mobile laboratories are stand-alone vehicles that operate separately from the drive point rig, thereby allowing efficient operation of both. Each mobile lab has a Varian 3400 temperature programmable gas chromatograph (GC) equipped with cryogenics capable of cooling the column to below 0°C using carbon dioxide. The chromatograph is connected to an Envirochem thermal desorber (Model 850) which accepts the glass sorption tubes used to collect the soil gas, atmospheric,

or the purged water or soil samples. Figure 4 is a schematic of the analytical apparatus. Helium flow is opposite to the flow direction of sample collection. The thermal desorber rapidly heats the sample sorption tube to $380 \pm 4^{\circ}\text{C}$ in 26 ± 2 seconds releasing the volatile organic compounds from the activated carbon. The released compounds are transferred from the desorber unit to the analytical columns via heated (250°C) nickel lines. The compounds are held in the cooled columns (cryofocused) at the start of the chromatographic run.

The carrier gas is ultra high purity helium at 10-20 ml/minute. The carrier gas flow is augmented with an additional 25 ml/minute helium before entering the photoionization detector (PID) to optimize response of both PID and Hall electrolytic conductivity (Hall) detectors.

2.3.2 GC Columns

A DB 624 Megabore column, 30 m x 0.53 mm (J&W Scientific) is used in the Varian 3400 chromatograph. The helium flow rate is adjusted to approximately 7.0 ml/minute. The temperature program varies with the client needs. A typical temperature program is as follows: the column temperature is held at 2°C for 3 minutes, then



MOBILE LABORATORY ANALYTICAL SCHEMATIC

Figure 4

programmed to 35°C at 15 C°/minute, no hold time, to 145°C at 8 C°/minute, no hold, to 230°C at 35 C°/minute.

Additional columns are available for the analysis of pesticides and classes of hydrocarbons other than aromatic and halogenated. Columns available include DB-WAX, 30m x 0.53mm, DB-5, 30m x 0.53mm, and DB 608, 15m x .53mm. All columns are obtained from J&W Scientific.

The GC is also equipped with a 1/8" x 18" Carbosphere column, 60/80 mesh, used to analyze for nonsorbable gases, such as methane, by direct injection. This column is operated at ambient temperatures.

2.3.3 Detectors

2.3.3.1 A photoionization detector (PID) equipped with a 10.2ev lamp (Tracor Model 703) is used.

2.3.3.2 A Hall electrolytic conductivity detector (HECD) (Tracor Model 700A) is also used. Operation conditions are as follows:

Reaction tube:	Nickel 1/16" OD
Reactor temperature:	900°C
Reactor base temperature:	250°C

Electrolyte:	n-propyl alcohol
Electrolyte flow rate:	0.7 ml/min
Reaction gas:	hydrogen at 35 ml/min.
Carrier gas plus make up:	helium at 32 ml/min.

2.3.3.3 Hydro Geo Chem also has available an Electron Capture Detector, ECD (Varian).

2.3.3.4 A Flame Ionization Detector, FID (Varian), is also provided for total hydrocarbon analyses.

2.3.4 Integrators

The mobile laboratories are equipped with Spectra Physics dual channel integrators (Model 4400) and Varian integrating printer/plotters.

2.3.5 Purge and Trap Apparatus

An in-house designed and built purging apparatus (Figure 5) is used in the analysis of soil and water samples. High purity, inert (He

PURGE APPARATUS FOR SHALLOW WATER SAMPLES

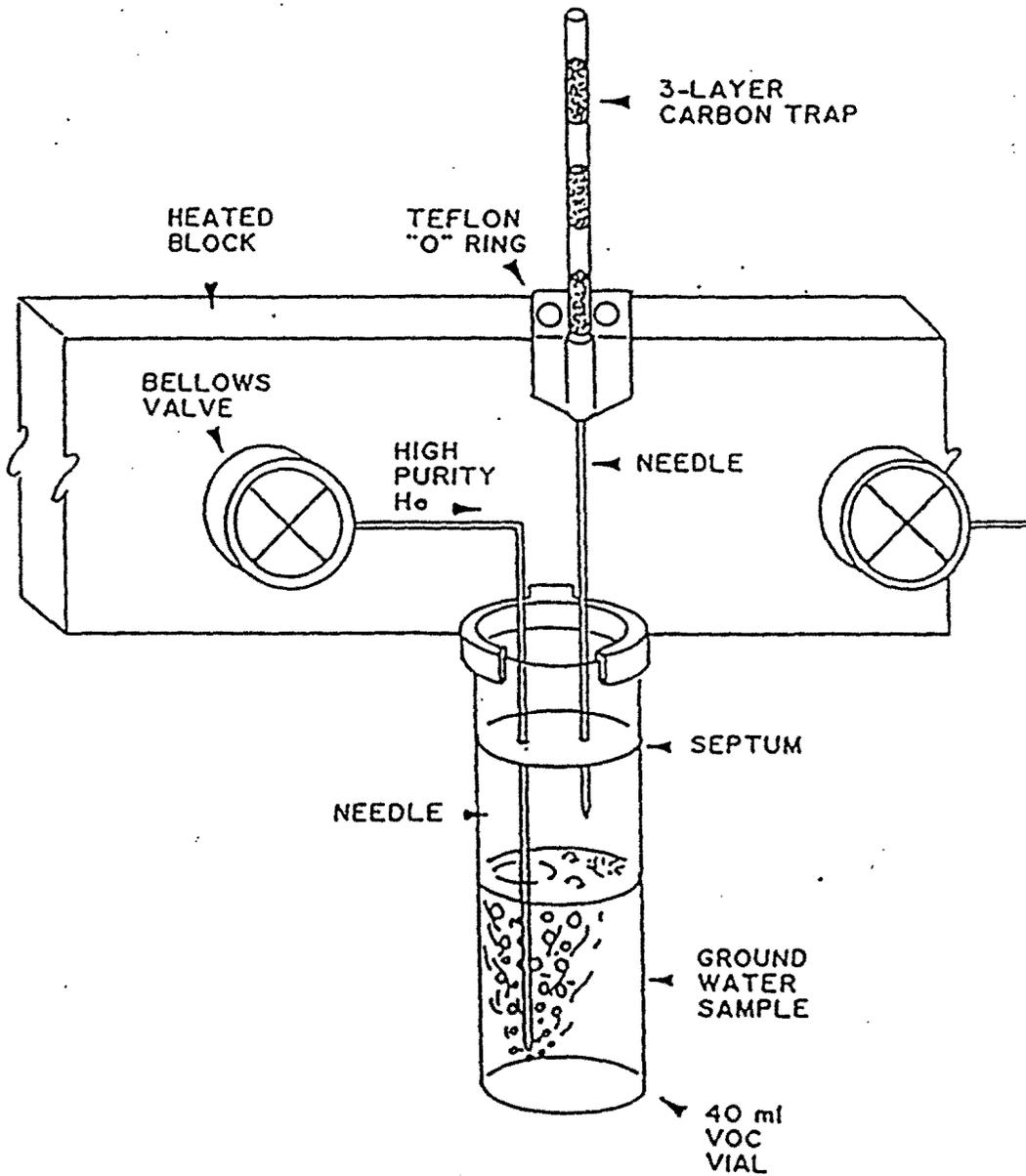


Figure 5

or N₂) gas is bubbled through the sample at 200 ml/min for 15 minutes. In addition, a temperature programmable Dynatherm Thermal Dynamic Stripper is also used for purging both soil and water samples. Purged sample components are trapped in activated carbon cartridges.

2.3.6 Standards and Reagents

2.3.6.1 Standards are obtained from certified gas mixtures or prepared from stock mixtures of neat reagent grade compounds. Stock mixtures are prepared by adding a measured aliquot of each compound to be analyzed to a preweighed septum sealed vial. The actual mass of each compound added is determined by weighing the vial. An aliquot volume of the final mixture is then weighed to establish density (weight/volume). Weighing is done on a 0.1 mg Mettler balance calibrated according to manufacturers guidelines with weights traceable to NBS standards. Certified mixtures include vinyl chloride in nitrogen, and methane in nitrogen purchased from Matheson Gas Products, Cucamonga, California.

2.3.6.2 A spiking solution containing two compounds to be used as internal standards are prepared as described in Section 2.3.6.1. The internal standards are selected such that they do not interfere with the compounds of interest. Typical compounds used as internal standards are fluorobenzene, 2-bromo-1-chloropropane, bromochloromethane, 1-

chloro-2-bromobutane, and 4-bromo-fluorobenzene. The internal standard is added to the calibration standards or samples and carried through the analytical procedure. The amount of internal standard is selected such that its concentration is 3 to 5 times greater than the expected range of concentrations found in the actual samples.

2.3.6.3. VOC-free water used in purging soil samples is prepared from distilled water degassed by boiling >1 hour.

2.3.6.4 Purge and Trap or HPLC grade methanol is used when analyzing soil samples.

2.4 Calibration

2.4.1. Calibration

2.4.1.1 For daily soil gas calibration standards, a measured volume of the standard mixture is injected into a nitrogen-filled 1-liter glass, gas bottle through a septum side port. After heating the bottle to achieve volatilization and mixing of the standards, measured volumes are extracted with a gas syringe and injected into a 200 ml/min helium gas stream leading to a carbon packed sorption cartridge. Internal standards, if utilized, will also be injected at this time. After 2 minutes,

this cartridge is inserted into the thermal desorber and analyzed exactly as the samples.

2.4.1.2 Standards used for soil and water analysis are prepared by injecting an aliquot of the stock mixture into methanol. An aliquot of the methanol solution will be injected into a 10.0 ml volume of water and purged in the same manner as soil or water samples. The aliquot of stock standard and methanol solution will depend on concentrations anticipated in the samples.

The amount of the standard stock solution used are dependent upon the required mass of analyte.

The standard will be injected at least three times at the beginning of the day to verify the instrument response. If the response varies by greater than $\pm 20\%$ appropriate measures will be taken to correct the circumstances causing the variability. Continuing calibration checks are performed after every tenth sample.

2.4.1.3 Spectra Physics calculates response factors when the external standard method is used as follows.

$$RF = A/C$$

where A = area of analyte to be measured

C = concentration of analyte, $\mu\text{g/l}$

Varian 3400 calculation of RF

$$\text{RF} = C/A \times 10000$$

2.4.1.4 The Spectra Physics calculates response factors when internal standards are used as follows.

$$RF_S = \frac{C_S}{A_S} \times \frac{A_{IS}}{C_{IS}}$$

where RF_S = response factor of components

A_S = area of components peak

C_S = amount of component used in the calibration sample, μg

A_{IS} = Area of the internal standard peak

C_{IS} = amount of internal standard used in the calibration sample, μg

2.4.1.5 Acceptable retention time window is ± 0.10 minutes from the average retention time derived from the daily calibration analyses.

2.5 Quality Control

2.5.1 System Blank

A randomly selected sampling cartridge is analyzed daily to detail interferences from cartridges or the analytical system. If interference is found at unacceptable levels, an unpacked cartridge is analyzed to determine whether the interference is due to the cartridge or to the analytical system. Appropriate measures are taken to eliminate such interferences.

2.5.2 Reagent Blanks

At the beginning of each day that soil or water samples will be analyzed, the chemist fills a sampling container with reagent water/methanol and proceeds to handle it as an actual sample is handled in order to demonstrate that the system, methanol, and water are interference-free. If VOC's are detected, a water blank will be analyzed to determine if the interferences are in the water or the methanol. Appropriate measures will be taken to eliminate the interferences.

2.5.3 Field Blanks

2.5.3.1 Soil Gas

Prior to each day's soil gas or atmospheric sampling, field blanks of the entire sampling apparatus are taken and analyzed to check background contamination in the sampling system and cartridges. Sampling cartridges are attached to both the inlet and outlet end of a sampling probe. The sample collected in the discharge end cartridge is representative of sampling train contamination only while the intake cartridge provides a measure of the atmospheric concentrations. Additional field blanks are collected prior to any reuse of recleaned sampling equipment.

2.5.3.2 Water

A sampling container will be filled with interference-free water in the field in the same manner as water samples are collected. This sample, now designated as a field blank, is returned to the laboratory for analysis. If VOC's are detected, sample collection procedure will be reviewed. If necessary, sampling equipment will be thoroughly decontaminated. One field blank will be collected and analyzed on each day that water samples are collected.

2.5.3.3 Soil

Collection of field blank soil samples is not feasible due to the nature of the matrix and because interference-free soil is ordinarily not available.

2.5.4 Duplicate Samples

Duplicate soil gas, atmospheric, or shallow groundwater samples are collected from each sampling location. Duplicate analyses are performed on at least 10% of the samples collected. Duplicate analyses are not performed on soil samples because it would require homogenization of the sample, tend to release volatiles from the sample, and therefore, limit the accuracy of the results.

2.5.5 Trip Blanks

2.5.5.1 Soil Gas

An unused sample cartridge is transported into the field with the sampling equipment. The trip blank cartridge is handled in the same manner as a sample, but a sample is not collected through this cartridge. The trip blank is returned to the lab with the other samples and

analyzed. If VOC's are detected, sample handling and transport procedures are subsequently reviewed.

2.5.5.2 Water

A sampling container is filled with water determined to be interference-free and taken into the field. The trip blank container is handled in the same manner as other water samples. The trip blank is then returned to the laboratory for analysis with the other samples. If VOC's are detected, sample handling and transport procedures are reviewed and sampling equipment is decontaminated as necessary.

2.5.5.3 Soil

Trip blanks for soil sampling are not used because uncontaminated soils without background levels of organics are not available and the trip blank procedures are not feasible.

2.5.6 Matrix Spike Duplicate

2.5.6.1 Soil Gas

During each standard calibration procedure for soil gas analysis, the activated carbon in the sample collection cartridges is directly spiked and thermally desorbed.

2.5.6.2. Water

Once a day a duplicate field sample is spiked with a calibration standard of known concentration. This spiked sample is then processed and analyzed in the same manner as all samples. The difference between the reported concentration per compound and the concentration of the spike are then compared to the previous analysis of the unspiked sample duplicate.

2.5.6.3. Soil

Soil sample spikes are provided on purged soil samples by injecting a compound of known concentration directly into the vessel containing methanol extract of the soil. The soil sample is then purged and trapped onto the carbon cartridges for thermal desorption analysis to evaluate purging efficiencies.

2.5.7 Chromatographic Information

2.5.7.1 System Parameters

On the first page of each day's chromatograms, the following system parameters are noted:

- A) Gas flows for H₂, He, N₂, and air
- B) Tank pressures for H₂, He, N₂, and air
- C) Temperatures
 - 1. Injector
 - 2. Columns
 - 3. Detector
 - 4. Thermal desorber oven
 - 5. Thermal desorber transfer lines
 - 6. Thermal desorber desorption temperature and duration
- D) Integrator parameters
 - 1. Attenuation
 - 2. Peak markers
 - 3. Baseline offset
- E) Column(s)
 - 1. Type
 - 2. Length and diameter
 - 3. Packing material
 - 4. Temperature
- F) Operator
- G) Date

If any system parameters change, the changes shall be noted.

2.5.8 Internal Quality Control

All chromatograms are reviewed internally by a chemist other than the chemist performing the analysis.

2.5.9 Outside Quality Control Audits

Samples are periodically sent to independent laboratories for analysis as a quality assurance check.

2.5.10 Sample Chain of Custody

All samples are labeled with the following information:

- 1) Sample identification number
- 2) Date and time of sample collection
- 3) Name of sampler

In addition to labeling the samples, a field data/chain of custody form is completed for each sample (Figure 6). At the time of sample collection, the field sampler signs the custody form and records the date, time and sampling conditions. The sample is then transferred to the laboratory, where the individual receiving the sample for analysis signs the original custody form and records the date and time. This Soil Gas

Field Data Sheet (Chain of Custody Form) is then filed in a notebook with the hard copy of the analytical results and eventually becomes part of the final report.

2.6 Procedures

2.6.1 Typical chromatographic equations are summarized in Section 2.7.

2.6.2 The system is calibrated daily as described in Section 2.4.1.

2.6.3 Soil Water Samples

A representative fraction, typically 20 ml of each cooled soil water sample will be transferred to a tared 40.0 ml VOC vial. The remaining volume is stored at less than 4°C. If less than 20 ml of the soil water sample is used, the volume will be brought to 25 ml with VOC-free reagent water prior to purging. Any surrogate or spiking

mixture will then be introduced by piercing the septum and injecting the mixture below the water surface. Following any additions, needle sparging will be carried out for 15 minutes under ambient lab temperatures using a purge gas flow rate of 200 ml/minute. The purged volatile compounds will then be trapped on a packed cartridge which is held at 40°C to minimize carry-over of water.

2.6.4 Soil Samples

Five grams of each soil will be transferred to a 40 ml VOC vial. Immediately following, five milliliters of HPLC-grade methanol will be added and the vial sealed. Any surrogate or spiking mixture will then be added by piercing the septum and injecting the mixture below the methanol surface. Following any additions, the soil/methanol mixture will be agitated to fully wet the soil with the extracting solution. After allowing the soil/methanol mixture to settle so that a particulate free layer forms, a measured aliquot will be transferred to a second 40.0 ml VOC vial containing 25 ml of VOC-free reagent grade water. The second vial will then be needle sparged for 15 minutes under ambient laboratory temperatures using a purge gas flow rate of 200 ml/minute. The purged volatile compounds will then be trapped on a packed cartridge which will be held at 40°C to minimize carry-over of water.

2.6.5 Gas Samples

The thermal desorption tubes on which the samples are collected are placed in the thermal desorber and heated to $380 \pm 4^{\circ}\text{C}$ with a helium flow of 20 ml/min. at the same time that the GC temperature program is initiated and data acquisition started. The trapped materials are desorbed and carried through the heated transfer lines to the GC columns where separation occurs (Figure 4).

2.7 Calculations

2.7.1 Each analyte in the sample chromatogram is identified by comparing the retention time of the suspect peak to retention times generated by the calibration standards on the appropriate detector. When applicable, the relative response of the alternate detector to the analyte is determined. The relative response should agree to within 20% of the relative response determined from the standards.

2.7.2 Quantitation is usually performed on the detector which exhibits the greater response if all detectors respond to an analyte. In cases where greater specificity or precision would result, the analyst uses his/her professional judgement in determining the alternate detector.

2.7.3 The concentration of the unknowns is determined by using the calibration curve or by comparing the peak height or area of the unknowns to the peak height or area of the standards as follows for external standards:

External Standards:

$$C = (A/RF)(1/SA)$$

or $C = (A \times RF/10000)(1/SA)$

where C = concentration of the analyte in sample in $\mu\text{g/L}$

SA = sample amount in L or kg

RF = relative response factor

Internal Standards:

$$C_{\mu\text{g/l}} = \left(\frac{IS}{SA} \right) \left(\frac{RF_S A_S}{RF_{IS} A_{IS}} \right)$$

where C $\mu\text{g/L}$ = concentration of the component of interest present in the sample

SA = sample volume or mass (L or kg)

IS = the amount of the internal standard added to the samples

RF_S = response factor of component's determined by calibration

A_S = area count of the components in the sample analysis

RF_{IS} = response factor of the internal standard is

1 by definition

A_{IS} = the area of the internal standard in the sample
analysis run

2.7.4 The results for the unknown samples are reported in $\mu\text{g/L}$. The results are rounded off to the nearest 0.01 $\mu\text{g/L}$ or 2 significant figures.

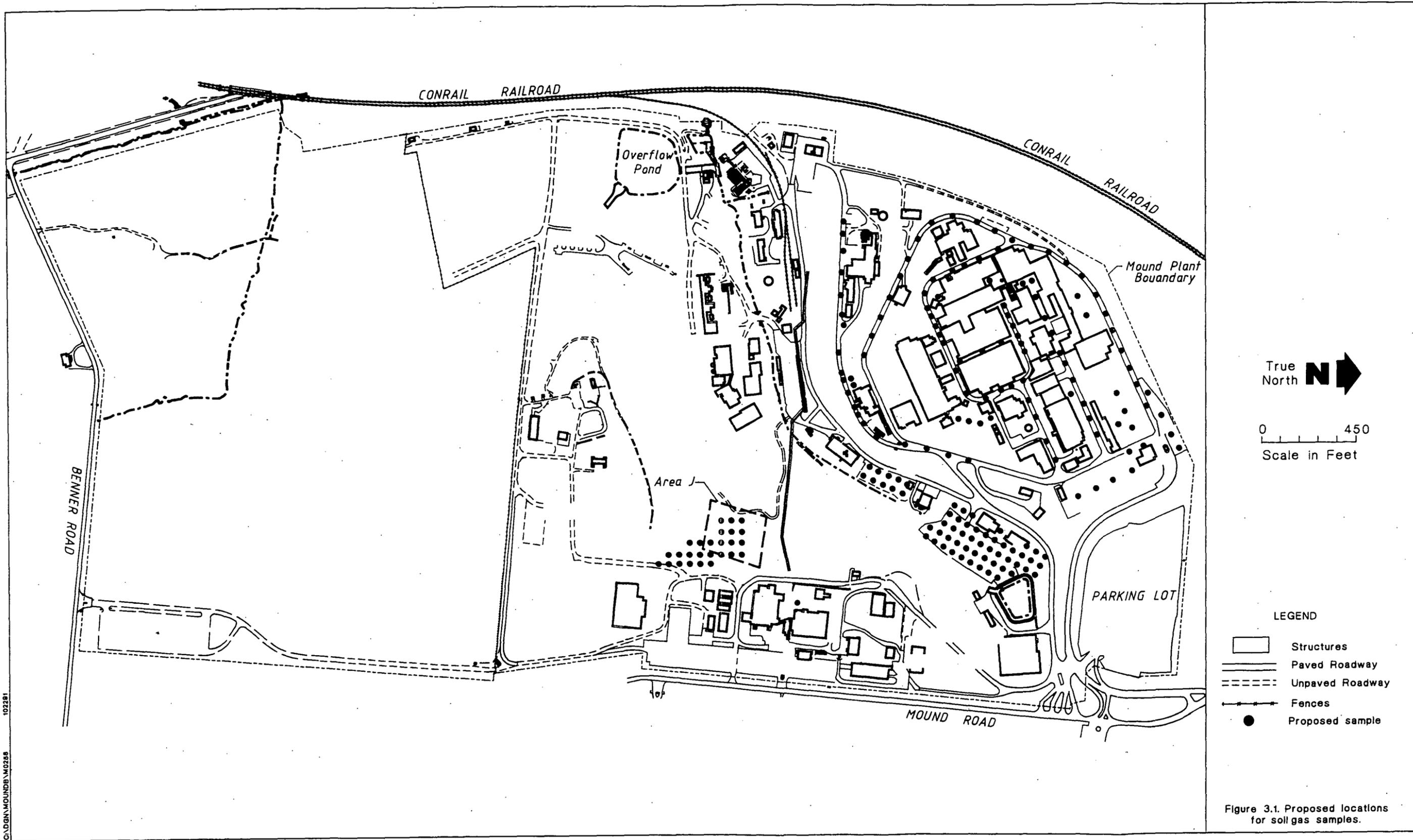


Figure 3.1. Proposed locations for soil gas samples.

C:\DGN\MOUND\M0285 102281