

DTIC FILE COPY

PHOTOGRAPH THIS SHEET

Best Available Copy

AD-A221 186

DTIC ACCESSION NUMBER

LEVEL

INVENTORY

ESL-TR-88-062

DOCUMENT IDENTIFICATION

SEPT 1989

DISTRIBUTION STATEMENT A

Approved for public release;
Distribution Unlimited

DISTRIBUTION STATEMENT

APPROVAL FOR	
NTIS	GRAMI
DTIC	TRAC
UNANNOUNCED JUSTIFICATION	
BY	
DISTRIBUTION/	
AVAILABILITY CODES	
DISTRIBUTION	AVAILABILITY AND/OR SPECIAL
0-1	

DISTRIBUTION STAMP

DTIC
ELECTE
MAY 07 1990
S E D

DATE ACCESSIONED

DATE RETURNED

80 05 04 137

DATE RECEIVED IN DTIC

REGISTERED OR CERTIFIED NUMBER

PHOTOGRAPH THIS SHEET AND RETURN TO DTIC-FDAC

AD-A221 186



ESL-TR-88-62

IN SITU SOIL DECONTAMINATION BY RADIO-FREQUENCY HEATING- FIELD TEST

H. DEV, J. ENK, G. STRESTY, J. BRIDGES,
D. DOWNEY

ILLINOIS INSTITUTE OF TECHNOLOGY
RESEARCH INSTITUTE
10 W. 35th ST
CHICAGO IL 60616

SEPTEMBER 1989

FINAL REPORT

JANUARY 1987—APRIL 1988

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED



AIR FORCE ENGINEERING & SERVICES CENTER
ENGINEERING & SERVICES LABORATORY
TYNDALL AIR FORCE BASE, FLORIDA 32403

20030211027

NOTICE

PLEASE DO NOT REQUEST COPIES OF THIS REPORT FROM
HQ AFESC/RD (ENGINEERING AND SERVICES LABORATORY).
ADDITIONAL COPIES MAY BE PURCHASED FROM:

NATIONAL TECHNICAL INFORMATION SERVICE
5285 PORT ROYAL ROAD
SPRINGFIELD, VIRGINIA 22161

FEDERAL GOVERNMENT AGENCIES AND THEIR CONTRACTORS
REGISTERED WITH DEFENSE TECHNICAL INFORMATION CENTER
SHOULD DIRECT REQUESTS FOR COPIES OF THIS REPORT TO:

DEFENSE TECHNICAL INFORMATION CENTER
CAMERON STATION
ALEXANDRIA, VIRGINIA 22314

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION:		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for Public Release Distribution Unlimited	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			
4. PERFORMING ORGANIZATION REPORT NUMBER(S) IITRI Project NO. C06666, C06676		5. MONITORING ORGANIZATION REPORT NUMBER(S) ESL-TR-88-62	
5a. NAME OF PERFORMING ORGANIZATION Illinois Institute of Technology Research Institute	5b. OFFICE SYMBOL (if applicable)	7a. NAME OF MONITORING ORGANIZATION USAF Space Division	
6a. ADDRESS (City, State, and ZIP Code) 10 W. 35th St. Chicago IL 60616		7b. ADDRESS (City, State, and ZIP Code) P. O. Box 92960 Worldwide Postal Center Los Angeles CA 90009	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION HQ AFESC	8b. OFFICE SYMBOL (if applicable) RDV	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER USAF/SD Contract No. F04701-86-C-0002	
6c. ADDRESS (City, State, and ZIP Code) Tyndall AFB FL 32403-6001		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO. 63	PROJECT NO. 2103
		TASK NO. 70	WORK UNIT ACCESSION NO. 99

11. TITLE (Include Security Classification)
In Situ Soil Decontamination by Radio-Frequency Heating-Field Test

12. PERSONAL AUTHOR(S)
H. Dev, J. Enk, G. Stresty, J. Bridges, D. Downey

13a. TYPE OF REPORT Final	13b. TIME COVERED FROM Jan 87 TO Apr 88	14. DATE OF REPORT (Year, Month, Day) September 1989	15. PAGE COUNT
------------------------------	--	---	----------------

16. SUPPLEMENTARY NOTATION
Availability of this report is specified on reverse of the front cover

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) Soil Decontamination, Radio-Frequency Heating Thermal Treatment, Fire Training Areas
FIELD	GROUP	SUB-GROUP	
09	03		
07	01		

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This report summarizes a successful pilot test of radio-frequency soil decontamination performed at an abandoned fire training area on Volk Field ANGB WI. The results of preliminary laboratory column studies are provided as well as a complete discussion of soil sampling and analytical methods. Approximately 500 cubic feet of soil was heated to a temperature of 150°C using radio-frequency energy. During the 12 days of heating, the hot vapors were collected from the soil surface and concentrated using a condenser and activated carbon. Final soil sampling showed excellent removal rates of fuel contaminants. Over 99 percent of the aromatic compounds, such as benzene, were removed from the soils. Less volatile aliphatics were reduced by over 95 percent. A complete summary of operating conditions and soil decontamination results are provided in this report.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ OTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL Mr. Doug Downey		22b. TELEPHONE (Include Area Code) (904) 283-2942	22c. OFFICE SYMBOL HQ AFESC/RDV

DD Form 1473, JUN 86

Previous editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE

SUMMARY

The radio-frequency (RF) soil decontamination process removes volatile hazardous waste materials through in situ heating of the soil and volatilization of the hazardous substances. The gases and water vapors generated by heating of the soil are recovered and collected from the heated zone and sent to an onsite gas cooling and treatment system. A concentrated condensate stream is produced containing water and the organic phases.

A field test was performed at the Volk Field Air National Guard Base (ANGB), Camp Douglas, WI, to prove the feasibility of the RF in situ soil decontamination process. The results of the field test show that 94 to 99 percent decontamination of a 500 ft³ block of soil was achieved during a 12-day treatment period.

The test site was located in a old fire-training area which had been used to conduct fire-training drills in simulated aircraft fires. It is estimated that approximately 50,000 gallons of unburned jet fuel remains in the sandy soil of the fire training area. The feasibility of the RF in situ decontamination process was demonstrated by heating a block of soil of dimension 6 feet by 12 feet by 7 feet to a temperature range of 150°-160°C. Analysis of numerous pre- and posttest soil samples has indicated that on the average 99 percent of the volatile aromatics and aliphatics had been removed from the 500 ft³ heated volume. On the average, 94 percent of semivolatile aliphatics and 99 percent of the semivolatile aromatics were also removed. Hexadecane, (b.p. 287.5°C) which was present in the soil, was analyzed to determine the removal of high boiling aliphatics; its concentration was reduced by 83 percent. These results show that substantial removal of high boiling contaminants can be achieved at temperatures significantly lower than their boiling point. This occurs due to the long residence time provided at lower temperatures and due to a steam distillation provided by the native moisture.

Migration of contaminants into and out of the heated zone was assessed by injecting a tracer into the soil and sampling for it in zones immediately outside the perimeter of the heated volume. HalonTM tracer, injected 4 feet outside the heated volume at a depth of 6 feet, was detected in the raw gases

leaving the heated zone, indicating that soil gases and liquids were moving into the heated zone from outside. A contaminant reduction of 70-76 percent was observed in the immediate area outside the heated zone. These results demonstrate there was no net migration of contaminant from the heated area to the surrounding soil.

Cost estimates were prepared for a full-scale system to treat fire training areas with typical dimensions of 96 feet by 96 feet to a depth of 8 feet. It is estimated that the treatment cost will vary between \$28 to \$60 per ton of soil. These estimates are substantially lower than the cost of soil remediation by excavation and incineration which could be as high as \$300 per ton.

The results of this study warrant optimization and full-scale demonstration of the technology so that it can be used on a commercial basis for the remediation of sites containing jet fuel, gasoline, and other similar contaminants. Full-scale testing is planned for FY 90.

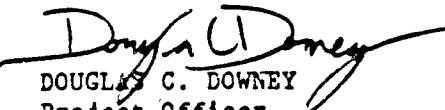
PREFACE

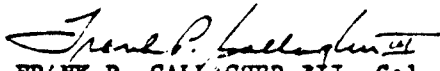
This report was prepared by the Illinois Institute of Technology Research Institute, 10 West 35th St, Chicago IL 60616-3799, under USAF Space Division Contract No. F04701-86-C-0002, for the Air Force Engineering and Services Center, Engineering and Services Laboratory (AFESC/EDVW), Tyndall AFB FL 32403-6001.

The report summarizes preliminary laboratory column studies and a pilot-scale field test of the radio frequency soil decontamination method. Laboratory testing began in Jan 87 and the field test was completed in Apr 1988.

This report has been reviewed by the Public Affairs (PA) office and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication.


DOUGLAS C. DOWNEY
Project Officer


FRANK P. GALLACHER III, Col, USAF, BSC
Director, Engineering and Services
Laboratory


F. THOMAS LUBOZYNSKI, MAJ, USAF, BSC
Chief, Environmental Engineering

TABLE OF CONTENTS

Section	Title	Page
I	INTRODUCTION.....	1
	A. OBJECTIVE.....	1
	B. BACKGROUND.....	1
	1. The Problem.....	1
	2. Radio Frequency Heating.....	2
	3. Prior Decontamination Feasibility Studies.....	3
	4. Process Cost Evaluation.....	4
	C. SCOPE.....	5
II	LABORATORY COLUMN TESTS.....	6
	A. BACKGROUND AND SUMMARY.....	6
	B. SOIL PREPARATION.....	7
	1. Preparation of Spiked Soil.....	7
	2. Preparation of Field Samples.....	9
	C. EXPERIMENTAL EQUIPMENT AND PROCEDURE.....	10
	D. PILOT-SCALE RESULTS.....	12
	1. Spiked-soil experiments with CBZ and TRCE.....	12
	2. Jet Fuel-Contaminated Samples.....	15
	E. CONCLUSIONS.....	22
	F. IMPLICATIONS FOR IN SITU TEST DESIGN.....	24
III	SITE CHARACTERISTICS.....	26
	A. SITE HISTORY.....	26
	B. SITE GEOLOGY.....	27
	C. SITE CONTAMINANTS.....	29
	D. SOIL DIELECTRIC PROPERTIES.....	29
IV	RF PROCESS DESIGN.....	38
	A. DESIGN OBJECTIVES.....	38
	B. DESIGN OF THE ELECTROMAGNETIC HEATING SYSTEM.....	40
	1. Electrode Array Design.....	40
	2. RF Transition Section.....	44
	3. Matching Network and Power Transmission System.....	44
	C. VAPOR CONTAINMENT AND HANDLING SYSTEM.....	48
	1. Vapor Barrier and Collection System.....	48
	2. Vapor Handling System.....	50
	D. TEMPERATURE MONITORING SYSTEM.....	52
	E. EMISSION MONITORING PLAN.....	54
	1. Monitoring of Electromagnetic Emissions.....	54
	2. Monitoring of Gaseous Emissions.....	56
	F. SITE LAYOUT.....	56
V	SOIL AND VAPOR SAMPLING.....	58
	A. SOIL SAMPLING.....	58
	1. Objectives.....	58

TABLE OF CONTENTS
(continued)

Section	Title	Page
	2. Soil Sampling Plan.....	58
	3. Composite Preparation and Soil Analysis.....	64
	4. Sampling and Analysis of Soil for EP-Toxicity.....	66
B.	VAPOR AND CONDENSATE MONITORING.....	67
	1. Gas and Vapor Stream Measurements.....	68
	2. Condensate Measurements.....	68
VI	FIELD TEST RESULTS.....	71
	A. FIELD TEST PROCEDURE AND CHRONOLGY.....	71
	B. SOIL TEMPERATURE DISTRIBUTION.....	72
	C. SOIL DECONTAMINATION RESULTS.....	80
	1. Removal of Volatile Contaminants.....	81
	2. Removal of Semivolatile Contaminants.....	88
	3. Removal from "Trench Area".....	93
	4. Tracer Experiments.....	93
	5. Contaminant Removal from 90-96 Inch Depth.....	95
	D. GAS AND CONDENSATE COLLECTION.....	95
	1. Raw Gas Analysis for Jet-Fuel Vapors.....	96
	2. Rate of Jet-Fuel Vapor Productions Vs. Collection.....	98
	3. Collection of Water and Hydrocarbons.....	98
	4. Analysis of Condensate for Total Organic Halides (TOX)...	98
	5. Gas Analysis by Draeger® Tubes.....	102
	6. Gas Flowrate Measurements.....	103
	E. RF SYSTEM PERFORMANCE.....	104
	F. RF EMISSIONS MONITORING.....	107
VII	CONCLUSIONS AND RECOMMENDATIONS.....	113
	A. CONCLUSIONS.....	113
	B. RECOMMENDATIONS.....	114
	REFERENCES.....	116
APPENDIX		
A.	ANALYSIS OF SOLVENTS AND JET FUEL IN SANDY SOIL.....	117
	A. NIELSEN-KRYGER STEAM DISTILLATION-EXTRACTION OF SOIL.....	119
	B. DETERMINATION OF TETRACHLOROETHYLENE RECOVERY EFFICIENCY....	121
	C. GC/ECD PROCEDURE FOR EXTRACT AND DISTILLATE ANALYSIS.....	122
	D. GC/MS ANALYSIS OF EXTRACTS AND DISTILLATES CONTAINING CHLOROBENZENE AND TETRACHLOROETHYLENE.....	131
	E. GC/MS PROCEDURES FOR EXTRACTS AND DISTILLATES CONTAINING JET FUEL.....	133
B.	PURGE AND TRAP PROCEDURE FOR VOLATILES.....	139
	A. SOIL EXTRACTION.....	140
	B. PURGE AND TRAP PROCEDURE.....	141

TABLE OF CONTENTS
(concluded)

Section	Title	Page
	C. GC/MS ANALYSIS AND CALIBRATION PROCEDURE.....	142
C.	GC/MS PROCEDURE FOR THE ANALYSIS OF SOIL EXTRACT FOR SEMI- VOLATILES.....	151
	A. GC/MS ANALYSIS AND CALIBRATION PROCEDURE.....	152
D.	EP-TOXICITY RESULTS FOR SOIL SAMPLES.....	157
E.	SOIL TEMPERATURE DISTRIBUTION DATA.....	165

LIST OF FIGURES

Figure	Title	Page
1	Pilot-scale Decontamination Setup for Solvents and Fuel.....	11
2	Time-temperature History for Pilot-scale Experiment 5.....	18
3	Time-temperature History for Pilot-scale Experiment 6.....	19
4	Chromatograms for Experiment 5.....	20
5	Chromatograms for Experiment 5.....	21
6	Soil Particle Size Distribution by Sieve Analysis.....	28
7	Relative Permittivity vs. Temperature for Volk Field Soil.....	34
8	Loss Tangent vs. Temperature for Volk Field Soil.....	35
9	Conductivity vs. Temperature for Volk Field Soil.....	36
10	Solid Wall, Totally Enclosed Guide; Solid-Wall Guide with Sidewall Removed; Discrete Electrode Realization.....	41
11	Electrode Array Design.....	43
12	RF Transition Section Layout Sketch.....	45
13	Design of the Installed RF Transition Section.....	46
14	Volk Field ANGB Fire Training Pit Demonstration Site Layout.....	49
15	Gas and Vapor Handling System.....	51
16	Thermocouple Layout.....	53
17	Pretest Soil Sample Hole Locations.....	59
18	Posttest Soil Sample Hole Locations.....	61
19	Vacuum-Lift Procedure for Posttest Soil Sampling.....	63
20	Raw Gas Sampling Train.....	69
21	Location of Temperature Measurement Boreholes..	73
22	Temperature Distribution Along Exciter Row, Depth 36 Inches.....	75
23	Temperature Distribution Along Plane DD at 9 Inch Depth.....	76

LIST OF FIGURES
(continued)

Figure	Title	Page
24	Temperature Distribution Along Midpoint (36 inches) of Plane DD...77	
25	Temperature Distribution Along Plane DD at 72 Inch Depth.....78	
26	Temperature Distribution at Ground Plant 3 (Row 3) Mid Point.....79	
27	Volatile Removal (Purge and Trap Data).....82	
28	Rate of Temperature Rise in Selected Exciter Electrodes.....83	
29	Rate of Temperature Rise in Selected Row 3 Electrodes.....84	
30	Before and After Concentration of Volatile Aliphatics.....86	
31	Before and After Concentration of Volatile Aromatics.....87	
32	Semivolatile Removal.....89	
33	Before and After Concentration of Semivolatile Aliphatics.....90	
34	Before and After Concentration of Hexadecane.....91	
35	Before and After Concentration of Semivolatile Aromatics.....92	
36	Removal of Volatiles from the "Trench" Area.....94	
37	Concentration of Jet Fuel Vapors in Raw Gas.....97	
38	Rate of Organics Production in Raw Gas and in Liquid Condensate...99	
39	Cumulative Water and Organic Collection vs. Time.....100	
40	Array Input Impedance; Actual vs. Predicted.....105	
41	RF Power Histogram.....108	
42	RFI Safety Measurement Locations.....110	
A-1	Nielson-Kryger Steam Distillation Apparatus.....120	
A-2	Tetrachloroethylene Recovery Efficiency by Steam Distillation....124	
A-3	GC Calibration Data for Tetrachloroethylene in iso-Octane.....127	

LIST OF FIGURES
(concluded)

Figure	Title	Page
A-4	GC Calibration Data for Tetrachloroethylene in Cyclohexane (high levels).....	129
A-5	GC Calibration Data for Tetrachloroethylene in Cyclohexane (low levels).....	130
A-6	Sample Calibration Curve.....	134
D-1	EP-Toxicity Test Report for "Trench" Area Sample A.....	159
D-2	EP-Toxicity Test Report for "Trench" Area Sample B.....	160
D-3	EP-Toxicity Test Report for "Trench" Area Sample C.....	161
D-4	EP-Toxicity Test Report for "Trench" Area Sample D.....	162
D-5	EP-Toxicity Test Report for "Trench" Area Sample E.....	163
E-1	Location of Temperature Measurement Boreholes.....	167
E-2	Temperature Distribution in Plane CC (Row 1) at 36-inch Depth....	168
E-3	Temperature Distribution Along Plane BB in Region 1 at 12-inch Depth.....	169
E-4	Temperature Distribution Along Exciter Row Depth 72 inches.....	170
E-5	Temperature Distribution Along Row 3 in Region 1 at 12-inch Depth.....	171
E-6	Temperature Distribution Along Row 3 in Region 1 at 72-inch Depth.....	172
E-7	Temperature Distribution Along Row 3 in Region 1 at 96-inch Depth.....	173
E-8	Temperature Distribution in Transverse Plane AA.....	174
E-9	Rate of Temperature Rise at the Center of the Heated Volume.....	175

LIST OF TABLES

Table	Title	Page
1	Pilot-Scale Experimental Conditions for Removal of CBZ and TRCE.....	13
2	Pilot-Scale Experimental Results (Spiked Soil).....	13
3	Residual Concentration of TRCE and CBZ as a Function of Soil Depth (Spiked Soil Exp.).....	16
4	Experimental Conditions for Removal of Jet Fuel.....	16
5	Comparison of Total Area Response.....	23
6	Pilot-Scale Decontamination of Soil Containing Jet Fuel (Treatment Temperature 150°-157°C).....	24
7	Description of Field Demonstration Test.....	39
8	Number and Location of Temperature Measurement Points.....	55
9	Emission Monitoring Plan.....	55
10	Summary of Soil Samples Taken.....	62
11	Target Compounds and Methods for Analysis.....	65
12	Summary of Internal, Surrogate, and Calibration Standards.....	67
13	Chronological Sequence of Field Test Events.....	71
14	Percent Relative Standard Deviation for Volatile Contaminants.....	85
15	Percent Relative Standard Deviation for Semivolatile Contaminants.....	93
16	Analysis of Total Organic Halides (TOX) in Condensed Phases.....	101
17	Power Consumption and Costs.....	107
18	RFI Safety Measurements Near/Far Field Applied Frequency = 6.78 MHz (ISM).....	111
19	Out-of-Band Electric Field Strength Near/Far Field Measurements Applied Frequency = 6.78 MHz (ISM) Applied Power = 35 kW.....	111

LIST OF TABLES
(concluded)

Table	Title	Page
A-1	Recovery Efficiency of Tetrachloroethylene from Spiked Soil.....	123
A-2	GC Operating Conditions.....	125
A-3	Results of Quality Control Sample Analysis.....	135
A-4	Average Response Factors for Calibration Compounds.....	136
A-5	Recovery Efficiencies for the Surrogate Standards Toluene-d ₈ and Decane-d ₂₂	137
B-1	Target Compounds for Pretest Purge and Trap Samples.....	143
B-2	Target Compounds for Posttest Purge and Trap Samples.....	144
B-3	Determination of Linear Response Range for the GC/MS System.....	145
B-4	Concentration of Calibration Standards Used for Pretest Soil Sample Analysis.....	146
B-5	Concentration of Calibration Standards Used for Posttest Soil Sample Analysis.....	147
B-6	Relative Response Factors for Pretest Purge and Trap Sample Analysis.....	148
B-7	Relative Response Factors for Posttest Purge and Trap Analysis.....	149
C-1	Calibration Standards for Pretest Soil Analysis for Semi- Volatiles.....	153
C-2	Calibration Standards for Posttest Soil Analysis for Semi- Volatiles.....	154
C-3	Average Response Factors for Pretest Soil Analysis for Semi- Volatiles.....	155
C-4	Average Response Factors for Posttest Soil Analysis for Semi- Volatiles.....	155
D-1	Results of EP-Toxicity Test (EPA Method 1310).....	158

SECTION I

INTRODUCTION

A. OBJECTIVE

An in situ soil decontamination field test was performed at the site of an old fire training pit located on Volk Air National Guard Base (ANGB), Camp Douglas, Wisconsin. The purpose of this test was to demonstrate the feasibility of the radio frequency in situ heating process for the decontamination of uncontrolled landfills and hazardous materials spill sites. Ninety-four percent of the aliphatic and 99 percent of the aromatic fraction present in the sandy soil of the fire training pit was removed. The results of this test have proven the feasibility of the in situ heating process.

Approximately 500 cubic feet of soil weighing 60,000 pounds was heated in situ for 7.5 days to a temperature of 150°C. The dimensions of the heated zone were 12 feet by 6 feet by 7 feet. The soil was maintained at this temperature for 4.5 days. The soil was decontaminated by the combined action of vaporization, steam stripping and steam distillation of the contaminants. A vapor barrier and collection system were used to collect and recover the gases and vapors for onsite treatment.

B. BACKGROUND

1. The Problem

Soils containing solvent and fuel contaminants are found at many Air Force installations. Typical examples are: tetrachloroethylene, dichloroethane, trichloroethylene, and various aromatic and aliphatic fractions of jet fuel. These materials boil at a temperature range of 80° to 230°C, and can be readily volatilized by increasing their temperature. These and other similar materials pose a continuous threat to the environment because the chlorinated hydrocarbons are extremely stable and degrade slowly in the environment. Chlorinated solvents and alkylbenzenes can migrate from the sites of original disposal and contaminate ground water resources.

There is increasing regulatory and public pressure for the cleanup of contaminated sites. Incineration of the excavated soil containing the contaminants is a proven process for the cleanup of such sites, however, it has several disadvantages: (1) Incineration is costly, (2) there is a risk of contaminant redispersion and personnel exposure during excavation, (3) there is a shortage of adequate incineration capacity which can treat large volumes of soil with little or no fuel value, and (4) incinerators produce large volumes of gases which require treatment before discharge.

At many contaminated sites the hazardous chemical species boil in the moderate temperature range of 80° to 300°C. For such materials, excavation and incineration of the soil at 2000°C represents overkill. A much more attractive alternative is to remove the chemicals from the soil in the vapor phase followed by condensation and ultimate disposal of a small quantity of the hazardous material by incineration or other suitable methods.

Other alternative in situ remediation processes are soil venting and bioremediation. The soil venting process can remove the more volatile compounds from soil as a mixture of vapors and air. This is achieved by drawing a vacuum in the contaminated zone through boreholes drilled in the soil. However, the concentration of the hazardous materials in the airstream is so small that it cannot be recovered economically for ultimate disposal. The in situ bioremediation processes often suffer from inefficient oxygen and nutrient delivery and undesired chemical reactions with compounds of the soil matrix.

2. Radio-Frequency Heating

The radio-frequency in situ heating method is a technique for rapid and uniform in situ heating of large volumes of soil. It can be used to increase the soil temperature to a range of 150° to 400°C, thus, volatilizing most of the hazardous compounds in the list defined in Section 101 (14) of Comprehensive Emergency Response Compensation and Liability Act of 1980 (CERCLA). The gases and vapor formed in the soil matrix can be recovered at the surface or through the electrodes used for the heating process. Because the process does not use heat transfer fluids or in situ combustion of fuels,

a concentrated gas stream can be recovered and the contaminants easily separated by condensation and carbon adsorption. Because the soil is uniformly heated, a more uniform decontamination of the heated zone can be achieved than could be obtained by applying a vacuum through a pattern of multiple boreholes. Another advantage of the in situ heating method is that it conditions the soil by increasing the permeability to gas flow by removing the soil moisture from the pores. This allows for uniform collection of the volatilized contaminants from the surface or from the electrode boreholes.

Radio-frequency heating is performed by the application of electromagnetic energy in the radio frequency band. The energy is delivered by electrodes implaced in holes drilled through the soil. The mechanism of heat generation is similar to that of a microwave oven and it does not rely on the thermal properties of the soil matrix. The power source for the process is a modified radio transmitter. The frequency of the applied power is selected from the industrial, scientific and medical (ISM) band, specifically set aside by the Federal Communications Commission (FCC). The exact frequency of operation is selected after evaluation of the dielectric properties of the soil matrix and the size of the area requiring treatment.

The RF heating process was originally developed for the recovery of hydrocarbonaceous resources from deposits of oil shale and tar sands. Several in situ heating experiments were performed (Reference 1) on such deposits ranging in size from 35 to 660 cubic feet, in which the resource was heated to an average temperature of 200° to 400°C. These field tests conclusively established the feasibility of in situ heating for various types of soil matrix.

3. Prior Decontamination Feasibility Studies

The feasibility of soil decontamination by thermal mechanisms such as vaporization, steam distillation and steam stripping was previously (Reference 2) established through laboratory and pilot-scale experiments. In the laboratory-scale experiments, soil spiked with tetrachloroethylene (TRCE) and chlorobenzene (CBZ) was decontaminated by treatment in the temperature range of 90° to 130° C for 4 hours. It was shown that 90 to 99 percent removal of

the contaminants is feasible. Mass balance closure of 75 to 104 percent was demonstrated in these studies.

In the pilot-scale studies a tall column of soil containing jet fuel was placed in a 7-foot tall, 2-inch diameter stainless steel pipe. It was shown that 90 to 99 percent removal of total aromatics and total nonaromatics fraction is feasible by treatment at 150° to 160°C for 14 to 40 hrs. The tall column studies were designed to show that the vaporized contaminants can be recovered from the upper surface of the soil bed. It was also shown in these experiments that higher boiling components such as pentadecane (boiling point 270.5°C) can be removed at lower temperature of 150° to 160°C provided a steam sweep can be established in the soil. In the pilot scale, the sweep was established by injecting preheated water at the base of the hot soil column. In this way, at least 94 percent of the high-boiling pentadecane was removed from the soil. In the field, such a sweep can be established by injecting water through the electrode holes.

The pilot studies also showed that a residual concentration profile does not develop along the length of the soil column after treatment.

4. Process Cost Evaluation

Preliminary cost studies have been performed (Reference 2) on the radio-frequency heating process. The cost of in situ treatment of a contaminated site was developed through a conceptual design of a treatment system. The design was used to estimate the capital, and operating cost of the process. It was assumed that a contaminated site will be cleaned up by the successive treatment of modules, each of 9200 square feet. The treatment depth was assumed to be 8 feet. The estimated treatment cost varies between \$1.5 to \$2.9 per 100 pounds of treated soil, because of the amount of native moisture present in the soil and the exact temperature of treatment. The study assumed a moisture and treatment temperature range of 5 to 20 percent, and 100° to 250°C, respectively. The capital cost for a 1.0-MW RF treatment system was estimated to be \$1.6 million and for a system that could treat 14 modules per year.

In another study (Reference 3), the cost of RF treatment of a site was compared with the cost of excavation and incineration in an approved facility. It was estimated the incineration process would be two to four times more expensive than the RF in situ treatment process.

C. SCOPE

The purpose of this phase of RF technology development was to field test a pilot unit on a contaminated Air Force site and carefully evaluate the soil decontamination process. A test site was selected at Volk Field ANGB, WI where 25 years of fire fighter training exercises had contaminated soils with JP-4, waste oils, and improperly disposed of solvents.

The pilot test was conducted on a 500 cubic feet soil block inside the old fire-training area. Following careful sampling of initial soil contamination, electrodes were placed in the soil and a vapor recovery system was set up over the test bed. The system was energized and soils were heated over a 12-day period. Contaminated soil vapors were condensed and organics separated from soil moisture. Analysis of over 80 soil samples in the treatment zone indicated that on the average 99 percent of the volatile aromatic and aliphatic contaminants were removed.

Following the test, a careful accounting of energy and operation costs was made to project a full-scale treatment cost of less than \$60 per ton of soil decontaminated. This successful pilot test also identified several potential improvements which will be incorporated in a new full-scale system and field tested in 1989.

SECTION II

LABORATORY COLUMN TESTS

A. BACKGROUND AND SUMMARY

A previous USAF/EPA-funded project (Reference 2), established the feasibility of thermal decontamination of soil containing tetrachloroethylene and chlorobenzene. These experiments were performed in the bench scale, using 75 grams of spiked sandy soil. The soil bed was no more than 2 to 3 inches deep. This made it relatively easy for the contaminant vapors to move up through the soil bed for recovery in the condenser. However, in the field, the vapors would have to move up to the surface through the thickness of the heated zone before collection at the surface.

The purpose of performing the laboratory column tests (pilot-scale tests) was, therefore, to demonstrate that at least 95 percent of the contaminants can be removed from tall soil beds by contaminant vaporization and recovery at the top surface. A 6-foot soil bed was used. This height was selected based on the preliminary designs of a field test at the Volk Air National Guard Base (ANGB), Camp Douglas, Wisconsin, in which it was planned to heat soil to a depth of 6 to 8 feet. Pilot-scale experiments were performed on soil spiked with contaminants, as well as that obtained from Volk ANGB. The field soil was contaminated with jet fuel. The spiked soil was prepared by adding solutions of CBZ and TRCE to clean sandy soil which was also obtained from Volk ANGB.

Results of the pilot-scale experiments using spiked contaminants have shown that it is feasible to remove greater than 99 percent of the contaminants from the soil. These experiments were performed on soil containing 0.5 to 36 ppm of TRCE and CBZ, in a temperature range of 130° to 160°C. It was also shown that the contaminants can be recovered at the top surface of the soil bed and that a residual concentration profile does not develop along the length of the heated 6-foot tall soil column.

Soil containing jet fuel (JP-4) was decontaminated by treatment in a temperature range of 152°-157°C for a period of 14 to 40 hours. Jet fuel has

a normal boiling point of 80°-230°C, but 90 to 99 percent decontamination was demonstrated in the treatment temperature range mentioned above. It was shown that high boilers such as pentadecane can be recovered more efficiently by providing a steam sweep created by water injection at the base of the heated bed. Without water injection, 75 percent removal of pentadecane was observed, but with water injection greater than 94 percent removal was achieved.

The results of the pilot-scale experiments have demonstrated the feasibility of in situ treatment of sandy soils containing fuels and solvents. A design was prepared for an in situ decontamination field test at Volk ANG, WI. In this design provision was made for injection of water into the heated soil to assist in the removal of heavy boilers, if required. The soil preparation methods, experimental procedures and results for the pilot-scale experiments are discussed in the following subsections.

B. SOIL PREPARATION

As mentioned above, experiments were performed by spiking clean soil as well as on contaminated soil containing jet fuel. The purpose of soil preparation was to adjust soil moisture content and to spike it with 10 ppm of each of TRCE and CBZ. No soil preparation was performed for experiments in which contaminated soil from the field was used.

1. Preparation of Spiked Soil

Four experiments were performed on spiked soil. In each experiment, 3.5 to 4 kilograms of soil were used. It was found that large quantities of soil could not be reliably spiked at 10 ppm level because of high volatility of the contaminants, which leads to very high contaminant loss. Evidence of this was provided through measurement of initial soil concentration and inability to close mass balance. It is suspected that contaminant loss was caused by vaporization at each of several material handling steps necessary to make the spiked soil. Two different methods were used to prepare the soil. In the first method, four 1-kilogram batches of soil were separately spiked, then mixed together. In the second method, one 5-kilogram batch was prepared for each experiment. In either case, the observed initial concentration of the spikes in soil ranged from 0 to 106.6 percent of the desired initial concentration level.

a. Soil Preparation by Method I

Four separate 1-kilogram batches of soil were made for each experiment. The moisture content of the batch was adjusted first, by measuring the initial moisture content of soil, by adding the required amount of hydrocarbon-free water to give approximately 5 percent moisture and by confirming the moisture content by reanalyzing samples obtained from each batch.

The contaminants were spiked after soil moisture content was adjusted. The soil was spiked with a known volume of a gravimetrically prepared solution of CBZ and TRCE in hexane. The volume of spiking solution used was adjusted to give an initial concentration of 10 ppm of each of the two contaminants.

Each time the soil was spiked with water or the contaminants, it was tumbled to homogenize the soil. A sealed glass or Teflon® jar containing approximately 1 kilogram of soil was placed horizontally on a ball-mill roller. Each batch was tumbled for a period of 2 hours. This method required the separate preparation of four batches of soil for each experiment. When this method was used, all the 4 batches were combined in a Teflon® bag and further mixed by tossing the soil inside the bag. The mixed soil was then loaded into the reactor. Two samples of soil were removed from the bag to determine the initial concentration of the contaminants. This method of soil preparation was used for Experiment 1 in which glass tumbling jars were used and for Experiment 2 which used Teflon® tumbling jars.

Several problems were encountered during the preparation of soil with the above method. These are:

- The soil forms a cake on the walls of the glass jar and does not tumble properly, even when the jar is hit with a mallet. This requires longer tumbling time to homogenize. This problem was solved by using the Teflon® jar.
- Because of considerable loss of the contaminant spike during the various preparation steps, the actual initial concentration of the soil was only 5 percent of the expected concentration based on the concentration of the spiking solution. This problem persisted even in the Teflon® jar.

b. Soil Preparation by Method II

To resolve the second problem, the soil mixing procedure was changed so that only one 5-kilogram batch of soil was prepared per experiment instead of four 1-kilogram batches per experiment. It was expected that by reducing the number of steps requiring transferring soil from one container to another, volatilization losses would be minimized. The 5-kilogram batches were prepared in a stainless steel V-blender. To decrease contaminant volatilization losses even further, the spiking solution solvent was changed from hexane to the higher boiling decane. And finally, the initial spiking levels were increased from 10 ppm to 50 ppm for each of the two contaminants. The modified method was used for Experiments 3 and 4.

The method of sampling the soil was also changed for Experiments 3 and 4. For Experiments 1 and 2, the samples were obtained from the Teflon® bag before loading the reactor. For Experiments 3 and 4, the samples were obtained from the funnel used for pouring the soil into the reactor. Four grab samples were taken at different times from the funnel as the soil was being packed. The four samples were combined in a sealed jar, tumbled and then split into two aliquots of 75 gram each. These two aliquots were analyzed to determine the initial soil concentration.

It was found that even with the new equipment and procedures, extensive loss of the spiked contaminants occurred as determined by an analysis of the soil samples. Because of the difficulties in preparing CBZ and TRCE spiked soil columns, a decision was made to continue column testing with soils from an actual contaminated site.

2. Preparation of Field Samples

Soil containing jet fuel was obtained from the Fire Training Pit located at Volk AFB. Soil was obtained by drilling holes through the contaminated zone with a bucket-auger. The samples were stored in 0.25-gallon glass jars with a Teflon®-lined screw cap. All samples were kept in an ice box until use. Each jar had approximately 1.0-1.5 kilogram of soil.

The experiments were performed by packing 3.5-4.0 kilogram of contaminated soil. As a result, more than one jar was needed to pack the reactor. Before packing the reactor, samples of soil were taken from each jar. Soil samples were obtained by inserting a glass tube all the way into the soil. The soil contained in the tube was transferred to a sealed Teflon® jar. All samples obtained from all jars used in each experiment were combined in the Teflon® jar and tumbled on a ball-mill roller. From the tumbled sample, two 150-gram aliquots were obtained for determination of initial soil concentration by the Nielson-Kryger steam distillation method.

C. EXPERIMENTAL EQUIPMENT AND PROCEDURE

The pilot-scale experiments were performed by packing 3.5-4.0 kilogram of contaminated soil in a stainless steel pipe reactor of diameter 1.5 inches and length 7 feet. External heating tapes, thermocouples, and insulation were added. The top of the reactor was connected to a glass water-cooled condenser equipped with a condensate recovery leg as shown in Figure 1. The condensate was collected in a chilled receiver. A Tenax® bed was attached to the gas outlet port of the condenser to trap any uncondensed vapors.

Two temperature controllers were used to control the temperature of the reactor as well as that of the vapor line connecting to the condenser.

In most experiments, water was injected at the base of the soil column. The purpose of water injection is to simulate a steam sweep through the soil column. In the field, a steam sweep will be established in the soil due to vaporization of native moisture in the heated zone. The sweep will be maintained due to intrusion of water from the surrounding unheated soil. Before water injection, the receiver was replaced with a new empty receiver. In one experiment, the soil was maintained at two different temperatures before steam injection. In this case, the distillate receiver was changed each time the temperature was changed and also before steam injection. This allowed separate quantification of the incremental amount of contaminants condensed as a result of changes in the operating conditions.

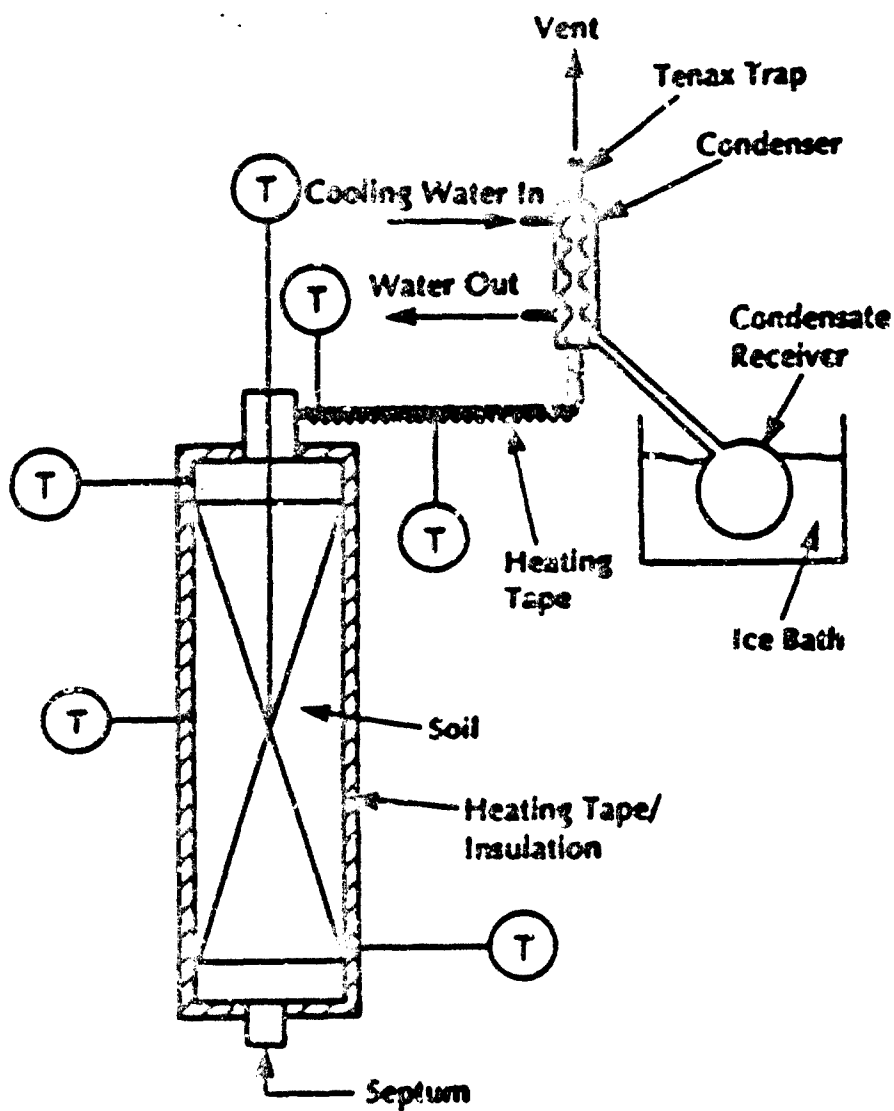


Figure 1. Pilot-scale Soil Decontamination Setup for Solvents and Fuel.

At the end of the experiment, the reactor was allowed to cool down before disconnecting the condenser. During the cool-down period, the vapor interconnect lines were kept heated. The condenser and the vapor lines were disconnected when the soil had returned to room temperature. The vapor lines, the interior of the condenser, the Tenax® trap, and the distillate line were thoroughly rinsed with hexane. These rinsates were combined with the distillate from the last receiver, which (in all the experiments) contained condensate recovered after water injection.

The cooled soil was removed from the reactor over a long mylar sheet. An attempt was made to obtain the concentration as a function of depth. This was done by moving the inclined open reactor over the mylar sheet. The direction of reactor movement was opposite to the direction of soil motion. In this fashion, a bead of soil was laid out over the mylar sheet. The length of the bead was measured and 75 gram samples were taken from the two extreme ends and the middle of the bead, representing the top, bottom, and middle of the packed soil. All the remaining soil was placed in a sealed glass jar, tumbled, and sampled to obtain an "average" sample for the entire bead. All the removal calculations are based on the analysis of the "average" soil sample.

D. PILOT-SCALE RESULTS

Results of pilot-scale experiments performed with soil spiked with CBZ and TRCE are given in the section below. Results of experiments performed with soil containing jet fuel are described on Page 15. Details of analytical procedures used for the analysis of contaminants in soil are given in Appendix A.

1. Spiked-Soil Experiments with CBZ and TRCE

The experimental conditions for four spiked-soil experiments are presented in Table 1. The results of Experiments 2, 3, and 4 are summarized in Table 2. Experiment 1 did not yield good results because the initial concentration of the TRCE was only 0.17 ppm and CBZ was below detection limit. This is probably related to the soil preparation problems described in a previous section. In Experiment 2, the initial TRCE concentration in soil was 0.57 ppm. Due to an oversight, CBZ was not spiked in this soil. The soil was

TABLE 1. PILOT-SCALE EXPERIMENTAL CONDITIONS FOR REMOVAL OF CBZ AND TRCE

Exp. #	Wt. of Soil, gm	Initial Concentration, µg/gm CBZ	Temperature, °C	Elapsed Time Before Water Injection, hrs	Elapsed Time From Water Injection to Shut-down, hrs	Water Injected, ml
1	3623.0	0.17	a.d.*	130	100.68	6.42
2	3654.0	0.36	a.p.**	137	49.65	7.07
3	3458.4	34.43	36.4	159	18.07	21.51
4	3433.0	9.3	10.66	128	15.67	16.4
5				153	30.42	15.92
						20

*a.d. = Not detected.

**a.p. = Not present.

TABLE 2. PILOT-SCALE EXPERIMENTAL RESULTS (SPINED SOIL)

Exp. #	Initial Wt. in Soil, (µg) CBZ	Distillate Before		Distillate After		Wt. Remaining in Soil (µg)		Removed from Soil, g		Mass Balance	
		Water Injection (µg)		Water Injection (µg)		in Soil (µg)		Soil, g		f	
		TRCE	CBZ	TRCE	CBZ	TRCE	CBZ	TRCE	CBZ	TRCE	CBZ
1	701.24	a.d.	15.43	a.d.	a.d.	NA	NA				
2	2106.2	a.p.	2607.3	a.p.	a.p.	a.d.	a.d.	100	124		
3	124,106	131,131	92,725	86,675	272.3	139.3	a.d.	a.d.	100	75.05	66.45
4	33,951.56	40,341.44	78,875	92,000							
		11	9.75	61	63.75	206.45	641.1	99.4	98.4	220.2	240.98

a.d. = Not detected.

a.p. = Not present.

NA = Not applicable.

heated to 137°C from room temperature at a rate of 4.4°C/hr. It was maintained at 137°C for a period of 50 hours. At the end of this time period, approximately 2600 microgram of TRCE were recovered as distillate. The distillate receiver was then replaced prior to water injection. Approximately 38 milliliters of water were injected. No additional TRCE was collected in the second distillate receiver. Analysis of soil taken from the reactor at end of the experiment showed that the concentration of TRCE had been reduced below the detectable limits.

In Experiments 3 and 4, the new method of soil preparation was utilized, using the stainless steel "V-blender." The target initial level of spiking was 50 ppm each of TRCE and CBZ. The actual initial concentrations in Experiment 3 were: TRCE 34.5 ppm and CBZ 36.4 ppm. In Experiment 3, the soil was heated to 151°C at a rate of 19°C/hr. The soil was maintained at 151°C overnight for a period of 18 hrs. At that time, it was observed that the temperature at the top and bottom of the reactor was approximately 30°C cooler than the middle. To increase the bottom and top temperatures, the temperature of the entire bed had to be raised to bring the temperature inside the center of the bed to 167°C. Water injection was begun as soon as the center temperature reached 167°C. Approximately 18 milliliter of water were injected. The amount of TRCE recovered as distillate prior to steam injection was 92,725 micrograms and the amount of CBZ recovered was 86,675 micrograms. After steam injection, the amount of TRCE and CBZ recovered was 272 and 139 microgram, respectively. The analysis of soil from the reactor after the experiment showed that the concentration of TRCE and CBZ had been reduced below the detectable limits. These results are summarized in Table 2.

In Experiment 4, the initial concentration of soil was 9.5 ppm TRCE and 10.7 ppm CBZ based on an analysis of two initial soil samples. The soil was initially heated to 123°-131°C at a rate of 17.1°C/hours. It was maintained in this range for a period of 15.7 hours. At the end of this time, the micrograms of CBZ. The temperature was raised to 153°C and maintained for a period of 30.4 hours. At the end of this time, only 11 micrograms of TRCE and 9.75 micrograms of CBZ were collected. This shows that there is no need to heat the soil to 150°C for these contaminants.

Approximately 20 milliliter of hydrocarbon-free water were injected, and the soil was kept at 153°C for 16 hours after steam injection. After steam injection, an additional 61 micrograms of TRCE and 64 micrograms of CBZ were collected in the distillate receiver. The soil analysis after the treatment was 0.06 ppm TRCE and 0.19 ppm CBZ.

Samples of soil were taken by emptying the reactor over a mylar sheet and forming a long bead of soil. The samples obtained from the two ends of the bed and from the middle were analyzed separately to estimate whether a vertical concentration profile had developed in the soil. Three such samples were obtained from all four experiments. The results of these samples are shown in Table 3. The conclusion is that a vertical residual concentration profile does not develop in the soil bed.

2. Jet Fuel-Contaminated Samples

Two experiments (5 and 6) were performed in the pilot-scale with soil contaminated with jet fuel. This soil was obtained from the Fire Training Pit located at Volk AHSB. The experimental conditions are summarized in Table 4.

In Experiment 5, the soil was heated to 157°C from room temperature at an average rate of 13.2°C/hr. It was maintained at 157°C for 14.4 hours. The time-temperature history for this experiment is shown in Figure 2. The experimental conditions were summarized in Table 4 (page 16).

In Experiment 6, the soil was heated to 152°C from room temperature at an average rate of 21.6°C/hr. The time-temperature history of this experiment is shown in Figure 3. The soil was maintained at 152°C for 17.4 hours. At the end of this time period, the distillate receiver was replaced to start the water injection. Approximately 54 milliliter of water were injected. The water injection was done over a period of 1.6 hours. After the water injection, the reactor was maintained at 157°C for 23 hours.

At the end of each experiment, the reactor was allowed to cool before disconnecting the condenser. During the cooling period, the vapor interconnect lines were kept heated. The condenser and the vapor line were disconnected when the soil had returned to room temperature. The vapor lines, the interior

TABLE 3. RESIDUAL CONCENTRATION OF TRCE AND CBZ AS A FUNCTION OF SOIL DEPTH (SPIKED SOIL EXP.)

Exp. #	Concentration in Spent Soil - Top		Concentration in Spent Soil - Middle		Concentration in Spent Soil - Bottom		Average Concentration in Spent Soil	
	TRCE	CBZ	TRCE	CBZ	TRCE	CBZ	TRCE	CBZ
1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2	n.d.	-	-	n.d.	n.d.	n.d.	n.d.	n.d.
3	n.d.	0.03 ^a	0.04	0.1	0.04	0.09	n.d.	n.d.
4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.06	0.18

n.d. = Not detected.

a = At detection limit.

TABLE 4. EXPERIMENTAL CONDITIONS FOR REMOVAL OF JET FUEL

Exp. No.	Weight of Soil, gm	Temp., °C	Elapsed Time		Water Injection, ml
			Before Injection, hrs	After Injection, hrs	
5	3068.0	157	14.4	0.0	0.0
6	4032.0	152	17.4	0.0	0.0
6	4022.0	157	0.0	23.0	54

of the condenser, the Tenax® trap, and the distillate line were thoroughly rinsed with hexane. In Experiment 5, the rinsates were combined with the distillate from the receiver. However, in Experiment 6, three distillate samples were prepared. These were: the distillate from the first receiver before water injection, the distillate from the second receiver after water injection, and finally, the rinsate of the vapor lines, the interior of the condenser, the Tenax® trap, and the distillate line.

Samples of treated soil were prepared in a similar way to that of Experiments 1 through 4, described earlier.

Qualitative and quantitative results of decontamination experiments performed on soil containing jet fuel are discussed below. In the subsection on qualitative results, the chromatograms of soil extract analysis before and after treatment are compared. In the section on quantitative results, analyses of soil for specific target compounds, total aromatics, and non-aromatics are presented.

a. Qualitative

The mass chromatograms for the analysis of initial and treated soil samples for Experiment 5 are shown in Figures 4(a) and 4(b). The X-axis in these figures starts at a time of 10 minutes. The order of elution of the various peaks is based on the normal boiling points of the various compounds. Thus, it is estimated that at 30 minutes, compounds with boiling point of 190°C should be eluting. Thirty-five minutes represents 220°C.

Figures 4(a) and 4(b) show that essentially complete cleanup (to detection limit levels) was achieved for all species eluting between 10 to 27 minutes. The large peak in Figure 4b at, approximately 26.5 minutes, is a standard.

Similar chromatograms for Experiment 6 are presented in Figures 5(a) and 5(b). In Experiment 6, water was injected and the total soak duration was longer, compared to that of Experiment 5. In Figure 5(b), the analysis of the treated soil is shown. The first five major peaks, counting from the origin, are standards.

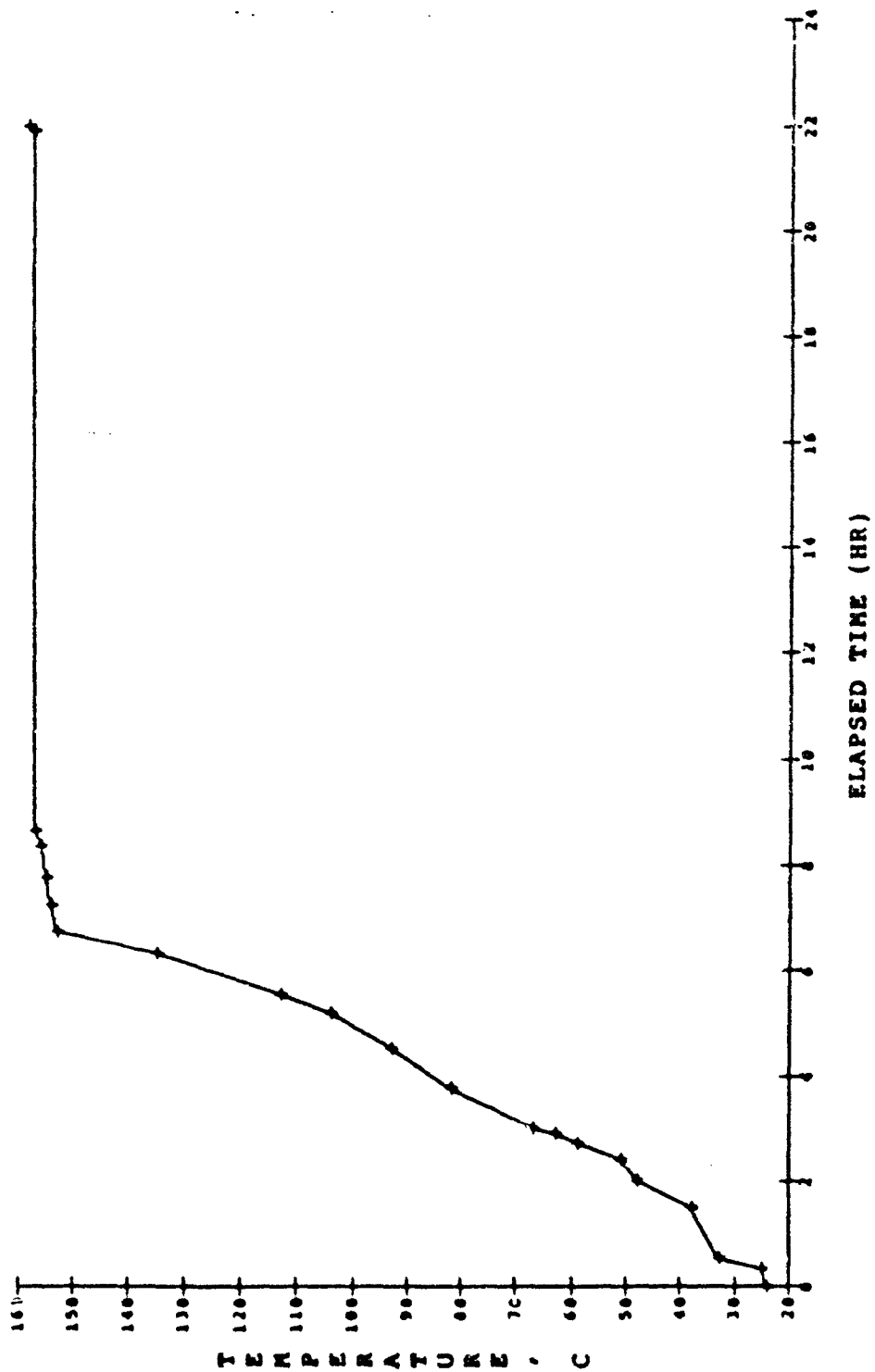


Figure 2. Time-Temperature History for Pilot-Scale Experiment 5.

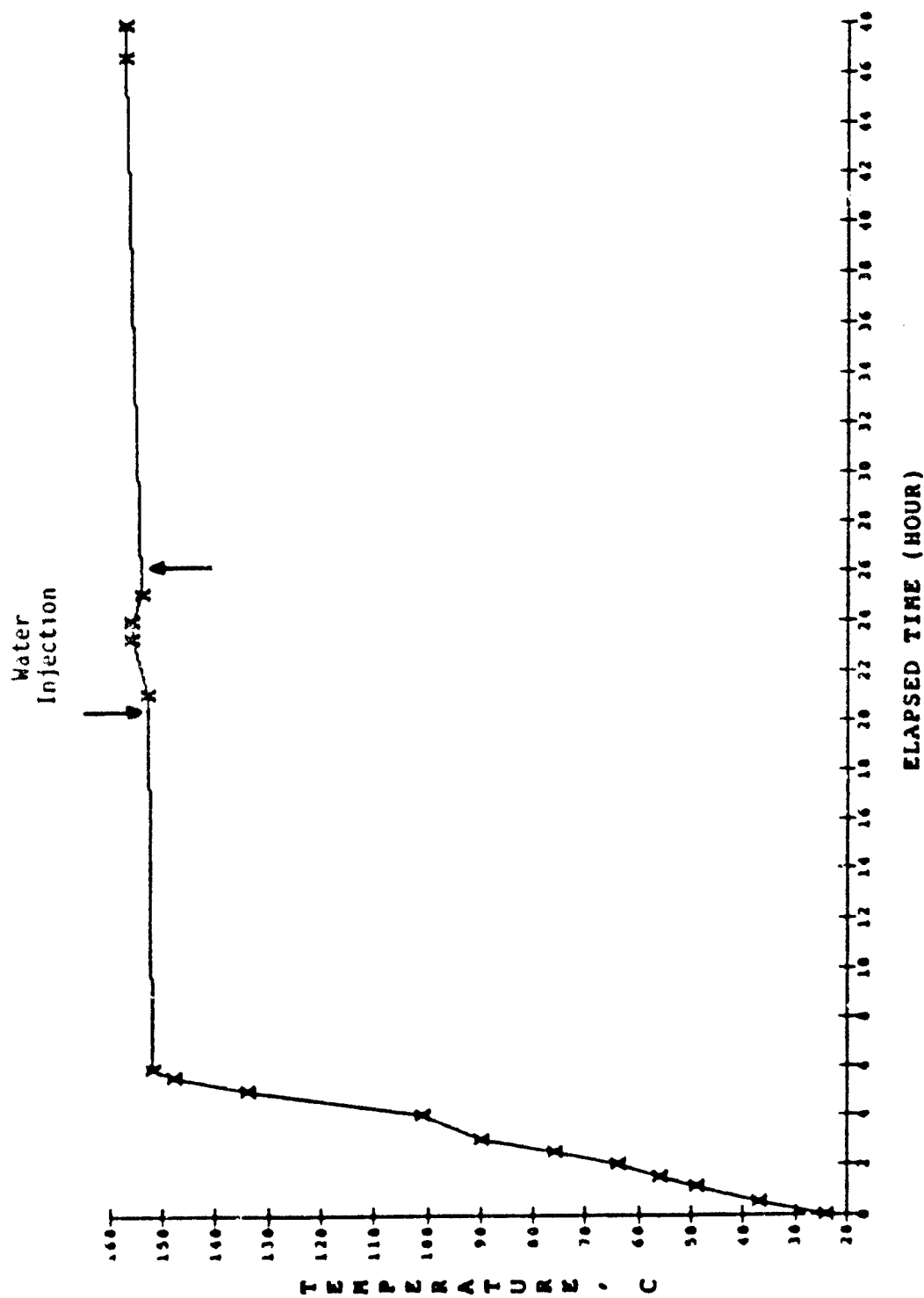


Figure 3. Time-Temperature History for Pilot-Scale Experiment 6.

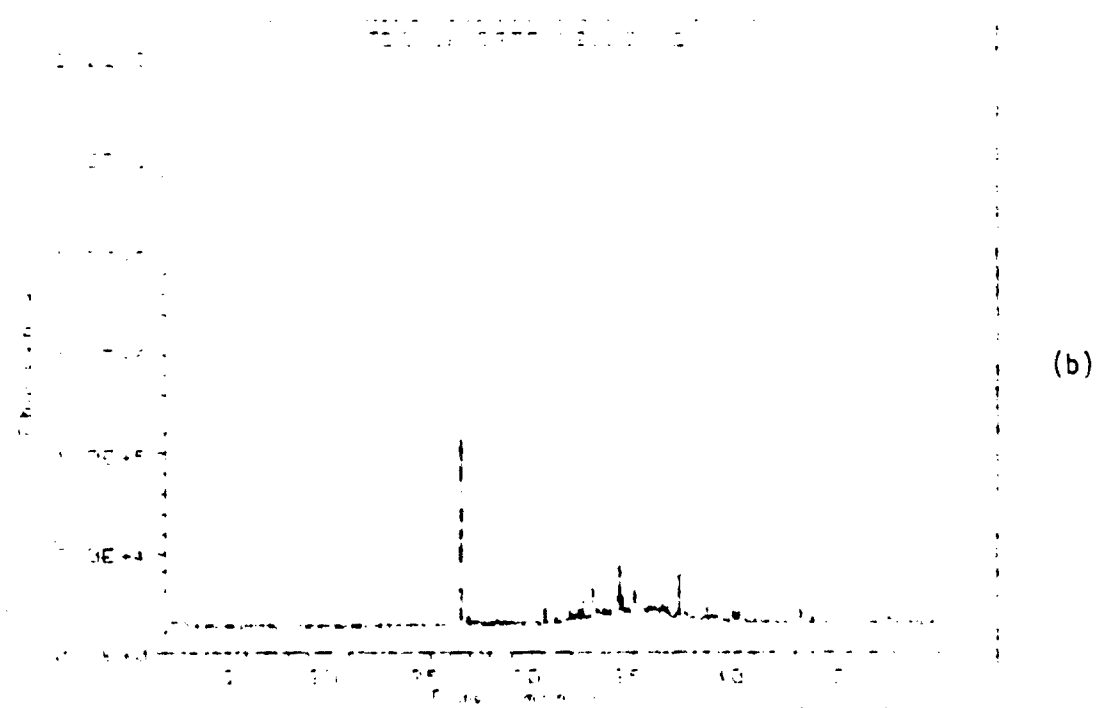
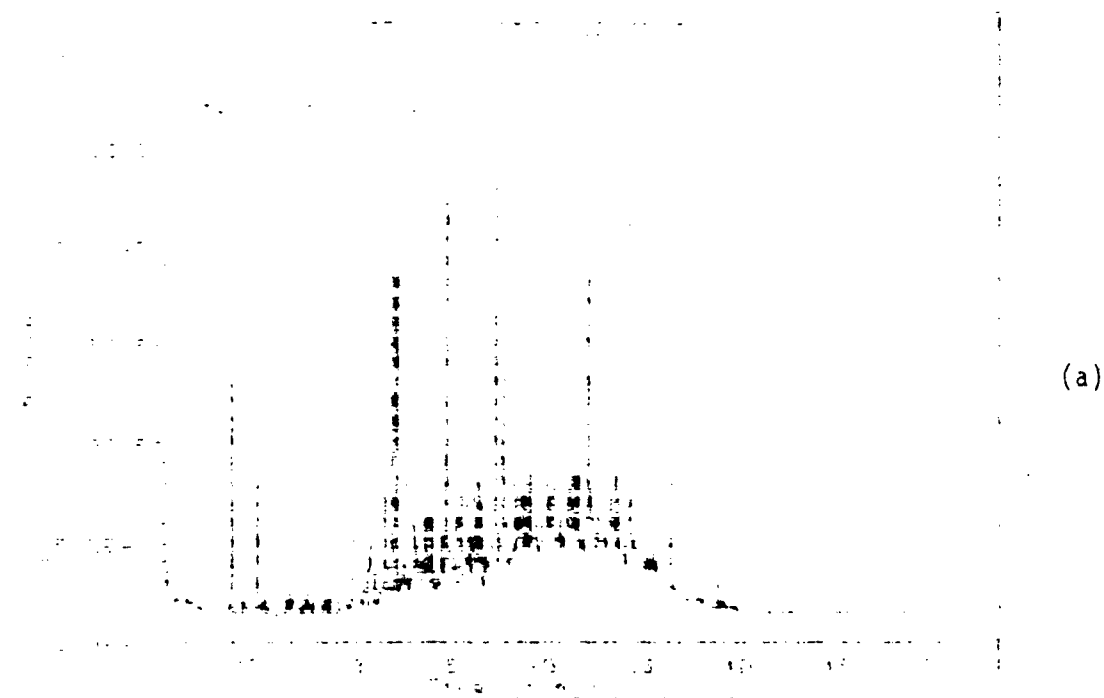


Figure 4. Chromatograms for Experiment 5. (a) Analysis of Initial Soil; (b) Soil Analysis After Treatment.

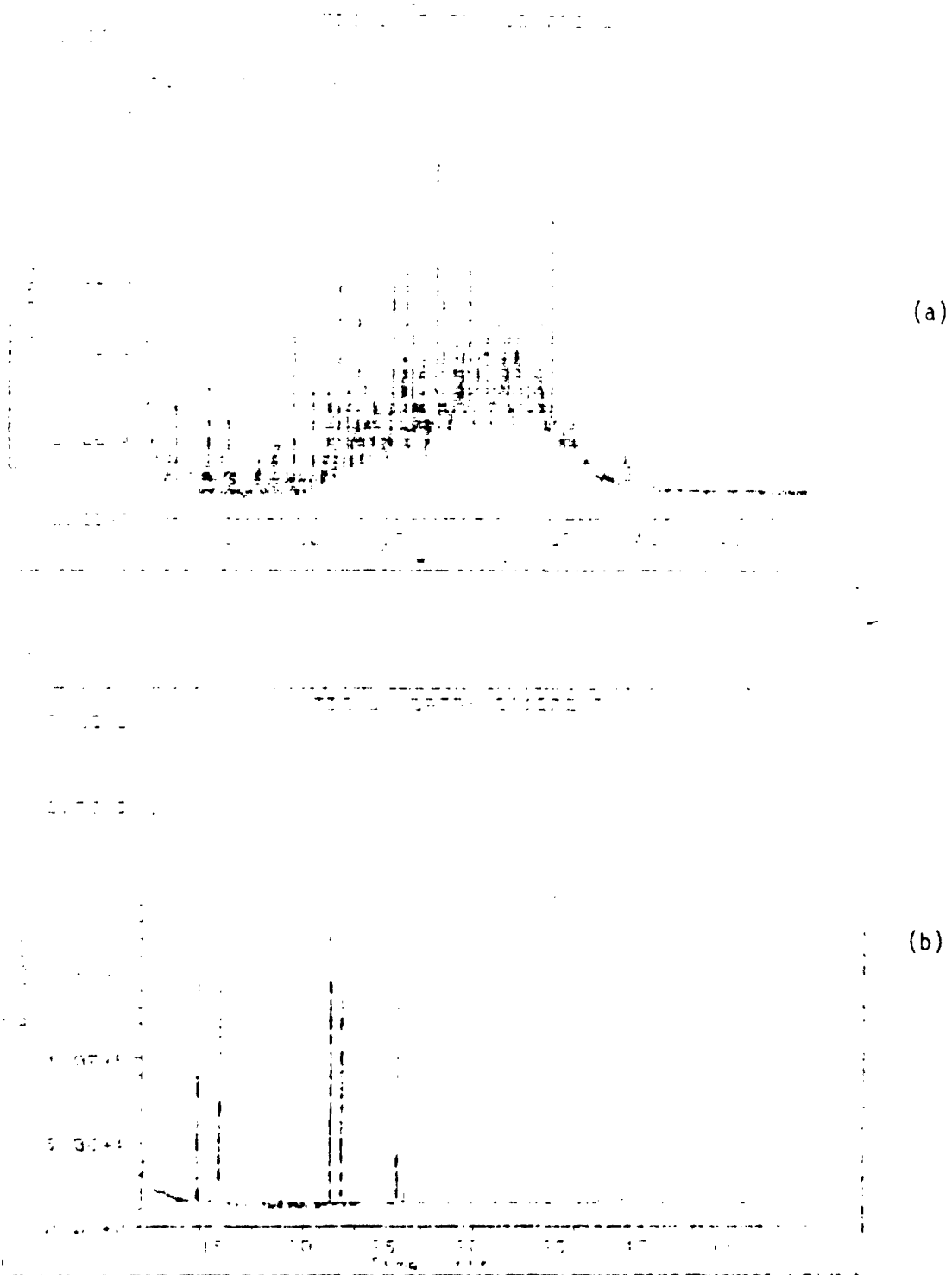


Figure 5. Chromatograms for Experiment 6. (a) Analysis of Initial Soil; (b) Soil Analysis After Treatment.

Experiments 5 and 6 showed that significant cleanup is feasible by heating the soil to 150°-157°C range followed by a soak period of 14 to 40 hours. The peak areas obtained by the analysis of treated and untreated soil extracts are compared in Table 5. Experiment 6 shows a relative total peak area response reduction of 99 percent; for Experiment 5, the reduction in area response was 90 percent.

b. Quantitative Results

The concentration of toluene, pentadecane, total aromatics, and a total nonaromatics in the soil before and after treatment was determined. This was done by a GC/MS analysis of a soil extract obtained by Nielson-Kryger steam distillation. These details are given Appendix A.

Table 6 compares the initial and final concentrations of each of the four contaminants measured for Experiments 5 and 6. Toluene was below the detection limit for both the soil samples of Experiment 5. It is estimated that the detection limit is approximately 3 ppm. In Experiment 6, toluene concentration was reduced from 4.3 ppm to below the detection limit.

When water was not injected, a 75 percent reduction of pentadecane was observed. When water was injected, pentadecane was reduced from 55 ppm to below the detection limit, a reduction of 94.5 percent. In both experiments, the total aromatics was reduced to BDL.

E. CONCLUSIONS

The pilot-scale experiments performed with CBZ and TRCE confirmed the results obtained in the bench-scale experiments (Reference 2). It was shown that both the contaminants can be removed from tall columns of soil in reasonable treatment time which can be economically provided under in situ conditions.

When a 6-foot tall column of soil containing jet fuel was thermally treated at 152°-157°C for 14 to 40 hours, the reduction in total contaminants ranged from 90-99 percent. Steam sweep generated through injection of water at the bottom of the bed significantly improved the removal of higher boiling

TABLE 5. COMPARISON OF TOTAL AREA RESPONSE

Exp. No.	Initial Soil				Treated Soil				
	Sample Weight, gm	Extract Volume, ml	Volume of injection, μ l	Area Response	Sample Weight, gm	Extract Volume, ml	Volume Injected, μ l	Area Response	Area Reduction, %
5	151.5	100	2.0	3.52×10^8	150.3	100	2.0	0.33×10^8	91
6	150.7	100	2.0	3.92×10^8	150.6	100	2.0	0.04×10^8	99

TABLE 6. PILOT-SCALE DECONTAMINATION OF SOIL CONTAINING JET FUEL
(TREATMENT TEMPERATURE 150°-157°C)

In Soil	Exp. 5	Exp. 6
Steam Sweep	No	Yes
Initial Toluene, ppm	BDL ^a	4.3
Final Toluene, ppm	BDL	BDL
Initial Pentadecane, ppm	104.7	55
Final Pentadecane, ppm	25.9	BDL
Initial Total Aromatics, ppm	385	479
Final Total Aromatics, ppm	BDL	BDL
Initial Total Non-Aromatics, ppm	4871	3407
Final Non-Aromatics, ppm	480	BDL
Initial Area Response	3.52×10^6	3.92×10^6
Final Area Response	0.33×10^6	0.04×10^6
% Area Response Reduction	91	99

^aEstimated detection limit based on 1,2-difluorotoluene (internal standard)

4-5 kilograms of soil in each experiment.

Residence time at final temperature: 14 to 40 hours

fractions in the fuel. For example, without water injection, approximately 75 percent removal of pentadecane was removed while water injection removed more than 94 percent.

The tall column experiments have shown that in situ heating followed by removal of vapors can be used for decontaminating soils under in situ conditions.

F. IMPLICATIONS FOR IN SITU TEST DESIGN

The pilot-scale experimental results have demonstrated that it should be feasible to decontaminate 6- to 10-foot thick beds of soil by using the in situ heating techniques. For the Volk Field site which has JP-4 jet fuel, solvents, degreasing agents, etc. in sandy soil, a treatment temperature range of 130°-160°C should be sufficient for at least 90 percent decontamination.

Results on high-temperature boilers such as pentadecane indicate that water injection may be necessary to achieve 90 percent removal. Water can be easily injected in the in situ field test by using selected electrodes as injection wells. Electrodes are implaced in bore holes drilled through the soil to apply RF power to the heated volume.

In the pilot-scale experiments, lateral movement of the heated contaminants was restrained by the cylindrical walls of the reactor. Under field conditions, no such physical barriers to lateral contaminant migration exist. Outward migration from the heated zone will, however, be mitigated by the presence of the cool soil surrounding the heated zone. In the cool zone, the pores will contain moisture and air. Due to the presence of moisture, the fractional permeability to gas flow will be greatly reduced, compared to the heated zone, where the moisture has been removed. Thus, the path of least resistance for the flow of contaminant vapors will extend vertically to the surface, through the heated zone, rather than laterally towards the cool zones.

Attempts at spiking large quantities of soil at desired initial concentration levels were unsuccessful. This indicates that severe difficulties will be encountered if attempts are made to treat large quantities of spiked soil in aboveground reactors to prove the feasibility of the in situ decontamination process. Without reliable initial base-line data, valid conclusions about the removal efficiency will not be possible.

SECTION III

SITE CHARACTERISTICS

An abandoned fire training area on an Air Force facility was selected for the field demonstration test. Historical data indicate that the site was used for over 25 years and routinely received waste oils, fuels and solvents which were deposited in the 100-foot (30 meters) diameter pit and ignited to simulate aircraft fires. An estimated 50,000 gallons (190 cubic meters) of waste hydrocarbons have soaked into the soils. Soil borings indicate a average total petroleum hydrocarbon concentration of 4000 mg/kg (ppm) extending down 12 feet (3.7 meters) to the groundwater. A more specific gas chromatography analysis shows an accumulation of heavy oils in the upper 12 inches (30 centimeters) with an increase in lighter fuel components with depth.

Soils in the site are homogeneous. Sieve analysis and penetration tests indicate that a medium grain sand extends from the surface to approximately 13 feet (4 meters) where fractured sandstone is encountered. The homogeneous contamination and soil found at this site were essential for an initial, controlled test of the RF technology. Future tests will address less homogeneous and clayey soils.

A. SITE HISTORY

An abandoned fire training area on Volk Field, Wisconsin was selected for this pilot test. Volk Field is located at Camp Douglas, Wisconsin approximately 90 miles northwest of Madison. The mission of this 2,300 acre base is to provide a realistic environment for military units to accomplish combat training in air and ground operations. Although the base has been used for military training since 1886, it has been an active Air National Guard base since 1947.

Records indicate that the fire fighter training area was established in the 1950's and used for over 25 years. Fire fighters were trained using simulated aircraft which was ignited using contaminated JP-4 fuels. Waste oils and solvents were also deposited in fire training areas to be burned. Unfortunately, not all fuels, waste oils, and solvents were consumed in the

fire. An estimated 50,000 gallons of these hydrocarbons have soaked into the sandy soils beneath this 100 feet diameter fire training area.

In 1984, the Air Force completed a survey of all hazardous waste/spill sites on Volk Field as a part of the Installation Restoration Program. The fire training area was identified as having a high potential for groundwater contamination and subsequent sampling has confirmed that considerable quantities of fuel and oils have contaminated soils and groundwater downgradient of the site. With the permission of the Air National Guard and Wisconsin Department of Natural Resources, the Air Force Engineering and Services Laboratory has used this site to test new soil decontamination technologies including the radio frequency thermal process.

B. SITE GEOLOGY

The geologic formations that underlie Volk Field are predominantly fine to coarse-grained sandstone with interbedded shale and overlying unconsolidated sand and small areas of silt and clay. Soils beneath the fire training area are 95 percent sand with 5 percent by weight finer than sand. The particle size distribution of the soil is shown in the sieve analysis in Figure 6. Mineralogically, soils are at least 98 percent alpha quartz with no clay as determined by X-ray diffraction.

The vertical permeability of the soil in the unsaturated zone was measured in a laboratory permeameter at 4×10^{-3} to 5×10^{-4} cm/sec. A shallow, but unconfined aquifer is found at 12-13 feet below the surface. The continual leaching of contaminants into this unconfined aquifer can only be stopped by effective soils decontamination. This site is excellent for decontamination research because of the homogeneous nature of the soils and good permeability. Although the Volk Field site must be considered a "best case" when compared to other geologic conditions, it offers a much more controlled soil volume for research. Future tests must address the more challenging problems of clay and more heterogeneous soils.

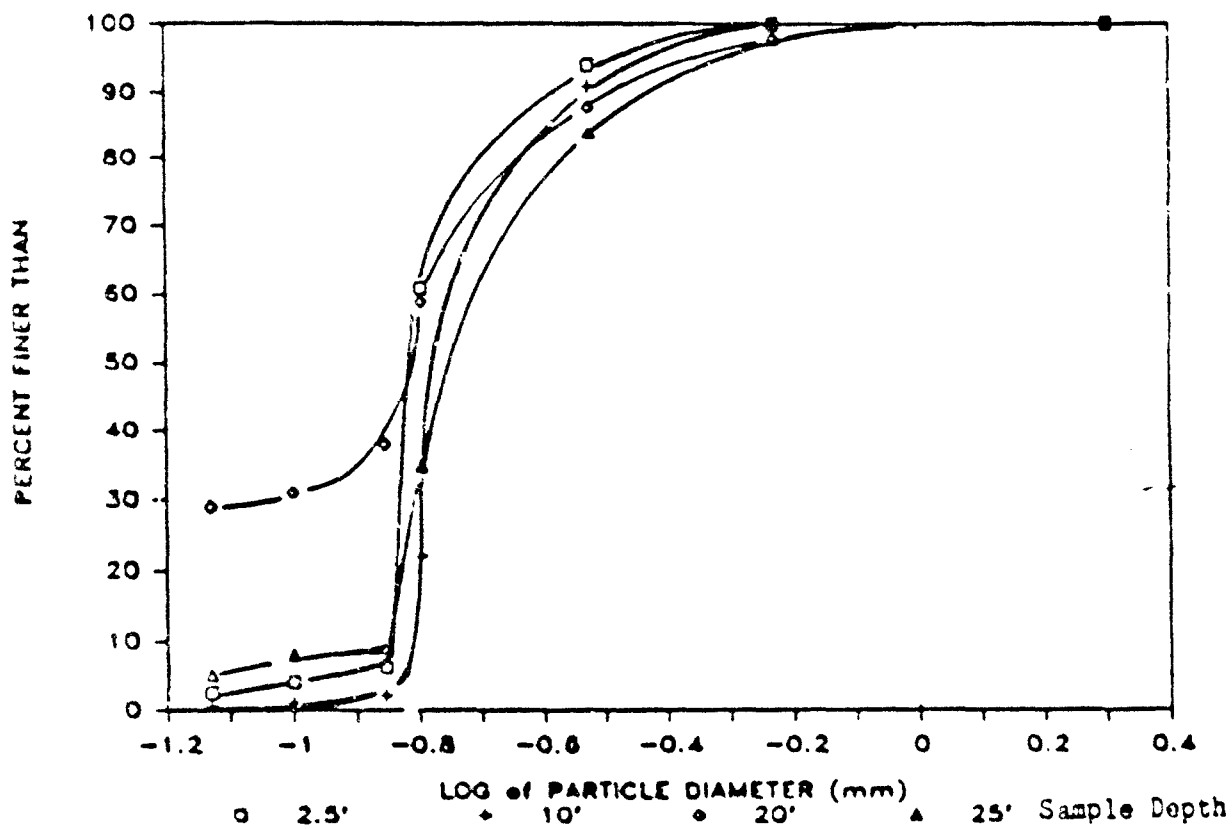


Figure 6. Soil Particle Size Distribution by Sieve Analysis

C. SITE CONTAMINANTS

The soil beneath the fire-training pit has been contaminated with waste oils, JP-4 jet fuel, and solvents used in maintenance functions around the base. The effect of such contamination on the soil is obvious when compared to nearby clean soil. The most obvious differences are color and lack of vegetation. The surface and near surface of the pit is black, cohesive, and free of any grass except at the edges of the pit. The pit emits an odor of fuel oil and surface oil has enough residual contamination to feel oily.

Soil borings in the fire training area indicate an average total petroleum hydrocarbon (EPA Method 418) concentrations of 4000 mg/kg extending down to the groundwater. A more specific gas chromatography analysis confirms an accumulation of heavy oils in the upper 12 inches and an increase in lighter fuel components with depth. Evidently the more water soluble hydrocarbons have migrated downward over time and have concentrated at the groundwater interface. A more detailed discussion of sampling techniques and specific contaminants identified is found in Section V. It should be pointed out that no detectable levels of chlorinated solvents (at 0.2 ppm level) were found in pretest soil samples. Previous groundwater sampling had identified TCE in the 100 ppb range.

D. SOIL DIELECTRIC PROPERTIES

In addition to quantifying the types of contaminants and the extent of contamination for a given target soil volume, an electromagnetic characterization of the soil is required for the design of a controlled uniform dielectric heating system. The electromagnetic or dielectric properties measured as part of this characterization are functions of the applied frequency and percent moisture of the target soil which is affected by the soil's temperature achieved during the heating process.

The exact frequencies chosen for heating a particular target soil volume partially depend on the volume's dimensions and the soil's electromagnetic (EM) characteristics. As a general rule, the higher the operating frequency, the more readily the material will absorb the energy. Too rapid absorption, however, can cause most of the energy to be absorbed near the source of the

applicator (electrode) at the expense of not heating the material further away. This affect can be lessened with lower operating frequencies. Since less power is absorbed near the source, better penetration results. There is a limit, however, to which the frequency can be decreased. Since the absorbing properties drop off with frequency, a reduced heating rate will result unless the amplitude of the EM fields is increased. The amplitude can be increased only up to a certain point, however, before corona discharge, spark-over, or electrical breakdown seriously affects the operation. Thus, the precise frequency of operation is ideally determined by the geometry of the volume and the electrical parameters or dielectric properties of the material.

In a practical sense, other limiting factors include frequency authorization and power source availability. The industrial, scientific, and medical (ISM) band frequency channels allocated for the dielectric heating process are 6.78, 13.56, 27.12, 40.68, 915, and 2,450 MHz. Because of the wide use of dielectric heating, relatively inexpensive tubes for power sources at the ISM band frequencies are available. Therefore, the selection of an ISM band frequency as the operating frequency is preferred.

The key dielectric parameters (relative dielectric constant, loss tangent, and conductivity) as a function of the applied frequency and the soil temperature in conjunction with the geometry of the material determine the appropriate selection of operating frequency and design of the electrode array for a specific controlled uniform dielectric heating application. The maximum allowable heating rate and the efficiency of energy distribution within the soil are then determined, based on the above and the dielectric/corona breakdown constraints of the soil.

Two additional design factors are considered in the selection of the operating frequency and the electrode array dimensions for a given application. The depth of penetration or skin depth of the electromagnetic wave as it enters the volume of soil to be heated and the corresponding wavelength of the applied frequency within the soil, both of which are dependent on the measured dielectric properties of the target soil, provide the basis for determining

the maximum allowable heating rates and energy distribution efficiency for a specified electrode array geometry. The skin depth of the electromagnetic wave is given by

$$\Delta^{-1} = \omega \left(\frac{\mu_0 \epsilon}{2} [(1 + \tan^2 \delta)^{1/2} - 1] \right)^{1/2} \quad (1)$$

where:

- Δ = skin depth, meters
- $\omega = 2 \pi f$; f = frequency, Hertz
- μ_0 = permeability of free space = $4 \pi \times 10^{-7}$ Henry/meter
- ϵ = permittivity of the material = $\epsilon_0 \epsilon_r$
- ϵ_0 = permittivity of free space = 8.854×10^{-12} Farad/meter
- ϵ_r = relative dielectric constant
- $\tan \delta$ = loss tangent.

Similarly, the wavelength at the applied frequency within the soil is given by

$$\lambda^{-1} = \frac{\omega}{2\pi} \left(\left(\frac{\mu_0 \epsilon}{2} [1 + \tan^2 \delta]^{1/2} \right)^{1/2} \right) \quad (2)$$

where: λ = wavelength in the soil, meter

For the design of a vertically emplaced, discrete electrode, tri-plate application array, as used for the field test of this technology, the depth of contaminant concentration establishes the desired length of the vertical tri-plate array. To ensure uniform dielectric heating within the boundaries of the tri-plate electrode array, it is necessary that the skin depth of the electromagnetic energy be large compared to all dimensions relative to the direction of energy propagation. Thus, both the vertical length of the electrode array and the electrode row spacing (spacing between the three plates) should be much less than the skin depth of the electromagnetic wave in the soil. Since the skin depth is inversely proportional to the frequency as well as the square roots of both the relative dielectric constant and loss tangent, the selection of an appropriate row spacing is accomplished at the highest

frequency of application desired and using dielectric parameter associated with the vaporization phase of water at approximately 100°C. The row spacing should also be less than one-half of the wavelength of the electromagnetic energy in the soil to assure single mode operation. Simultaneously, knowing the length or depth of the tri-plate array, also limits the highest frequency for appropriate electromagnetic energy application. The wavelength of this frequency in soil should be less than 4 times the electrode length at temperatures above 110°C or the water vaporization phase, so as not to create excessive standing wave patterns along the length of the vertical tri-plate array. This is desired to assure that the electromagnetic energy is distributed in a uniform sense both horizontally and vertically. Finally, the design of the surface areal extent of the vertical tri-plate is governed by the method of efficient electromagnetic energy coupling and the available power capacity. Further, the electrode spacing within any given row should be less than one-half of the row spacing to ensure adequate confinement of the electromagnetic energy within the desired volume to be heated.

Once the array geometry is defined, along with the selection of one or more appropriate frequencies of operation, the operational power delivery requirements are determined from the dielectric parameter data and voltage breakdown constraints. The power absorbed by the soil is given by

$$P = \omega \epsilon_0 \epsilon_r \tan \delta (E)^2 \quad (3)$$

where:

P = power absorbed (W)

E = electric field intensity, rms (volt/meter)

The geometry of the tri-plate array and the measured soil dielectric properties as a function of the selected operating frequency and soil temperature are utilized to provide a prediction of the electrode array's input impedance as a function of the heating process. This prediction, along with the input power requirements, provides the basis for defining the matching network requirements throughout the heating process.

The dielectric properties of soil samples obtained from Volk Field ANGB were measured as part of an earlier phase of this program, (Reference 4) by placing the material inside a coaxial transmission line sample holder and measuring the complex input impedance at the front of the line. The sample holder allowed for measurement of the input impedance over a frequency range of 0.5 to 20.0 MHz for sample temperatures up to 250°C. Calculation of the dielectric parameters of the material under test using this sample holder are valid for those materials whose permittivities are in the intermediate to low range. This corresponds to soils with lower concentrations of water.

The dielectric parameters of three sandy soil samples obtained from Volk Field ANGB were measured using this procedure. Each test was performed on a small sample of approximately 200 grams of soil packed within the low-frequency/low-permittivity sample holder to a density as close to field conditions as possible. The samples consisted of a clean sandy soil (Sample 1) taken from an uncontaminated area at Volk Field, and two contaminated soil samples extracted from depths of 42 to 46 inches and 84 to 89 inches at the Fire Training Pit located at Volk Field (Samples 2 and 3, respectively). Figures 7 through 9 illustrate the relative permittivity, loss tangent, and conductivity, respectively as functions of temperature and frequency for soil sample 3. All samples tested were held at a temperature of approximately 105°C for 1 hour to provide ample time for the vaporization and removal of the moisture within the sample.

All of the dielectric parameter curves, illustrated in Figures 7 through 9 have the same rapid decrease or "roll-off" beginning after the temperature has reached 110° to 115°C. This corresponds to the water in the sample being vaporized and allowed to escape. The dielectric data obtained from the two contaminated samples were essentially the same as those presented here with

VOLK FIELD TEST #3 (Sample #17 - IIT2; Depth = 84"-89")
FOR VARIOUS FREQUENCIES

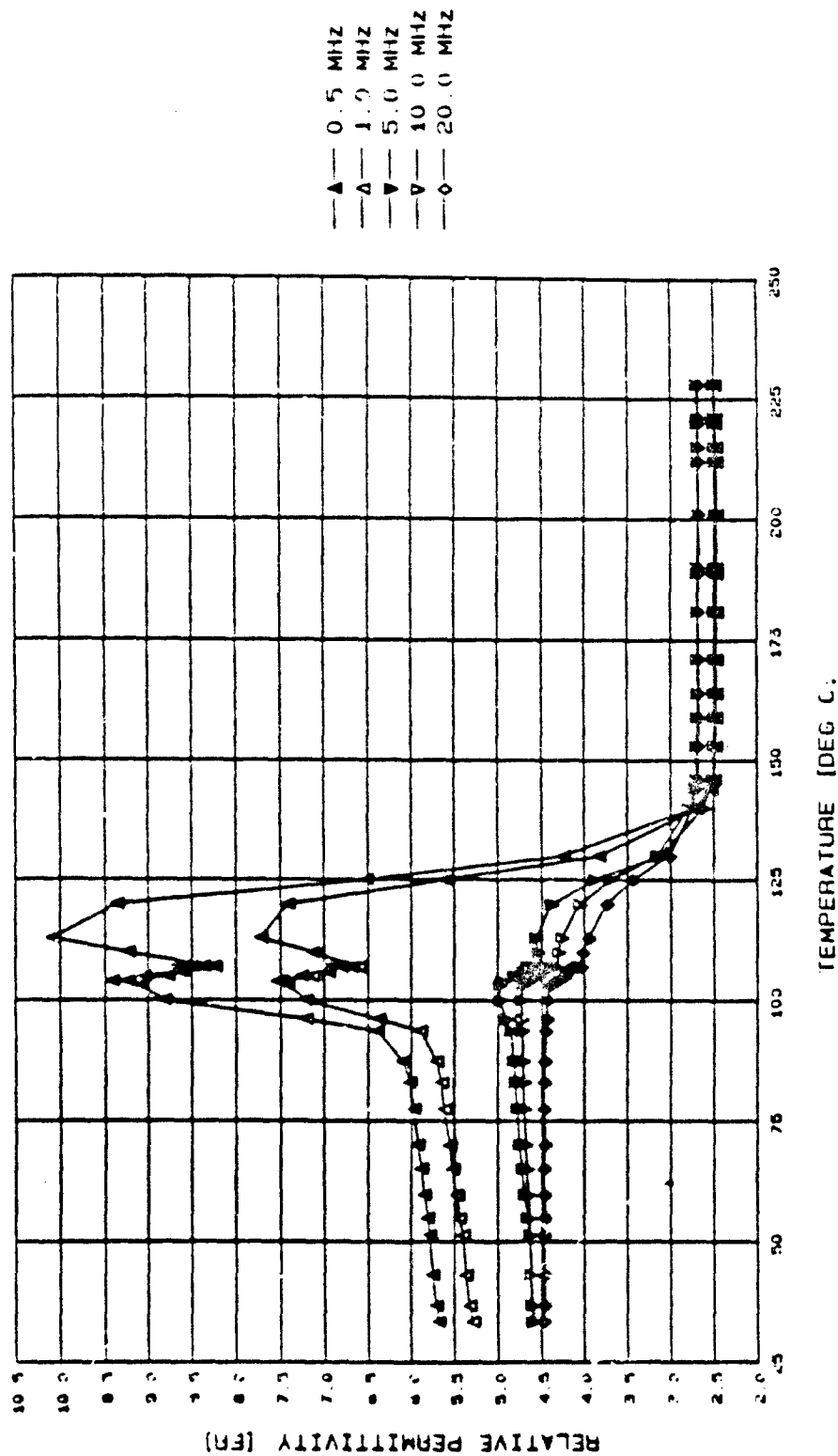


Figure 7. Relative Permittivity vs. Temperature for Volk Field Soil.

VOLK FIELD TEST #3 (Sample #17 - IIT2, Depth = 84"-89")
FOR SELECT FREQUENCIES

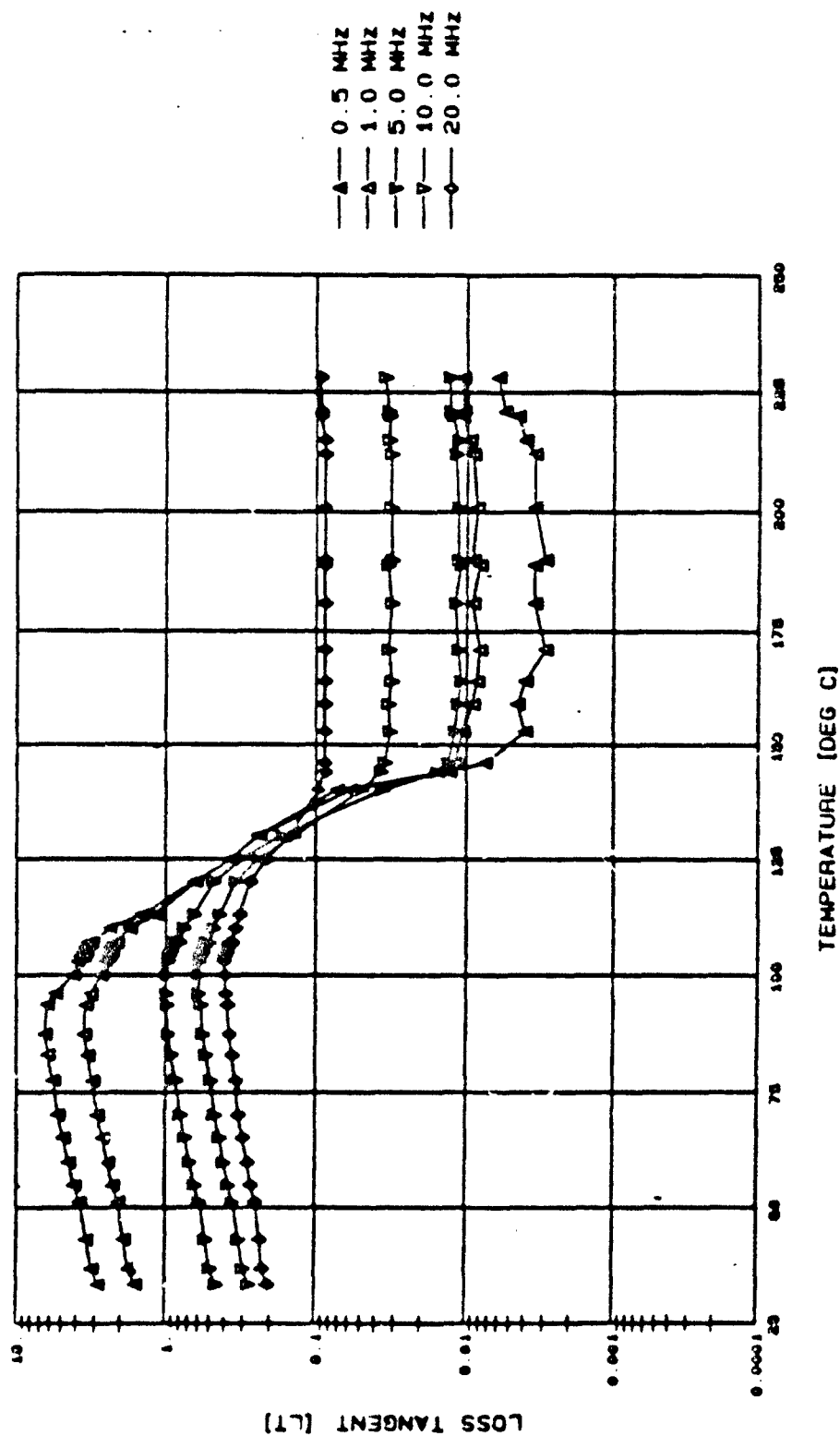


Figure 8. Loss Tangent vs. Temperature for Volk Field Soil.

VOLK FIELD TEST #3 (Sample #17 - 1112, Depth - 84"-89")
FOR SELECT FREQUENCIES

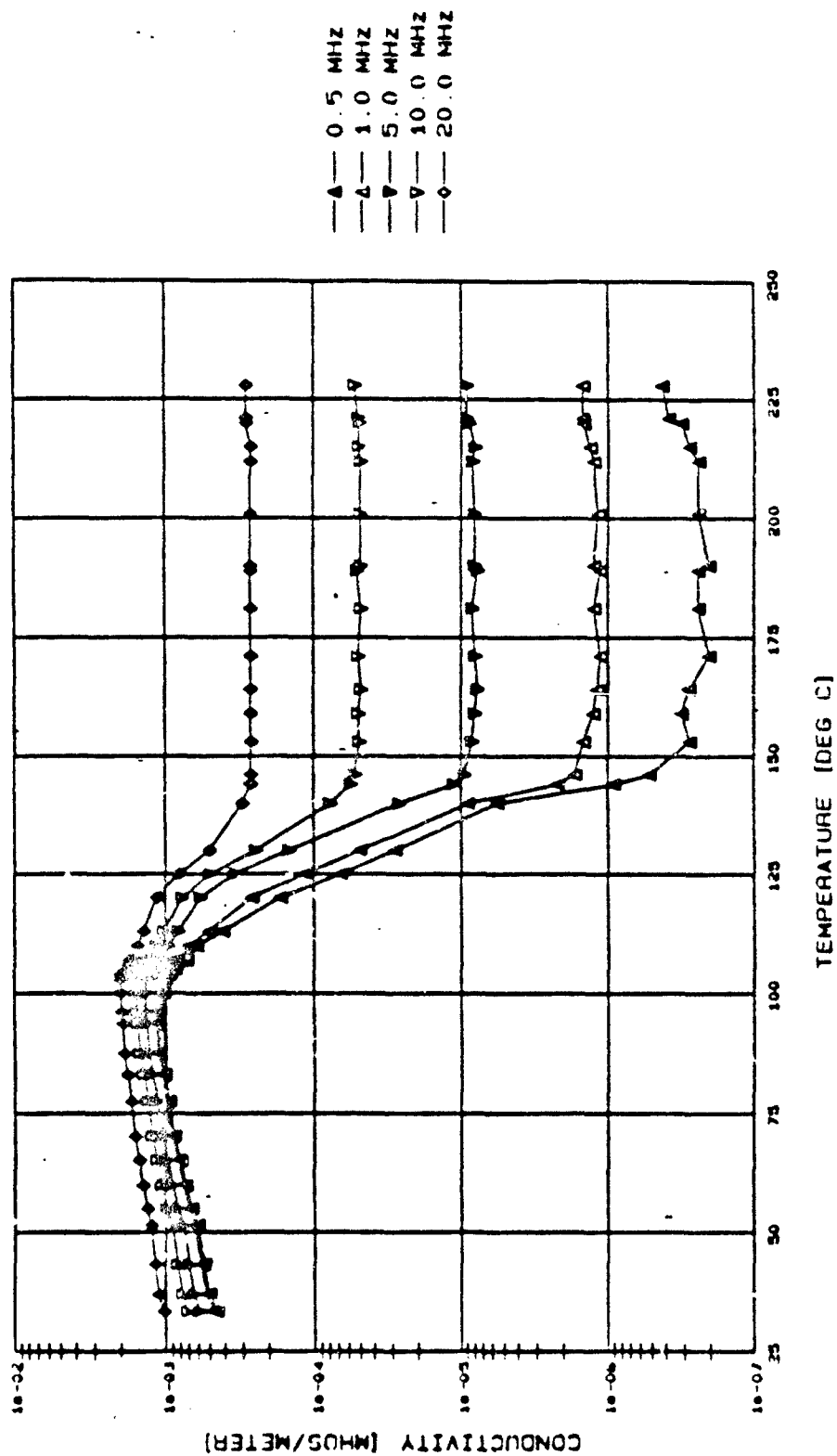


Figure 9. Conductivity vs. Temperature for Volk Field Soil.

little or no difference in the dielectric parameters for the two depths sampled. An analysis of the clean soil, however, indicated much lower values for the loss tangent and conductivity for all frequencies as compared to the contaminated samples. The representative data presented here on Sample 3, show that, at temperatures of 150 to 160°C, the soil will be absorbing a small percentage of the power absorbed during the water vaporization phase. The power absorbed at 150°C in contaminated soil appears significantly greater than in the clean soil. This is most likely attributed to the ability of the remaining contaminants to absorb the electromagnetic energy. This represents the difference between the contaminated samples at 150°C being able to absorb 1 percent or less of the power absorbed at 100°C and the clean sample at 150°C being able to absorb 0.1 percent or less.

The ability of the soil to withstand the high-intensity electric fields that will exist within the electrode array and at its edges was evaluated by conducting a dielectric breakdown test as part of the earlier phase of this program, on approximately 200 grams of the Volk Field clean soil (Sample 1). The results of this test, although qualitative, indicate that for the maximum applied power densities planned for the field test (approximately 2.5 kW/cubic meter) no dielectric breakdown is anticipated. During the breakdown test, as the moisture was evaporated from the soil sample, the impedance of the sample increased significantly so as to make matching difficult. At the termination of the test, approximately 95 percent of the moisture had been evaporated. RF energy, although only a few percent of that applied from the source, was still being absorbed by the soil sample as noted by the continuing temperature increase.

SECTION IV

RF PROCESS DESIGN

A. DESIGN OBJECTIVES

The primary objective of this project was to conduct an in situ radio frequency heating test at a contaminated site to demonstrate at least 90 percent reduction in the overall level of contaminants present in the site. In an earlier related program, (Reference 2) a candidate site for the experiments was identified at the Volk ANG8, Camp Douglas, Wisconsin. The design of the RF heating system was performed to achieve the following objectives:

- Heat 500 ft³ of sandy soil having a depth of 6 to 8 feet
- Demonstrate heating to a temperature range of 150° to 160° C
- Maintain temperature for a period of 4 days
- Evaluate contaminant migration effect from the heated zone
- Evaluate migration of species from outside of the heated zone into the heated zone
- Evaluate the efficiency of the vapor collection and treatment system
- Determine AC power consumed by the RF power source and obtain cost estimates. Extrapolate this information to more efficient power sources.

In situ heating of soil requires three major subsystems. These are: the electromagnetic heating system, the vapor containment and handling system, and the temperature monitoring system. The design of each of these subsystems is described in the following sections, while the description of the field test is summarized in Table 7.

TABLE 7. DESCRIPTION OF FIELD DEMONSTRATION TEST

Dimensions: Area 6 ft x 12 ft	72 ft ²
Ground Electrode Depth	8 ft
Exciter Electrode Depth	6 ft
Volume Heated, 6 ft x 12 ft x 7 ft	504 cu ft
Mass Heated	69,000 lbs
Water in Place @ 5% by Weight	3,450 lbs
Hydrocarbons in Place @ 1,600 ppm*	110 lbs
Chlorinated Hydrocarbons in Place at 3 ppm*	0.21 lbs
Average Water Boil-Up Rate	1.4 gph
Average Rate of Power Delivery:	
Below 100°C	30 kW
At 100°C	30 kW
Above 100°C (After Water Boil Off)	10 kW
Time to Reach 100°C	2.5 days
Time at 100°C	4 days
Time to Reach 150°C from 100°C	2.5 days
Time at 150°C	4 days

* Based on analysis performed by SAI, Inc.

B. DESIGN OF THE ELECTROMAGNETIC HEATING SYSTEM

The basic configuration of the EM heating system consists of three major components, the first being the triplate electrode array embedded into the contaminated soil. This electrode array is connected to the EM power system via a triplate to coaxial transition section. The second major component is the coaxial transmission line interconnecting the RF load or triplate array to the matching network and continuing from the matching network to the RF power source. The last major component of the EM heating system consists of the power source itself, which for the Volk Field test, consisted of a 40 kW HF band radio transmitter that was trailer mounted. Details of the component design are discussed in the following sections.

1. Electrode Array Design

The triplate transmission line is an example of a field-confining guide, simply described as a coaxial cable whose conductors have been flattened from their normal concentric circular configuration. Figure 10 illustrates the evolution of the solid triplate line to the discrete electrode configuration used for the Volk Field test. Because of the large size of the individual electrode planes compared to their separation, the field between the inner conductors and the outer conductors is essentially uniform, except near the edges of the inner conductor plane. Soil, or any lossy dielectric placed within this electric field will be heated in a nearly uniform and controlled manner.

The data obtained from the dielectric parameter measurement experiments, were used to design the electrode array and predict the variation in its input impedance as a function of the average soil temperature during the heating process. Based on the data obtained and on experience in heating, the dielectric power absorption for soil temperatures of 125° to 130°C could be expected to be achieved with little difficulty. Final temperatures in the 150° to 160°C range, for a type of soil found at the Fire Training Pit at Volk Field AFB, appeared obtainable but posed additional electrical design difficulties. These difficulties included the need for the designing of separate impedance matching network components for both the lower

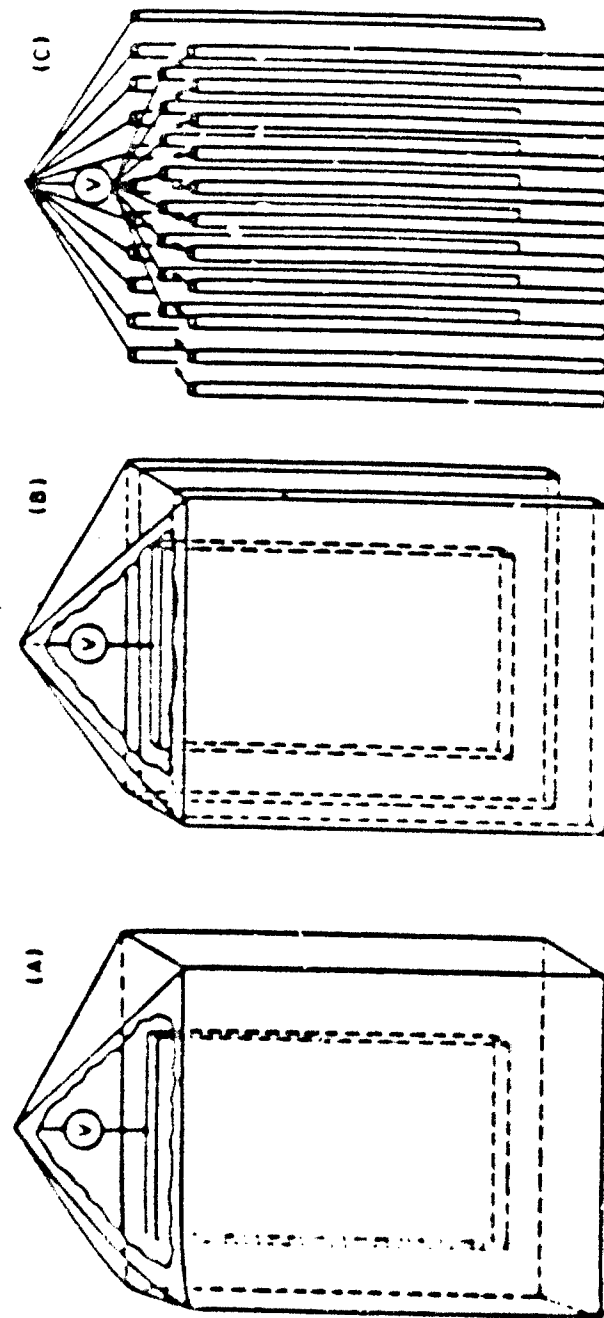


Figure 10. (A) Solid Wall, Totally Enclosed Guide; (B) Solid-Wall Guide with Sidewall Removed; (C) Discrete Electrode Realization of (B).

temperature and higher final temperature range. Provisions were also included in the original design to use a dual-frequency electrode array in the event that at the higher final temperatures the input impedance to the electrode array presented too great a mismatch for even the second matching network to accommodate. Two ISM band frequencies (6.78 and 13.56 MHz) were identified for use during the Volk Field heating demonstration.

A 6 feet by 12 feet area of the fire training pit was heated using the triplate electrode array shown in Figure 11. In the top left hand corner of the figure a plan view of the electrode array is shown. Three electrode rows were implaced in drill holes bored through the soil with a hand bucket auger. In each row there were 13 copper electrodes, spaced 1-foot apart. The length of each row was 12 feet. The spacing between each of the three rows was 3 feet as shown in View AA of Figure 11. The depth of the two outer rows of electrodes was 8 feet while the center row depth was 6 feet. The two outer electrode rows are called the ground planes and the center row is called the exciter plane.

All the electrodes in each row were electrically connected together with brass straps, as shown in the Views BB and CC of Figure 11. Five electrodes in each row were 3 inches longer than the other electrodes, so that they could pass through the vapor barrier and connect to the RF power feed transition section. The ends of all the electrodes were capped with end-caps to prevent moisture from entering the electrodes. If moisture is allowed inside the electrodes a heat-pipe is set up within each electrode.

The test plan called for a 1-foot wide trench outside the electrode array be excavated and backfilled with clean soil from the site. The purpose of this trench was to assess the migration of contaminants from the heated zone to the surrounding soil. Trench excavation was not possible because a severe cave-in of the treatment zone occurred. It was decided to obtain soil samples from the trench area before and after the test to determine whether contaminant migration had occurred and to conduct a Halon® tracer test to determine soil gas flow direction.

2. RF Transition Section

The RF transition section is a carefully designed geometric structure used to change the shape of the coaxial RF power cable into a triplanar RF cable which provides power to the electrode array. Such a structure possesses specific electrical waveguide properties which confine the RF radiation within a shielded zone, while uniformly distributing power to the electrodes.

Figure 12 shows a layout sketch of the RF transition section. An artist's conceptual sketch of the RF transition section as installed is shown in Figure 13. A 6 1/8-inch diameter coaxial input is translated downwards, by copper and brass solid metal structures, to the electrode connection points. A door allowing access within the transition region was provided to facilitate innerconnection of thermocouples installed in the center row electrodes. These metal thermocouples must be disconnected while the RF power is being applied.

3. Matching Network and Power Transmission System

The RF matching network can handle high power and is used to obtain optimum conditions for power transmission between the RF power source and the soil-loaded electrode array. Optimum power transfer is accomplished by matching the impedance of the array to the output impedance of the power source. When these impedances are mismatched the load does not absorb all the generated power. Instead, some of the EM energy is reflected back to the power source. The reflected energy is dissipated as heat in the matching network components and the power transmission cable. Mismatches are detected by measuring the forward power and the reflected power at the output of the RF power source. Variable components of the matching network are then adjusted to reduce the reflected power to zero and to obtain a matched condition.

Two matching network designs were used for the demonstration test conducted at Volk Field AAGB. Both designs relied on available high power RF capacitors in both series and shunt configurations and the use of a portion of coaxial cable functioning as a line stretcher between the electrode array and

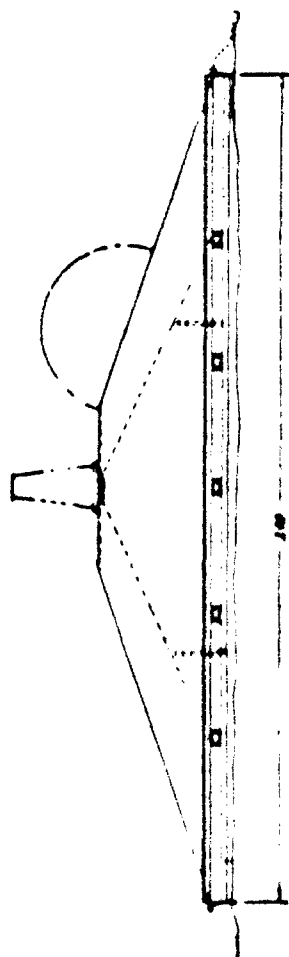
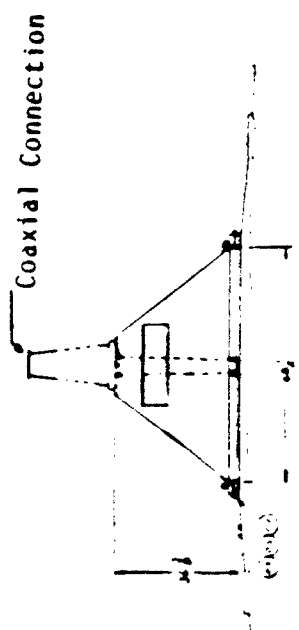
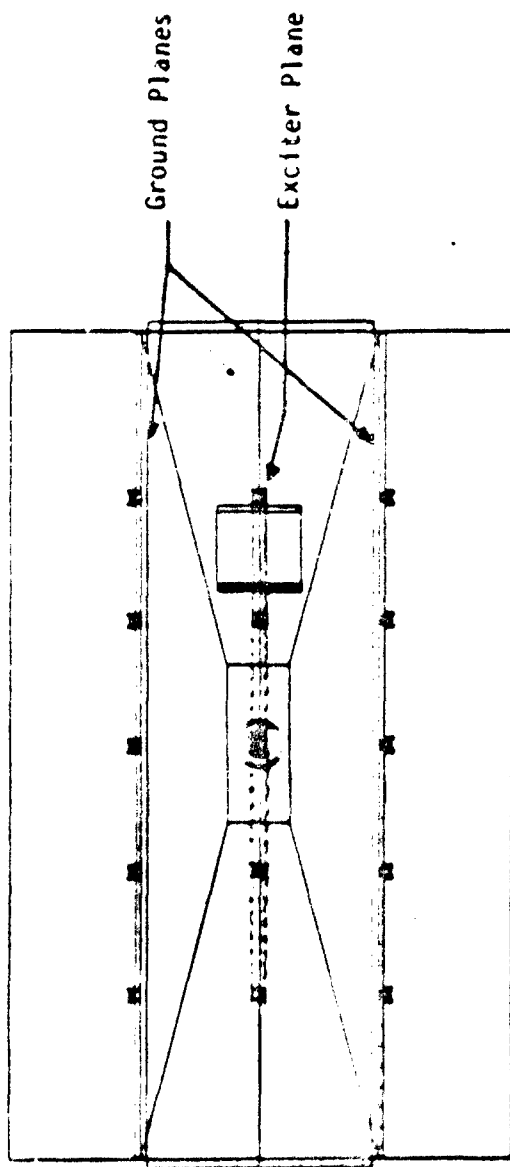


Figure 12. RF Transition Section Layout Sketch.

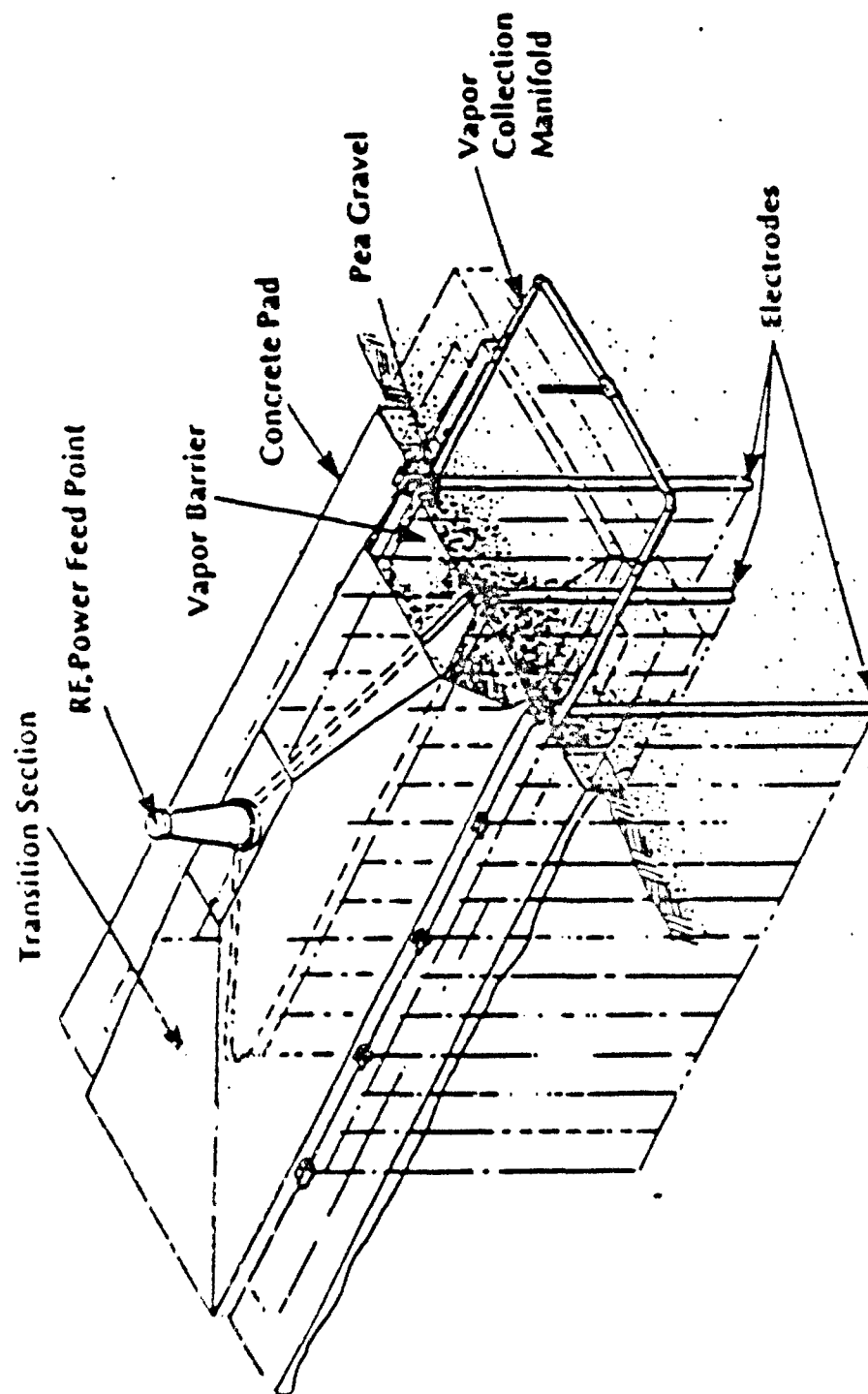


Figure 13. Design of the Installed RF Transition Section.

the variable capacitors. One matching network configuration was designed for operation when the electrode array input impedance generated a standing-wave ratio (SWR) of no greater than 15:1. The second matching network configuration covered an SWR range of 15:1 to 35:1.

The term standing-wave ratio (SWR), used to quantify the impedance mismatch between a load and its coupling source impedance, is defined as follows:

$$\rho = \frac{Z_L - Z_0}{Z_L + Z_0} \quad (4)$$

and

$$SWR = \frac{1 + |\rho|}{1 - |\rho|} \quad (5)$$

where ρ = reflection coefficient
 Z_L = complex load impedance
 Z_0 = characteristic impedance presented to the load
 SWR = standing wave ratio

The vaporization of the bulk of the moisture within the soil occurs while the electrode array SWR remains below 15:1, and corresponds to a final average soil temperature in the 115° to 130°C range. As the average soil temperature is elevated from 130°C to 160°C, the SWR increases from 15:1 to approximately 35:1 for the single frequency case. The components required to achieve these higher SWR ratings in and of themselves require component cooling. This cooling is supplied by means of circulating deionized water through the plates and fins of the variable capacitors for this matching network configuration.

All the components involved in both of the matching network configurations were comprised of IITRI available equipment and equipment available for use on this program as a result of a DOE cooperative program evaluating the RF heating of tar sands.

The coaxial transmission line section used as the line stretcher in both matching network configurations required pressurization with dry nitrogen to no greater than one atmosphere gauge pressure, and a maximum pressurization to 5 psig using Freon 116. The Freon 116 provided additional cooling while at the same time maintaining voltage withstand capability within the coax transmission line.

An IITRI designed and fabricated in-line coaxial impedance meter section was installed in both of the matching network configurations. Data obtained from the in-line impedance meter provided real-time tracking of the array's input impedance.

The 40 kW HF band radio transmitter capable of operating at either of the two ISM band frequencies, 6.78 or 13.56 MHz functioned as the power source for the demonstration test at Volk Field. The output of the RF power source was connected to the matching network via 3-1/8-inch rigid and 3-inch flexible coaxial transmission line. Both forward and reflected power meters were installed between the RF power source and the input to the matching networks to provide process data as well as input to the matching network control functions. Figure 14 provides an overview of the EM heating systems interconnection for the demonstration test at Volk Field ANGB.

C. VAPOR CONTAINMENT AND HANDLING SYSTEM

As the soil is heated in situ, gases and vapors form as a result of contaminant vaporization and boiling of water. These vapors were collected at the surface of the heated zone for treatment in a vapor cooling and condensation system. A vapor barrier was designed to contain the vapors in the zone above the surface of the heated area and to facilitate their collection.

1. Vapor Barrier and Collection System

The design of the vapor barrier and the collection system is shown in Figure 13 (Page 46). The vapor barrier was made from 0.125-inch thick silicon rubber sheet. This sheet was stretched over the heated zone from end to end and allowed to extend on to a concrete pad poured around the perimeter of the heated zone. The "picture-frame" concrete pad and the rubber sheet are

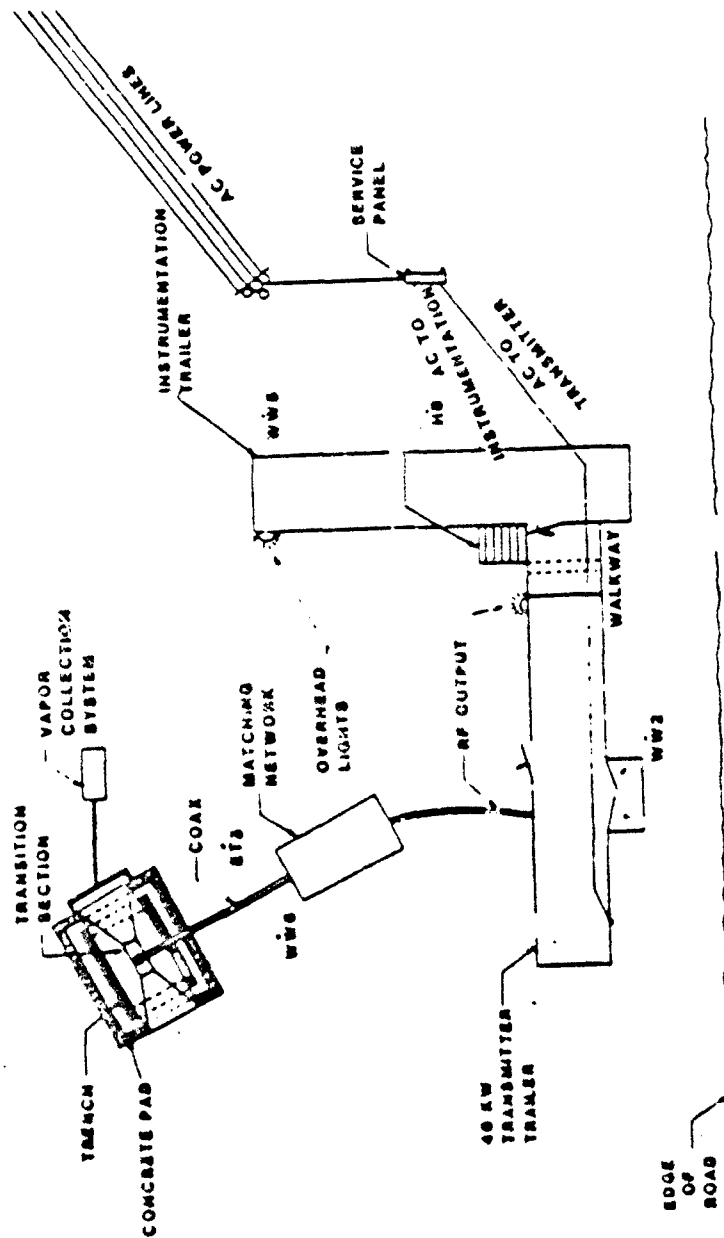


Figure 14. Volk Field AFB Fire Training Pit Demonstration Site Layout.

illustrated in Figure 13. The concrete pad was 12 inch wide, and varied in thickness from 3 to 6 inches, to provide a true horizontal top surface to which the rubber sheet was bonded with a silicon adhesive. A layer of pea gravel was poured inside the concrete pad. Two horizontal, perforated gas collection lines were placed in the pea gravel and manifolded together as shown in Figure 13 (Page 46). The gas collection lines were electrically bonded to the ground plane electrodes with brass straps. The gas collection lines were made from copper. This was done as a matter of convenience only since spare electrode tubing material was available.

A 2-inch thick layer of thermal insulation was placed over the vapor barrier to reduce heat loss from the heated zone and to reduce condensation of the vapors in contact with the inner surface of the barrier.

2. Vapor Handling System

The purpose of the vapor handling system was to remove the vapors from the collection zone below the vapor barrier and to cool and condense the vapors in an air-cooled heat exchanger. The uncondensed gases were treated through a carbon bed before release to the atmosphere. Figure 15 is a flow diagram of the vapor handling system. The gas collection manifold was heat-traced and connected to the inlet of a finned-tube, air-cooled heat exchanger designed to reject 41,000 Btu/hr. This is approximately twice the anticipated heat load, based on the condensation and subcooling of 11.4 lb/hr of steam from 150°C to 27°C. The outlet of the heat exchanger was connected to a stainless steel separator drum in which condensed hydrocarbons and soil moisture were separated from the gas stream. A Teflon® demister pad was installed in the drum to remove any liquid particulate carryover in the gas stream. The liquid collecting in the separator was siphoned into a condensate collection drum.

The outlet of the separator was connected to a carbon bed containing 150 pounds of type EPL carbon supplied by Calgon. The outlet of the carbon bed was connected to two 20 cfm blowers which were used to remove the vapors from the collection zone and transport them through the vapor handling system. The vapor collection zone below the silicon rubber vapor barrier was maintained at a vacuum of 0.5 to 0.6 inches of H₂O.

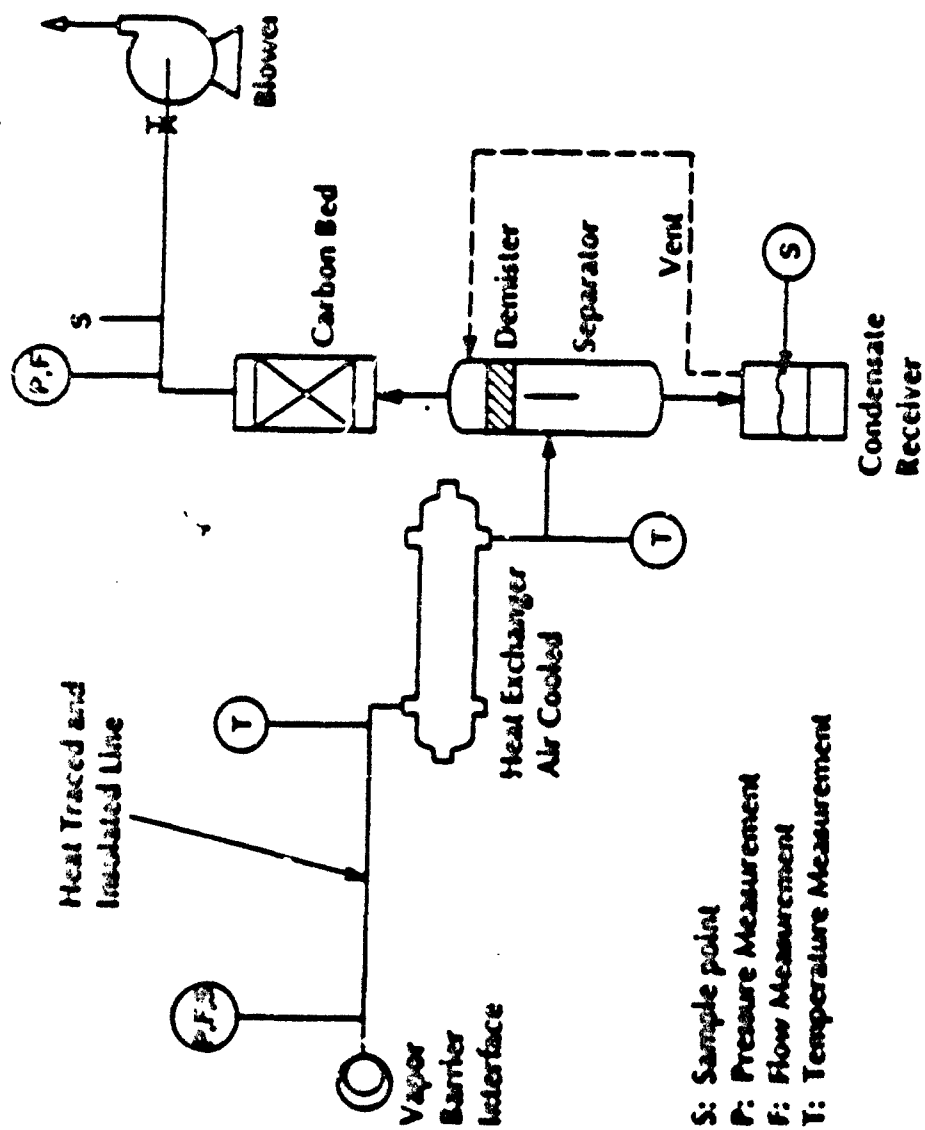


Figure 15. Gas and Vapor-Handling System.

D. TEMPERATURE MONITORING SYSTEM

The purpose of the temperature monitoring system was to allow mapping of the spatial temperature distribution within the heated volume as a function of time. For this purpose, thermocouples were attached to the inside walls of selected electrodes, and fluid filled thermowells were placed in boreholes at various depths to allow for temperature measurements. Like the microwave oven, metallic probes may not be placed in the center of the heated volume. Thus, thermocouples were not placed between the electrode rows. Thermocouples can be placed in the electrodes itself without causing electrical difficulties. The location of the various temperature measurement points is illustrated in Figure 16. The plan view of the electrode array is shown in the upper left corner of Figure 16. This view shows the locations of all electrodes containing thermocouples, and all bore hole locations in which thermowells were placed.

The distribution of the temperature measurement points was based on the assumption that the temperature profile will be symmetric about the center of the array. Accordingly, one quadrant of the array was more densely packed with the measurement points while the other quadrants had a lower density, sufficient to confirm the assumption of symmetry. The upper right hand quadrant in the plan view of Figure 16 was heavily instrumented.

View AA is a cross section of the heated zone which indicates the burial depth of the thermowells in boreholes labelled B1, B2, and B3. View AA also shows the location of a thermocouple buried at a depth of 3-feet in the trench area outside the heated zone.

View BB of Figure 16 illustrates the location and depths of thermocouples and thermowells in the plane of the exciter row. Twelve thermocouples were attached to the exciter row electrodes. In addition, four thermowells were placed on the same plane outside the heated zone to determine the rate at which heat is lost to the surrounding cool soil.

View CC of Figure 16 illustrates the thermocouple and thermowell distribution in each of the two ground planes. In each of the ground planes, 14 thermocouples were distributed among the 13 electrodes. Two thermowells

were also placed in the same plane outside the heated zone as shown in View CC.

The location and number of the various temperature measurement points are summarized in Table 8. Temperature data were obtained from 25 different locations when the RF power was on. Every 6 to 8 hours the RF power was switched off to measure temperature from the exciter plane locations as well as from the thermowells. When the RF power was off an additional 24 measurement points were added to the basic 25 points available when the power is on.

E. EMISSION MONITORING PLAN

Two types of emissions were monitored during the test. These were emissions of RF energy from the test array and emissions of chemicals in the gas stream discharged from the carbon bed shown in Figure 15. The emission control and monitoring activities are summarized in Table 9.

1. Monitoring of Electromagnetic Emissions

Near and far field electromagnetic (EM) measurements will be made at and around the test area. Near field refers to the immediate vicinity of the test site. Far field refers to locations 0.25 to 1 mile from the test site. The far field locations were selected in consultation with the base communications personnel. The purpose of these measurements is to ensure that the radiated RF power levels are below permissible FCC and Air Force standards.

These measurements will be made by turning on the RF power to the array at low levels before the test. The emission measurements will be made at two different low power levels. Should the level of emitted RF radiation exceed the permissible standards, then corrective action will be taken to reduce the emission levels to acceptable limits and base communication personnel's approval will be obtained. The near and far field measurements will be repeated shortly after the power input to the array reaches the normal operating levels around 30 kW.

TABLE 8. NUMBER AND LOCATION OF TEMPERATURE MEASUREMENT POINTS

Location	RF Power On	RF Power Off
Row 1	14 T/C	14 T/C
Row 2	None	12 T/C
Row 3	5 T/C	5 T/C
Between Rows 2 and 3	2 F/O	9 F/O
In the "Trench" Area	1 T/C	2 F/O
		1 T/C
Outside Heated Zone and "Trench" Area	2 T/W	2 T/W
		4 F/O
Total	25	49

T/C = Imbedded thermocouple

F/O = Thermowell for fiberoptic temperature sensor

T/W = Thermowell for thermocouple

TABLE 9. EMISSION MONITORING PLAN

Prior to Test

- Measure background and emitted RF radiation at low and high power levels.

During and After Test

- Cool vapor stream to 27°C using air or water-cooled heat exchanger.
- Separate liquids from uncondensed gases/vapors.
- Save liquids in drums. Measure rate of production of water and hydrocarbon layers.
- Pass gases through carbon bed.
- Measure flow rate of gas stream.
- Take periodic gas samples for analysis of total hydrocarbons via FID monitor, and/or Draeger tubes.
- Analyze water and hydrocarbon layers.
- All collected condensate will be saved in drums for later ultimate disposal.

2. Monitoring of Gaseous Emissions

Gases and vapors formed during the field test will be collected through the vapor collection line as shown in Figure 14. On this figure the location of three gas sampling points is indicated by the symbol SP. These points are the inlet to the heat exchanger, and the inlet and outlet of the carbon bed. The total gas flow rate leaving the carbon bed will be measured.

The purpose of these analyses is to obtain quantitative data on total hydrocarbons in the gas streams. Another purpose of the field analyses of the gas stream is to obtain semiquantitative information on how the concentration of the hydrocarbon components in the gas stream is changing with time. This information will be used as a measure of the progress and performance of the soil decontamination system as soil samples cannot be readily obtained from the heated zone while heating is in progress.

A portable FID probe will be inserted into the gas lines at the three locations shown in Figure 15 to determine the total hydrocarbon concentration of the gas stream. (The portable FID probe could not be used in the field because it was found that the high boiling hydrocarbon components would condense in the instrument. Instead, gas samples were collected in chilled gas wash bottles and the samples were analyzed in Chicago on a GC equipped with flame ionization detector.)

In addition to these measurements, a lower-explosion limit monitor will be used to determine the flammability of the atmosphere near the test site, and around the vapor barrier, and in the transition zones.

F. SITE LAYOUT

A description of location of the fire training pit in which the in situ decontamination field test was conducted is presented in Section III. Figure 14 (Page 49) illustrates the site layout. The experiment was located in the northwest section of the fire-training pit. Two 40-foot trailers were brought onsite and parked as shown in the Figure. A minor amount of earth moving was done under the instrumentation trailer to level the ground. A new AC power line was installed to provide power for the test. This power line is capable of supplying 500 KVA of three-phase power. Watt-hour meters were installed on

a panel mounted to the transformer pole to separately account for the power used by the RF source and other AC power loads at the site. The matching network and the vapor handling system were skid-mounted and placed outside near the test area, as shown in the Figure. Locations of selected existing groundwater monitoring wells near the test area are also shown in Figure 14. A thermocouple was dropped in monitoring Well WW 6 to monitor the groundwater temperature increase, if any, during the test.

SECTION V

SOIL AND VAPOR SAMPLING

A. SOIL SAMPLING

1. Objectives

The objectives of obtaining soil samples from the heated zone were:

- determine the pre- and posttest average concentrations of contaminants in the depth range of 6-72 inches
- determine average concentration of the contaminants in three depth intervals: 6-12 inches, 30-42 inches, and 60-72 inches
- determine migration, if any, of contaminants into and out of the heated zone
- determine decontamination efficiency in terms of percent reduction of contaminant concentration

These objectives were met by developing a sampling plan for extensive sampling of the heated zone before and after the test.

2. Soil Sampling Plan

A pre- and posttest soil sampling plan was developed for determining the average contaminant concentration before and after the test. The pretest sample hole locations are illustrated in Figure 17, which shows the plan view of the 6 feet by 12 feet heated area. The three electrode rows are shown with 13 electrodes in each row. The entire area was subdivided into eight cells, each of dimensions 3 feet by 3 feet. It was decided to take samples from three different types of sample locations. Samples of Type 1 were taken around the perimeter of the heated zone, as indicated by open circles in Figure 17. Samples of Type 2 were taken from the 8 o'clock position relative to the center of each of the eight 3 feet by 3 feet cells. Samples of Type 3 were taken from the 2 o'clock position relative to the center of each of the eight cells. There were 8 sample holes for each type, with a total of 24 sample holes in the heated area. To determine the concentration in three different depth intervals, three samples were taken from each sample hole.

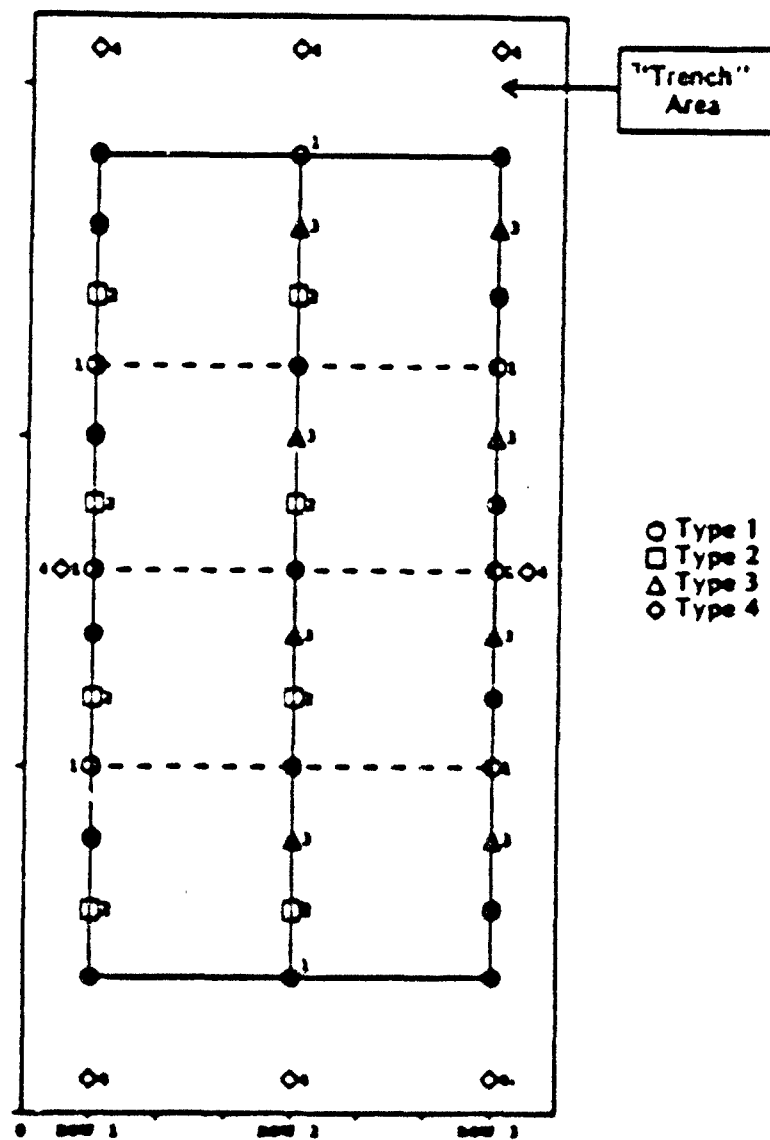


Figure 17. Pretest Soil Sample Hole Locations.

These samples were obtained from the depth intervals of 6-12 inches, 30-42 inches, and 60-72 inches. Thus, there were 72 different soil samples obtained from the heated zone.

The average overall concentration was determined by making composite samples. All samples of the same type and from the same depth interval were combined in the composite sample. For example, all samples of Type 1 taken from the depth interval of 30-42 inches were combined to make a single composite, thereby reducing the 72 samples from the test area to nine composite samples. Each of these nine samples were analyzed according to methods summarized in Section V.A.4.

It was mentioned in Section IV that the electrode array design required the formation of a 1-foot wide, 6-foot deep trench around the perimeter of the heated area. The purpose of this trench was to assist in determining outward contaminant migration from the heated zone. This would have been accomplished by backfilling the trench with clean soil before the treatment. Posttest sampling and analysis of the soil in the trenched area were planned to determine whether outward contaminant migration had occurred. The trench could not be made because of severe cave-in problems encountered at the time of excavation. It was decided not to continue with the excavation; instead, samples of the "trench" area soil were obtained from eight different holes. These are the Type 4 holes in Figure 17. Like the other holes, samples were obtained from the same three different depth intervals and composite samples were prepared. Thus, 24 different trench area samples were obtained which were reduced to three composite samples, one for each depth.

The posttest sampling plan is illustrated in Figure 18. As with the pretest plan, three different types of samples were obtained from the heated area. To determine the degree of outward contaminant migration, Type 8 samples were taken from four holes in the "trench" area. Table 10 summarizes the number of samples taken and composites made for both the pre- and posttest samples.

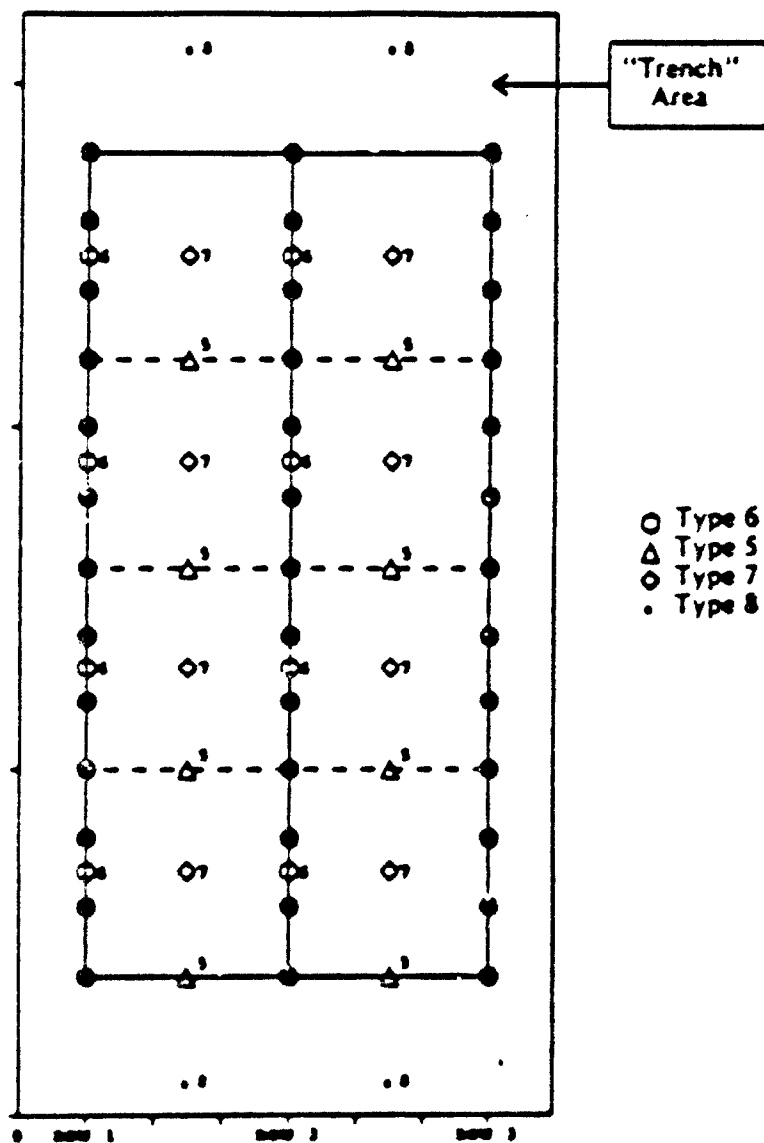


Figure 18. Posttest Soil Sample Hole Locations.

TABLE 10. SUMMARY OF SOIL SAMPLES TAKEN

Parameter	Pretest	Posttest
Type of sample locations	4	4
Sample hole per type	8	8*
Depth intervals per hole	3	3
Number of samples	96	84*
Number of composites	12	12

*Only 4 sample holes from "trench area" (Type 8).

2. Soil Sampling Procedure

A hand bucket auger was used to start the soil-sample hole to a depth representing the top of the sample interval. Then a lined split-core barrel sampling tool was inserted in the hole and driven into the soil with a sledge hammer. When the core barrel had progressed down through the sampling interval, the barrel was removed from the hole and the sample was recovered. All samples were sealed in the field in the plastic liner tube used inside the split core barrel. The samples were preserved in an ice-box and shipped to IITRI laboratories in Chicago for the preparation of composites and their analysis.

The above sampling technique was used for all 96 pretest samples obtained from the trench and heated areas. However, the core barrel could not be used for the sampling of soil after the test. It was found that the soil had become free flowing, like dry beach sand, and no core recovery was possible, despite attempts with core retainers designed for free-flowing soils and for sludge sampling. A new technique, based on vacuum lift of the soil, had to be devised in the field to obtain the posttest samples. This technique is illustrated in Figure 19.

First, a 6-inch deep starter hole was made in the soil by means of a bucket auger. Then, a 3-foot segment of 2.5 inch black iron pipe was placed in this hole. A vacuum hose was placed inside the black iron pipe, and the soil was suctioned out by means of a "wet-dry shop-vac." The soil samples was

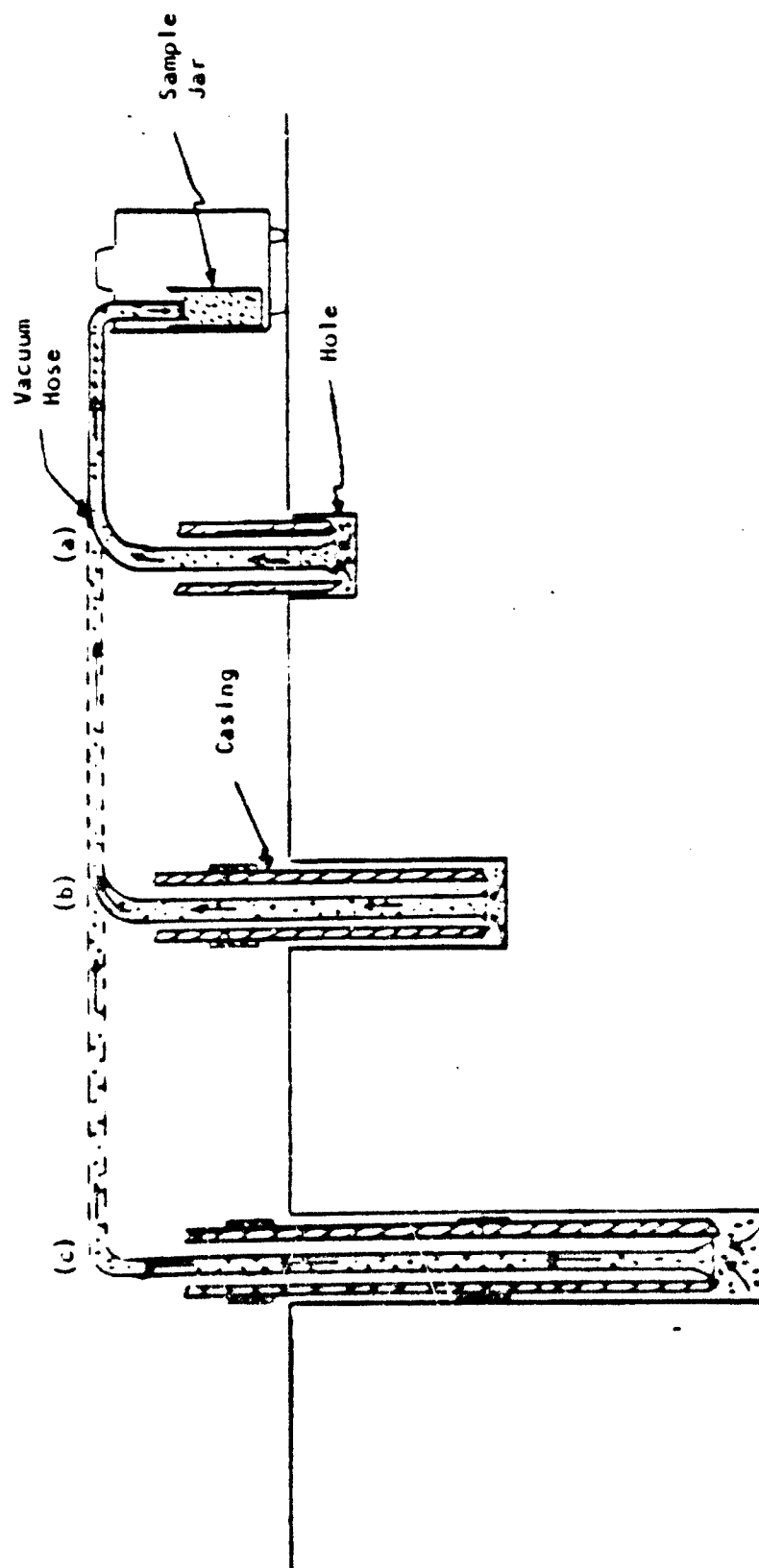


Figure 19. Vacuum-Lift Procedure for Posttest Soil Sampling.

recovered in a glass sample jar placed inside the "shop-vac" cannister. A minor modification was made to the shop-vac to ensure that the soil was discharged directly into the glass jar.

As soil was removed by the application of the vacuum, it was found that the black iron pipe was driven further into the hole. Using this procedure and adding additional 3-foot segments of pipe, it was possible to extend the hole to depths of 90 inches. Samples of soil were taken by placing the glass jar in the vacuum cannister whenever the bottom of the black iron pipe was moving through the desired sampling interval.

For the posttest samples, the length of two of the three sample intervals had to be reduced from 12 to 6 inches. Thus, samples were taken from 6-12 inch, 30-36 inch, and 60-66 inch intervals. Unlike the pretest samples, the posttest samples were taken with a diameter of 2.5 inches. It was found that if the samples were taken over the entire 12-inch length, large quantities of sample were recovered than required or necessary. Due to this reason and for operational convenience, it was decided to reduce the length of two sample intervals from 12 to 6 inches.

3. Composite Preparation and Soil Analysis

Selected individual soil samples obtained in the field were combined together to make composite samples. All samples from the same depth interval which belonged to the same type of sample location were combined together. In general, eight samples were combined per batch to make the composite. The mixing was done by rolling the soil in a 1-gallon glass jar placed on a roller. The mixing was performed in a cold room maintained at 4°C. After rolling the jar for 30 minutes, three samples of soil were removed. Then the jar was rolled for an additional 5 minutes and three more samples were removed. Duplicate samples were submitted for the analysis of moisture, volatiles, and semivolatiles. The analytical scheme is briefly summarized in the following subsection.

Composite samples of soil were submitted for the analysis of moisture, volatile aliphatics and aromatics, and semivolatile aliphatics and aromatics. Table 11 summarizes the types of analyses performed. The moisture and 104°C

TABLE 11. TARGET COMPOUNDS AND METHODS FOR ANALYSIS

Compound	Method of Determination
Moisture and 104°C volatiles	Weight loss in oven
Volatile aromatics	Purge and trap with GC/MS
Volatile aliphatics	Purge and trap with GC/MS
Selected chlorinated and other solvents	Purge and trap with GC/MS
Semivolatile aromatics	Steam distillation with GC/MS analysis of extract
Semivolatile aliphatics	Steam distillation with GC/MS analysis of extract
Hexadecane	Steam distillation with GC/MS analysis of extract

volatiles were obtained by observing the weight loss in a heated oven. Samples were heated at 104°C for 16 hours, cooled in a desiccator, and weighed. The same sample was replaced in the 104°C oven for an additional period of 1 hour, cooled, and reweighed to make sure that no further weight loss occurred.

Volatile aromatics and aliphatics were determined by a purge and trap analysis of a methanol extract of the soil sample. Samples were extracted by shaking 2.0 grams aliquots of soil with 40 milliliters of methanol in a wrist-action shaker. Surrogate standards were added to the soil prior to extraction. After extraction, the soil was separated from the methanol by centrifuging at 4000 rpm for 15 minutes. The extract was removed from the centrifuge tube and transferred to a 25 milliliter volumetric flask. Before adjusting the volume to 25 milliliters, an internal standard spike was added to the same flask. The extract was analyzed by removal of the volatile hydrocarbons from the liquid phase in a purge and trap device. The desorbed hydrocarbons from the trap were analyzed on a GC/MS system.

Information regarding the internal and surrogate standards is presented in Table 12. For purge and trap analysis, the surrogate standard was d-benzene and bromopentafluorobenzene was the internal standard. Aliphatics

were quantified by summing up the peak areas of those components having an ion with $m/e = 71$. Aliphatics were calibrated against octane. Aromatics were quantified by summing up the peak areas of those components having an ion with $m/e = 91$. Aromatics were calibrated against toluene.

Details of the analytical procedure are presented in Appendix B.

The soil was analyzed for semivolatile aliphatics and aromatics by extraction in a Nielson-Kryger steam distillation-extraction device. The prepared extract was injected into a GC/MS system for analysis. The concentration of hexadecane was also determined to track the removal of high boilers during the in situ decontamination experiment. As summarized in Table 12, surrogate and internal standards were spiked before analysis. The surrogate standard was d-decane and the internal standard was d-napthalene. The aliphatics were calibrated against hexadecane, ($m/e = 71$) while the aromatics were calibrated against d-toluene ($m/e = 98$). The details of the Nielson-Kryger extraction procedure are described in Appendix A, while the GC/MS procedures are described in Appendix C.

4. Sampling and Analysis of Soil for EP-Toxicity

Samples of soil were obtained from the trench area before excavation. These samples were analyzed by EPA standard method 1310 (Reference 5) to determine whether the trench area soil was hazardous, as per EP-Toxicity test, particularly for lead. This information was obtained to determine how the excavated soil would be disposed of if it was hazardous because of high concentrations of heavy metals.

Ten holes were made in the proposed trench area, and samples were retrieved from depth intervals of 6-12 inches and 30-36 inches. Samples were also obtained at depth intervals of 66-72 inches from four of these 10 holes. Samples from corresponding depth intervals were combined to make three composite samples. These samples were submitted to an outside laboratory for analysis of heavy metals by EPA method 1310. In addition to these composite samples, one sample from 6-12 inch depth and another from 30-36 inch depth were also submitted. The analysis report is attached in Appendix D, which

TABLE 12. SUMMARY OF INTERNAL, SURROGATE AND CALIBRATION STANDARDS

	Purge & Trap Data	Steam Distillation Data
Internal	Bromopenta fluoro benzene (m/e = 246)	d ₈ - naphthalene (m/e = 136)
Surrogate	d - benzene (m/e = 84)	d - decane (m/e = 50)
Aliphatics	Calibrated against octane (m/e = 71)	Hexadecane (m/e = 71)
Aromatics	Calibrated against toluene (m/e = 91)	d ₈ - toluene (m/e = 98)

shows that none of the submitted samples are hazardous due to the EP-Toxicity test procedure.

8. VAPOR AND CONDENSATE MONITORING

Vapor and condensate samples were collected and flow rates measured during the in situ decontamination field test. These data were obtained to monitor the progress of soil decontamination as well as to determine the amount, if any, of hydrocarbon emissions from the vapor-handling system. Because of electrical constraints and safety considerations, it is not feasible to obtain representative samples of soil from the heated zone during the treatment period. For this reason, the progress of soil decontamination can be indirectly monitored by measuring the flow rate and concentration of raw gases leaving the vapor collection zone.

Although it was not the purpose of this project to demonstrate an optimum vapor-handling system, it was considered desirable to monitor its performance by determining the rate of water and hydrocarbon condensation, the total gas flow through the system, etc. To meet the above objectives, a vapor and condensate monitoring plan was developed and executed during the field test. This plan is described below with the help of Figure 15 (Page 51), which shows the various sampling and measurement points.

1. Gas and Vapor Stream Measurements

Gas and vapor stream measurements were made at two locations. These locations are:

- raw gases in the manifolded vapor collection line
- residual gases leaving the carbon bed.

The measurements made at each location are described below.

a. Measurements at the Vapor Collection Manifold

Raw gases leaving the collection zone above the heated soil surface were analyzed by sampling in the heat traced manifold conveying gases to the inlet of the heat exchanger. A gas sample was withdrawn from the manifold through a heated sample line. The gases were cooled and moisture and hydrocarbons were collected in two cold traps placed in a dry ice/acetone bath. Figure 20 shows the sampling train used for obtaining these samples. The condensed sample was recovered from the traps and analyzed on GC/FID system and calibrated against a JP-4 jet fuel sample. These results were converted to JP-4 concentration in the raw gas manifold.

The total flow rate of the gases in the raw gas manifold was measured by means of a pitot tube. Static pressure in the manifold was also obtained at the same time.

The raw gases were also analyzed by inserting Draeger tubes into the raw gas line. Draeger tubes for petroleum hydrocarbons (equivalent octane), benzene, and trichloroethylene were used.

b. Measurements at the Outlet of the Carbon Bed

The total gas flow leaving the carbon bed was measured by inserting a pitot tube in the line. At the same time, static pressure measurement was also obtained. The gas stream was analyzed for petroleum hydrocarbon concentration by inserting a Draeger tube into the gas line.

2. Condensate Measurements

Condensate produced by cooling the gas stream was collected in drums, as shown in Figure 15. The liquid level in the drum was monitored as a

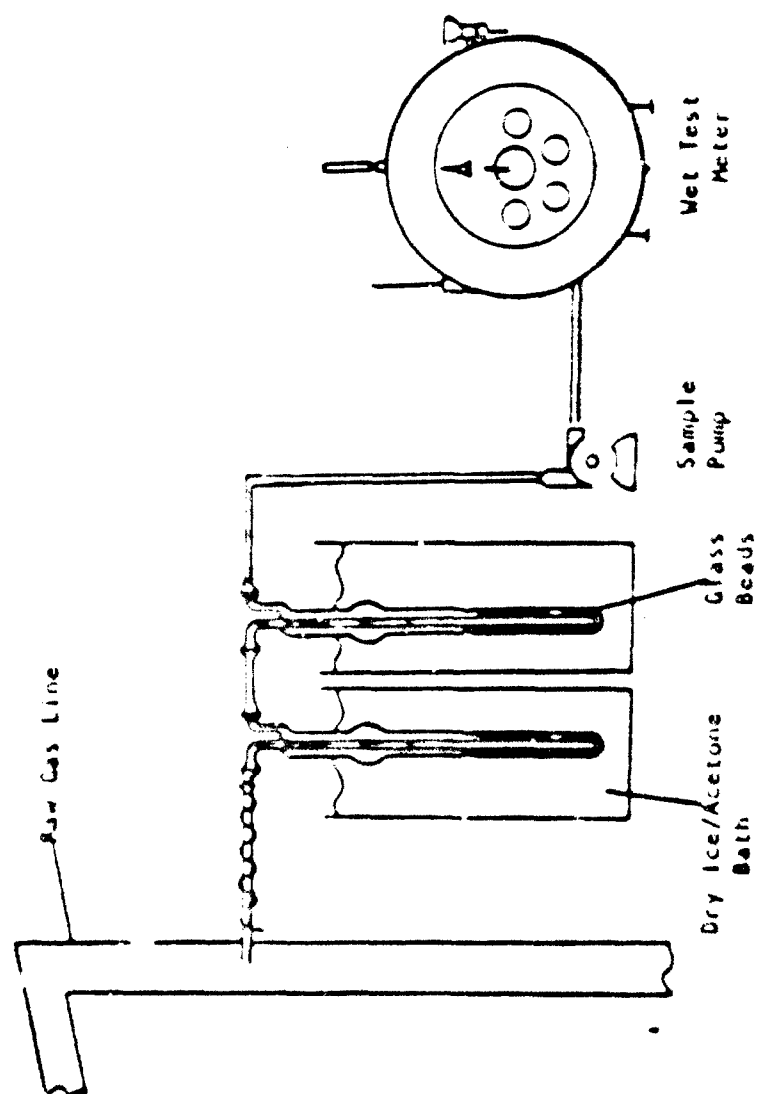


Figure 20. Raw Gas Sampling Train.

function of time. The levels of aqueous phase and the floating hydrocarbon layer were separately monitored by inserting a dipstick coated with a dye sensitive to the presence of the water layer. The location of the water/hydrocarbon interface was detected by means of the dye. The volume of the hydrocarbon layer was then determined by taking the difference between the total liquid height and the height of the water/hydrocarbon interface.

Samples of the condensate phase were obtained from each drum. The aqueous and hydrocarbon layers from selected samples were analyzed for total organic halides (TOX) by EPA standard method 9020 (Reference 5).

SECTION VI
FIELD TEST RESULTS

A. FIELD TEST PROCEDURE AND CHRONOLOGY

The sequence of events during the various field tasks is summarized in Table 13. Before field activities began, a briefing was given to Wisconsin Department of Natural Resources to obtain permission to perform the test. Based on their comments appropriate changes were made in the test monitoring and sampling plan. In August 1987, soil samples were obtained from the proposed trench area to determine whether the soil is hazardous as per the EP-Toxicity procedure for heavy metals.

TABLE 13. CHRONOLOGICAL SEQUENCE OF FIELD TEST EVENTS

Activity	Dates
• Briefing to Wisconsin DNR	6/16/87
• Sampling for EP-Toxicity test	8/5
• Pretest sampling	9/9-9/10
• Site preparation	10/6
• Decontamination test started	11/3
• Reached 100°C	11/5
• Tracer studies	11/11
• Grab samples taken	11/12
• Test termination	11/15
• Posttest sampling	12/3-12/9
• Site closure	12/11

Pretest soil sampling from the heated zone was performed in September 1987. These samples were returned to Chicago for analyses in IITRI laboratories. The site preparation was begun on October 6, 1987 with the installation of AC power lines. The complete heating system was installed and ready to run by November 2, 1987. The test was begun on November 3, 1987.

The average temperature of the test-volume had reached 100°C by November 5. By November 11th, the test volume had achieved the desired average temperature of 150-160°C. At this time Halon® tracer studies were performed to determine whether soil gas was migrating into the heated zone from the surrounding cool zones. On November 12, approximately 30 hours after achieving the desired final temperature, four grab samples were obtained from the heated zone to assess the progress of soil decontamination. These grab samples were obtained from a depth of approximately 12 inches, just inside each of the two ground planes. The results of grab sample analyses indicated that 84 percent of the aliphatics and 99.1 percent of the aromatics had already been removed from the ground plane area. Because of these excellent results, it was decided to terminate the test, in accordance with the test plan, after 4 days at the final temperature. The test was terminated on November 15, 1987. The vapor-handling system was turned off on November 16, because of concerns that continued venting would promote the migration of contaminated soil gas into the "clean" test volume.

The heated zone was allowed to cool down until December 2, 1987. Posttest sampling was performed between December 3 and December 9. During the same period, the equipment was disassembled and packed for shipment to Chicago. The site was closed on December 11, 1987.

B. SOIL TEMPERATURE DISTRIBUTION

As described in Section IV, a total of 49 temperature measurement points were available in the heated zone and the trench area. Figure 21 is a plan view of the heated array which shows the various sections and planes in which temperature distribution data is available. Temperature data are presented with the help of Figure 21.

In Figure 21, the plane BB runs through the excitor row of electrodes and the two outer thermowell holes. One hole is in the "trench area," 1 foot away from the last excitor electrode and the second hole is 2.5 feet removed from the last excitor electrode. Similarly, plane CC runs through the ground plane Row 1. The Plane DD defines temperature distribution along the row of three thermowell holes placed halfway between excitor row and ground plane Row 3.

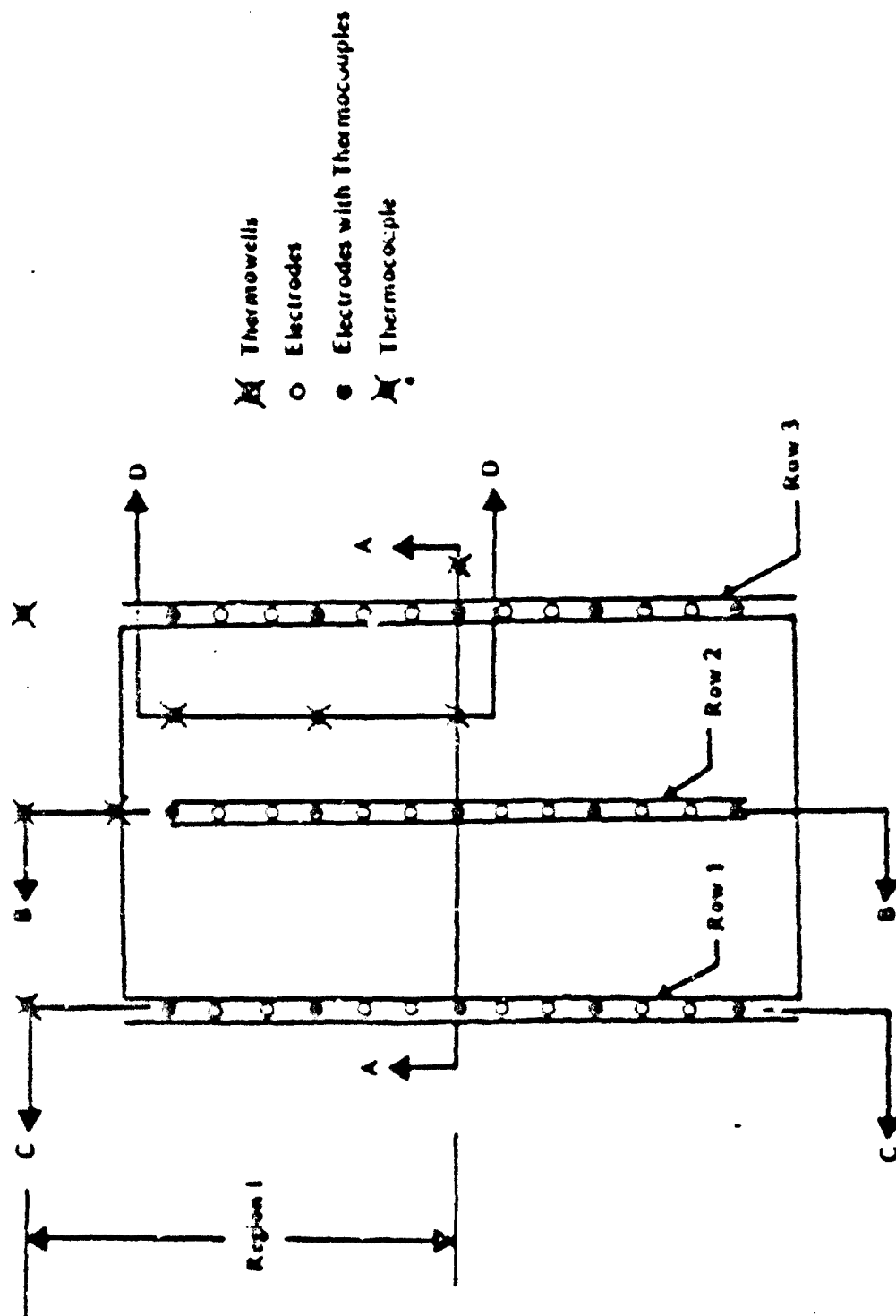


Figure 21. Location of Temperature Measurement Boreholes.

Temperature data in the transverse plane AA is available at a depth of 36 inches below the surface. This includes a temperature monitoring point located in the "trench area" outside Row 3.

The soil temperature data at the midpoint (depth 36 inches) of plane BB is shown in Figure 21, as a function of distance along the electrode row. The location of the first and last electrodes is marked by 0 and 12 feet, respectively. The location of the two thermowells is at 13 and 14.5 feet from the first excitor-row electrode. This figure shows a generally symmetric temperature profile about the center of the electrode plane, marked by the seventh electrode located at 6 feet. Two hot spots, one centered on the first electrode and the other on the 13th electrode, are seen as originally predicted in the design of the array. In the unheated zone beyond 12 feet a sharp drop in soil temperature is observed, nevertheless the soil was heated by conduction. The temperature in the trench area reached 100-105°C range by 144 hours and stayed at that level through the end of the test.

The next three figures present the temperature data in plane DD at three different depths. Figure 23 shows the temperature along plane DD at the depth of 9 inches. As shown, the near surface temperatures at locations between the excitor and ground planes reached a temperature of 165°C by the end of the test.

A similar temperature distribution was observed at the depth of 36 inches in plane DD, as illustrated in Figure 24. Temperatures at a depth 72 inches in plane DD are shown in Figure 25. These data show that the maximum temperature attained at depth of 72 inches was in the range of 150-157°C.

The temperature distribution along the plane of Row 3 is illustrated in Figure 26. These data show that at a depth of 36 inches, the ground plane electrodes had achieved a temperature of 95°C after 3 days of heating. By the time the test ended, these temperatures had gone to 100-105°C range.

Additional temperature distribution data are presented in Appendix E.

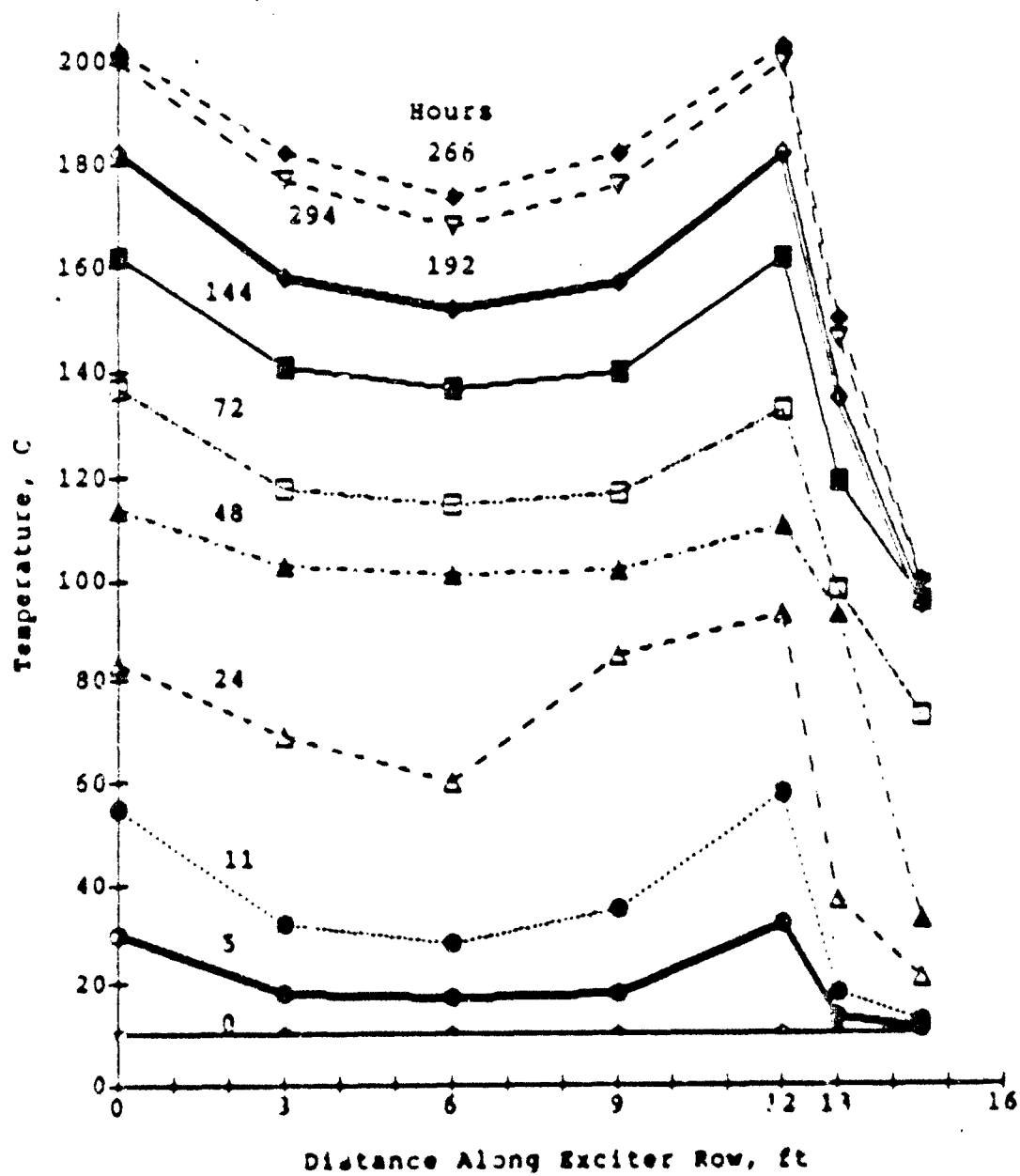


Figure 22. Temperature Distribution Along Exciter Row
Plane BB, Depth 36 inches.

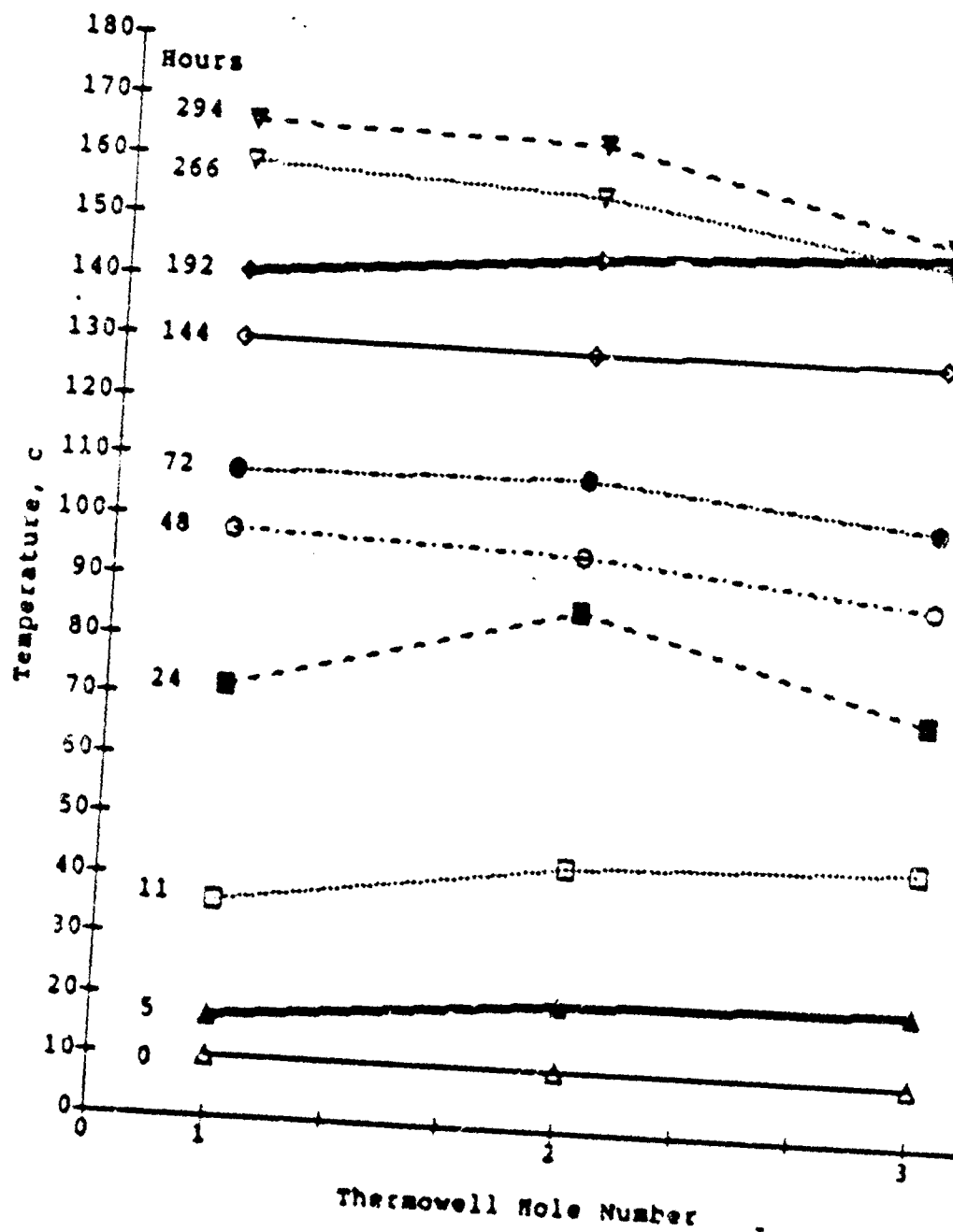


Figure 23. Temperature Distribution Along Plane 00 at 9 inch Depth.

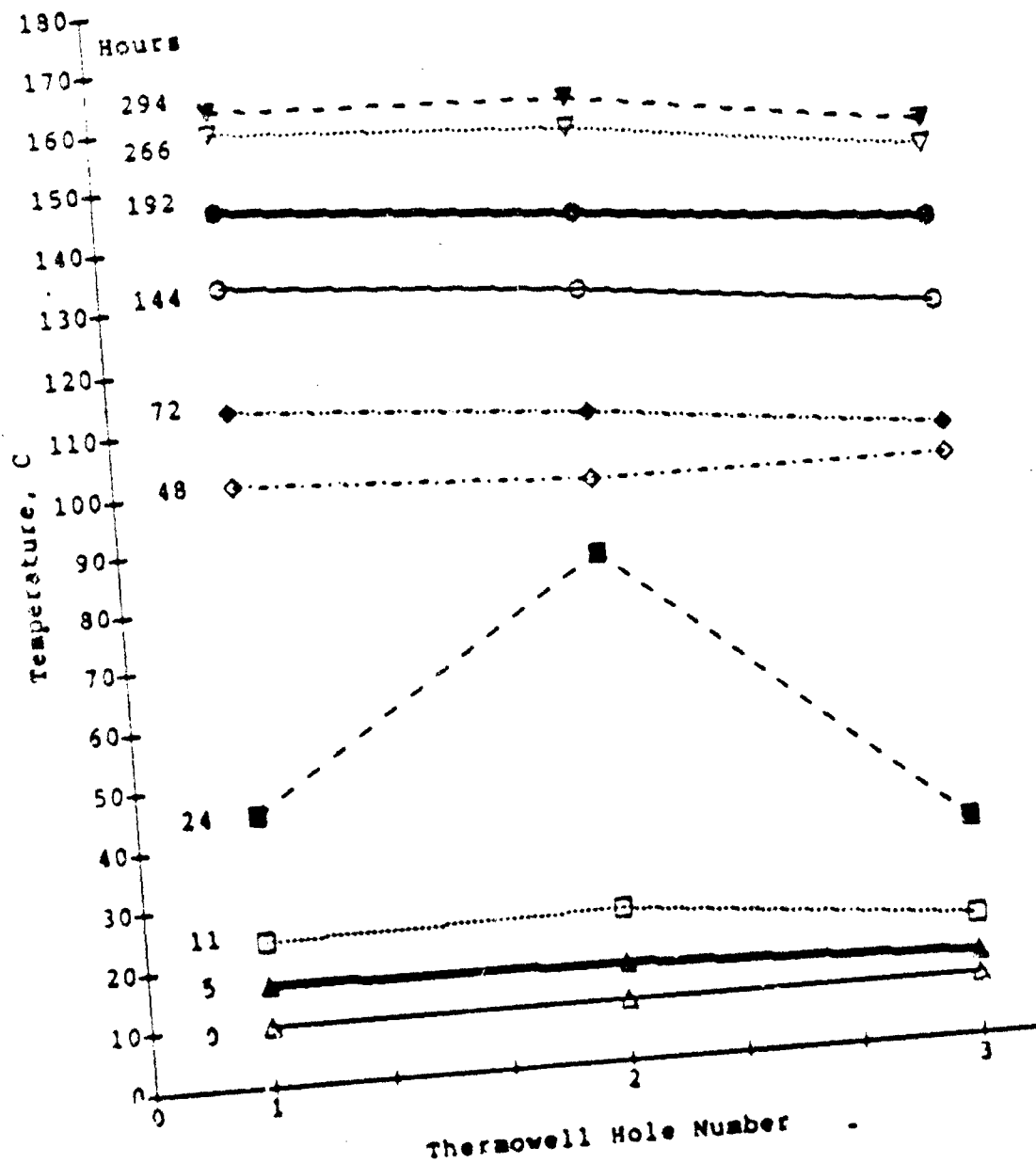


Figure 24. Temperature Distribution Along Midpoint (36 inch) of Plane D0.

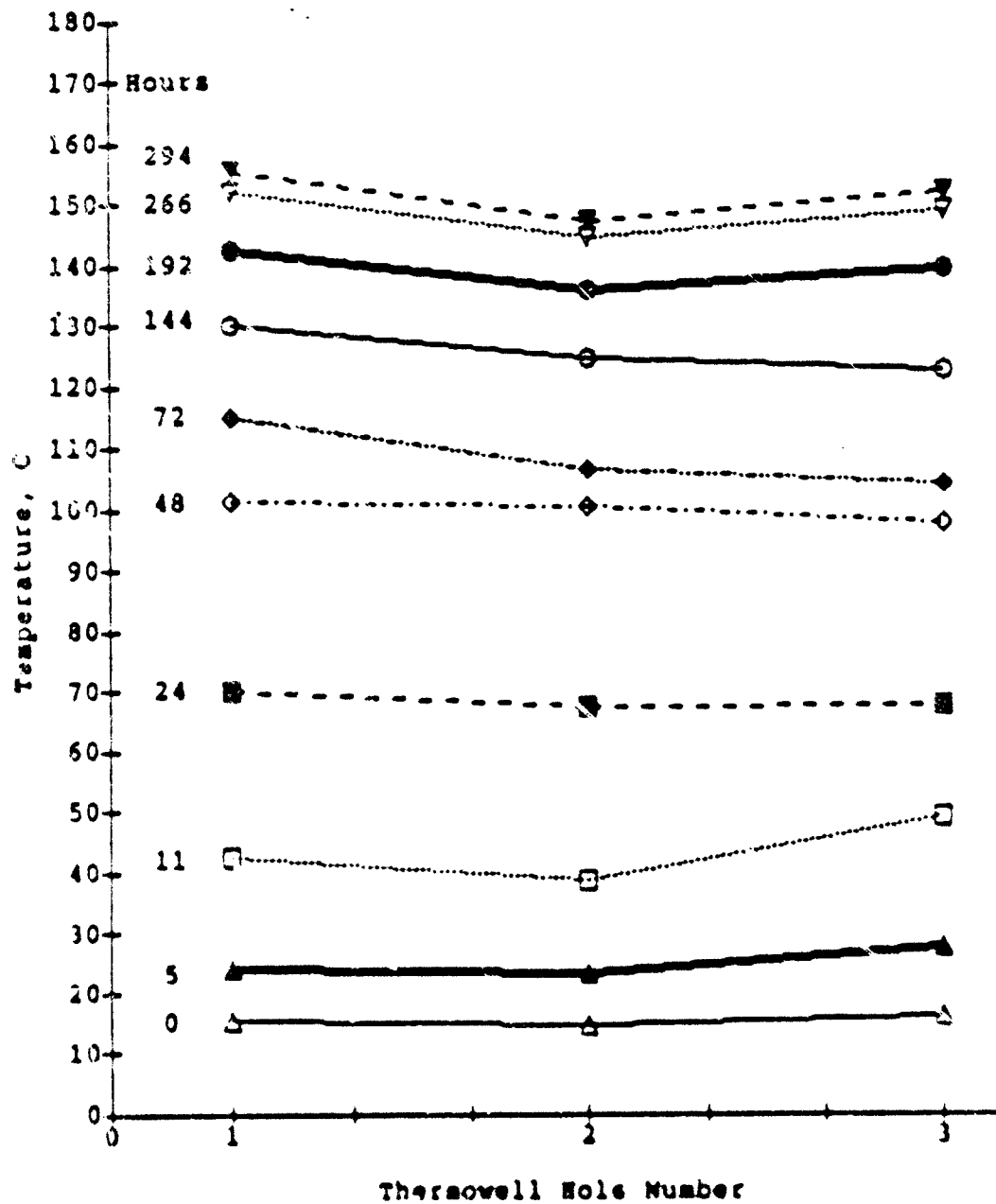


Figure 25. Temperature Distribution Along Plane 00 at 72 Inch Depth.

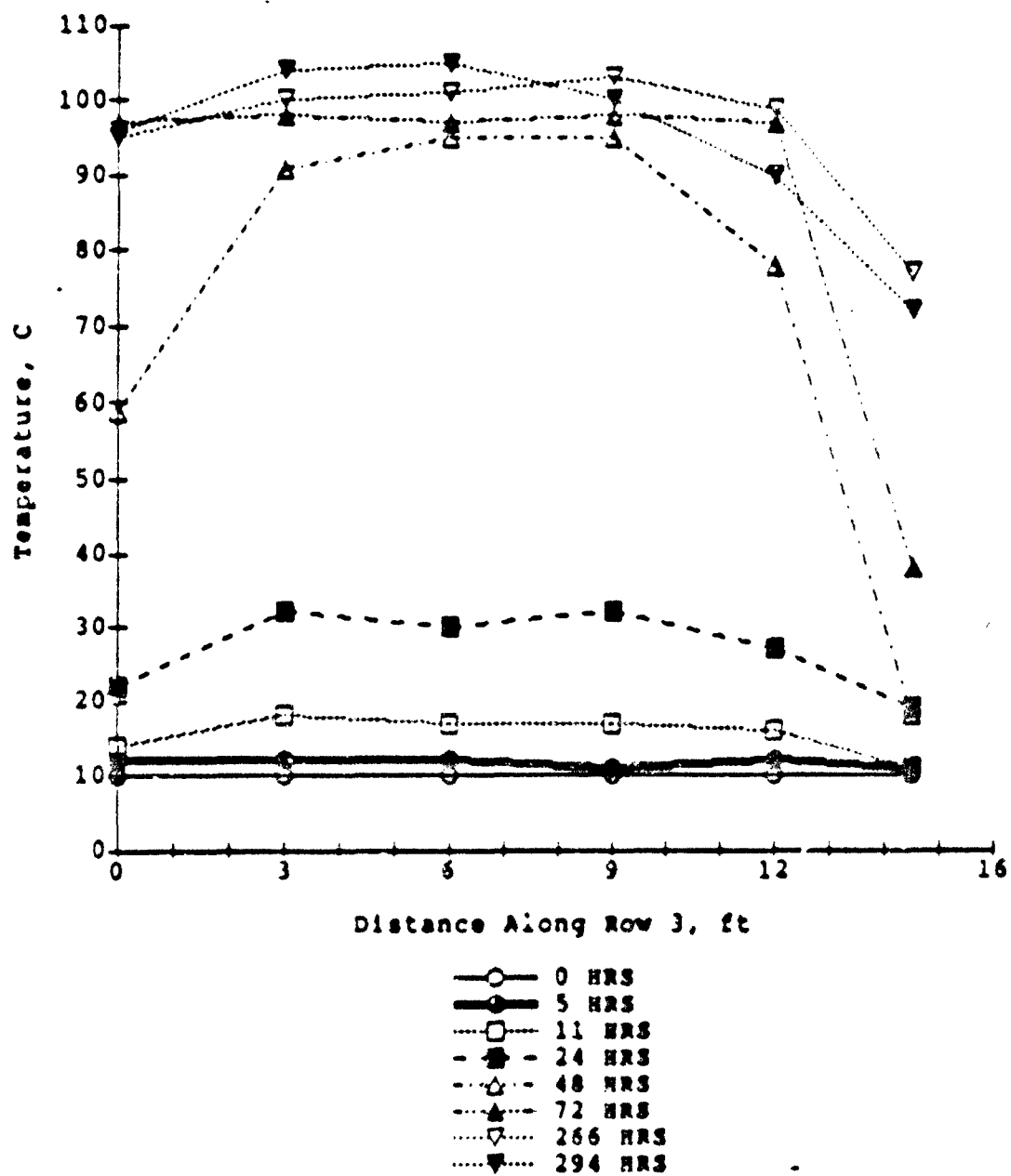


Figure 26. Temperature Distribution Along Ground Plane 3 (Row 3) Midpoint.

C. SOIL DECONTAMINATION RESULTS

Soil samples, obtained according to the plan described in Section V, were analyzed to determine the initial and final concentrations of the contaminants. Purge and trap analyses were performed on extracts of composite soil samples as described in Section V. Volatile aromatic and aliphatic concentration was obtained by this analysis. Steam distillation of composite soil samples was performed to determine the concentration of semi-volatile aromatics, aliphatics, and hexadecane in the soil before and after treatment.

As discussed in Section V, 72 different soil samples were obtained from three different depth intervals from each of 24 different sample holes located in the heated zone. These 72 samples were combined together to give nine different composite samples. Each composite sample was extracted and analyzed in duplicate.

The average concentration of the contaminants in the heated zone was calculated by the following equations; average concentration of contaminant ($\bar{X}$) in the test volume is given by:

$$\bar{X} = \frac{1}{mn} \sum_{i=1}^n \sum_{j=1}^m X_{ij} \quad (6)$$

where n: is the total number of composite samples (nine)

m is the number of individual samples analyzed from each composite sample (two)

X_{ij} concentration of contaminant from each sample.

The standard deviation of the composites is given by:

$$S = \left(\frac{1}{n-1} \sum_{i=1}^n (\bar{X}_i - \bar{X})^2 \right)^{1/2} \quad (7)$$

where $\bar{X}_i = \frac{1}{m} \sum_{j=1}^m X_{ij}$

The decontamination efficiency is given by:

$$\text{Decontamination Efficiency} = \frac{\bar{X}_{\text{pretest}} - \bar{X}_{\text{post test}}}{\bar{X}_{\text{pretest}}} \times 100 \quad (8)$$

The above equations can also be used for the determination of average concentrations and removal efficiencies for the "trench" area samples and in each of the three different depth intervals from which soil samples were obtained.

1. Removal of Volatile Contaminants

The percentage removal of volatile aliphatics and aromatics was determined from the equations presented above and the purge and trap analyses data of individual composite samples. In Figure 27 the percentage removal is presented for the entire heated zone, as well as for each of the three depth intervals. The overall removal for aliphatics was 99.3 percent while that for aromatics was 99.6 percent.

Examination of the data in individual depth intervals indicates that in the near surface zone of 6-12 inches, aliphatics removal was 98.2 percent. The removal of aliphatics increases with depth, as shown in Figure 27. The aromatics removal was fairly uniform with the depth of the heated zone, ranging from 99.2 to 99.9 percent.

The low aliphatics removal in the near surface regions could be due to lower final temperature in the near surface areas, as shown by Figure 28, in which temperature at selected exciter plane locations is plotted as a function of time. These data show that at a depth of 12 inches, the temperature was significantly lower than at 72 inches. Similar data is shown for selected ground plane locations in Figure 29. Prior site characterization work done by USAF has indicated that there is a concentration of high boiling hydrocarbons in the near surface zones. These probably represent the heavy lubricating oils improperly disposed of at the site prior to fire training drills.

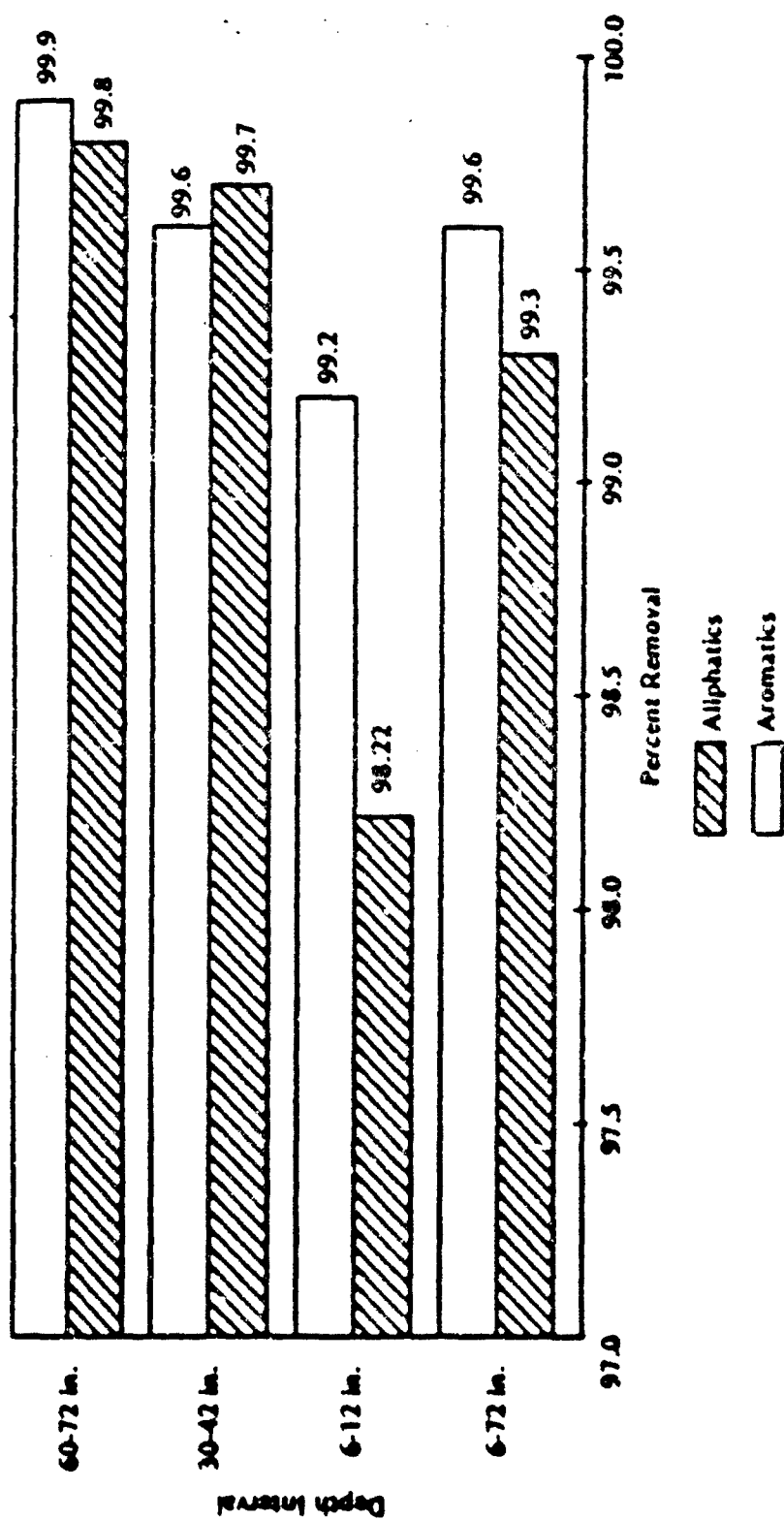


Figure 27. Volatile Removal (Purge-and-Trap Data).

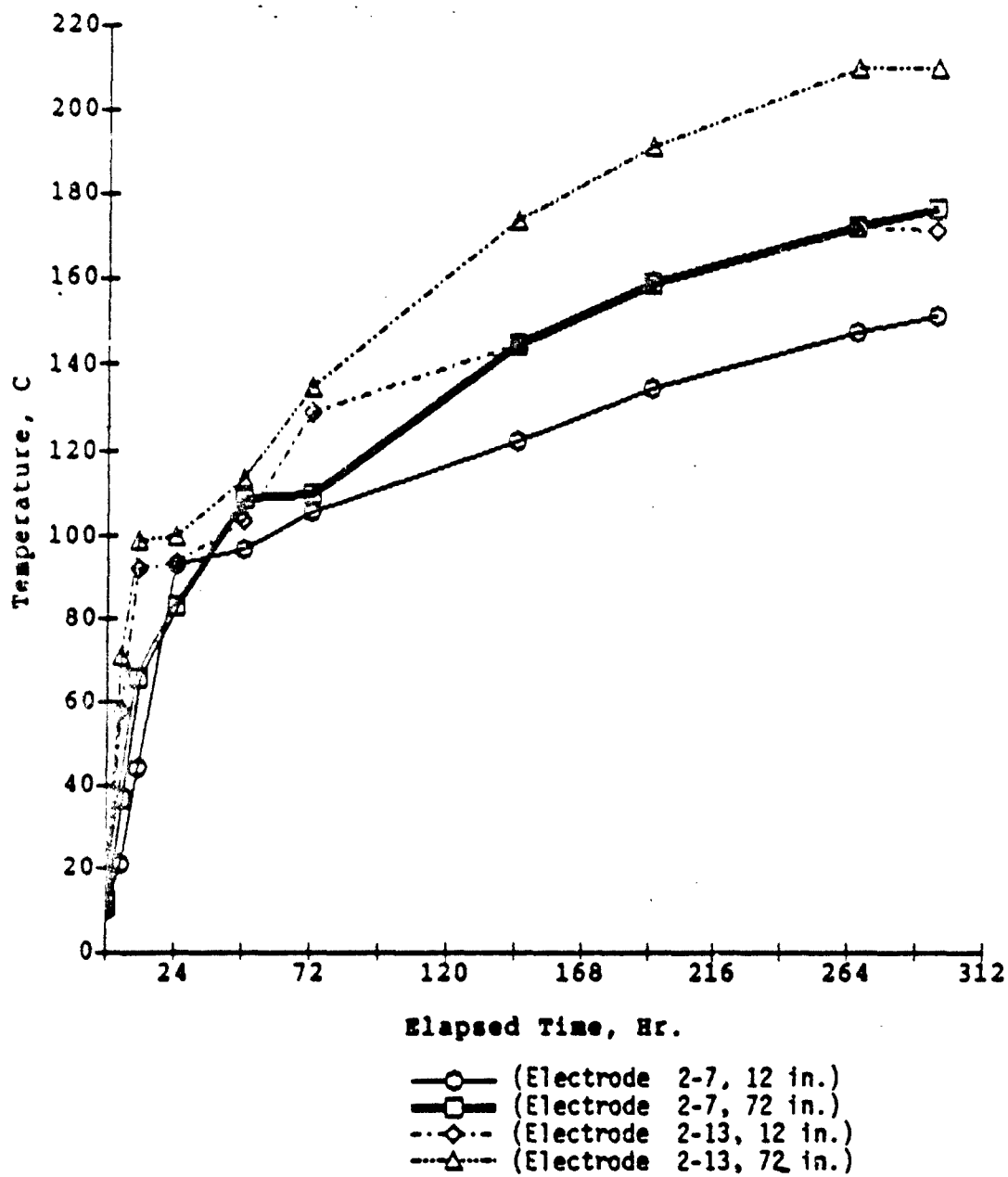


Figure 28. Rate of Temperature Rise in Selected Exciter Electrodes.

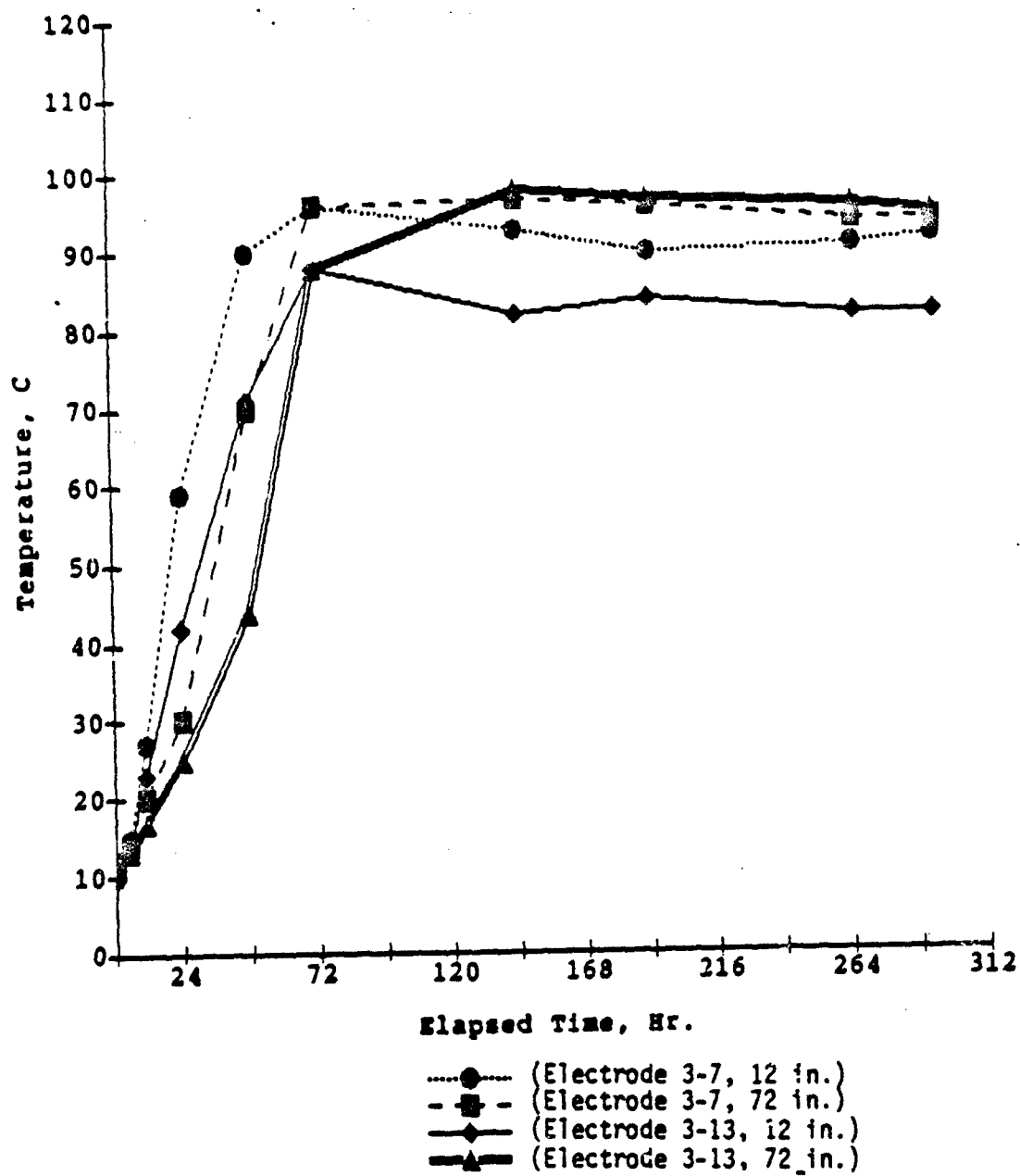


Figure 29. Rate of Temperature Rise in Selected Row 3 Electrodes.

Another reason for lower recovery from the near surface cool zones is that 24 hours after the RF power was switched off, the vapor collection fans were also switched off to minimize inward migration of fluids from the surrounding cool zones. In absence of the vacuum, vapors rising up from the deeper, hotter zones could easily condense in the surface regions instead of being collected through the gas collection manifolds placed on the soil surface.

The before and after concentration of volatile aliphatics in the heated zone is illustrated as a function of depth in Figure 30. The initial aliphatics concentration in the near surface zones is higher by a factor 1.3 to 1.6 than the concentration at deeper locations.

The final average concentration of aliphatics in the entire heated zone was 28 ppm. A range of 8 to 65 ppm was observed in the depth range of 6 to 72 inches. The before and after concentrations of volatile aromatics are shown in Figure 31 as a function of depth. The initial average aromatic concentration was 212 ppm, and the final average concentration in the heated zone was less than 1 ppm.

The standard deviation of the before and after concentrations was calculated by using Equation (4). The relative standard deviations were then calculated based on the average concentrations as determined by Equation (3).

The relative standard deviation for the analysis of volatiles is presented in Table 14.

TABLE 14. PERCENT RELATIVE STANDARD DEVIATION
FOR VOLATILE CONTAMINANTS

Depth Interval, in.	Pretest		Posttest, %	
	Aliphatics	Aromatics	Aliphatics	Aromatics
6-72	24.7	28.3	103	129
6-12	12.4	11.3	-	-
30-42	11.7	13.2	-	-
60-72	11.9	13.4	-	-

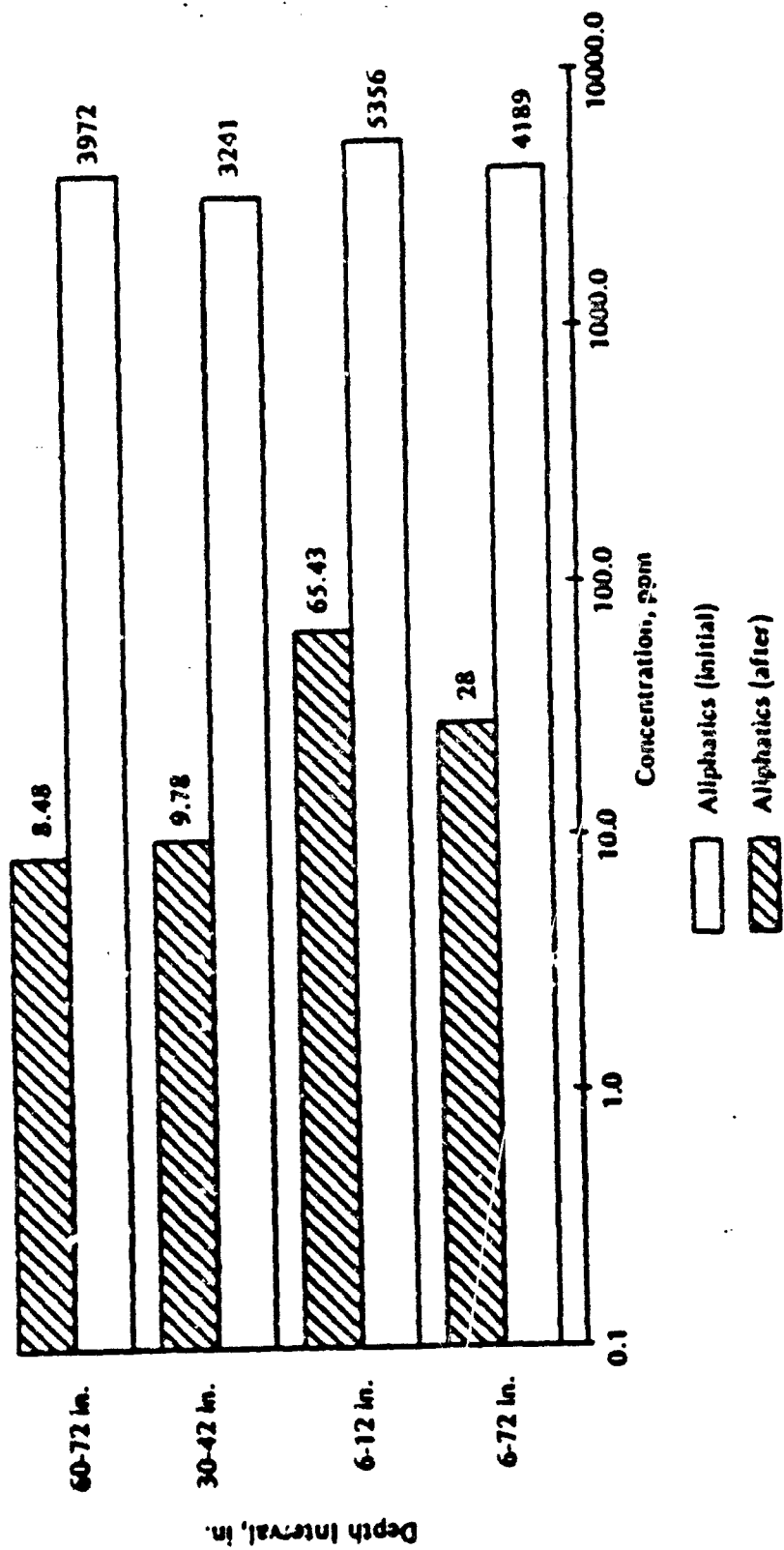


Figure 30. Before and After Concentration of Volatiles Aliphatics.

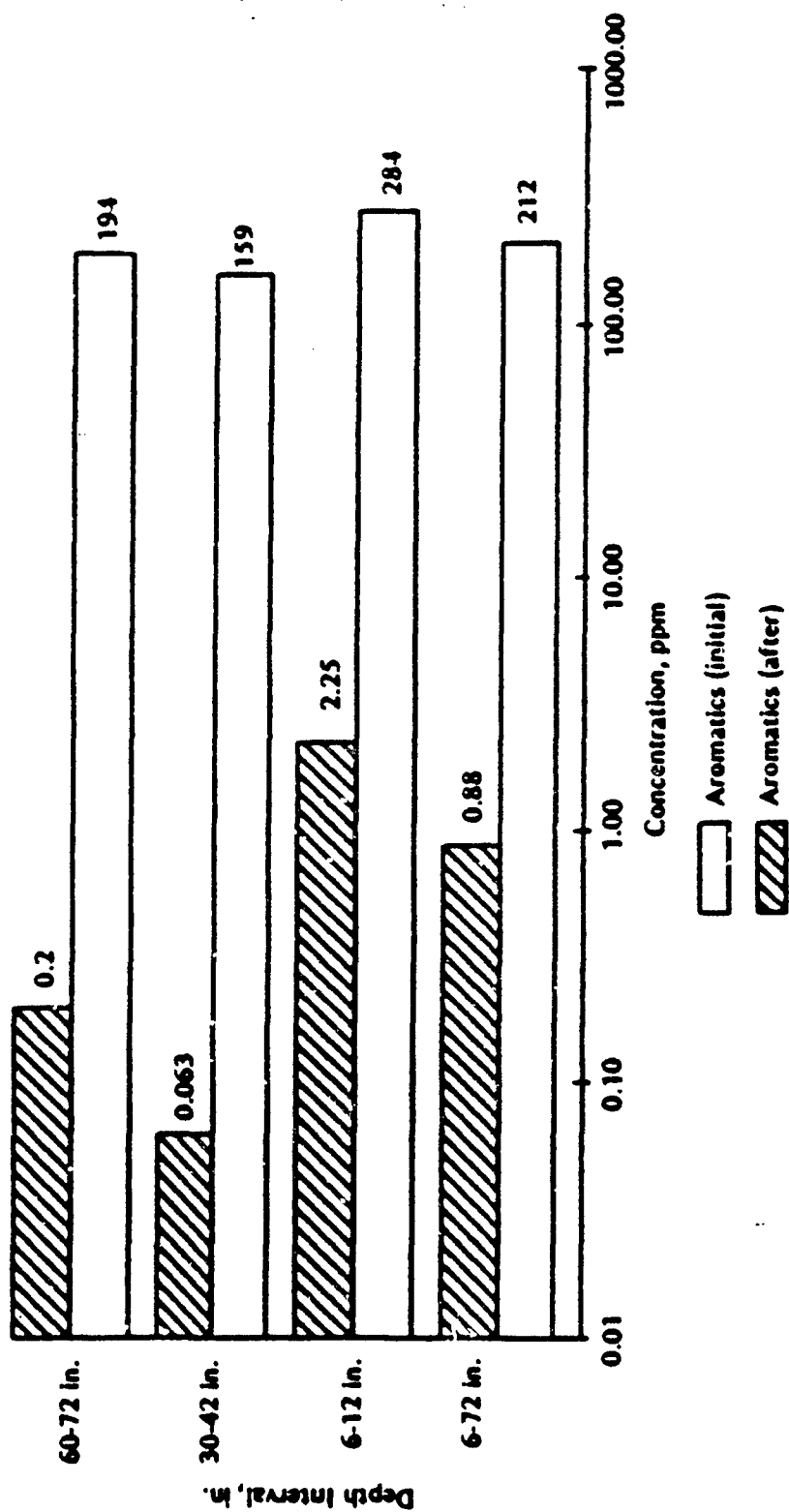


Figure 31. Before and After Concentration of Volatile Aromatics.

The data for relative standard deviation includes the total variability in the analysis due to all sources of error including spatial distribution variations in the soil. For posttest samples, the overall standard deviation in the depth interval of 6-72 in. is available, but sufficient data for estimating relative standard deviation in the three depth intervals is not available.

2. Removal of Semivolatile Contaminants

Semivolatiles were analyzed by steam-distillation of the composite soil samples as described in Appendices A and C. Concentrations for aliphatics, aromatics and hexadecane were obtained.

The percentage removal of semivolatiles is illustrated in Figure 32. The overall removal for aliphatics, hexadecane, and aromatics was 94.3, 82.9 and 99.1 percent, respectively.

As noted earlier, the removal of aliphatics from the near surface zones was significantly less than the overall removal from the entire heated zone. The reason for this is the lower near surface soil temperature as discussed in the previous subsection.

The before and after concentrations for aliphatics, hexadecane and aromatics is illustrated in Figures 33 through 35. The average final concentrations for aliphatics, hexadecane, and aromatics were 94.5, 5.4, and 2.3 ppm, respectively. These data illustrate how a high-boiler like hexadecane (n.b.p 287.5°C) can be removed even at temperatures much below its boiling point. This has occurred due to the long residence time at lower temperatures and due to the effect of steam and gas sweep generated by the vaporization of lower boiling material present in the soil.

The standard deviation of the before and after concentrations of the semivolatile contaminants was calculated by Equation (4). The relative standard deviations are presented in Table 15.

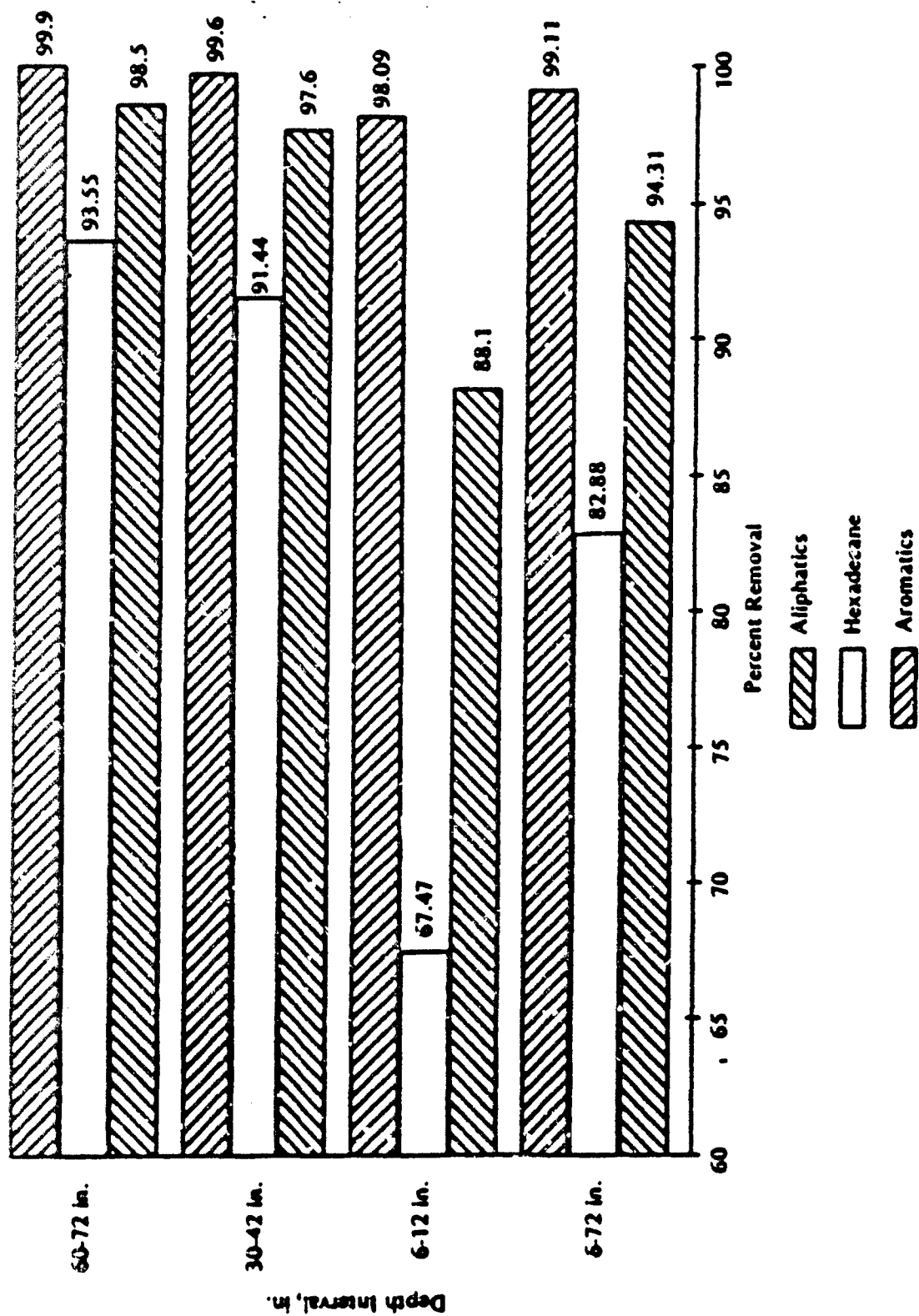


Figure 32. Semivolatile Removal.

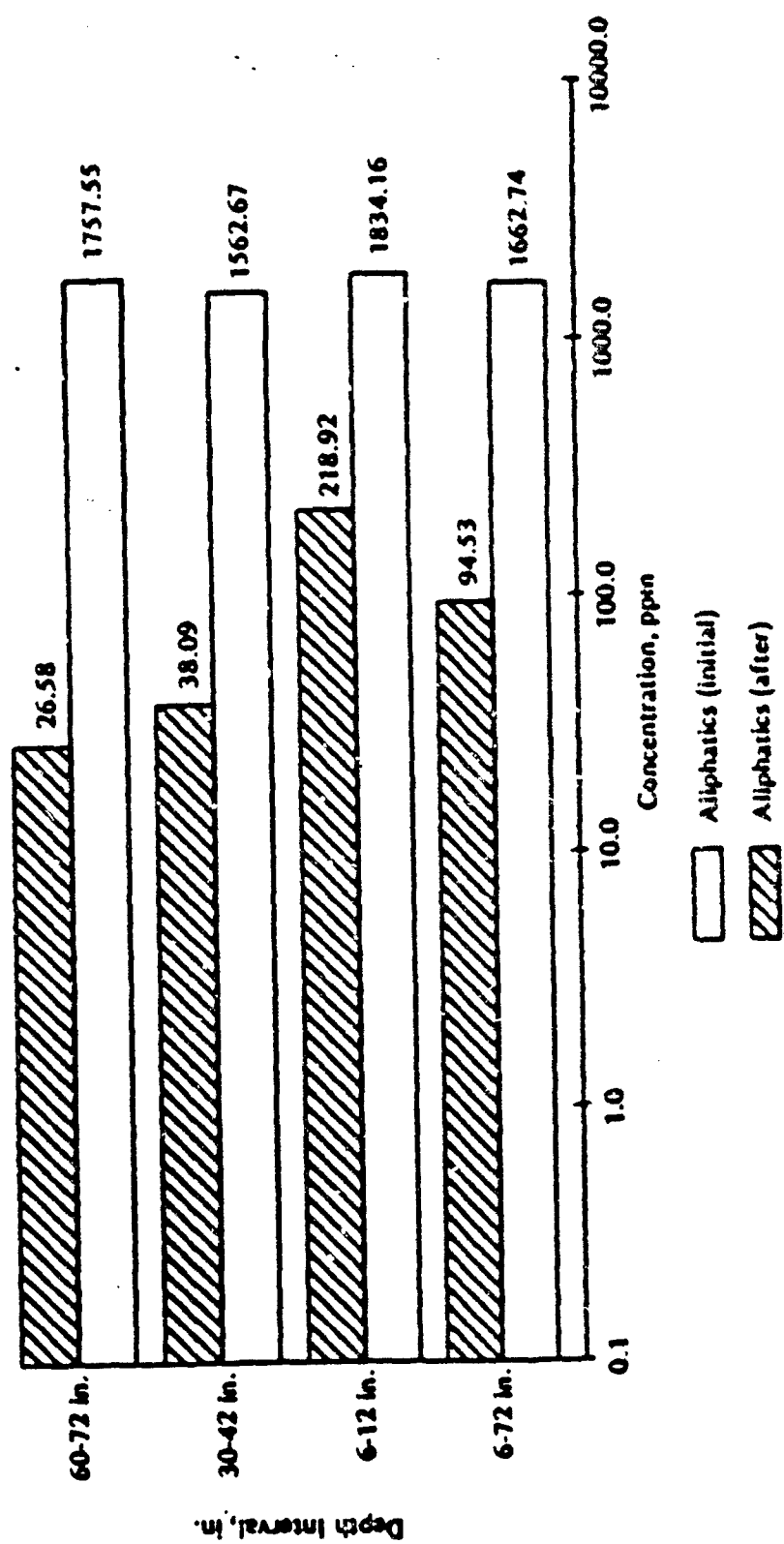


Figure 33. Before and After Concentration of Semivolatile Aliphatics.

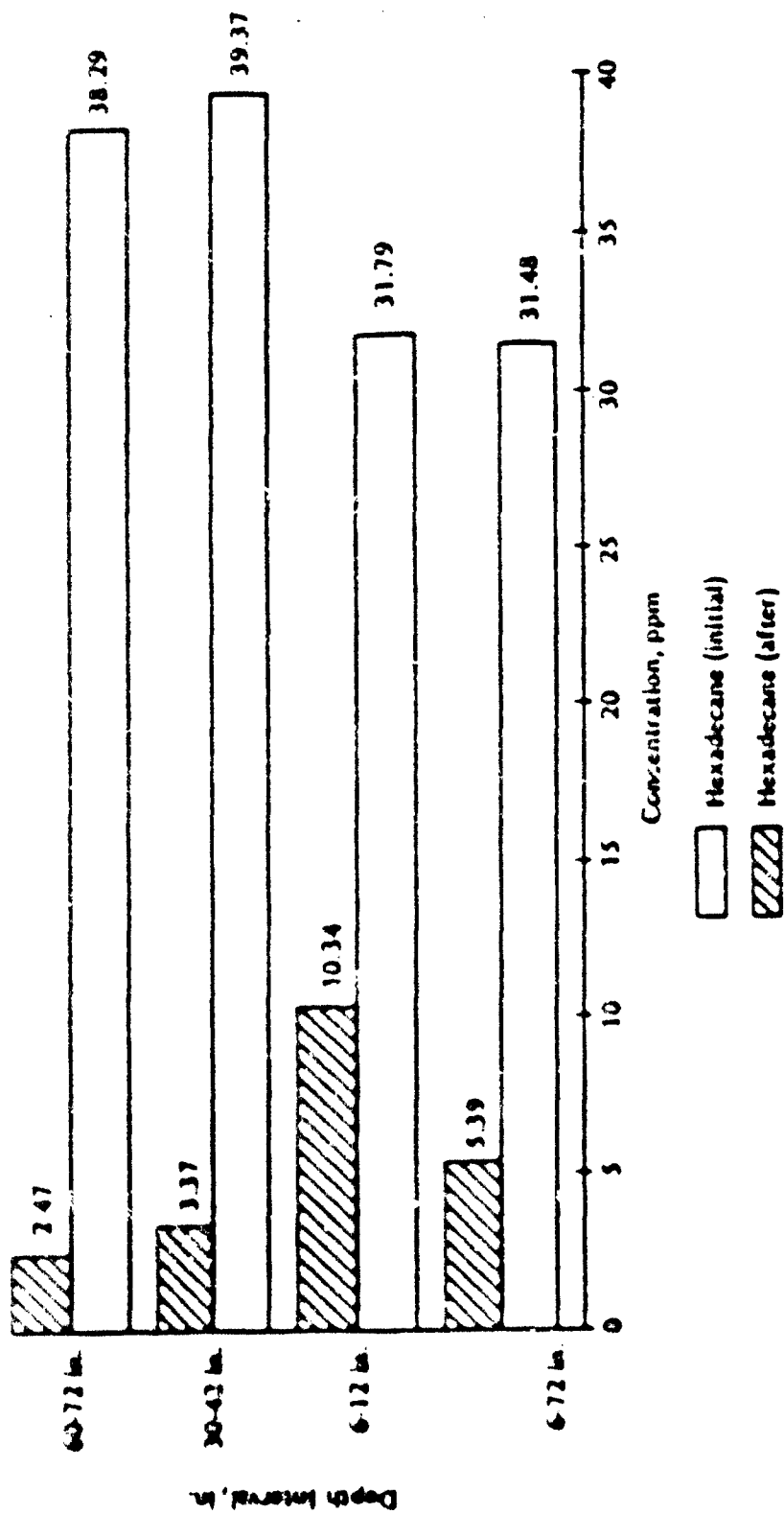


Figure 34. Before and After Concentration of Hexadecane.

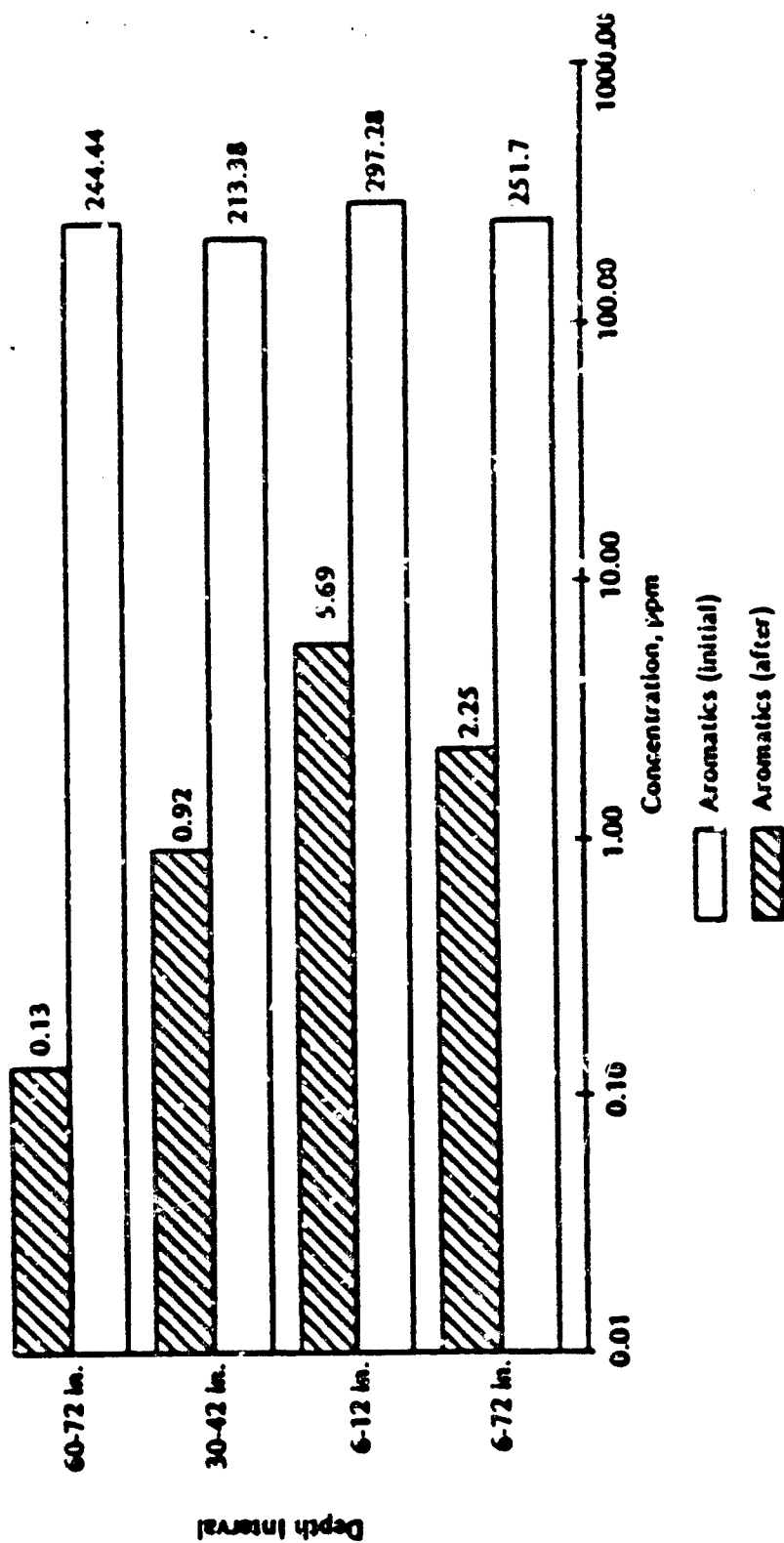


Figure 35. Before and After Concentration of Semivolatile Aromatics.

TABLE 15. PERCENT RELATIVE STANDARD DEVIATIONS
FOR SEMIVOLATILE CONTAMINANTS

Depth Intervals, in.	Pretest			Posttest		
	Aliphatics	Aromatics	hexa- decane	Aliphatics	Aromatics	Hexa- decane
6-72	18.2	25.6	28.7	121.5	103.4	77.8
6-12	7.8	29.7	20.8			
30-42	9.3	16.0	15.7			
60-72	28.5	30.9	23.3			

The data for the relative standard deviation includes the total variability in the analysis due to all sources of error including spatial distribution variations in the soil. For the posttest samples, overall standard deviation in the depth interval of 6-72 in. is available, but sufficient data for individual depth intervals is not available.

3. Removal From "Trench Area"

Soil samples obtained from the "trench" area were analyzed by the purge and trap procedure to determine the before and after concentration in this area. The purpose of performing these analyses was to determine whether there was any net migration of contaminants from the heated zone into the soil in the immediate vicinity of the heated zone. Contaminant removal from the "trench" area is shown in Figure 36, as a function of depth. The overall aliphatics removal was 76.8 percent and the aromatics removal was 70.6 percent. These data indicate that there was no net migration into the "trench" area from the heated zone.

4. Tracer Experiments

A tracer was injected 4 feet outside the ground plane Row 1, at a depth of 5 feet. The purpose was to determine whether the tracer fluid migrates towards the heated zone. The raw gases in the gas collection manifold were sampled and analyzed as a function of time to determine the presence of the tracer in the heated zone.

Dibromotetrafluoroethane, sold under the brand name of Halon® 2402 by Great Lakes Chemical Corporation was used as the tracer. Halon® 2402 is a

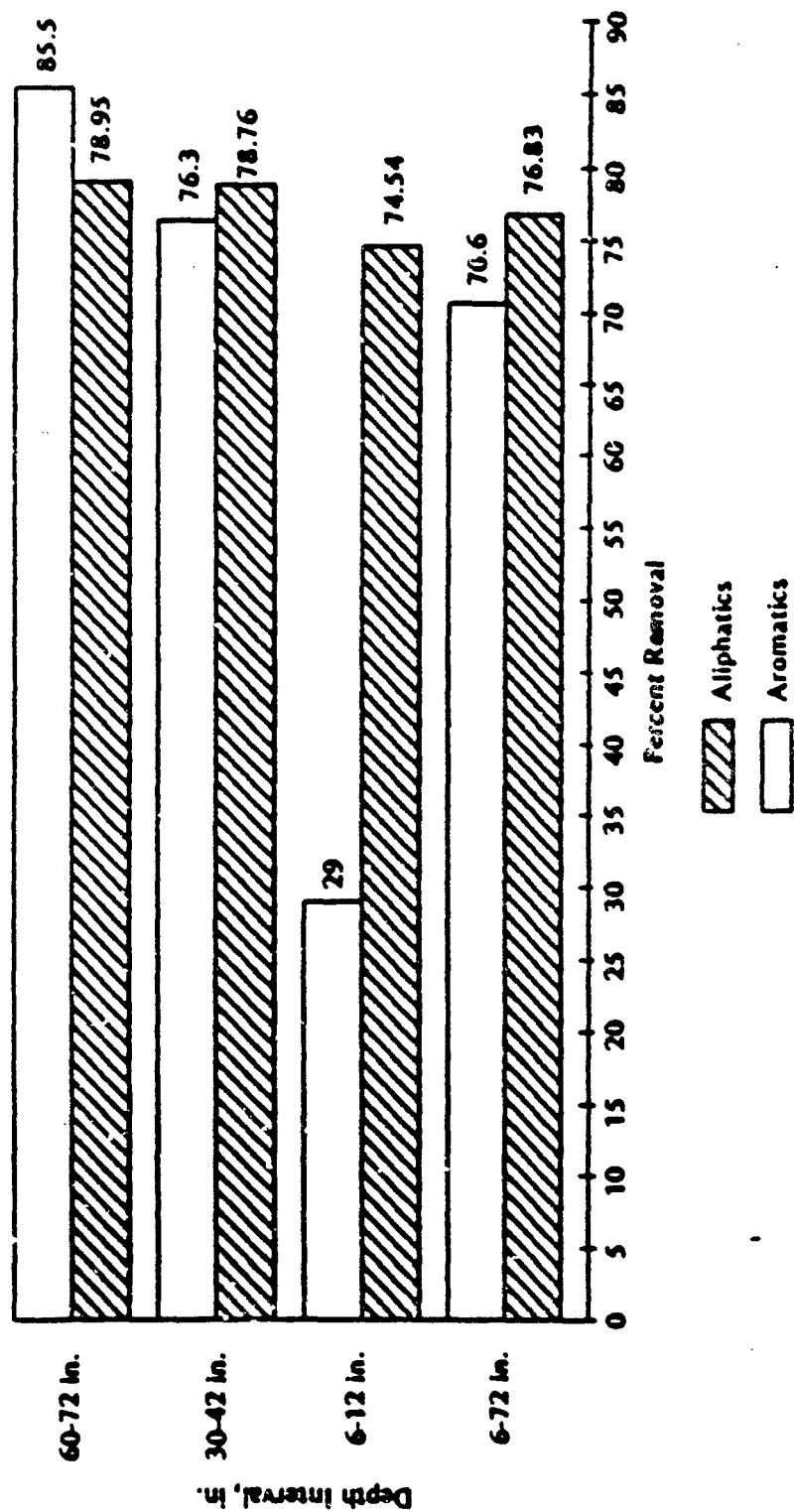


Figure 36. Removal of Volatiles from the "Trench" Area.

liquid at room temperature, boiling at 47.3°C. The vapor is 8.97 times heavier than air. Approximately 5 milliliters of the tracer liquid was injected in a hole 4 feet outside Ground Plane Row 1 and at a depth of 6 feet. The presence of Halon® 2402 was monitored in the raw gas stream leaving the vapor barrier by injecting gas samples in a GC equipped with an electron capture detector. A strong Halon® peak was detected 107 minutes after introduction of Halon® into the soil outside the heated volume. Gas samples taken 5 and 48 minutes after introduction of the tracer did not show any Halon® present in the raw gases leaving the soil surface.

The results of the tracer experiment and the analysis of pre- and posttest "trench" area samples show that there was no net migration of contaminants from the heated zone into the immediate vicinity of the test volume. In fact, tracer experiments showed that fluids from as far as 4 feet outside the heated zone were migrating into the heated volume.

5. Contaminant Removal from 90-96 Inch Depth

Soil samples were taken from three holes in the heated zone at extra deep locations of 84 to 96 inches. The purpose of these samples was to determine whether contaminant removal had occurred below the heated zone as a result of heat conduction. These samples were analyzed for volatile hydrocarbons by the purge and trap procedure. The results show that 92 percent of the aliphatics and 95 percent of the aromatics were removed from the extra deep locations. These results show that contaminant migration in the downward direction did not occur.

D. GAS AND CONDENSATE COLLECTION

Raw gases were collected from the covered surface of the heated zone by means of a suction applied to the gas collection pipes. The gases and vapors were transported to the gas cooling and condensation system described in Section IV B. During the course of the field test the following measurements were made on the gas and condensate streams.

- analysis of raw gas leaving the vapor barrier for jet-fuel
- rate of water and liquid organic production

- analysis of condensate phases for total organic halides
- analyses of gases with Draeger tubes for selected contaminants
- measurement of gas flow rates with pitot tube

The data and results of the above measurements and analyses are presented in the following subsections.

1. Raw Gas Analysis for Jet-Fuel Vapors.

A sample of raw gases leaving the collection zone below the vapor barrier was passed through two gas wash bottles placed in dry-ice/acetone bath to trap the moisture and condensibles. The wash bottles were filled with 20 milliliters of acetone.

The collected liquids were removed from the wash bottles and transferred to a 50 milliliters volumetric flask. The wash-bottles were rinsed with fresh acetone which was then transferred to the 50 milliliter volumetric flask. The volume was adjusted to 50 milliliters with acetone.

During gas sampling the gas flow rate was set with a diaphragm pump which had been calibrated against a wet-test meter. The line conveying the gas sample to the wash bottles was heat traced and insulated to prevent condensation of vapors. The temperature of the gas sample line was maintained between 136-145°C.

The liquid sample was injected into a GC equipped with a flame ionization detector (FID). The FID response was calibrated against jet-fuel. The concentration of jet-fuel vapors in the raw gas stream was reported as mg/L.

The concentration of jet fuel in raw gases leaving the heated zone is shown in Figure 37, as a function of elapsed time. The first raw gas sample was obtained 110 hours after the heating was started. At this time there was 13 mg/L of jet-fuel vapors in the gas stream, while the temperature in the center of the deposit was 114°C. The jet fuel concentration continued to increase with time until it reached a range of 30-36 mg/L. The data scatter in Figure 37, shows that until the end of the test there was no evidence of the jet-fuel concentration decreasing with time.

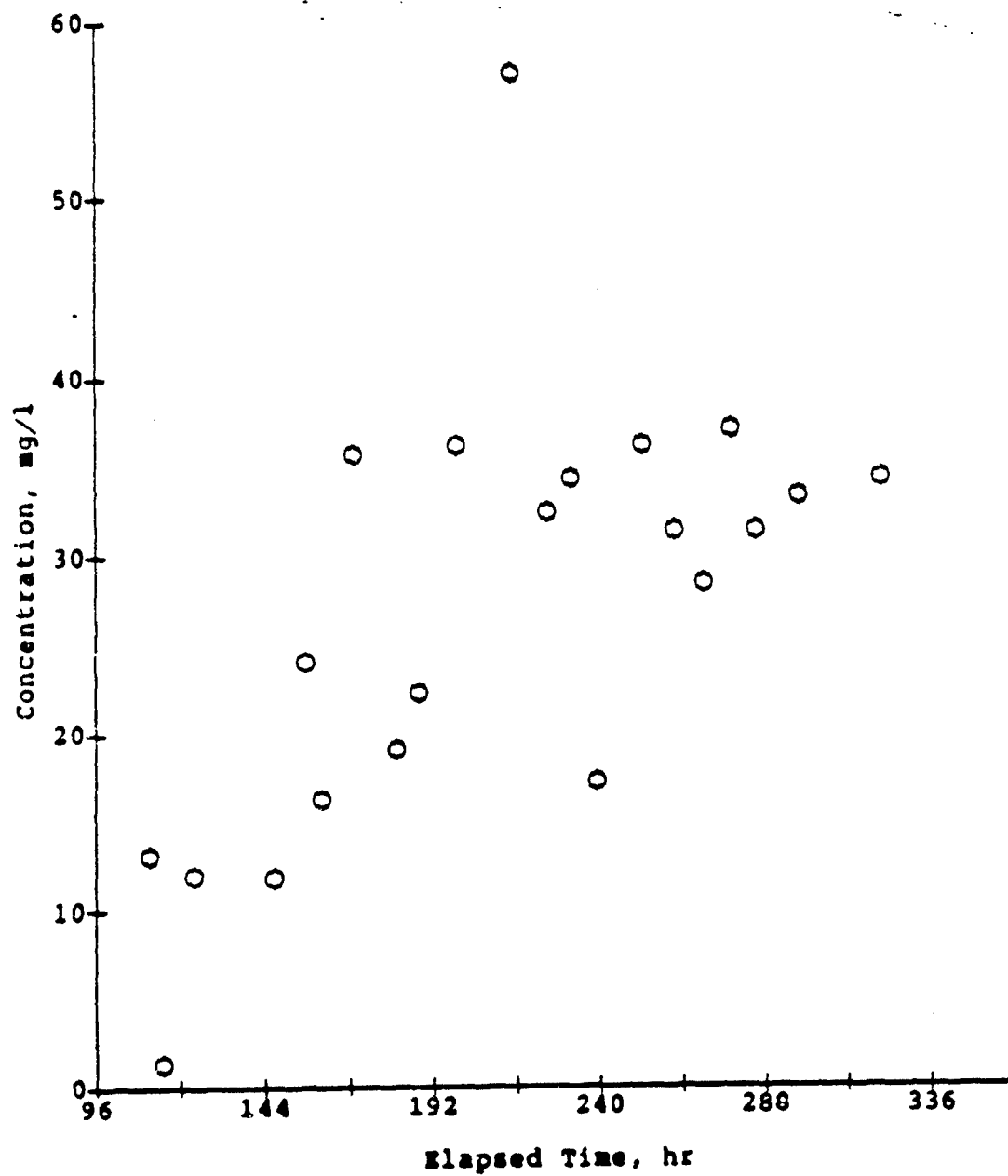


Figure 37. Concentration of Jet-Fuel Vapors in Raw Gas.

2. Rate of Jet-Fuel Vapor Productions Vs. Collection

The term production is used to denote the mass-flow rate of jet-fuel vapors in the raw gas stream leaving the heated zone. The term collection is used to denote the rate of collection of the condensed hydrocarbon phase in the condensate drum. When the condensation efficiency is 100 percent the production and collection rates should be equal. Figure 38, is a scatter diagram showing the JP-4 vapor production and collection rate in pounds per hour. Approximately 30 to 40 percent of the produced JP-4 vapors were collected by condensation in the air-cooled heat exchanger. The condensation efficiency can be improved by using a larger air-cooled or water cooled heat exchangers in the vapor handling system.

3. Collection of Water and Hydrocarbons

The amount of water and hydrocarbon phases collected in the condensate drum is plotted as a function of time in Figure 39.

Totals of 1620 pounds of water and 174 pounds of organic layer were collected over the course of the test. The data in Figure 39, show the rate of water and organic collection was steady over the course of the treatment duration.

The initial average moisture content of the test volume was 4.0 percent. It is estimated that the soil initially contained 2760 pounds of moisture. The residual moisture content in the soil was 0.21 percent, representing a removal of 94.8 percent. The amount of water condensed therefore represents 59 percent of the water removed by vaporization.

4. Analysis of Condensate for Total Organic Halides (TOX)

During the course of the test eight 30-gallon drums of condensate were collected. Samples of water and hydrocarbon phase were taken for TOX analysis from four of the eight drums. Each phase was separately analyzed for organic halides by USEPA standard method number 9020. The results of these analyses are shown in Table 16. The results show that the highest aqueous phase TOX concentration of 26 ppb occurred in the first two drums of water. No hydrocarbon phase was present in these drums. The TOX concentration in the

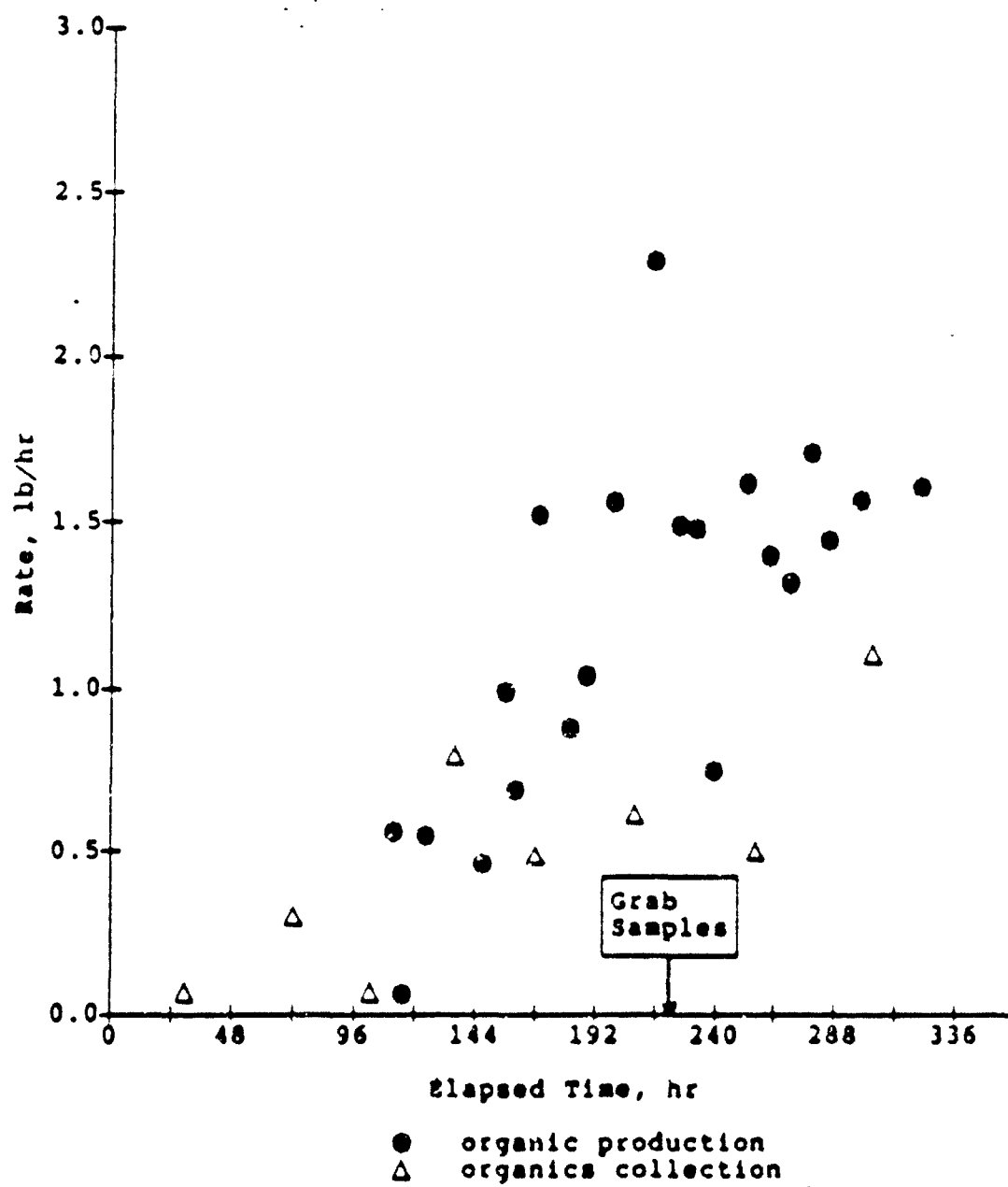


Figure 38. Rate of Organics Production in Raw Gas and in Liquid Condensate.

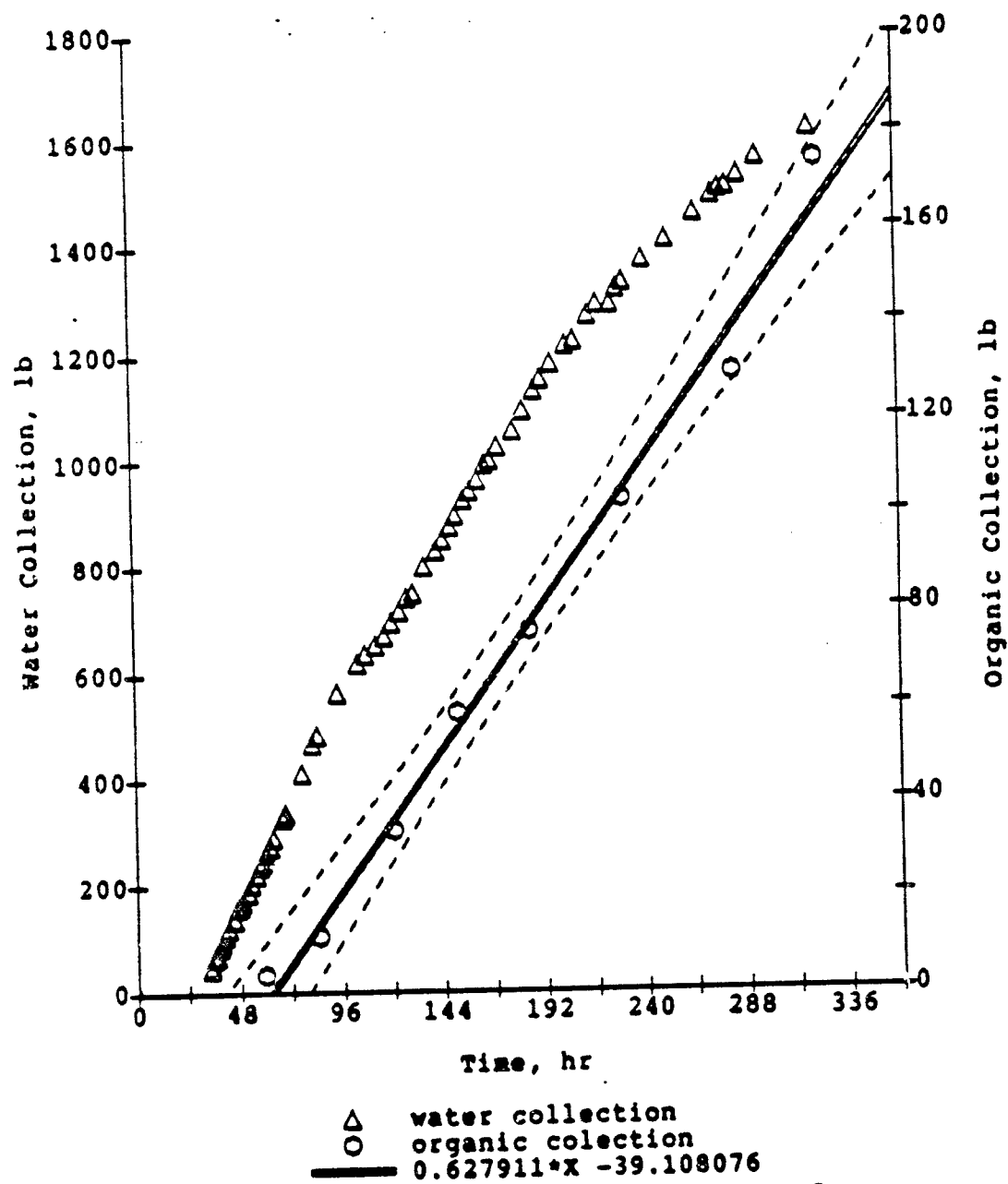


Figure 39. Cumulative Water and Organic Collection vs. Time.

TABLE 16. ANALYSIS OF TOTAL ORGANIC HALIDES (TOX) IN CONDENSED PHASES

Drum No.	TOX Analysis ^a	
	$\mu\text{g/mL}$ in Hydrocarbon Phase	$\mu\text{g/L}$ in Aqueous Phase
1	N.P.	25
2	N.P.	26
5	3.8	12
6 ^b	N.P.	14
6	3.7	10

^a $1 \mu\text{g/L} = 1 \text{ ppb}$; $1 \mu\text{g/mL} = 1 \text{ ppm}$; N.P. = hydrocarbon phase not present.

^b New drum.

condensed water phase decreased steadily during the test as shown by the data trend. In drums 5 and 6 halogenated organics were present at a level of 3.8 ppm in the organic phase. These results show that the TOX concentration in the condensate phases is below the 100 ppm threshold level at which the condensate would be considered hazardous due to the presence of halogenated organics.

5. Gas Analysis by Draeger® Tubes

Draeger® tubes were used to obtain an estimate of the concentration of benzene, trichloroethylene, and petroleum hydrocarbons in the gas streams. These measurements were made in the raw gas manifold as well as at the suction to the blowers, downstream of the carbon bed.

Trichloroethylene was never detected by the Draeger® tubes at any time in the raw gas manifold. Benzene was detected in the range of 5-90 ppm in the raw gases leaving the vapor barrier during the first 46 hours of the heating cycle. In subsequent measurements, benzene's presence could not be positively confirmed with the Draeger® tubes due to apparent interference with other materials present in the gas stream.

The Draeger® tubes for petroleum hydrocarbon were used extensively during the heating cycle. Its results are calibrated in terms of ppm, equivalent n-octane. This tube responds to other aliphatic hydrocarbons present in the gas stream (Reference 6). Due to multiple components present in the raw gases leaving the vapor collection zone, the results of the hydrocarbon Draeger® tubes cannot be used for reliable quantitative work. For example, the tube indicates 2500 n-octane if the gas line contains 1000 ppm of n-hexane. On the other hand, if the gas line contains 1000 ppm n-nonane, the Draeger® tube indication would be only 500 ppm n-octane. Thus, as the molecular weight increases beyond that of octane, the tube response in equivalent n-octane decreases; and as the molecular weight decreases below that of n-octane, the response increases. The addition or removal of even one CH_2 group from the hydrocarbon chain has significant effect on the response of the tube. The tube response is also highly dependent upon the structure of the hydrocarbon chain. For example, 1500 ppm of iso-octane would be indicated

as 2500 ppm n-octane. Because of the cross sensitivity of the tube, the actual total hydrocarbon content in the raw gas manifold may be higher than the readings registered by the Draeger® tube. This is so because in the raw-gas manifold the concentration of higher molecular weight hydrocarbons would be high as opposed to the concentration after cooling and condensation.

On the other hand, the readings of the tube taken downstream of the carbon bed would be higher than the actual hydrocarbon concentration, because the tube gives a higher reading in response to low molecular weight hydrocarbons, which would be expected at the outlet of the carbon bed.

The hydrocarbon Draeger® tube measurements were made on raw gases leaving the collection zone. These measurements ranged between 500 to 1500 ppm equivalent n-octane from the 4th day of the heating cycle to the end of the test.

Draeger® tube measurements on the gas stream leaving the carbon bed indicated a concentration in the range of 200 to 2000 ppm. There was only one data point out of 21 with a concentration of 2000 ppm. As discussed above, these concentrations are probably much higher than the true hydrocarbon concentration in the outlet gas line.

6. Gas Flowrate Measurements

The total gas flow rate in the raw-gas manifold and at the suction of the blowers were measured by inserting a pitot tube in the gas lines. The actual gas flowrate in the raw-gas manifold was found to vary between 12.7 and 16.8 cubic feet/minute. The average rate was 15.1 cfm with a standard deviation of ± 1.1 cfm. The standardized flowrate at 70°F and 1 atm was in the range of 9.7 to 12.6 scfm. The average rate was 11.5 scfm with a standard deviation of ± 0.8 scfm.

The actual flowrate in the blower suction ranged from 11 to 18.7 cfm. The range of standard flowrate at 70°F and 1 atm pressure at the blower suction was 10 to 18.2 scfm. The average rate was 15.3 scfm with standard deviation of ± 2.4 . The apparent discrepancy between the standard flowrates at the two locations is probably due to errors in the pitot tube measurement procedure and due to gas leaks into the blower suction.

E. RF SYSTEM PERFORMANCE

The operational performance of the RF heating system used for the Volk Field ANGB demonstration test was evaluated by monitoring the RF power absorbed by the array, the AC power consumed by the RF power source, by tracking the electrode array's input impedance and by continuously adjusting the matching network to achieve the most efficient energy distribution between the source and the array.

Both the forward and reflected power at the output of the RF power source were continuously monitored throughout the test with measurements made not less than once per hour. By periodically adjusting the variable components of the matching networks, the reflected power to the RF power source was maintained at zero. The three-phase AC power consumed by the RF power source was periodically measured during the test. Finally, by recording the measurements obtained from the IITRI designed in-line impedance meter, changes in trends in the input impedance to the electrode array were tracked as a function of process temperature and time. By monitoring the trends in this impedance, a qualitative assessment of the performance of the RF heating system was maintained in conjunction with measured soil temperature data and by comparing the actual electrode array input impedance measurements to those predicted. The Smith chart illustrated in Figure 40 shows the degree of agreement between the actual measured electrode array input impedance and that predicted during the design phase of this project. The trends in the two illustrated curves are, for the most part, identical with the exception of their initial starting point which was affected by the lower percent moisture concentration exhibited in the actual field test soil as compared to that used in the predictions. Based on the RF heating systems performance for this target soil, it was determined, during the operation of this test, that the use of a single applied RF frequency of 6.78 MHz would be sufficient. The continuous tracking of the electrode array's input impedance showed good agreement to the initial predictions which had identified the sole use of this frequency. In addition, the maximum SWR achieved by the electrode array at the final average soil temperature of approximately 160°C did not exceed the 35:1 range. As a result,

FREQUENCY = 6.78 MHZ
NORMALIZED TO 50 OHMS

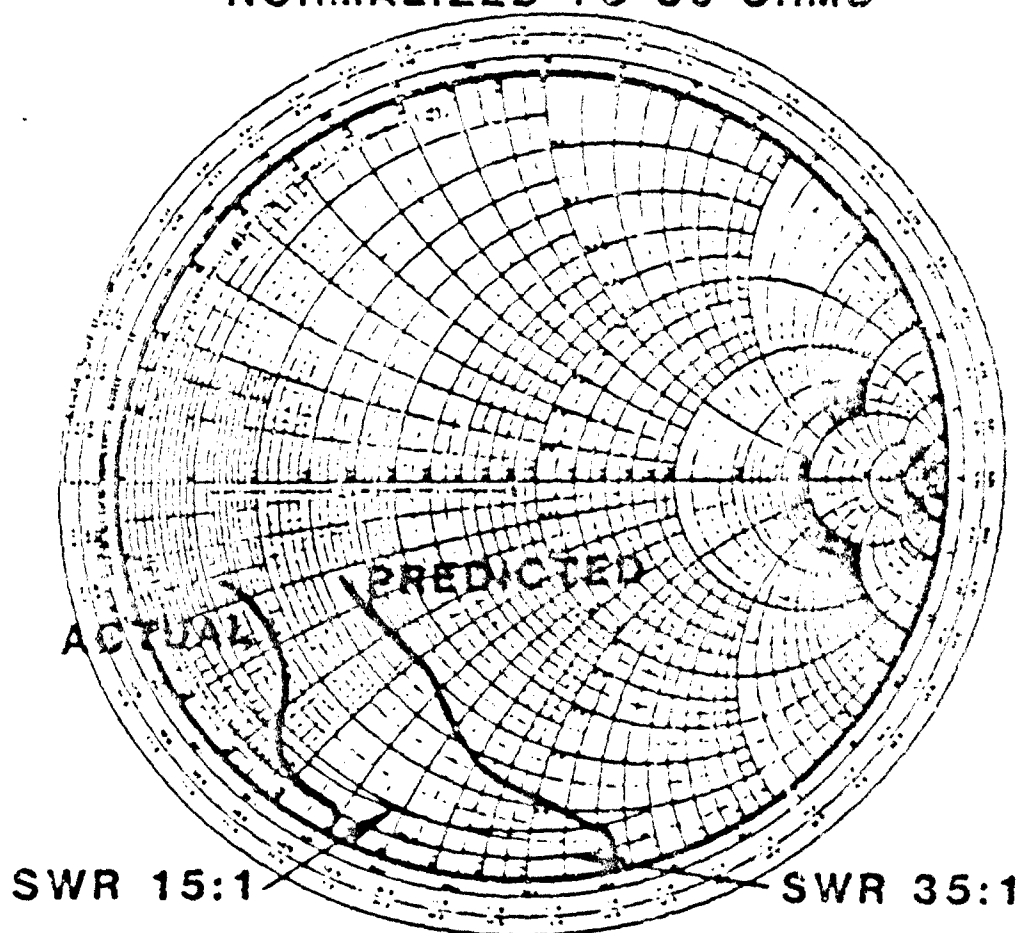


Figure 40. Array Input Impedance; Actual vs. Predicted.

none of the matching network components from either of the two configurations utilized during the test required operation at their stress points.

Both of the matching network configurations originally envisioned during the design phase of this demonstration test were required even though a single frequency of applied RF energy was utilized. The initial heating, to an average soil temperature of approximately 120°C with a corresponding electrode array SWR of approximately 15:1, utilized a matching network consisting of variable high power RF capacitors coupled with a coaxial line stretching section that was capable of operation for a low SWR range. This configuration required little or no enhanced voltage stand-off pressurization of the coax line stretching section. No additional deionized water cooling was required by the variable capacitors of this matching network configuration. It became apparent that as the moisture within the soil was vaporized and the average soil temperature went beyond 120°C, that the capacity of the initial matching network configuration would be exceeded. At this point, a second matching network configuration consisting of similar components but capable of handling higher SWR ranges was installed. This configuration required the pressurization of the coaxial line stretching section with Freon 116 to a pressure of 5 psig. Additionally, deionized water cooling was required for one of the variable high power RF capacitor elements of this matching network configuration. An external cooling unit circulating deionized water was connected for this purpose. The changeover between the two matching network configurations occurred at the end of the fourth day of heating. Use of this second higher SWR range matching network configuration was continued throughout the balance of the heating.

An average RF power level of 35 kW was applied for the first 4 days of heating. This corresponded to the time and energy required to achieve an average soil temperature of 100°C and included the vaporization of the majority of the soil volume's moisture content. The average soil temperature at the end of the first 4 days of heating was approximately 120°C. An average RF power level of approximately 20 kW was applied for the final 8 days of heating. This corresponded to achieving an average soil temperature of approximately 150°C (4 days) and maintaining this temperature during a soak

period of approximately 4 days. Figure 41 illustrates a histogram of the RF power level applied during the demonstration test as well as cumulative curves of the RF energy absorbed by the electrode array and the required three-phase AC power consumed by the RF power source. Table 17 summarizes this information and includes an estimated cost per unit volume for the electrical energy required to decontaminate soils of this type. It should be noted that this demonstration test utilized an existing HF band radio frequency transmitter whose efficiency is in the 40 to 45 percent range. Newer units or power amplifiers are available today that can provide between 70 and 80 percent power conversion efficiency. Use of one of these newer units would have the impact of reducing the cost per cubic yard from the listed \$45 to approximately \$25 per cubic yard.

TABLE 17. POWER CONSUMPTION AND COSTS

RF Energy Absorbed	6580 KWHR
3 ϕ AC Energy Consumed	15480 KWHR
Efficiency*	43%
Volume Heated	504 cu. ft. (19 cu. yd.)
Mass Heated	35 tons
AC Energy Costs	5.5¢/KWHR
Cost for RF Energy Generation	\$850
Cost per Unit Volume	\$45 per cu. yd.

*Based on utilizing the IITRI available
AN/FRT-6 HF band RF transmitter.

F. RF EMISSIONS MONITORING

Near and far-field electromagnetic measurements were made at and around the test area. Near-field refers to the immediate vicinity of the test site; far-field refers to locations 1/8 to 1/2 mile from the test site. All far-field locations were selected in consultation with Volk Field ANGB base communication personnel. The purpose of these measurements was to ensure that any radiated RF power levels were below permissible FCC and Air Force

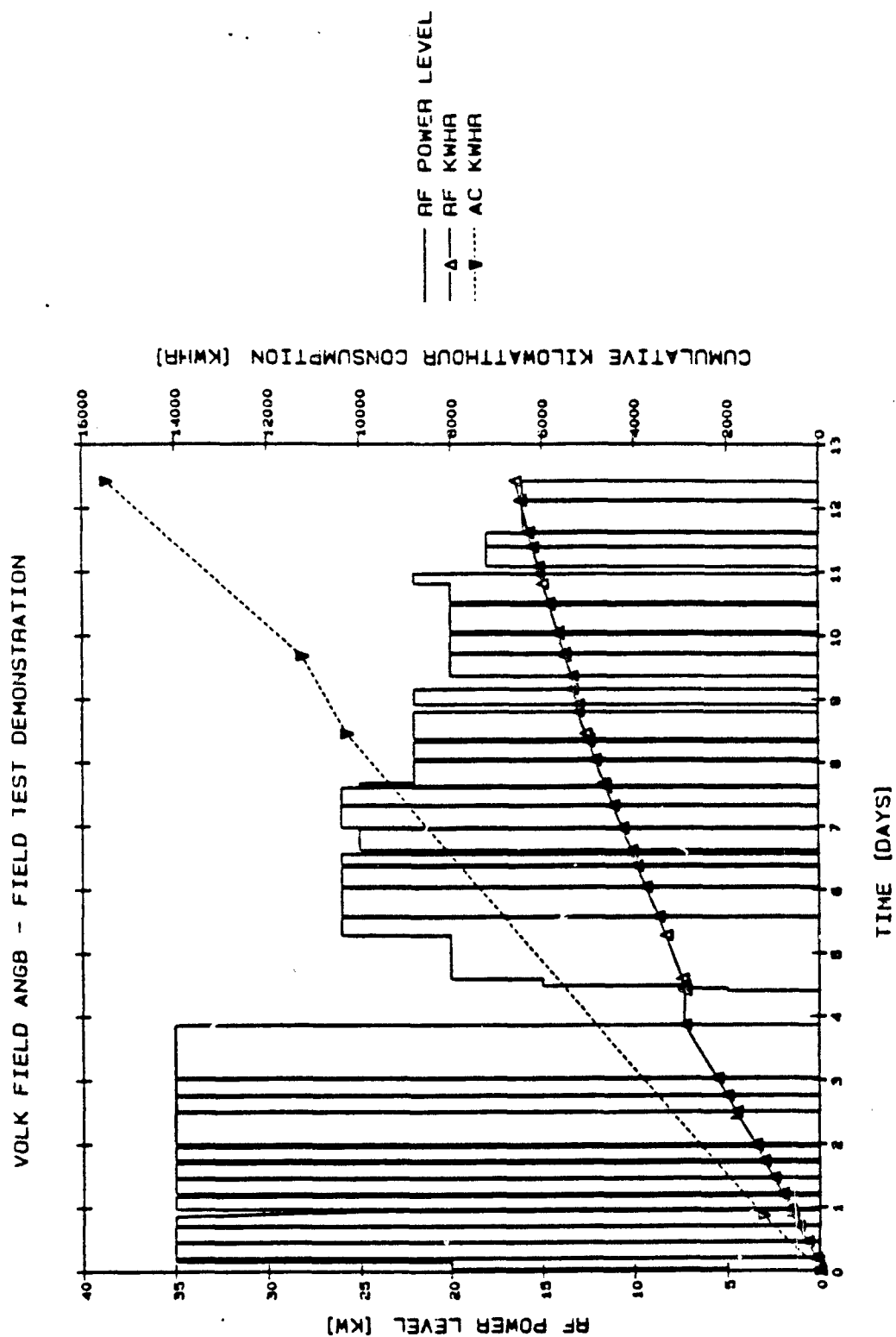


Figure 41. RF Power Histogram.

standards, that no interference was generated with base communications, and that no personnel safety problem areas existed.

These measurements were made in two distinct phases. The first phase or series of measurements was conducted before the initiation of the actual test by applying low RF power levels to the electrode array and monitoring both near and far-field radio frequency interference (RFI) electric field intensity values in order to identify any potential problem areas. The second series of measurements were conducted throughout the test at intervals of approximately every 3 days. These near and far-field RFI measurements were made while full power was being applied to the electrode array. Ambient field levels were measured by momentarily turning off the RF source to the electrode array at each measurement point or location.

Figure 42 illustrates an overview of the Volk Field fire training pit site layout. This figure illustrates the locations of the RFI safety measurement points monitored throughout the test. Both radiated RF electric field strength and ambient electric field strength are illustrated. Table 16 contains the maximum measured electric field strengths for both near and far-field RFI safety measurements. Also illustrated in this table are the appropriate limits identified by the American National Standards Institute (ANSI) and the National Institute of Occupational Safety and Health (NIOSH) for near field continuous exposure to electric fields at this frequency of operation. The maximum measured electrical field strength of 28 volts per meter is more than six times below the minimum of these two ratings. In addition, no electromagnetic interference was experienced by any of the air national guard base communication staff throughout the duration of the heating experiment.

Table 18 identifies the electric field strength as measured at a variety of distances away from the electrode array. These values of 30 millivolts per meter at a distance of 200 meters away and 4 millivolts per meter at 800 meters away meet all FCC requirements for operations utilizing ISM band frequencies. Table 19 identifies out-of-band electric field strength measurements for a principal applied frequency of 6.78 MHz. Out-of-band refers to measurements at frequencies other than the operating frequency directly generated as

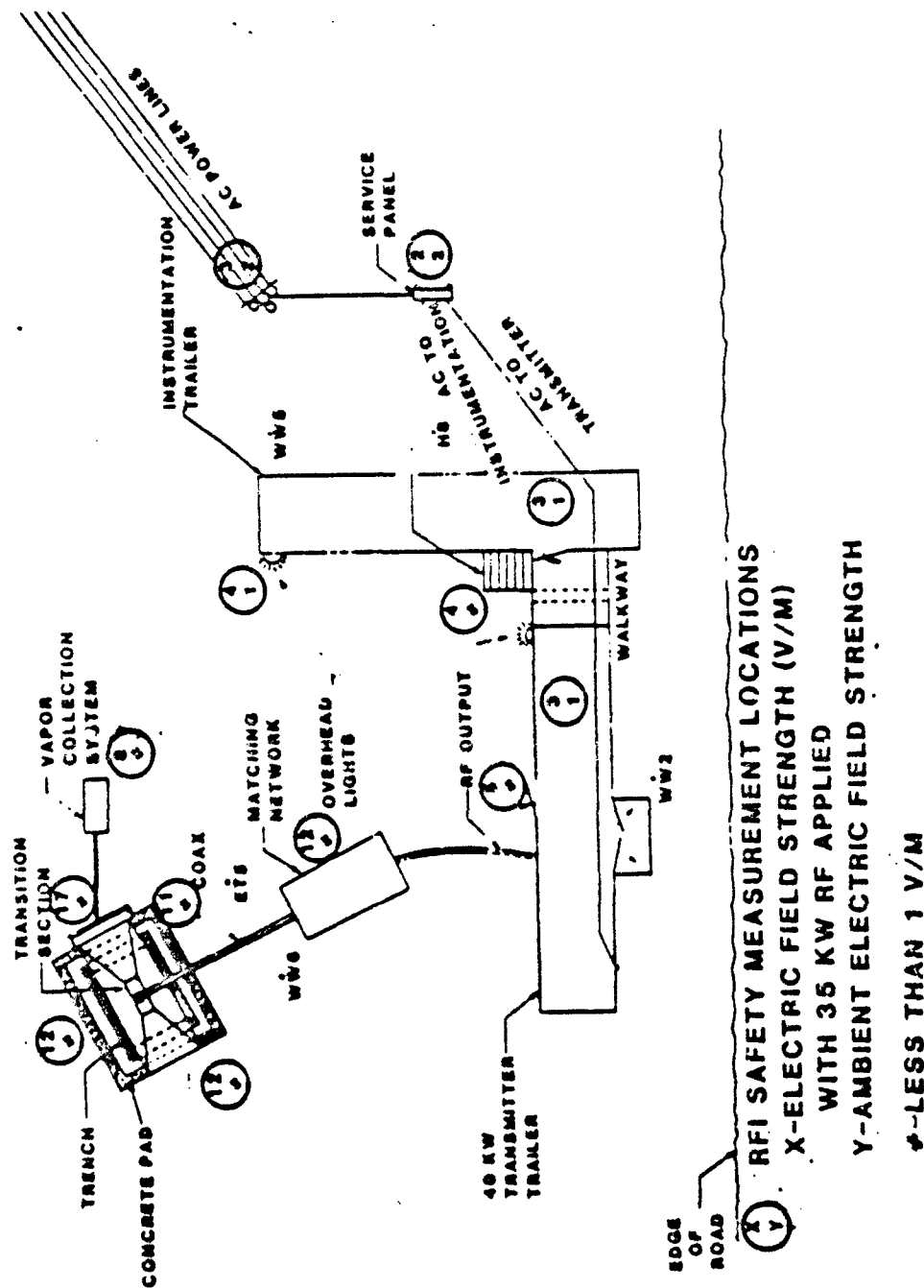


Figure 42. RFI Safety Measurement Locations.

TABLE 18. RFI SAFETY MEASUREMENTS NEAR/FAR FIELD APPLIED
FREQUENCY = 6.78 MHz [ISM]

Distance From Array [Meters]	Electric Field Strength* [Volts/Meter]	Personnel Safety Continuous Exposure Electric Field Standards [Volts/Meter]	
		ANSI	NIOSH
5	28.0	272.	192.
200	0.030		
800	0.004		

* Applied Power = 25 kW
SWR = 28:1

TABLE 19. OUT-OF-BAND ELECTRIC FIELD STRENGTH NEAR/FAR FIELD MEASUREMENTS
APPLIED FREQUENCY = 6.78 MHz [ISM] APPLIED POWER = 35 kW

Distance from Array [Meters]	Electric Field Strength* [Micro Volts/Meter]	FCC Out-Of-Band Electric Field Strength Limits [Micro Volts/Meter]
5	175.	---
300	3.	210.
800	< 1.	10. [1600 meter limit]

part of its operation (harmonics, case radiation, etc). From the table it is seen that the out-of-band electric field strengths, as measured in and around the Volk Field test site, were more than 10 times lower than the acceptable FCC limits.

The fact that all RFI measurements near and far-field met personnel safety limits and were within permissible standards, indicates that more than sufficient efforts were employed during the design, fabrication and installation of this field demonstration test. The steps taken towards these design and fabrication procedures are easily scaled up to full-scale site decontamination projects.

SECTION VII

CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The feasibility of in situ soil decontamination by RF in situ heating of soil was successfully demonstrated in this project through a field test. It was shown that sandy soil containing unburnt waste oils, solvents and jet fuel can be cleaned up and 94 percent of the aliphatics and 99 percent of the aromatics removed by heating the soil to a temperature range of 150°-160°C. It is expected that additional removal of aliphatics can be achieved by increasing the treatment duration or by increasing the temperature.

Analysis of soil samples as a function of depth indicates that uniform decontamination of the contaminated zone had occurred to depths of 6 feet.

It was shown that high boiling contaminants such as hexadecane can be removed at temperatures much below their normal boiling point as a result of long residence time and gas sweep provided by native moisture boiling out of the soil.

The reduction of contaminant concentration in the zone immediately outside the heated zone indicates that no net migration of contaminants from the heated area had occurred. The presence of a vaporized liquid tracer, injected outside the hot soil, in the hot gases leaving the treatment zone indicates that soil gas and liquids migrate from the surrounding cool zones into the hot zone where they are volatilized and recovered.

The cost estimates for the full-scale system for the treatment of soil indicate that the RF process is substantially less expensive than excavation and incineration of the contaminated soil. It was estimated that the treatment cost per ton by the RF process would be in the range of \$29 to \$60. Treatment by incineration of soil is expected to cost \$180-310 per ton of soil.

The RF in situ process offers additional significant advantages over conventional and even alternative technologies under development for the remediation of waste sites.

The RF process is a true in situ process. It is estimated that only 0.5 percent of the volume treated will require excavation or drilling in order to install the electrode array. As a result, the process minimizes the risk of contaminant redispersion and exposure of operating personnel.

Effluent gas production in the RF process is minimal; it is estimated that it would be 1000 times less than processes based on incineration. This is because the RF process does not rely on in situ combustion of fuels or injection of air for heating of the soil. A concentrated liquid stream is produced which contains water and the condensed organic materials, which can be readily disposed off in conventional treatment systems.

B. RECOMMENDATIONS

The results of this field test have shown that in situ soil decontamination by RF heating is indeed feasible. These results warrant process optimization and full-scale demonstration so that the process can be made available for commercial application for hazardous waste site remediation.

The purpose of process optimization is to reduce the overall treatment cost, develop hardware designs which can be readily implemented in the field, and extend the process to other types of soil, such as clay, through laboratory pilot-scale experiments. Specifically, the optimization studies need to address the following issues:

- Evaluate cost benefits of combining the RF in situ heating with the soil venting process on soil decontamination.
- Evaluate cost cutting strategies through an in situ pre-heating step which utilizes the same electrode array but uses 60 Hz AC heating instead of RF heating. With AC heating, the power source capital cost can be reduced.
- Evaluate technical merits and limitations of various combinations of AC and RF heating with soil venting.
- Develop engineering designs of an efficient vapor treatment system.
- Develop designs for reusable vapor barriers which can be easily installed in the field.

- Eliminate the RF transition section and replace with a simplified RF power feed system which is less bulky, cheaper and easier to install than the existing transition.
- Extend the process to clayey soils through laboratory pilot-scale experiments.

The full-scale commercial design of the treatment system should be done after the various optimization issues listed above have been resolved. A full-scale decontamination of a fuel- or solvent-contaminated site should be done to demonstrate the optimized design.

REFERENCES

1. Krystansky, J., et al. RF Heating of Carbonaceous Deposits. DOE Report DOE/ER/10181-1, IIT Research Institute, Chicago, IL, March 1982.
2. Dev, H. et al. Radio Frequency Enhanced Decontamination of Soils Contaminated with Halogenated Hydrocarbons. Final Report, IITRI Project No. C06600, USEPA Co-operative Agreement CR 811529, Hazardous Waste Engineering Research Laboratory, USEPA, Cincinnati, OH, June 1988.
3. Dev, H., Bridges, J. E., and Sresty, G. C. Decontamination of Hazardous Waste Substances from Spills and Uncontrolled Waste Sites by Radio Frequency In Situ Heating. EPA-600/D-84-077. USEPA, Cincinnati, OH, 1984, p. 34.
4. Dev, H., Enk, J., Bridges, J. E., and Condorelli, P. High-Temperature Thermal Treatment of Soils with Radio Frequency Heating, Final Report IITRI Project No. C06600, EPA Grant No. CR-811529-01-0, IIT Research Institute, Chicago, IL, June 1986.
5. Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 2nd Ed. Office of Solid Waste and Emergency Response, USEPA, Washington, DC, USEPA Report No. SW-846, April 1984.
6. Operating Instructions, Draeger Tube: Petroleum-Hydrocarbons, 100/a. Drägerwerk AG, Lubeck, Germany, 5th Ed., February 1986.

APPENDIX A

ANALYSIS OF SOLVENTS AND JET FUEL IN SANDY SOIL

APPENDIX A

ANALYSIS OF SOLVENTS AND JET FUEL IN SANDY SOIL

In this appendix, the procedures for the analyses of tetrachloroethylene, chlorobenzene, and jet fuel in soil are described. Analysis of these contaminants in soil is a two-step procedure. The first step consists of extraction and removal of the contaminant from the soil into an organic solvent. The second step consists of analysis of the extract phase for the contaminant of interest.

During the course of this program, distillates were produced by condensation of vapors formed during the thermal-treatment experiments described in Section II of the main report. After drying, the distillates were analyzed for contaminants using procedures of the second step mentioned above.

For all experiments, the extraction step was performed by steam distillation of the soil in a Nielson-Kryger distillation apparatus. But two different methods were used for the analysis of distillate depending upon the type of contaminants present.

When the only contaminant present in soil was tetrachloroethylene, the extracts as well as the distillates were analyzed on a gas chromatograph equipped with an electron capture detector (ECD). When tetrachloroethylene and chlorobenzene were present together or when jet fuel was present in soil, the extracts as well as distillates were analyzed on a GC/MS system.

The extractions and analytical methods and procedures described in this appendix are only applicable to the pilot-scale experimental results presented in Section II of the report. The Nielson-Kryger steam distillation procedure was used along with a different GC/MS procedure for the analysis of semivolatiles in the pretest and posttest results presented in Section VI of the report. The analytical procedures for the samples associated with the field test are described in Appendices B and C.

A. NIELSON-KRYGER STEAM DISTILLATION-EXTRACTION OF SOIL

1. Equipment

A Nielson-Kryger steam distillation condenser (sold by Ace Glass, Inc.); 500 ml Pyrex® round-bottom flask, disposal glass pipettes for preparing Tenax® traps, prewashed Pyrex® glass wool plugs.

2. Reagents

TA grade, 60/80 mesh Tenax®; pesticide grade iso-octane; HPLC grade hexane, anhydrous sodium sulfate, ACS grade; and hydrocarbon free distilled water.

3. Sample Preparation

Contaminated soil needs no special preparation prior to steam distillation.

4. Procedure

Steam distillation of the soil was performed to remove the contaminants in the soil and to transfer them to a liquid phase that could then be analyzed on a gas chromatograph (GC) using an electron capture detector (ECD), or a GC/MS system.

Approximately 75 gms of the soil sample was placed in the 500 ml round bottom flask, 250 ml of hydrocarbon (HC) free water was added and the flask was attached to the Nielson-Kryger condenser as shown in Figure A-1. The condenser was charged with approximately 10 ml of HC free water followed by approximately 10 ml of hexane. The two liquids separate into two phases in an annular zone in the lower part of the condenser. The heavier water phase fills the liquid withdrawal tube and partially fills the water overflow tube. A disposable glass pipet filled with approximately 1 gm of Tenax® was attached to the condenser opening at the top.

When the water, soil, and contaminant mixture is brought to boil in the flask, the vapors rise up through the vapor-riser and condense on the walls of the condenser. The condensate collects in the annular space which was prefilled with hexane/water. The condensate separates into two phases

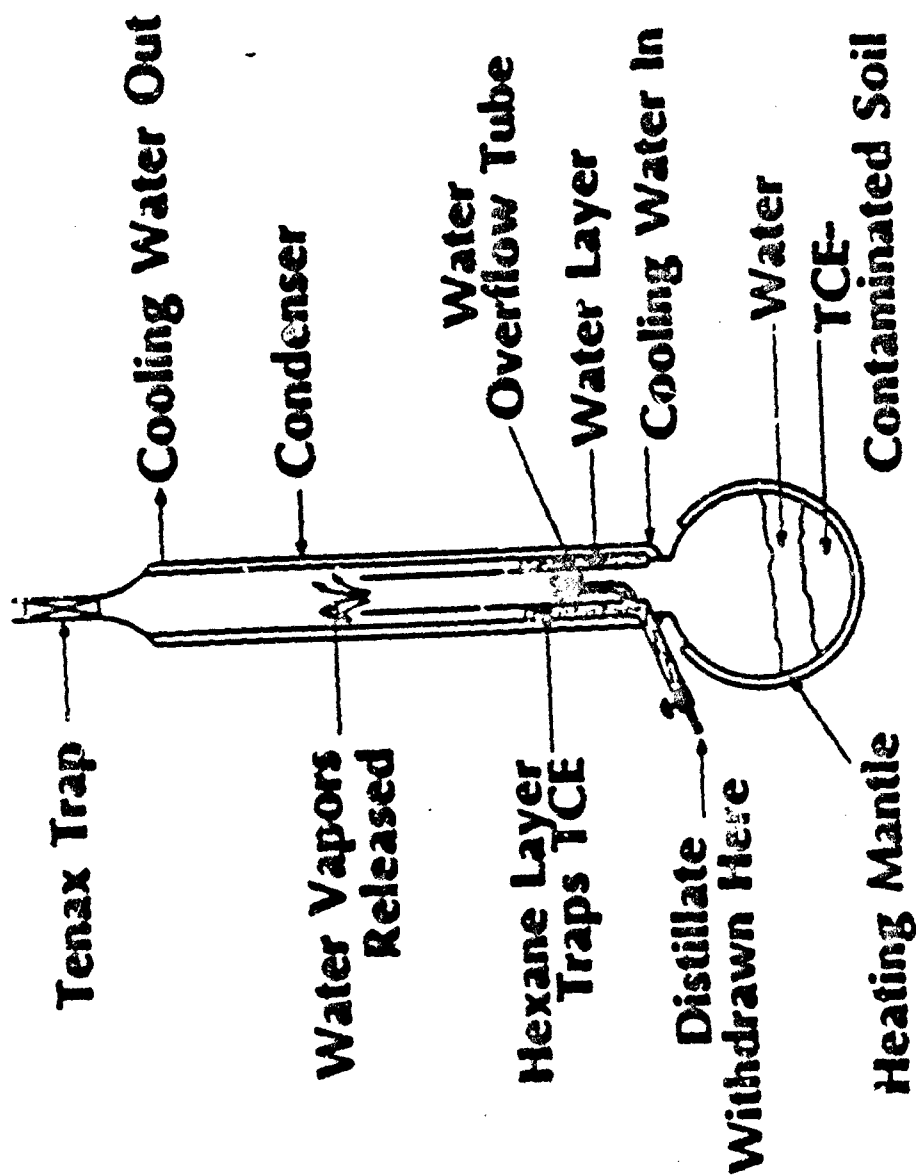


Figure A-1. Nielson-Kryger Steam Distillation Apparatus.

with simultaneous extraction of the contaminant into the hexane phase. As the steam distillation proceeds, the water layer increases in volume until it begins to reflux through the overflow tube. The steam distillation was performed for a period of 16 hours on each soil sample, at the end of which the heating was discontinued and the apparatus allowed to cool to room temperature.

The water and hexane phase were recovered into a separatory funnel. The Tenax® trap was removed from the condenser and flushed with pure hexane (10 ml) to remove any trapped contaminant. The condenser was rinsed with water and hexane to remove any unrecovered droplets of water. The washings were also added to the separatory funnel. After allowing the two phases to separate, the water phase was drained from the funnel and the remaining hexane phase was combined with the hexane from the Tenax® wash and dried through a bed of anhydrous sodium sulfate. The dried hexane phase was brought up to a volume of 100 ml in a volumetric flask. Two to three drops of triple distilled mercury was added to the recovered extract to remove sulfur impurities.

B. DETERMINATION OF TETRACHLOROETHYLENE RECOVERY EFFICIENCY

The purpose of this evaluation was to determine what fraction of tetrachloroethylene originally contained in soil could be recovered during steam distillation and the subsequent sample preparation steps. The recovery of tetrachloroethylene was expected to be less than 100 percent for several reasons: (1) strong irreversible adsorption of tetrachloroethylene on the soil, (2) evaporative losses, and (3) sample loss during transfer, phase separation, drying, etc.

Of the three reasons mentioned above, adsorption of tetrachloroethylene in soil was expected to account for most of the recovery losses. Evaporative losses were minimized in the proposed techniques by two precautions: the use of Tenax® traps to minimize loss of uncondensed tetrachloroethylene through the condenser and the use of ice baths to chill the distillate during preparatory steps like phase separation, drying, etc. Sample losses during distillate transfer, phase separation, drying was minimized by thoroughly rinsing

all glassware with fresh hexane. The rinsate was combined with the distillate prior to the drying step.

Steam distillation of tetrachloroethylene and water mixture in the absence of soil showed essentially the same recovery efficiency as for steam distillation of spiked soil. Thus, irreversible adsorption of tetrachloroethylene on the sandy soil used in this study was presumed to have negligible effect upon recovery efficiency.

The recovery efficiency for tetrachloroethylene was determined by the steam distillation of spiked soil. The recovery efficiency was found to range from 86 to 107 percent. In Table A-1 the data on recovery efficiency is presented. In Figure A-2 the recovery efficiency is plotted as a function of true concentration of tetrachloroethylene in soil. The data in this figure show that the recovery efficiency is independent of the tetrachloroethylene concentration in soil. An average recovery efficiency of 97.1 percent was obtained.

1. Procedure for Spiking Soil

Spiked soil samples were prepared by adding to the soil a known volume of a tetrachloroethylene solution in iso-octane. The concentrations of the solution were determined by GC analysis. Each recovery efficiency experiment was performed using 75 gms of soil containing 5.7 percent moisture in a 500 ml Pyrex® glass round bottom flask. A known volume of spiking solution was added to the soil, the flask was sealed and the spiked soil was tumbled for a period of 3 hours by attaching the sealed flask to a rotovap. To prevent vaporization of tetrachloroethylene during tumbling the rotating flask was placed in an ice bath. After 3 hours of tumbling the flask was attached to the Nielson-Kryger distillation condenser. The recovery experiments were performed in the same flask in which the soil was spiked and tumbled.

The steam distillation and sample preparation were performed according to the procedure described above.

C. GC/ECD PROCEDURE FOR EXTRACT AND DISTILLATE ANALYSIS

As mentioned earlier, two different procedures were used for the analysis of extracts and distillates. The GC/ECD procedure was used when the only

TABLE A-1. RECOVERY EFFICIENCY OF TETRACHLOROETHYLENE FROM SPIKED SOIL^a

No. of Times Extracted	No. of Times Analyzed on GC	Concentration of Spiked Soil, ppm						
		1150	93.0	910	500	40.0	250	50
1	1	97.0	101.1	95.6	87.7	100.2	98.9	97.6
	2	93.9	99.9	106.4	97.3	95.8	95.3	98.0
	3	95.4		96.7		97.7	95.4	--
	4			94.4		97.9		--
2	1	95.6	98.5				99.7	97.5
	2	92.4	97.7				96.2	97.3
	3	96.3					95.0	90.4
	4							--
Average		95.1	99.3	98.3	92.5	97.9	96.8	97.6
Std deviation		1.68	1.51	5.50	1.80	2.03	--	4.88
S Std deviation		1.8	1.5	5.6	1.8	2.1	4.8	8.7
n		6	4	4	7	4	6	2
							2	4

^aTyndall AFB soil steam distilled

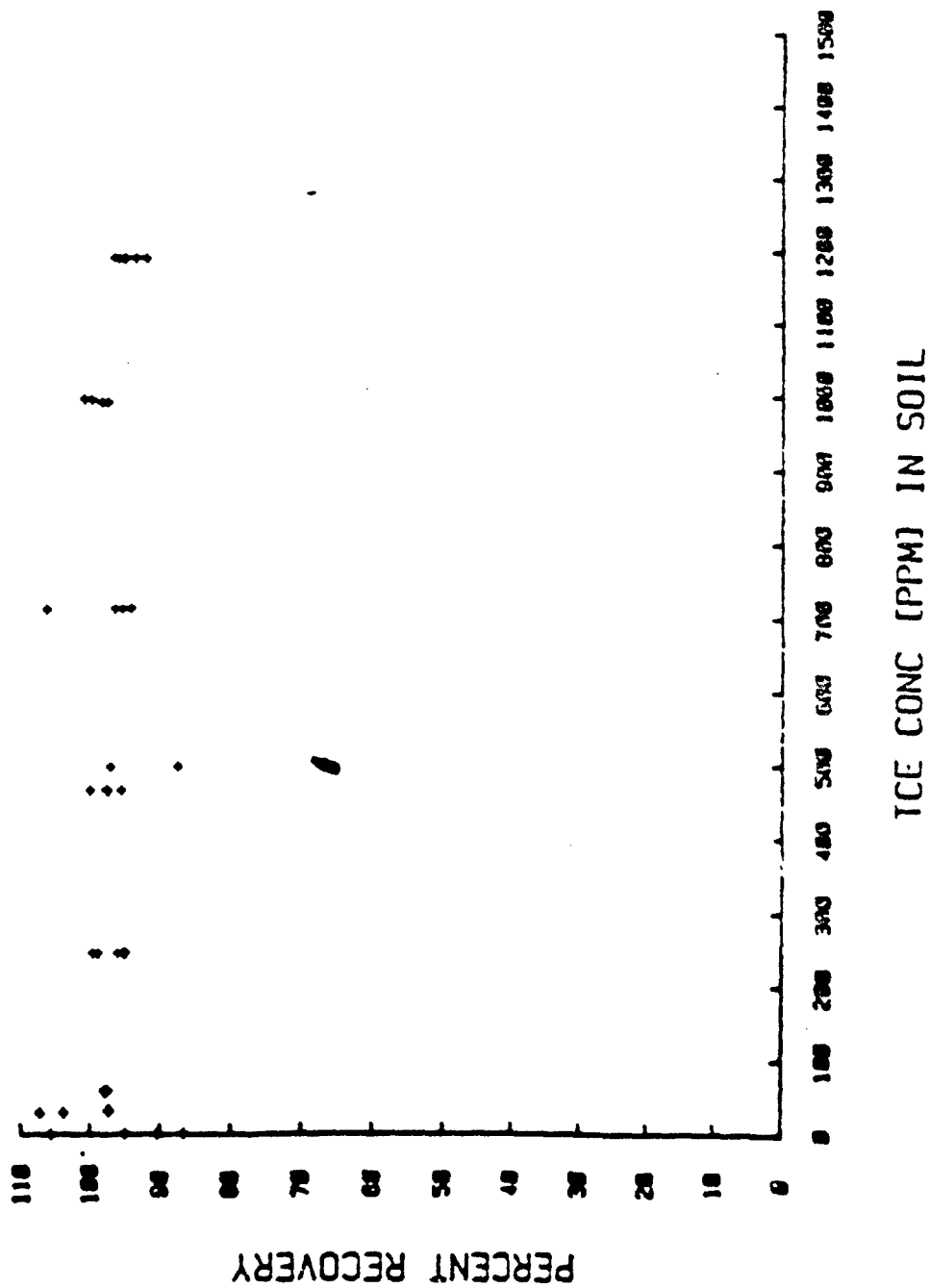


Figure A-2. Tetrachloroethylene Recovery Efficiency by Steam Distillation.

contaminant present in soil was tetrachloroethylene; when chlorobenzene or jet fuel was present a GC/MS procedure was used.

1. Equipment

A Hewlett-Packard 5840 gas chromatograph equipped with a Ni^{63} electron capture detector was used for the analysis. The separation was performed on a 2-meter long, 1/8-inch I.D. stainless steel column packed with 4 percent OV-101 supported on 80/100 mesh chromosorb G, AW-DMCS supplied by Supelco, Inc. The GC operating conditions are summarized in Table A-2.

TABLE A-2. GC OPERATING CONDITIONS

Temperatures:	
Oven	70°C (isothermal)
Injector	175°C
Detector	200°C
Carrier Gas:	5% CH ₄ in 95% Argon 20 ml/min
Analysis time	25 minutes
Average retention time of tetrachloroethylene	5.8 ± 0.1 minutes

2. Sample Injection Procedure

Approximately 1.5 to 4.0 μl of the prepared sample (or calibration standard) was injected into the GC with a 10 μl syringe. The solvent flush injection method (1.5 μl air, 2 μl of hexane, 1 μl air, sample, 1.0 μl air) was used. Between injections the syringe was cleaned by rinsing 10 times each in three different bottles of MeOH and five times each in two different hexane bottles.

3. Calibration Procedure

The GC was checked with three different calibration standards every day. The area response was compared with the full calibration curve to check for deviations and linearity. If the daily GC check showed a deviation of

more than 10 percent, then the GC was recalibrated. Fresh calibration standards were prepared every week. When not in use, the calibration standards were kept under refrigeration at a temperature of -20°C . Before use, each standard was allowed to warm up to room temperature and checked for integrity and volume loss.

Calibration standards were prepared by volumetric dilution of a stock solution prepared gravimetrically from 99.9 percent HPLC grade tetrachloroethylene. Fresh stock solutions were prepared on a monthly basis.

Solvent blanks were performed on the pesticide grade iso-octane. These blanks showed a small response at the same retention time as that of tetrachloroethylene. It was determined that for soil experiments at 1000 ppm level, the solvent blank concentration would be a factor of 400 less than that of the most dilute sample to be analyzed. For this reason, iso-octane was used as a solvent. However, in the course of the program, it was decided to perform experiments with soil containing 10 ppm of the contaminant. It was determined that for such experiments iso-octane could not be used as a solvent for preparing calibration standards. It was estimated that the solvent blank concentration would be less than the response of the most dilute sample by only a factor of 15. Therefore, various solvents were screened until HPLC grade cyclohexane was found to give no interfering response with tetrachloroethylene. Solvent blanks were also performed on other solvents used in this program such as hexane and methanol. It was verified that these materials did not interfere with tetrachloroethylene.

Multipoint calibration curves were obtained by injection of prepared standards and plotting the mass of tetrachloroethylene injected against the area response on the GC.

Figure A-3 shows a typical calibration curve for standards prepared in iso-octane. A second order polynomial was fitted to the data. The equation of the fitted curve was:

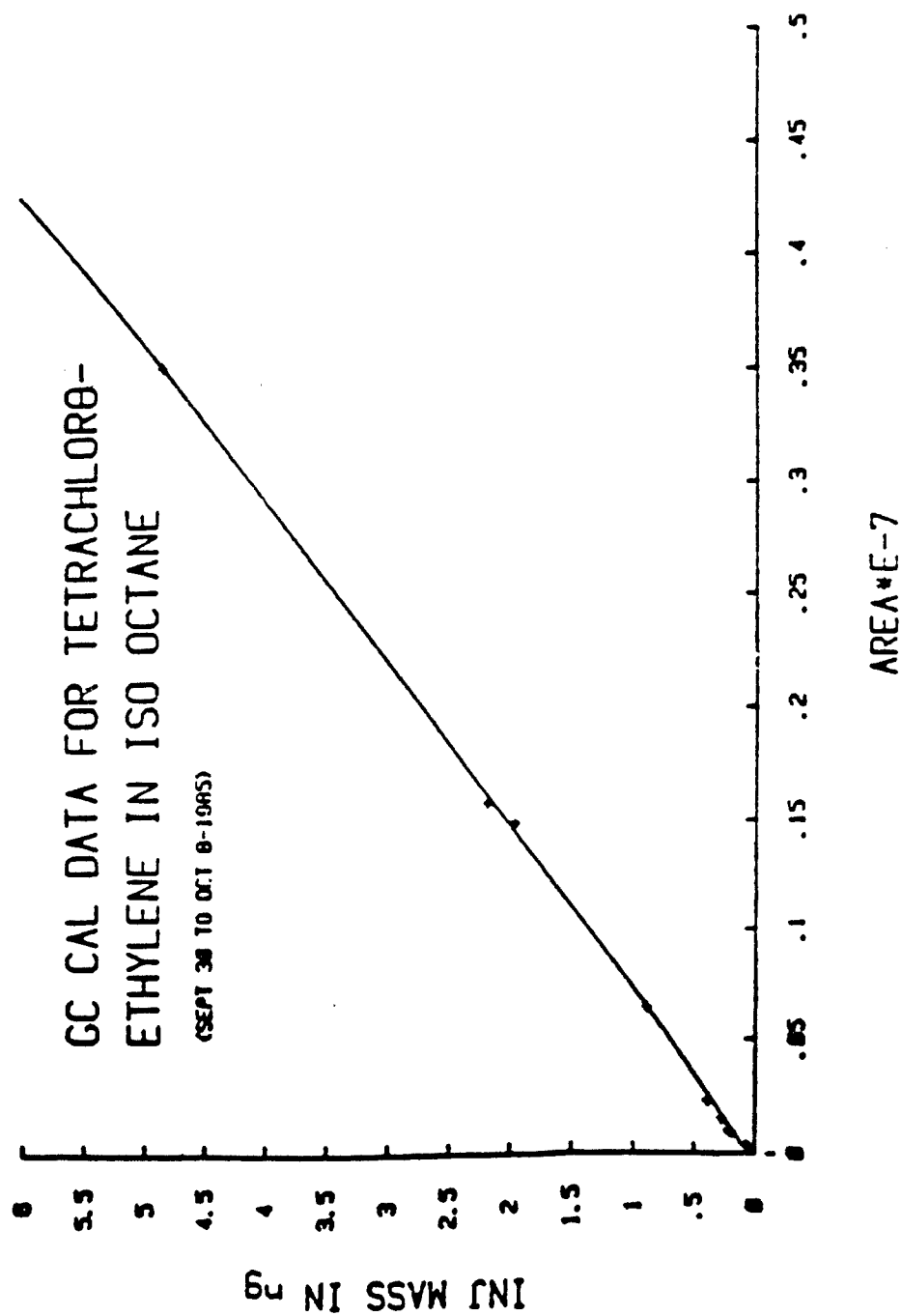


Figure A-3. GC Calibration Data for Tetrachloroethylene in iso-Octane.

$$Y = 2.59 x^2 + 12.7 x + 0.0624 \quad (A-1)$$

where Y is the amount of tetrachloroethylene injected, nanograms
x is the scaled area response obtained by dividing the
actual area response by 10^7 .

The r^2 value for the above fit is 0.99945.

Figures A-4 and A-5 give calibration curves for standards prepared in cyclohexane at high and low levels, respectively. The data from each of these calibration curves was fitted to second-order polynomials.

The equation obtained for the high-level calibration data of Figure A-4 was:

$$Y = 5.38 x^2 + 10.40 x + 0.115 \quad (A-2)$$

The r^2 value for this fit is 0.9972.

The equation obtained for the low-level calibration data of Figure A-5 was:

$$Y = -89.4 x^2 + 16.63 x + 0.0122 \quad (A-3)$$

The r^2 value for this fit is 0.9916. Correlation of high and low level data separately allowed simple regression equations to be fitted to the data while still minimizing the error of the correlations.

4. Calculation Procedure

The following procedure was used for calculating the concentration of tetrachloroethylene in soil. This was done by analyzing the concentration of tetrachloroethylene in the distillate obtained from steam distillation of the

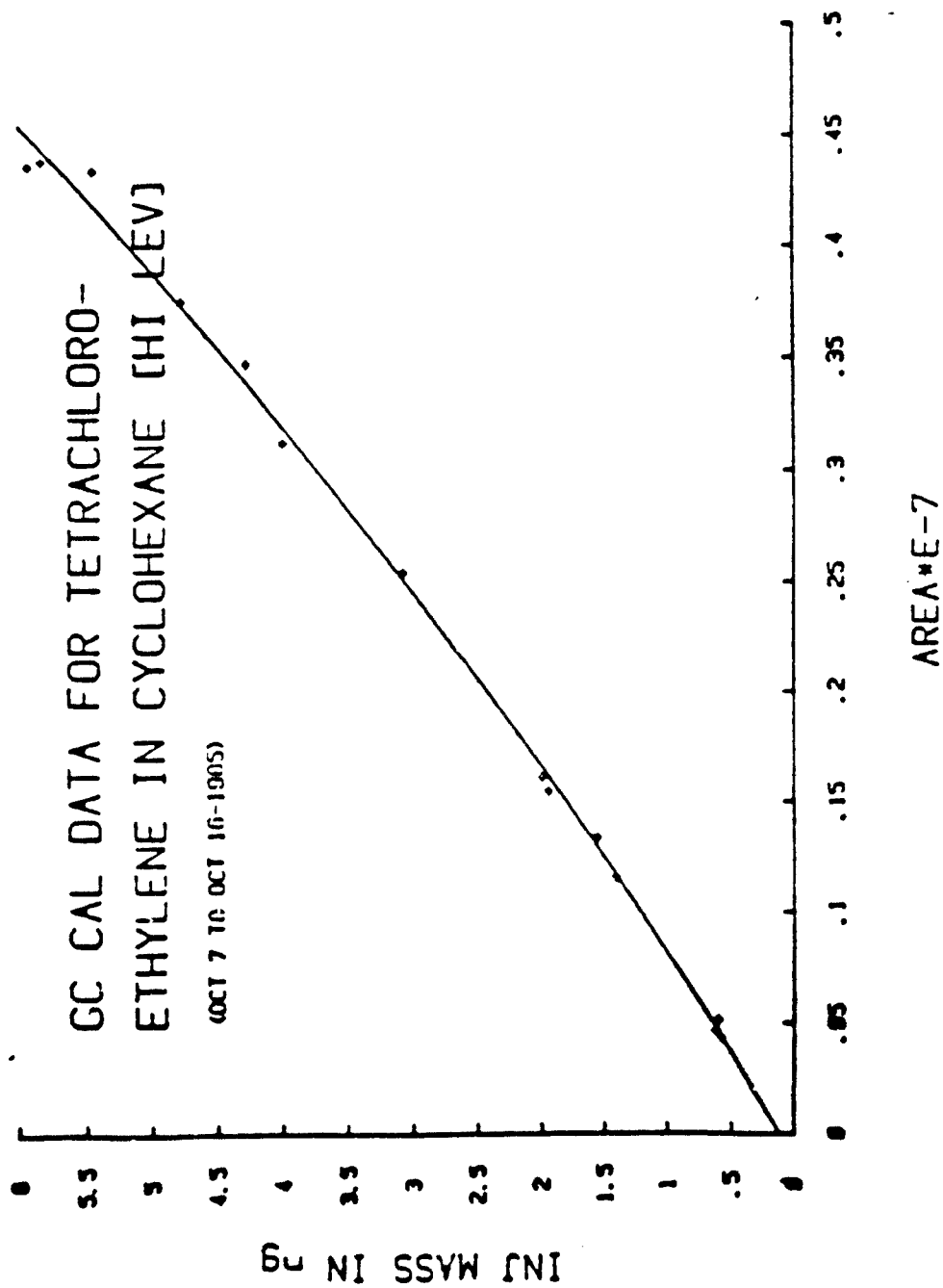


Figure A-4. GC Calibration Data for Tetrachloroethylene in Cyclohexane (High Levels).

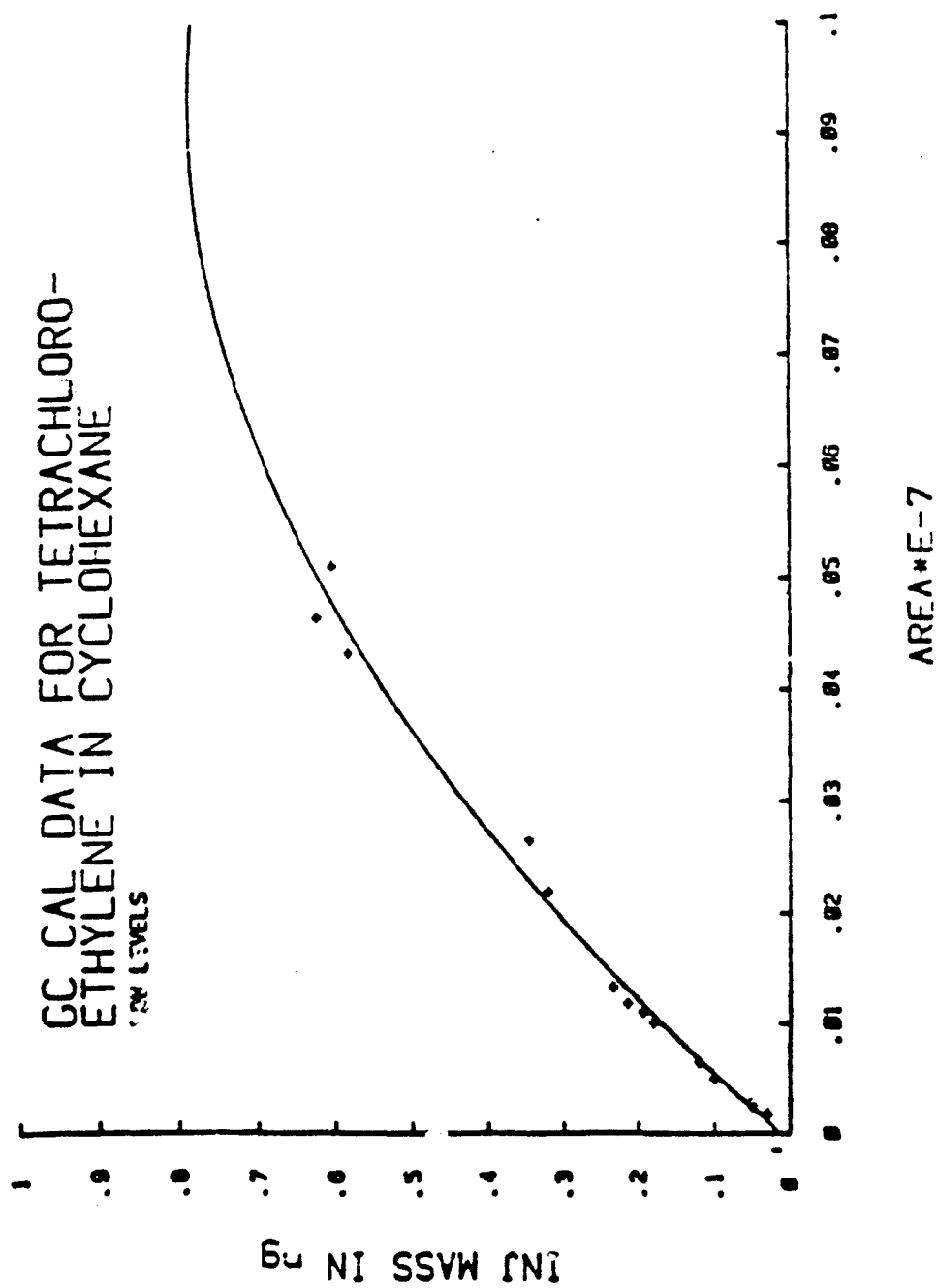


Figure A-5. GC Calibration Data for Tetrachloroethylene in Cyclohexane (Low Levels).

soil. The total weight of tetrachloroethylene recovered in the distillate was corrected by the weight of soil extracted to obtain the apparent concentration in the soil. A recovery efficiency correction was used to obtain the true concentration of tetrachloroethylene in soil.

Let the area response of the tetrachloroethylene peak be A_x when V_x μ l of a prepared soil extract (obtained by the steam distillation of a soil sample) is injected. Then the concentration of the tetrachloroethylene in the soil can be calculated by the following equations.

$$C_x = \frac{Y_x}{V_x} \frac{(DF) V_e}{M_s} \left(\frac{1}{\epsilon}\right) \text{ ppm} \quad (\text{A-4})$$

$$Y_x = 10^{-3} a_1 A_x^{\frac{1}{2}} \quad (\text{A-5})$$

- where C_x = concentration of tetrachloroethylene in soil, ppm
 Y_x = weight of tetrachloroethylene injected on column, ng
 V_x = volume of sample injected, μ l
 DF = dilution factor if prepared sample is diluted before injection
 V_e = volume of extract made by steam distillation, ml
 M_s = weight of soil extracted, gms
 ϵ = recovery efficiency
 a_1 = parameters of the second order polynomial fitted to the GC calibration curve (obtained from equation 1, 2, or 3)
 A_x = scaled area response of the tetrachloroethylene peak.

D. GC/MS ANALYSIS OF EXTRACTS AND DISTILLATES CONTAINING CHLOROBENZENE AND TETRACHLOROETHYLENE

The purpose of this experiment was to analyze the soil sample extracts for the presence of TRCE and CBZ, and to quantify the amount detected. This procedure can be carried out by using a gas chromatograph with an electron capture detector. Initially, this method was used for the analysis but there

were difficulties in the detection of CBZ due to its relatively low electron capture response. Therefore, combined gas chromatograph/mass spectrometry (GC/MS) was chosen as the analysis technique.

1. Equipment

Samples and standards were analyzed using combined capillary column GC/MS. The gas chromatograph, a Hewlett-Packard 5890, was equipped with a 30 m x 0.33 mm i.d. BP1 bonded phase vitreous silica (S.G.E. Australia) capillary column. Samples were injected in the splitless mode, with the column programmed from 40°C (isothermal for 5 min) up to 220°C at 4°/min. The carrier gas (He) flow rate was set at 0.83 ml/min. The column was coupled directly to the ion source of the mass spectrometer, a HP 5970A mass selective detector (MSD). The MSD was operated in the selected ion monitoring (SIM) mode.

Two masses for each of the compounds of interest, TRCE and CBZ, were selected for monitoring during the SIM experiment. The masses monitored for TRCE were 163.8 and 165.8 amu and 77.0 and 111.9 amu for CBZ. The process leading to the selection of these masses involved analyzing a standard solution containing TRCE and CBZ in the cyclic scan mode, scanning the mass range from m/e 34 to m/e 200. The mass spectrum of each compound was examined and the two masses having the highest intensity were selected for the SIM mode.

The parameters for the SIM experiment included setting up two time windows (one window for each of the 2 sets of masses). The times chosen for these windows were based on the retention times of the two compounds. The dwell time on each mass was 200 msec. The total cycle time was 0.5 sec/cycle. The multiplier applied voltage was set at 2.0 KeV. The data acquired were stored using the HP 9816S computer equipped with a 15 Mbyte hard disc drive. (The HP 9816S is the controller for the GC/MS system. It also receives the data from the detector and presents the data in a suitable form to the user.)

2. Quantification

An external standardization method was chosen in order to quantify the experimental samples submitted for analysis. A series of standard solutions in the concentration range 0.06 to 6.0 ng/μl for TRCE and CBZ was prepared. These solutions were used to calibrate the instrument. A calibration curve,

similar to the one illustrated in Figure A-6, was generated and used to determine the levels of TRCE and CBZ present in the experimental samples. To ensure instrument stability, a calibration standard was analyzed each work-day prior to sample analysis. If the value of the calibration standard was not within 10% of the working curve, then the instrument was recalibrated. The experimental sample concentration for each compound was determined from the calibration curve, using the standard program in the data system.

3. Quality Control

Quality control samples were periodically submitted along with the experimental samples for analysis in order to verify that the results reported were reliable.

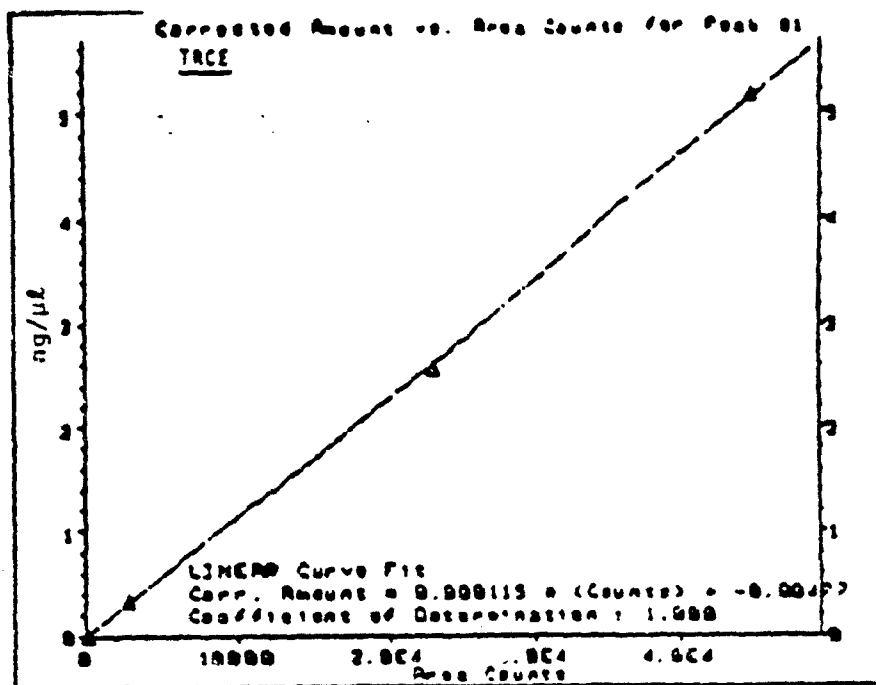
A summary of the results for the analysis of one set of QC samples is in Table A-3. The results reported are based on one analysis of each sample. The values obtained based on the analysis results between concentration range 6.0 to 0.20 ng/ μ l are within a 10% relative error. The percent relative error is higher for those concentrations which begin to fall outside the linear range of the calibration curve. Also, at the lower end of the calibration range, the concentrations begin to approach the lower detection limits of the instrument. In order to improve the results of the values obtained at the low concentration level, an additional calibration curve was prepared in the concentration range 0.2 to 0.02 ng/ μ l. All other samples were prepared to lie within the linear range of the calibration curve.

4. Detection Limits

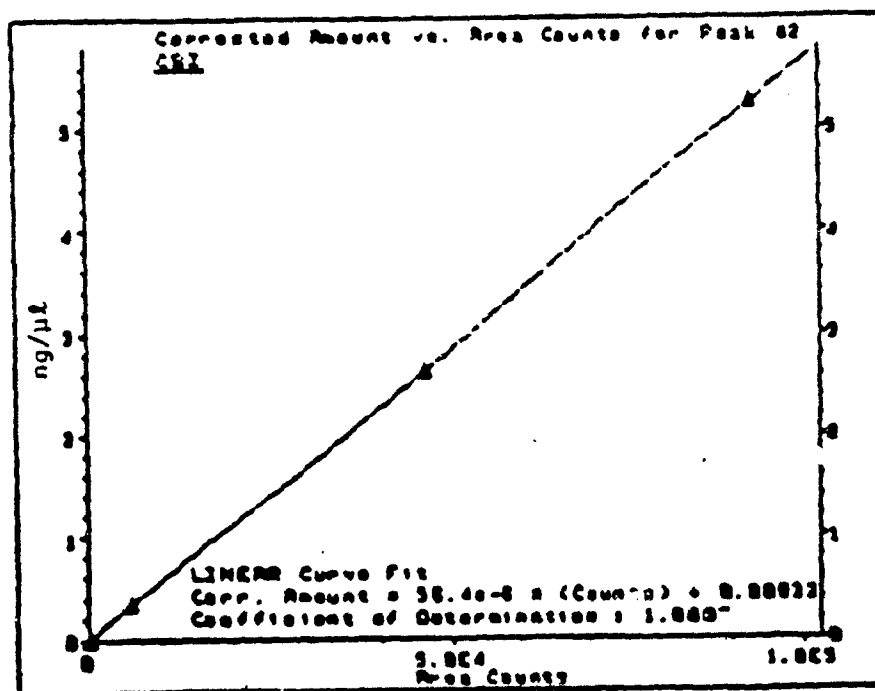
The detection limit is estimated to be 0.038 ng/ μ l for TRCE and 0.020 ng/ μ l for CBZ. These concentrations are based on a peak which is assumed to be 2.5 x the background noise. Converted to soil basis, this corresponds to 51 ppb for tetrachloroethylene and 27 ppb for chlorobenzene.

E. GC/MS PROCEDURE FOR EXTRACTS AND DISTILLATES CONTAINING JET FUEL

The purpose of this analysis was to determine the concentration of total aromatics, total nonaromatics, toluene and pentadecane in extracts obtained by Nielson-Kryger steam distillation of soil containing jet fuel.



(a)



(b)

Figure A-6. Sample Calibration Curve for: (a) Tetrachloroethylene (TRCE) and (b) Chlorobenzene (CBZ).

Toluene was chosen to represent the aromatics present in the samples, while pentadecane ($C_{15}H_{32}$) was selected to represent the high boiling compounds. Combined gas chromatography/mass spectrometry (GC/MS) was used as the analysis technique.

TABLE A-3. RESULTS OF QUALITY CONTROL SAMPLE ANALYSES

Sample	GC/MS Code	Expected Amt TRCE, ng/ul	Calculated Amt TRCE ng/ul	% Error	Expected Amt CBZ, ng/u	Calculated Amt CBZ, ng/ul	% Error
VS10-27-86 I	TB61028	6.195	5.590	9.76	4.340	3.740	13.8
VS10-27-86 II	TB71028	2.478	2.500	0.89	1.736	1.650	4.95
VS10-27-86 III	TB21029	0.620	0.611	1.45	0.434	0.405	6.68
VS10-27-86 IV	TB91028	0.248	0.264	6.45	0.174	0.182	4.60
VS10-27-86 V	TB41029	0.0991	0.127	28.1	0.0694	0.0864	24.5
VS10-27-86 VI	TB101029	0.0496	0.0665	34.1	0.0347	0.0487	40.3

(Note: TRCE = tetrachloroethylene, CBZ = chlorobenzene)

$$\% \text{ Error} = \frac{\text{Expected Amt} - \text{Calculated Amt}}{\text{Expected Amt}} \times 100\%$$

1. Equipment

Samples and standards were analyzed using combined capillary column GC/MS. The gas chromatograph, a Hewlett-Packard (HP) 5890, was equipped with a 30 m x 0.33 mm i.d. BPI bonded phase vitreous silica (S.G.E. Australia) capillary column. Samples were injected in the splitless mode, with the column programmed from 40°C (isothermal for 5 min.) up to 220°C at 6°/min. The carrier gas flow rate was set at 0.83 ml/min. The column was coupled directly to the ion source of the mass spectrometer, a HP 5970A mass selective detector (MSD). The quadrupole mass filter of the MSD was scanned from m/e 20 to m/e 250 at a scan rate of 0.9 scans/sec during the run. The multiplier applied voltage was set at 1.8 KeV. The data acquired were stored using the HP9816S computer equipped with a 15 M-byte hard disc drive.

2. Quantification

An internal standardization method was used to quantify the experimental samples. The soil extracts were spiked prior to extraction with a standard mixture containing the surrogate standards, toluene- d_8 , octane- d_{10} , and decane- d_{22} , and also the internal standard, m-fluoriodobenzene (FIB). After extraction, the hexane extract was spiked with the internal standard 4-fluoro-2-iodotoluene (IFT). An aliquot of this extract was analyzed by GC/MS. The distillate samples were spiked with the internal standard IFT immediately prior to analysis.

Relative response factors (RRF) were determined from the analysis of a standard solution containing the internal standards and the surrogate standards (toluene- d_8 and decane- d_{22}) representative of the two target compounds of interest. Response factors were calculated from the following equation:

$$RF = \frac{(A_x) (Q_{IS})}{(A_{IS}) (Q_x)}$$

where A_x = total peak area of the analyte

A_{IS} = total peak area of the specified internal standard (IFT)

Q_{IS} = concentration of the internal standard (ng/ μ l) (IFT)

Q_x = concentration of the analyte in the standard (ng/ μ l)

The standard solution was analyzed in duplicate and an average response factor was generated for each compound of interest. The values are summarized in Table A-4.

TABLE A-4. AVERAGE RESPONSE FACTORS FOR CALIBRATION COMPOUNDS

Compound	Average Response Factor With Respect to IFT
Toluene- d_8	1.45 $\pm$ 0.01
Decane- d_{22}	1.96 $\pm$ 0.01

The average response factors for toluene- d_8 and decane- d_{22} were used in the quantification of toluene and pentadecane present in the samples.

The quantification of the total aromatics was based on the total area sum of those peaks with the characteristic m/e 91 in the full GC/MS profile. This area response value was subtracted from the total area response for the entire chromatographic envelope to determine the nonaromatic contributions to the amount of material present in the soil.

3. Recovery Efficiencies

The recovery efficiency for the soil extraction experiments was determined by comparing the actual amount of the surrogate standards found in the samples to the expected amount based on the known amount spiked onto the soil sample prior to extraction. The actual amount of the surrogate standard was determined from the relationship:

$$\text{ng}/\mu\text{l} = \frac{(A_1) (I_s)}{(A_{1s}) (RF)}$$

where A_1 = peak area of the surrogate

A_{1s} = peak area of the internal standard

I_s = the concentration of internal standard in the sample (ng/ μ l)

RF = the response factor determined from the standard analysis (reported in Table A-4)

The actual amount was then compared to the expected amount and a percent recovery was calculated. The results are summarized in Table A-5.

TABLE A-5. RECOVERY EFFICIENCIES FOR THE SURROGATE STANDARDS
TOLUENE- d_8 AND DECANE- d_{22}

Expt. No.	Initial Soil		Treated Soil	
	Toluene- d_8	Decane- d_{22}	Toluene- d_8	Decane- d_{22}
6	58%	96%	93%	90%

Note: Recovery efficiencies were not calculated for Experiment 5 because these soil samples were not properly spiked with the surrogate standards.

Good peak response was obtained for the spent soil samples.

4. Detection Limits

The detection limit was defined as the minimum detectable concentration required to produce a signal 2.5 times the average background signal. Based on the signal response for the internal standard IFT, the detection limit was determined to be 4 ng/ul.

APPENDIX B

ANALYSIS OF VOLATILE HYDROCARBONS IN SOIL
BY PURGE AND TRAP PROCEDURE

APPENDIX B

ANALYSIS OF VOLATILE HYDROCARBONS IN SOIL BY PURGE AND TRAP PROCEDURE

In this appendix the procedures for the analysis of volatile aromatic, aliphatic and selected chlorinated hydrocarbons is given. Composite samples of soils prepared by combining pre- and posttest soil samples were analyzed by the purge and trap procedure. The method for the preparation of composite samples was discussed in Section V.A.4 on page 64. The purge and trap analysis is a three-step procedure. These steps are:

- Extraction of soil with methanol
- Purge and trap of the soil extract
- Desorption of trapped contaminants and injection into the GC/MSD system.

The procedure for each of the three steps is described below.

A. SOIL EXTRACTION

1. Equipment

Fifty milliliter glass centrifuge tubes with Teflon-lined screw caps, wrist action shaker, laboratory centrifuge, 10 and 25 milliliter volumetric flasks, 20 milliliter pipette, 0-100 microliter adjustable automatic pipette, analytical balance.

2. Solvents and Standards

Purge and trap grade methanol supplied by Burdick and Jackson, 99.5 percent d_6 -Benzene supplied by Stohler Isotope Chemicals, Innerberg, Switzerland; Bromopentafluorobenzene (BPFB) supplied by Organic Chemicals Division, SCM Corporation.

3. Preparation of Standards

The surrogate standard, d_6 -benzene, was prepared in methanol at a concentration of approximately 880 $\mu\text{g/ml}$. This standard was prepared gravimetrically in a 10-milliliter volumetric flask. The standard was stored in 8-milliliter amber-colored vial with Teflon-lined screw cap. The internal standard, bromopentafluorobenzene was prepared in methanol at a concentration of approximately 400 $\mu\text{g/ml}$. The surrogate standard was spiked on the soil prior to extraction. The internal standard was spiked to the extract.

4. Procedure for Soil Extraction

Approximately 2.0 grams of soil was accurately weighed into a centrifuge tube. Using the 100 microliter automatic, adjustable pipette, 50 microliter of the surrogate standard was spiked on the soil in the centrifuge tube. The equivalent concentration of the surrogate standard in the soil was 22 ppm. Then, using a 20 milliliter pipette, 40 milliliters of methanol was added to the centrifuge tube. The tube was capped and placed on a wrist action shaker for a period of 15 minutes. After extraction, the tube was placed in a centrifuge for a period of 15 minutes at 4000 rpm.

The supernatant liquid was transferred to a 25 milliliter volumetric flask till it was partially full. Then 50 microliters of the internal standard, BPFB, was spiked into the flask. The volume in the flask was adjusted to 25 milliliters with the clear supernatant liquid from the centrifuge tube. The prepared extract was stored under refrigeration in a 30-milliliter amber-colored vial. The equivalent concentration of the internal standard in the sample was 0.77 $\mu\text{g/ml}$.

B. PURGE AND TRAP PROCEDURE

1. Equipment

Semiautomatic purge and trap concentrator, Model No. LCS-3, supplied by Tekmar Company, 100 microliter syringe, 5 milliliter gas tight syringe, and syringe cleaner.

2. Sample Concentration by Purge and Trap Procedure

For either standards or unknown sample extracts, 100 microliters of the prepared sample was added to 5 milliliter of water placed in the sampler of the purge and trap device. The water was purged with zero grade helium for 11 minutes, followed by a 4 -minute dry purge. The desorption cycle and the GC/MS injection sequence are started simultaneously. For the first 5 minutes of the desorption step, the GC column is maintained at 30°C, then heated to 220°C at a rate of 4°C/minute, and held at the final temperature for 3.5 minutes. The desorbed volatile components were conveyed through a heated line to the carrier gas input of the GC/MS injector port. This was achieved by routing the GC/MS carrier gas through the desorb gas inlet port of the purge and trap device. In the purge and dry-purge mode the desorb gas was bypassed by the six-position valve in the purge and trap device, and conveyed directly to the carrier gas input of the GC/MS injector port.

At the end of the desorb cycle, the bake cycle was initiated during which the traps are heated to 180°C for a period of 7 minutes. At the end of the bake period, the instrument is allowed to cool until the trap temperature falls below 30°C.

C. GC/MS ANALYSIS AND CALIBRATION PROCEDURE

1. Equipment

Hewlett Packard 5970A mass selective detector interfaced with Hewlett Packard 5890 gas chromatograph; 30 meters long, 0.33 millimeters internal diameter, B1 bonded phase vitreous silica, capillary column supplied by SGE, Australia. The GC column was directly coupled to the ion source of the mass spectrometer.

2. Solvents and Standards

Purge and trap grade methanol, supplied by Burdick and Jackson; hydrocarbon free, deionized millipore water; methylene chloride, chloroform, 1,1,1-trichloroethane, benzene, cyclohexane, trichloroethylene, toluene, octane, tetrachloroethylene, chlorobenzenes, BPFB, and d_6 -benzene.

3. GC/MS Operating Conditions

The GC oven was temperature programmed to hold the initial temperature at 30°C for 5 minutes, followed by a temperature ramp of 4°C/minute to 220°C where the temperature was held for 3.5 minutes. The injection port temperature was 250°C. The quadrupole mass filter was scanned from $m/e = 45$ to $m/e = 260$ at a scan rate of 0.5 scans/sec during the full scan analysis. The full scan analysis was used to select the ions to be monitored in the single ion mode for each of the 9 target compounds. The analysis of the unknown samples was done in the single ion mode based on the selected ions. Table B-1 provides a list of target compounds for the pretest soil samples and their corresponding ion mass ratio.

TABLE B-1. TARGET COMPOUNDS FOR PRETEST
PURGE AND TRAP SAMPLES

Compound	m/e
1,1,1-Trichloroethane	97
Benzene	78
Trichloroethylene	97
Toluene	91
Octane	71
Tetrachloroethylene	164
BPFB*	246
d_6 -Benzene	84
Chlorobenzene	112

*BPFB: Bromopentafluorobenzene

During the analysis of the pretest soil samples, many of the chlorinated species were not detected in the soil. Therefore, these targetted compounds were dropped during the posttest soil sample analyses. Table B-2 gives the list of target compounds for the posttest soil samples. The d_6 -benzene was used as a surrogate to assess recovery efficiency, while BPFB was used as an internal standard.

TABLE B-2. TARGET COMPOUNDS FOR POSTTEST
PURGE AND TRAP SAMPLES

Compound	m/e
Toluene	91
Chlorobenzene	112
Octane	71
BPFB*	246
d ₆ -Benzene	84

*BPFB: Bromo pentafluorobenzene

In addition to the target compounds, the total aliphatics were estimated by summing the area contributions of all compounds containing a m/e = 71 fragment. The total aromatics were estimated by summing the area contributions of all compounds containing a m/e = 91 fragment. The area response of the aliphatics was converted to a equivalent octane concentration by using the relative response factor for octane and the aromatics were reported as equivalent toluene.

4. Determination of GC/MS Linear Range

The linear response range of the GC/MS system was determined for each of the targetted compounds listed in Table B-1 by injecting three different standards in the range of 1.0 to 10 ng/ul. Table B-3 provides the information on the results of the linearity studies. A straight line was fitted to each curve and the coefficient of determination, r, was determined. The results show that the system response is linear in the range of 1 to 10 nanogram on column.

5. Calibration of the Analytical System

The analytical system comprises of the purge and trap device connected to the sample injection port of the GC/MS system. The calibration of the system was done by running three different calibration standards to determine the relative response factors for each targetted compound. The concentration of the calibration standards was such that when 100 microliters were injected

TABLE B-3. DETERMINATION OF LINEAR RESPONSE RANGE FOR THE GC/MS SYSTEM
(All Based on 1.0 μ l Injection)

Component	Mass on Column ng Y	Area Response X	Intercept C	Slope m	r ²
Methylene chloride	1.06	46,326	0.607	9.09×10^{-6}	1.000
	3.18	287,629			
	10.60	1,098,204			
Chloroform	0.895	38,693	1.37	8.44×10^{-6}	0.963
	2.69	58,005			
	8.95	900,417			
Trichloroethane	1.17	110,280	0.274	9.91×10^{-6}	0.998
	3.51	302,716			
	11.71	1,158,744			
Benzene	1.05	344,606	0.145	3.15×10^{-6}	0.998
	3.15	883,737			
	10.49	3,300,588			
Trichloroethylene	0.88	98,267	0.256	8.05×10^{-6}	0.998
	2.64	269,812			
	8.78	1,063,554			
Toluene	1.04	193,824	0.273	4.99×10^{-6}	0.998
	3.12	522,304			
	10.40	2,039,958			
Octane	0.98	122,848	0.241	8.23×10^{-6}	0.996
	2.95	290,548			
	9.84	1,172,305			
Tetrachloroethylene	0.97	125,710	0.107	7.44×10^{-6}	1.000
	2.92	366,859			
	9.74	1,297,209			
Chlorobenzene	1.11	182,196	0.294	5.72×10^{-6}	0.998
	3.32	479,595			
	11.06	1,889,588			
BFBP	1.19	190,094	0.388	5.81×10^{-6}	0.996
	3.57	484,113			
	11.89	1,988,500			

into the sampler of the purge and trap device, the amount delivered on-column of the GC was in the range of 1 to 10 nanogram. In all analyses, a 10:1 splitter was used to split away the sample prior to delivery on column. Table B-4 lists the concentration of the target compounds in the three different standards.

TABLE B-4. CONCENTRATION OF CALIBRATION STANDARDS USED
FOR PRETEST SOIL SAMPLE ANALYSIS

Component	Standard Number		
	310	33	31
	Concentration, ng/ μ l		
Methylene chloride	1.06	0.32	0.106
Chloroform	0.90	0.27	0.090
Trichloroethane	1.17	0.35	0.117
Benzene	1.05	0.31	0.105
Trichloroethylene	0.88	0.26	0.088
Toluene	1.04	0.31	0.104
Octane	0.98	0.30	0.098
Tetrachloroethylene	0.97	0.29	0.097
Chlorobenzene	1.11	0.33	0.111
BPF8	1.19	0.36	0.119
d ₆ -Benzene	0.95	0.29	0.095
Approx. mass on column for 100 μ l injection with 10:1 split, ng	10	3.0	1.0

For posttest samples, two standard solutions were made. These solutions contained only five components as listed in Table B-5. All the other components were eliminated because these were not detected in the pretest soil samples.

TABLE B-5. CONCENTRATION OF CALIBRATION STANDARDS USED FOR POSTTEST SOIL SAMPLE ANALYSIS

Component	Standard Number	
	1	2
	Concentration, ng/μl	
Toluene	.520	.260
Chlorobenzene	.553	.277
Octane	.492	.246
BPFB	.594	.297
d ₆ -Benzene	.475	.238
Approx. mass on column for 100 μl injection with 10:1 split, ng	5	2.5

The relative response factor, RF, for any component X was calculated with the following equation:

$$RF_X = \frac{A_X}{W_X} \frac{W_{IS}}{A_{IS}}$$

where A_X = area response for component x

W_X = mass on column for component x, ng

W_{IS} = mass on column for the internal standard (BPFB), ng

A_{IS} = area response for the internal standard.

The pretest soil samples were analyzed in the period 9/30/87 through 10/8/87. In this time interval, standards were injected daily to determine the day-to-day variation in the component response factors. Table B-6 lists the data on daily determination of response factors; it also provides the average, standard deviation (SD), and percent relative standard deviation for all the components. The overall average response factors were used in the calculations of unknown sample concentrations.

The response factors for posttest soil samples were developed by injecting the two standards described in Table B-5. At least one standard was injected every day when the posttest samples were being analyzed. The average

TABLE B-6. RELATIVE RESPONSE FACTORS FOR PRETEST PURGE AND TRAP SAMPLE ANALYSIS

Date	GC/MS Run No.	Methylene-chloride m/e=84	Trichloro-ethane m/e=97	Benzene m/e=78	Trichloro-ethylene m/e=97	Toluene m/e=91	Octane m/e=71	Tetrachloro-ethylene m/e=164	Chloro-benzene m/e=112	BPFB m/e=246	d ₆ -Benzene m/e=84
9/25/87	318	0.498	0.348	1.008	0.258	1.288	0.669*	0.361	0.667	1.0	0.537*
	338	0.490	0.449	1.454	0.245	1.560	0.225	0.383	0.791	1.0	0.977
	3108	0.491	0.502	1.653	0.301	1.834	0.342	0.503	1.011	1.0	1.294
9/28/87	31C	0.786	0.964	2.106	0.356	2.593	0.216	0.597	1.071	1.0	1.694
	33C	0.641	0.753	2.091	0.398	2.372	0.359	0.508	1.207	1.0	1.673
	310C	0.604	0.782	2.578	0.317	3.568	0.392	0.615	1.350	1.0	1.994
9/29/87	330	0.766	0.964	2.107	0.358	2.497	0.341	0.597	1.071	1.0	1.694
	310D	0.507	0.564	1.693	0.229	1.735	0.236	0.546	1.016	1.0	1.347
	310	0.708	0.725	1.947	0.181	2.129	0.127	0.412	0.911	1.0	1.010
10/3/87	33E	0.670	0.577	2.340	0.312	3.681	0.296	0.389	1.271	1.0	1.350
	310E	0.314	0.785	2.019	0.222	3.046	0.293	0.473	0.984	1.0	1.616
	31E	0.957	0.916	1.944	0.129	2.517	0.268	0.413	0.985	1.0	1.209
10/6/87	33F	0.637	0.643	2.172	0.294	3.343	0.314	0.488	1.013	1.0	1.543
	310F	0.633	0.619	2.653	0.367	2.326	0.341	0.513	1.347	1.0	1.644
10/8/87	Average	0.640	0.685	1.983	0.283	2.464	0.288	0.486	1.050	1.0	1.465
	S.D.	0.138	0.190	0.433	0.077	0.739	0.073	0.085	0.196	-	0.297
	RSD, %	21.6	27.8	21.8	27.1	30.0	25.2	17.4	18.6	-	20.2

*Excluded in calculation of average Rf.

response factors over all the calibration standard runs were used for data reduction. Table B-7 gives a summary of the average response factors, standard deviation and relative standard deviation for each component.

TABLE B-7. AVERAGE* RESPONSE FACTORS FOR POSTTEST PURGE AND TRAP ANALYSIS

Component	Response Factor	Standard Deviation	Relative Standard Deviation
d ₆ -Benzene	1.506	0.147	9.8
Toluene	3.066	0.483	15.7
Octane	0.295	0.041	13.9
Chlorobenzene	1.276	0.186	14.6

*Average of 10 calibration runs, with two different standards.

6. Data Reduction

The GC/MS data was used for the calculation of contaminant concentration in soil. All concentration data was reported on a moisture-free basis. The following equations were used to determine the concentration of individual components.

The amount of component x delivered on column was calculated by

$$W_x = \frac{A_x}{(RF)_x} \frac{W_{is}}{A_{is}}$$

where A_x = area of component x

$(RF)_x$ = response factor of component x

W_{is} = mass of internal standard, BPFB, in 100 μ l injection volume, ng

A_{is} = area of internal standard.

The concentration of component X in the extract is given by

$$C_x = \frac{W_x}{100} \frac{(ng)}{(\mu l)}, \text{ ng}/\mu l \text{ or } \mu g/ml$$

The concentration of component x in soil is given by

$$S_x = \frac{40 C_x}{S_w f_s}, \text{ } \mu\text{g/gm or ppm}$$

where C_x = concentration, $\mu\text{g/ml}$ of the extract

S_w = mass of soil extracted in 40 milliliters of MeOH, gm

f_s = fraction dry soil in the sample.

The recovery of surrogate, d_6 -benzene was calculated by determining the mass on column, W_x , and comparing it to the known amount of surrogate spiked into the soil prior to extraction.

In the posttest samples, it was found necessary to make corrections for solvent blank background levels. Three different runs were made in which 100 of methanol solvent was injected into the purge and trap device and analyzed as if a sample extract was injected. For each run, the area response for total aromatics, total aliphatics, and toluene was determined. A blank correction was made by subtracting the average blank areas from the corresponding area response of the real sample. The reason for making this correction for posttest samples is due to the low levels of the contaminants present in the treated soil, such that the area response of the component in the sample was comparable to the blank response.

APPENDIX C

**GC/MS PROCEDURE FOR THE ANALYSIS OF SOIL EXTRACT
FOR SEMIVOLATILES**

APPENDIX C

GC/MS PROCEDURE FOR THE ANALYSIS OF SOIL EXTRACT FOR SEMIVOLATILES

All the jet fuel contaminated soil samples obtained from the field were analyzed for semivolatiles aromatics, aliphatics and hexadecane. Hexadecane was selected as a marker compound for the upper end of the range, since it has a normal boiling point of 290°C. The purge and trap procedure is not recommended for the determination of semivolatiles, due to poor recovery efficiency of materials with boiling points in excess of 120°C.

Prior to GC/MS analysis, the soils were subjected to a steam distillation, liquid-liquid extraction to remove the contaminants from the soil and transfer them to a suitable solvent. The extraction was performed in a Nielson-Krieger steam distillation, extraction apparatus described in Section A of Appendix A. The extraction procedure for soil samples is also described in Appendix A. In this appendix, the GC/MS analytical procedure used for the analysis of semivolatiles in the soil extract is described. The GC/MS procedures presented herein are to be distinguished from those presented in Appendix A. The procedures presented here are for field samples containing several hydrocarbon contaminants whereas those presented in Appendix A are for soils spiked with two contaminants.

A. GC/MS Analysis and Calibration Procedure

1. Equipment

The GC/MS system used for this analysis was the same as that used with the purge and trap system. Please see Appendix B, Section C.

2. Solvents and Standards

HPLC or better grade hexane; d_{22} -decane, 99.3 atomic percent deuterated, supplied by MSC Isotopes, Merck Frost, Montreal, Canada; d_8 -naphthalene, 98+% deuterated, supplied by Aldrich Chemical Company; d_8 -toluene, 99+% deuterated, supplied by Aldrich Chemical Company; and n-hexadecane, 99+% pure, ASTM grade.

3. GC/MS Operating Conditions

The GC oven was temperature programmed to hold the initial temperature at 30°C for 5 minutes after direct injection of 1.0 microliter of the sample. The oven was then heated at 4°C/minute to 250°C. The injection port was maintained at 250°C. The solvent delay was 15 minutes. Like the purge and trap analysis, the GC/MS data was acquired for specific target ions representing d_8 -naphthalene, d_{22} -decane, n-hexadecane, and also for determining all species with $m/e = 71$ and 91 fragments. The area of all components with a $m/e = 71$ fragment was used to estimate the concentration of total aliphatics which were reported as equivalent hexadecane. The area of all components with $m/e = 91$ fragment was used to estimate the concentration of total aromatics which were calibrated against the equivalent ion $m/e = 98$ from d_8 -toluene.

4. Calibration of the GC/MS System

The calibration of the GC/MS system was done by injection of standards containing the target compounds, surrogate and internal standards at two levels. Table C-1 gives the composition of the standards used for determination of the relative response factors used for analysis of the pretest soil samples. d_8 -Naphthalene was used as an internal standard.

TABLE C-1. CALIBRATION STANDARDS FOR PRETEST SOIL ANALYSIS FOR SEMIVOLATILES

Component	Ion	Composition, ng/ μ l	
		STD 1	STD 2
Toluene- d_8	98	141.0	14.1
Hexadecane	71	140.0	14.0
Napthalene- d_8	136	152.0	15.2
Decane- d_{22}	57	146.0	14.6

For the posttest soil samples, the calibration standards are described in Table C-2.

TABLE C-2. CALIBRATION STANDARDS FOR POSTTEST SOIL ANALYSIS FOR SEMIVOLATILES

Component	Ion	Composition, ng/ μ l	
		STD 1	STD 2
Toluene-d ₈	98	83.75	41.88
Hexadecane	71	86.25	43.12
Napthalene-d ₈	1336	79.75	39.88
Decane-d ₂₂	57	81.75	40.88

The pretest samples were analyzed in the period 11/4/87 through 12/1/87. In this time interval, the standards listed in Table C-1 were used for determination of the relative response factor. A total of 16 calibration standard injections were made. The response factors were calculated by the following equation:

$$(RF)_x = \frac{A_x}{W_x} \frac{W_{is}}{A_{is}}$$

where A_x = area of component x

W_x = mass of component x in 1.0 μ l injection of the standard

W_{is} = mass of the internal standard in 1.0 μ l injection, ng

A_{is} = area of the internal standard.

In Table C-3, the average response factors, standard deviation, and relative standard deviation based on 16 injections of the two standards listed in Table C-1, is given.

In Table C-4, the average response factors, standard deviation, and relative standard deviations for the analysis of posttest soil samples is given. These data are based on the injections of the two calibration standards listed in Table C-2. The average is based on four different injections of the standards between 2/9/88 through 2/12/88 during which all the posttest samples were analyzed for semivolatiles.

TABLE C-3. AVERAGE RESPONSE FACTORS FOR PRETEST SOIL ANALYSIS FOR SEMIVOLATILES

Component	Ion	Response Factor	Standard Deviation	Relative Standard Deviation %
Toluene-d ₈	98	0.639	0.0599	9.365
Hexadecane	71	0.467	0.0244	5.23
Napthalene-d ₈	136	1.000	-	-
Decane-d ₂₂	57	0.532	0.0827	15.54

TABLE C-4. AVERAGE RESPONSE FACTORS FOR POSTTEST SOIL ANALYSIS FOR SEMIVOLATILES

Component	Ion	Response Factor	Standard Deviation	Relative Standard Deviation %
Toluene-d ₈	98	0.539	0.041	7.62
Hexadecane	71	0.378	0.057	15.1
Napthalene-d ₈	136	1.000	-	-
Decane-d ₂₂	57	0.5139	0.0675	13.1

5. Data Reduction

The GC/MS data was used for the calculation of contaminant concentration in soil. All concentration data was reported on a moisture-free basis. The following equations were used to determine the concentration of individual components.

The amount of component x in the injected sample was calculated from

$$W_x = \frac{A_x}{(RF)_x} \frac{W_{1s}}{A_{1s}}, \text{ ng}$$

where A_x = area of component x

$(RF)_x$ = response factor of component x

W_{1s} = mass of internal standard, d₈-napthalene in 1.0 µl injection, ng

A_{1s} = area of internal standard.

The concentration of component x in the extract is given by

$$C_x = \frac{W_x \text{ (ng)}}{1.0 \text{ (}\mu\text{l)}} ; \text{ ng}/\mu\text{l or } \mu\text{g/ml}$$

The concentration of component x in soil is given by

$$S_x = \frac{V_t C_x}{S_w f_s} , \mu\text{g/gm or ppm}$$

where C_x = concentration of the extract, $\mu\text{g/ml}$

S_w = mass of soil extracted, gm

V_t = total volume of extract, always 100 ml

f_s = fraction dry soil in the sample.

APPENDIX D

RESULTS OF EP-TOXICITY TEST PROCEDURE
ON "TRENCH" AREA SOIL SAMPLES

APPENDIX D

RESULTS OF EP-TOXICITY TEST PROCEDURE ON "TRENCH" AREA SOIL SAMPLES

Samples of soil were obtained from the trench areas prior to excavation. These samples were analyzed by EPA Standard Method 1310 (Reference 4) to determine whether the trench area soil was hazardous as per EP-toxicity test, particularly for lead and heavy metals. This information was obtained to determine how the excavated soil would be disposed off if it was hazardous due to high concentrations of heavy metals.

Ten holes were made in the proposed trench area, and samples were retrieved from depth intervals of 6-12 in., and 30-36 in. From four of these 10 holes, samples were also obtained from a depth interval of 66-72 in. Samples from corresponding depth intervals were combined together to make three composite samples. In addition to these composite samples, one sample from 6-12 in. depth and another from 30-36 in. depth were also submitted. These samples were analyzed by an outside laboratory for heavy metals by EPA Method 1310. Table D-1 is a summary of results of five samples submitted for EP-toxicity test. The results show that the trench area soil is not hazardous for heavy metals as per the EP-toxicity test procedure.

TABLE D-1. RESULTS OF EP-TOXICITY TEST (EPA METHOD 1310)

Sample I.D.	Type	Depth in.	EP-Toxicity Concentration (mg/L)							Se (1.0)	Ag (5.0)	Max Allow- able
			As (5.0)	Ba (100.0)	Cd (1.0)	Cu (5.0)	Pb (5.0)	Hg (0.2)				
A	Composite	6-12"	<0.001	<1.0	<0.10	<0.10	<0.10	<1x10 ⁻⁴	<0.001	<0.10		
B	Composite	30-36	<0.001	<1.0	<0.10	<0.10	<0.10	<1x10 ⁻⁴	<0.001	<0.10		
C	Composite	66-72	<0.001	<1.0	<0.10	<0.10	<0.10	<1x10 ⁻⁴	<0.001	<0.10		
D	Point	6-12"	<0.001	<1.0	<0.10	<0.10	<0.10	<1x10 ⁻⁴	<0.001	<0.10		
E	Point	Hole #77 30-36	<0.001	<1.0	<0.10	<0.10	<0.10	1x10 ⁻⁴	<0.001	<0.10		
		Hole #10T										

SUBURBAN LABORATORIES, Inc.

4140 LITT DRIVE

HILLSDALE, ILLINOIS 60162 - 1183

EARL I. ROSENBERG
President

August 25, 1987

H. R. THOMAS, JR.
Director

Illinois Institute of Technology Research Institute
10 West 35th Street
Chicago, Illinois 60616

Attention: Mr. Marsh Dev

Re: P.O. #64450

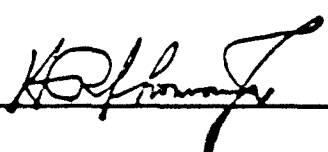
Sample Received: 8/13/87

Source: S/L #7-8008 - Combination Field Soil Sample A, 8/5/87

	E. P. Toxicity (mg/l)
(+) Arsenic	< 0.001
Barium	< 1.0
Cadmium	< 0.10
Chromium, Total	< 0.10
Lead	< 0.10
Mercury	< 0.0001
(+) Selenium	< 0.001
Silver	< 0.10

(+) by RCA

(< = less than)

ANALYSIS CERTIFIED BY: , Director (HRT/ak)

Members of American Society of Microchemists
American Chemical Society • American Society for Microbiology
Water Pollution Control Federation • Institute of Food Technologists
Certifications: NABSA #178 • IL Dept. of Public Health #1713 • Amer. Sales Trade Assn. • F.D.A. Reg. #140896 • IL EPA #100101

Figure D-1. EP-Toxicity Test Report for "Trench" Area Sample A.

SUBURBAN LABORATORIES, Inc.

4140 LITT DRIVE

HILLSDALE ILLINOIS 60162 - 1163

EARL I. ROSENBERG
President

August 25, 1987

H. R. THOMAS, JR.
Director

Illinois Institute of Technology Research Institute
10 West 35th Street
Chicago, Illinois 60616

Attention: Mr. Marsh Dev

Re: P.O. #64450

Sample Received: 8/13/87

Source: S/L #7-8009 - Combination Field Soil Sample B, 8/5/87

	<u>E. P. Toxicity</u> <u>(mg/l)</u>
(+) Arsenic	< 0.001
Barium	< 1.0
Cadmium	< 0.10
Chromium, Total	< 0.10
Lead	< 0.10
Mercury	< 0.0001
(+) Selenium	< 0.001
Silver	< 0.10

(+) By BCA

ANALYSIS CERTIFIED BY:

Director (RET/ch)

Members of American Society of Mass Spectrometry
American Chemical Society • American Society for Microbiology
Water Pollution Control Federation • Institute of Food Technology
Contributors: U.S.D.A. 61703 • U.S. Dept. of Public Health 617105 • Assoc. State Trade Assn. • F.D.A. Reg. 61410270 • N. EPA 610270

Figure D-2. EP-Toxicity Test Report for "Trench" Area Sample B.

SUBURBAN LABORATORIES, Inc.

4148 LITT DRIVE

HILLSDALE, ILLINOIS 60162 - 1183

EARL I. ROSENBERG
President

August 25, 1987

H. R. THOMAS, JR.
Director

Illinois Institute of Technology Research Institute
10 West 35th Street
Chicago, Illinois 60616

Attention: Mr. Marsh Dev

Re: P.O. #64450

Sample Received: 8/13/87

Source: S/L #7-6010 - Combination Soil Sample C, 8/5/87

	E. P. Toxicity (ug/l)
(+) Arsenic	< 0.001
Barium	< 1.0
Cadmium	< 0.10
Chromium, Total	< 0.10
Lead	< 0.10
Mercury	< 0.0001
(+) Selenium	< 0.001
Silver	< 0.10

(+) By BCA

ANALYSIS CERTIFIED BY:

Director (ERT/ch)

Members of American Society of Water Scientists
American Chemical Society • American Society for Microbiology
Water Pollution Control Federation • Institute of Food Technology
Certifications: U.S.D.A. 01702 • U.S. Dept. of Public Health 017102 • Amer. Spice Trade Assn. • F.D.A. Reg. #1412295 • U.S. EPA #105700

Figure D-3. EP-Toxicity Test Report for "Trench" Area Sample C.

SUBURBAN LABORATORIES, Inc.

4130 LITT DRIVE HILLSIDE, ILLINOIS 60162 - 1163

EARL ROSENBERG
President

August 25, 1987

H. R. THOMAS, JR.
Director

Illinois Institute of Technology Research Institute
10 West 35th Street
Chicago, Illinois 60616

Attention: Mr. Marsh Dev

Re: P.O. #64450

Sample Received: 8/13/87

Source: S/L #7-8011 - Combination Soil Sample D, 8/5/87

	E. P. Toxicity (ug/l)
(+) Arsenic	< 0.001
Barium	< 1.0
Cadmium	< 0.10
Chromium, Total	< 0.10
Lead	< 0.10
Mercury	< 0.0001
(+) Selenium	< 0.001
Silver	< 0.10

(+) By HCA

ANALYSIS CERTIFIED BY:

Director (MRT/ch)

Members of American Chemical Society • American Society for Microbiology
American Chemical Society • American Society for Microbiology
Water Pollution Control Federation • Institute of Food Technologists
Certifications: U.S.E.A. #1788 • U.S. Dept. of Public Health #17135 • Assoc. State Trade Assoc. • P.O.A. Reg. #1418278 • IL EPA #148791

Figure D-4. EP-Toxicity Test Results for "Trench" Area Sample D.

SUBURBAN LABORATORIES, Inc.

4140 LITT DRIVE

HILLSDALE, ILLINOIS 60162 - 1183

EARL ROSENBERG
President

August 25, 1987

H R THOMAS, JR.
Director

Illinois Institute of Technology Research Institute
10 West 35th Street
Chicago, Illinois 60616

Attention: Mr. March Dev

Re: P.O. #64450

Sample Received: 8/13/87

Source: S/L #7-8012 - Combination Soil Sample E, 8/5/87

	E. P. Toxicity (mg/l)
(+) Arsenic	< 0.001
Barium	< 1.0
Cadmium	< 0.10
Chromium, Total	< 0.10
Lead	< 0.10
Mercury	< 0.0001
(+) Selenium	< 0.001
Silver	< 0.10

(+) By RCA

ANALYSIS CERTIFIED BY:

Director (HRT/ck)

Member of American Society of Testing and Materials
American Chemical Society • American Society for Microbiology
Water Pollution Control Federation • Institute of Food Technology
Certification: U.S.D.A. #1702 • IL Dept. of Public Health #17125 • Amer. Water Works Assn. • F.D.A. Reg. #1410226 • IL EPA #100101

Figure D-5. EP-Toxicity Test Results for "Trench" Area Sample E.

(The reverse side of this page is blank.)

APPENDIX E

SOIL TEMPERATURE DISTRIBUTION DATA

APPENDIX E

SOIL TEMPERATURE DISTRIBUTION DATA

A total of 49 temperature measurement points were available in the heated zone and the "trench" area. Figure E-1 is a plan view of the heated array which shows the various planes and sections in which temperature data are available. In Figures E-2 through E-8 temperature data in the ground planes and the exciter planes are presented. Figure E-9 shows the rate of temperature rise at a point in the center of the heated volume.

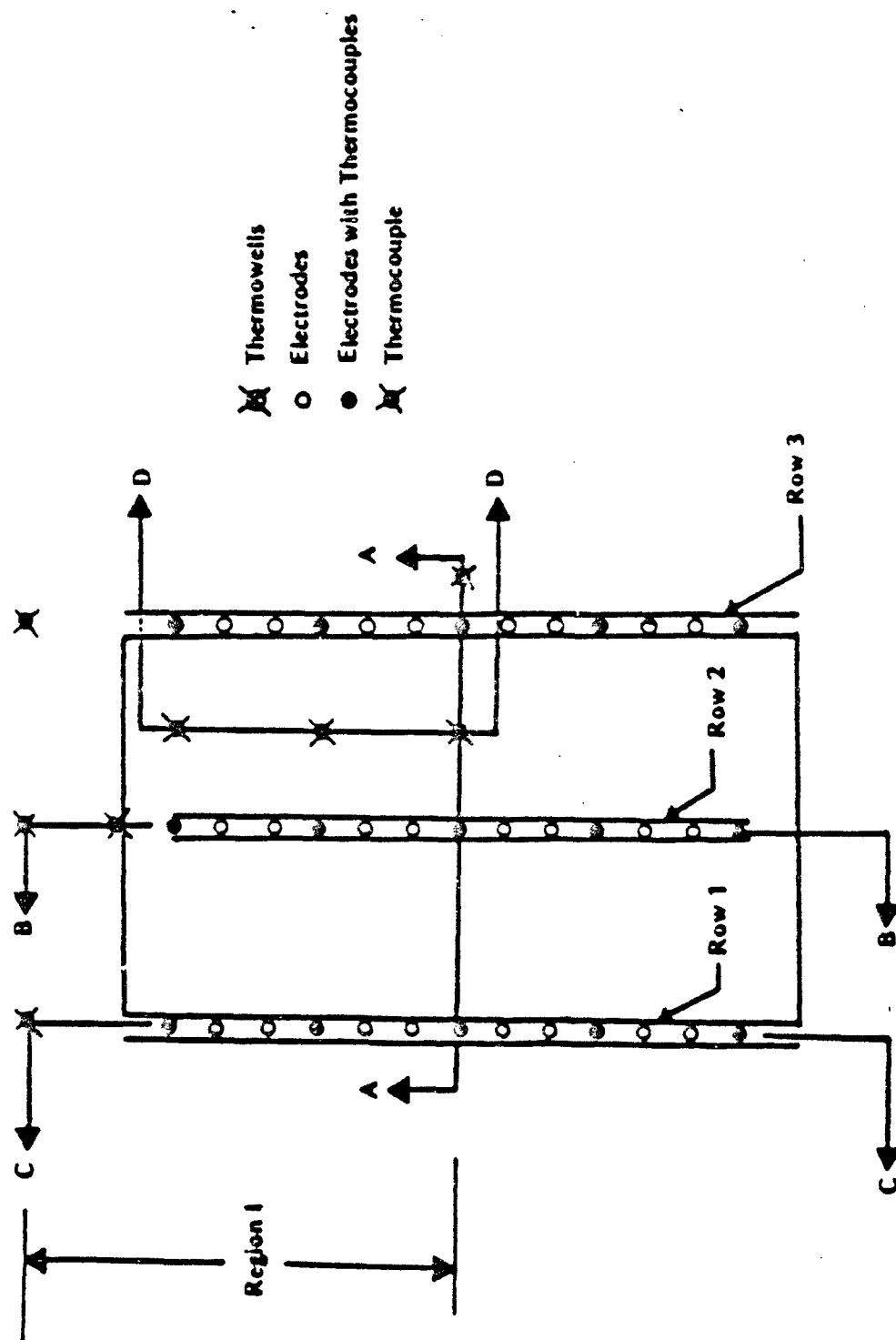


Figure E-1. Location of Temperature Measurement Boreholes.

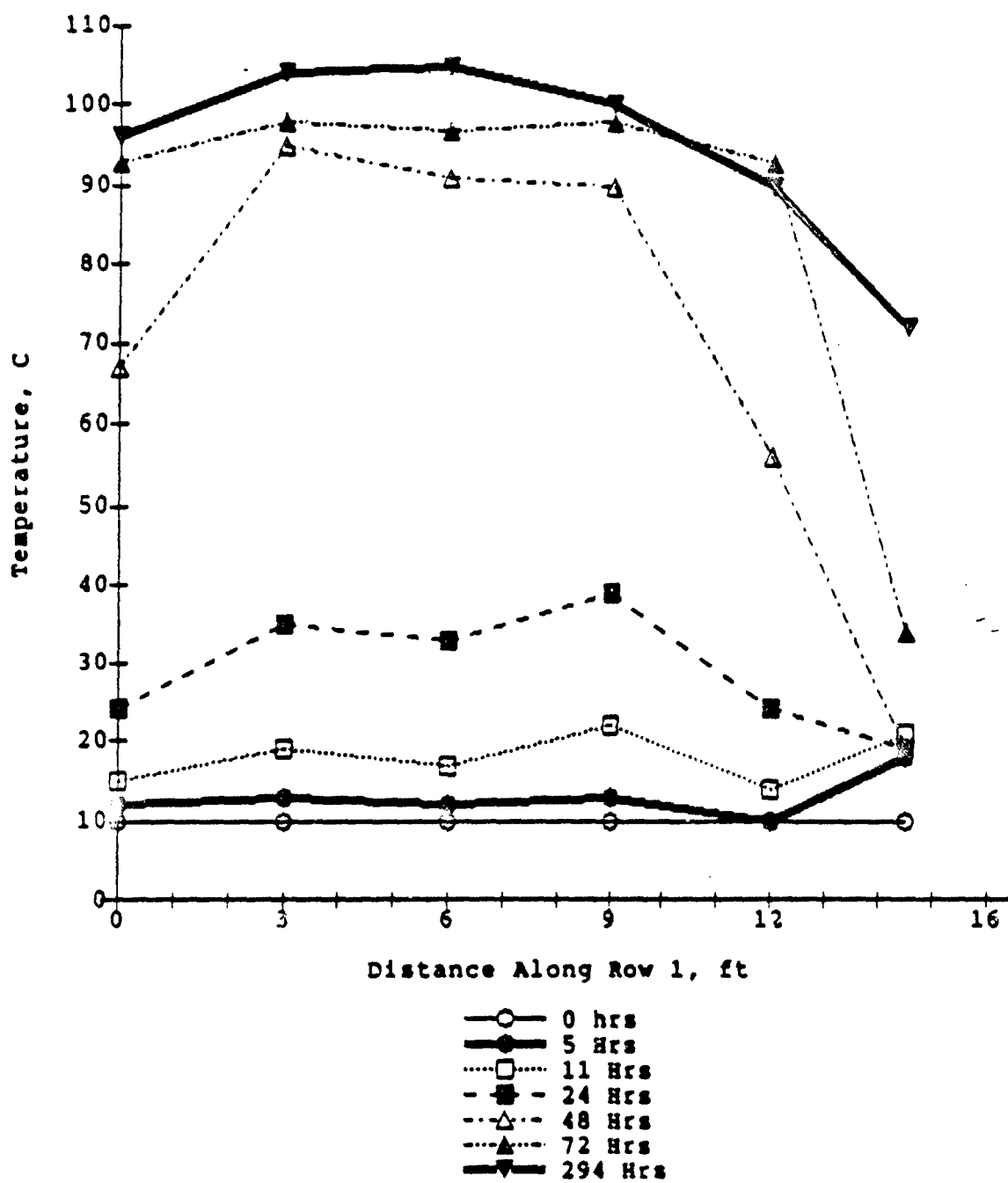


Figure E-2. Temperature Distribution in Plane CC (Row 1) at 36-inch Depth.

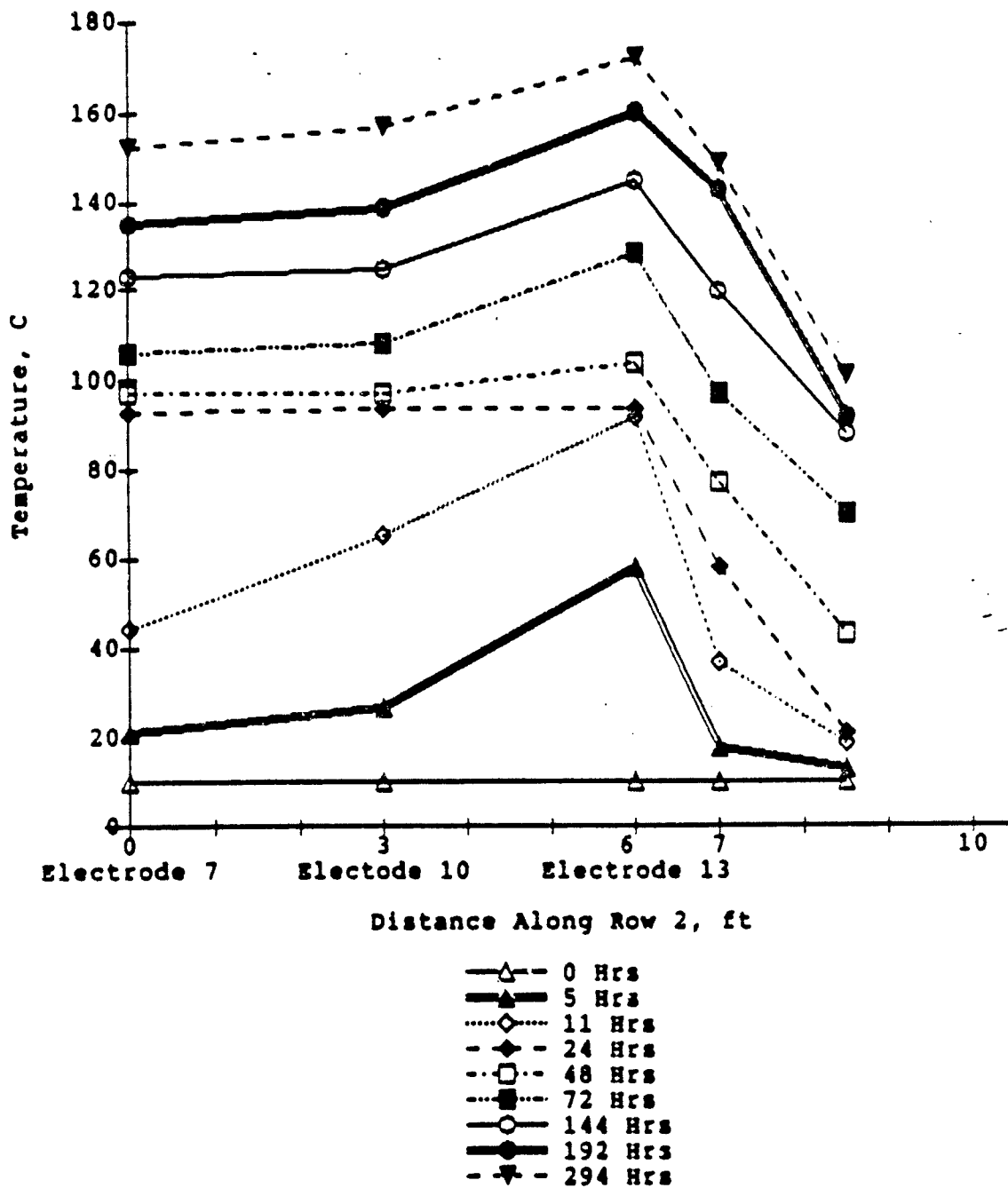


Figure E-3. Temperature Distribution Along Plane BB in Region 1 at 12-inch Depth.

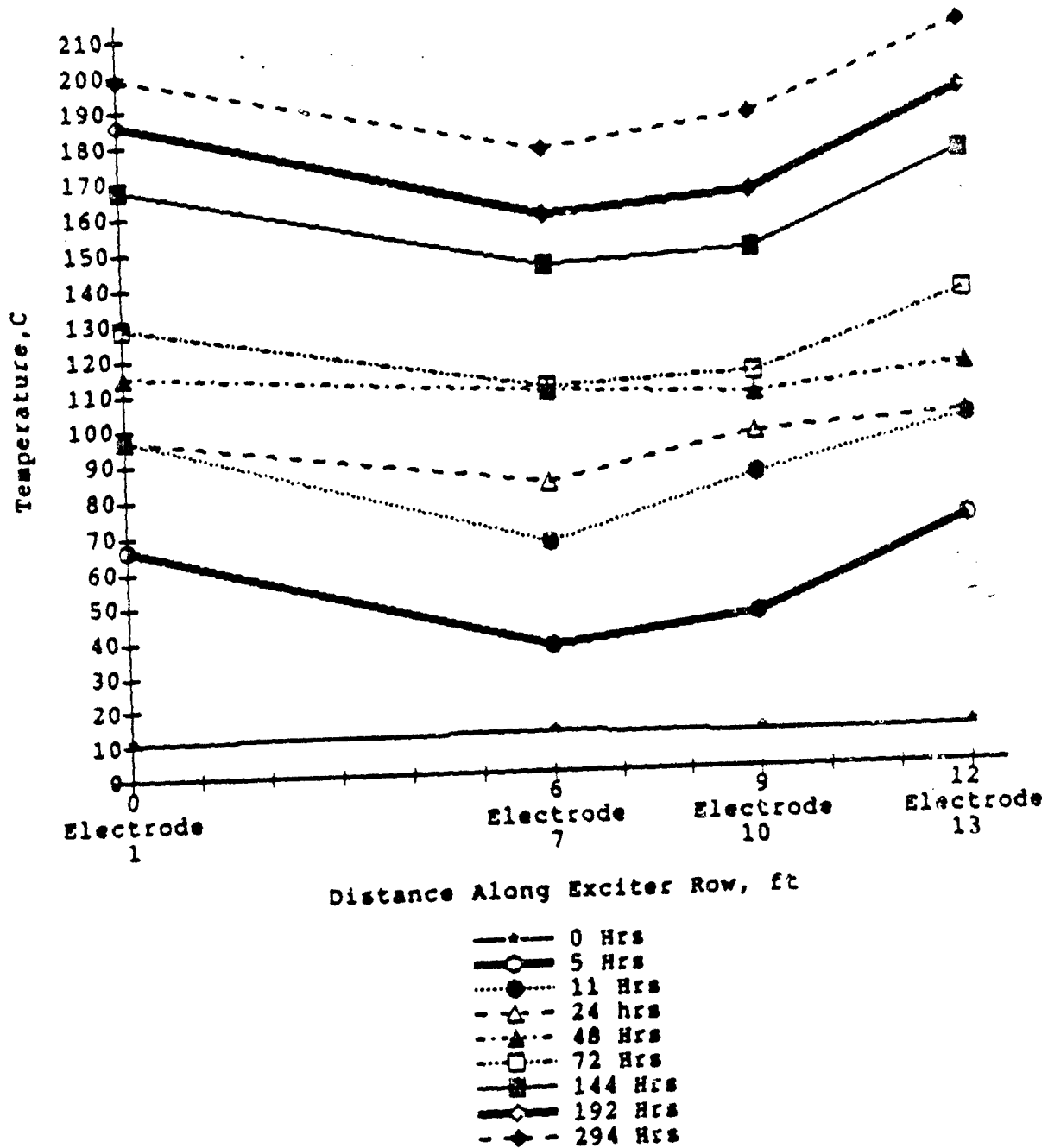


Figure E-4. Temperature Distribution Along Exciter Row
Depth 72 inches.

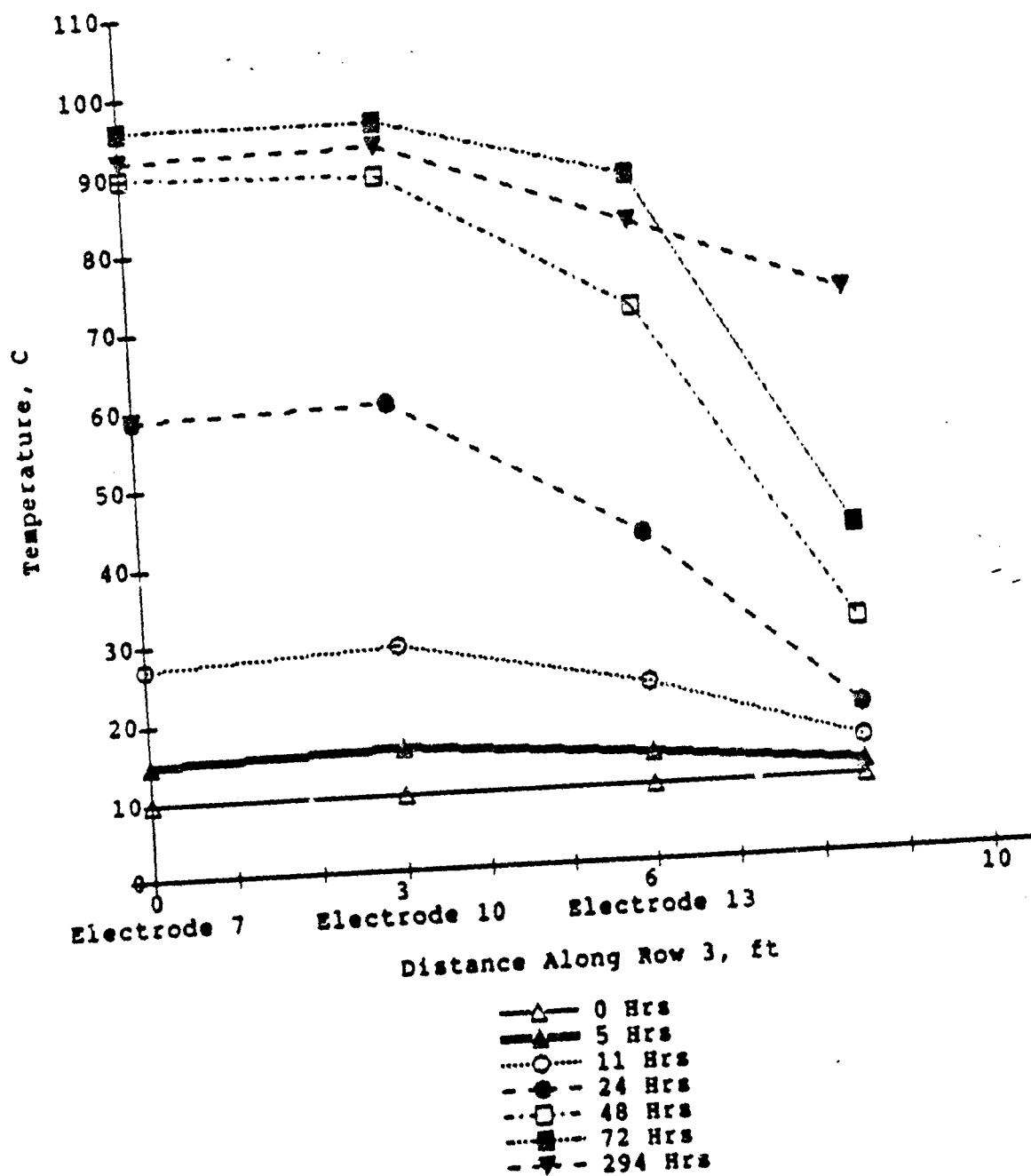


Figure E-5. Temperature Distribution Along Row 3 in Region 1 at 12-inch Depth.

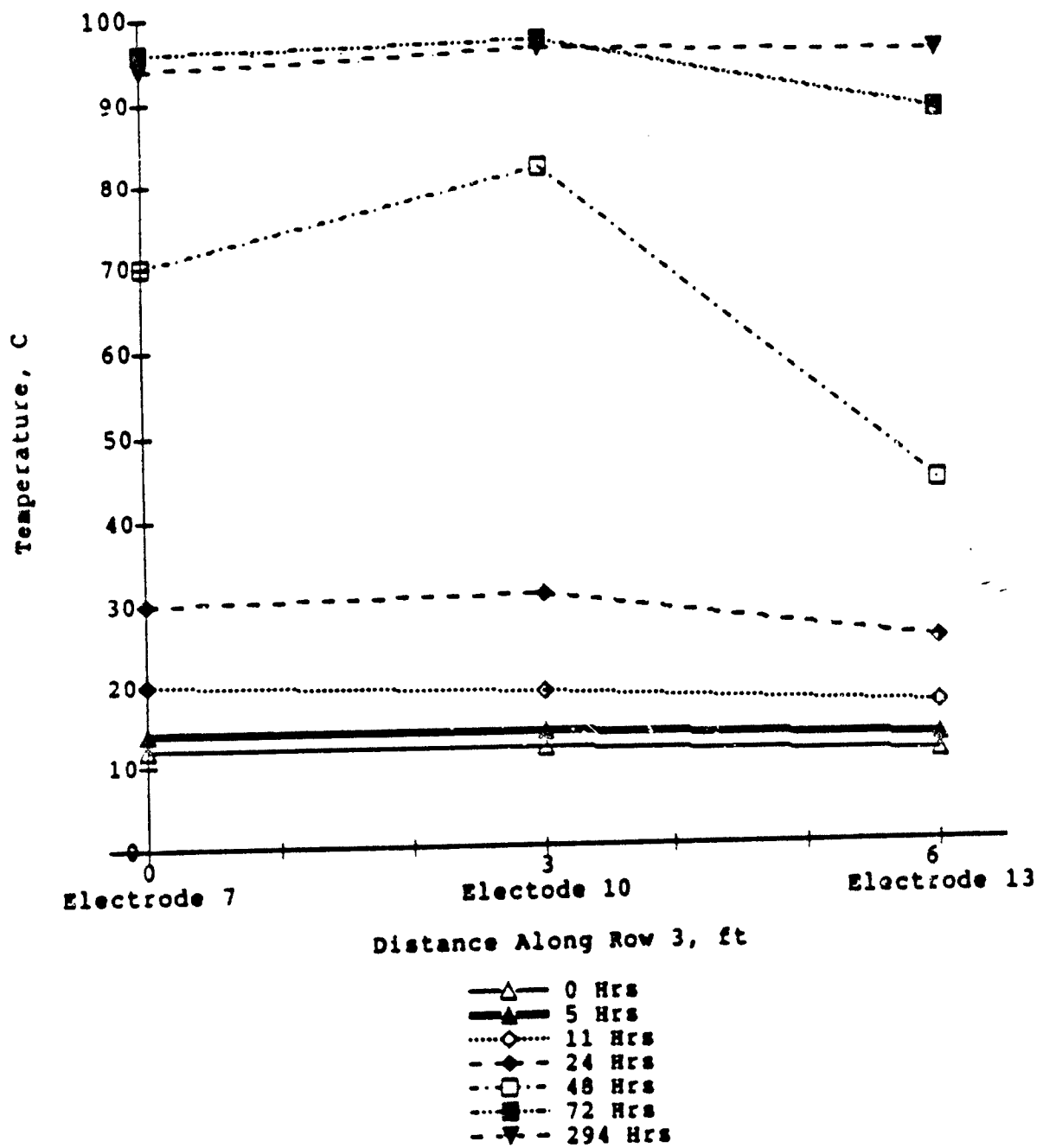


Figure E-6. Temperature Distribution Along Row 3 in Region 1 at 72-inch Depth.

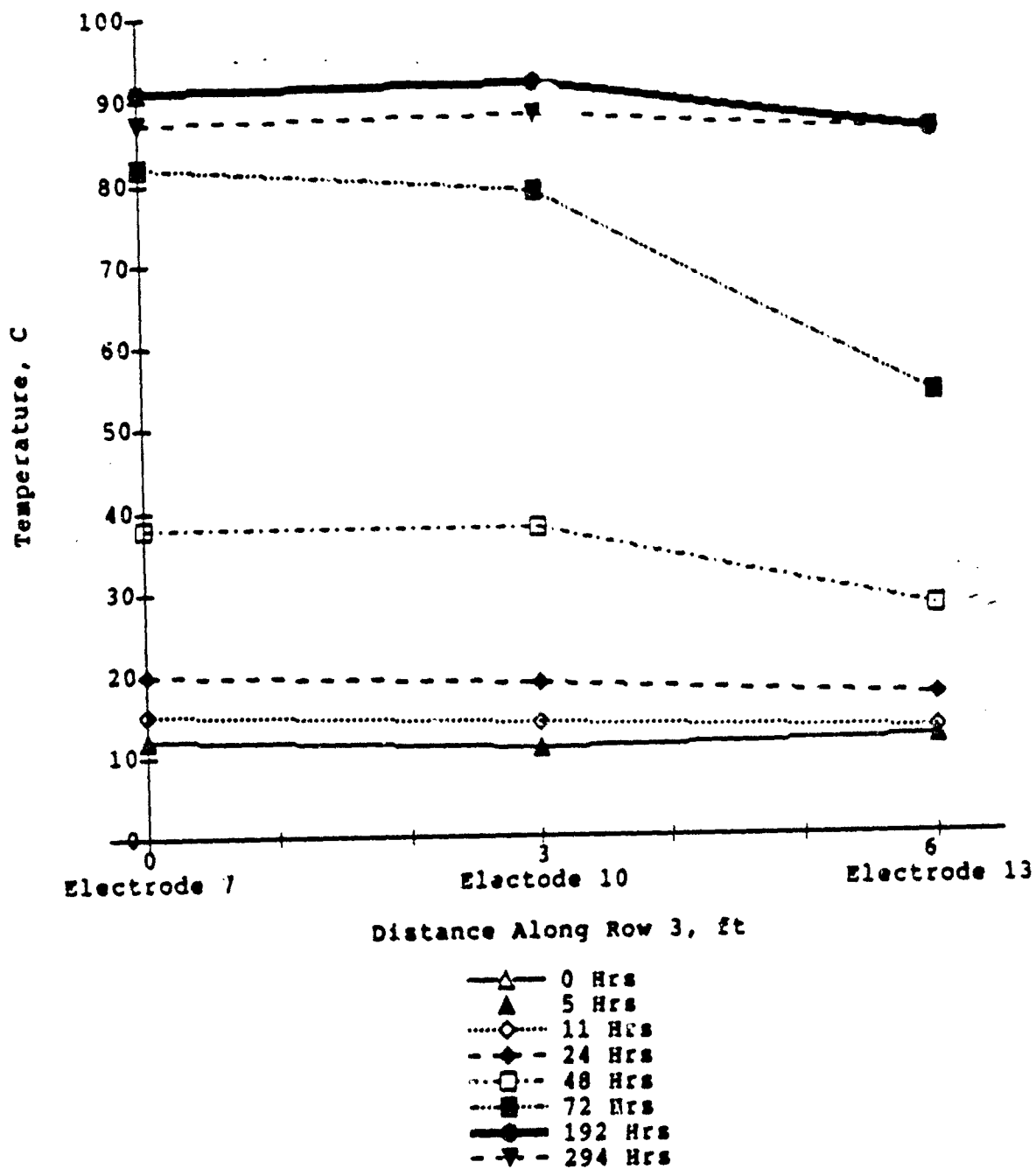


Figure E-7. Temperature Distribution Along Row 3 in Region 1 at 96-inch Depth.

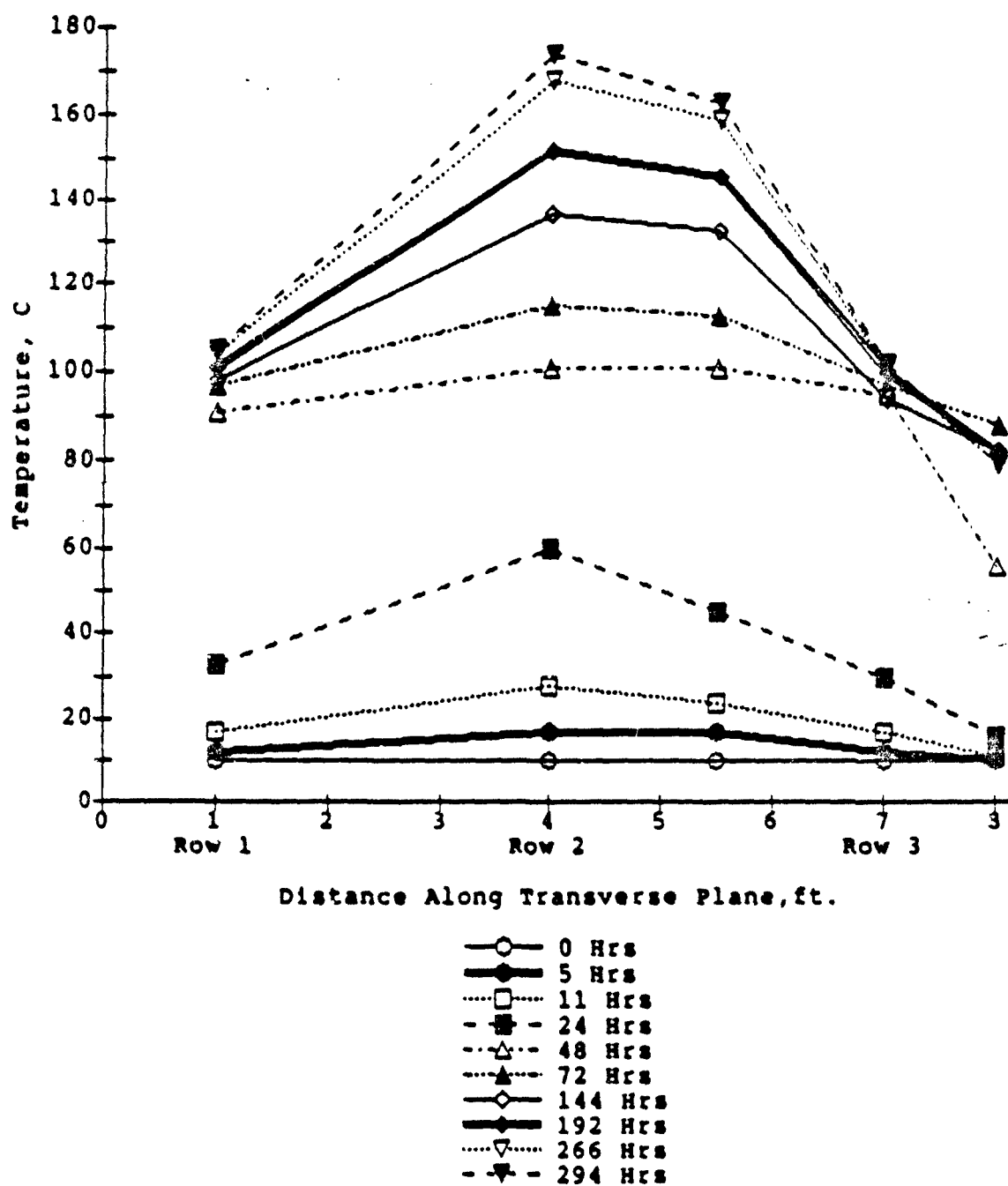


Figure E-8. Temperature Distribution in Transverse Plane AA.

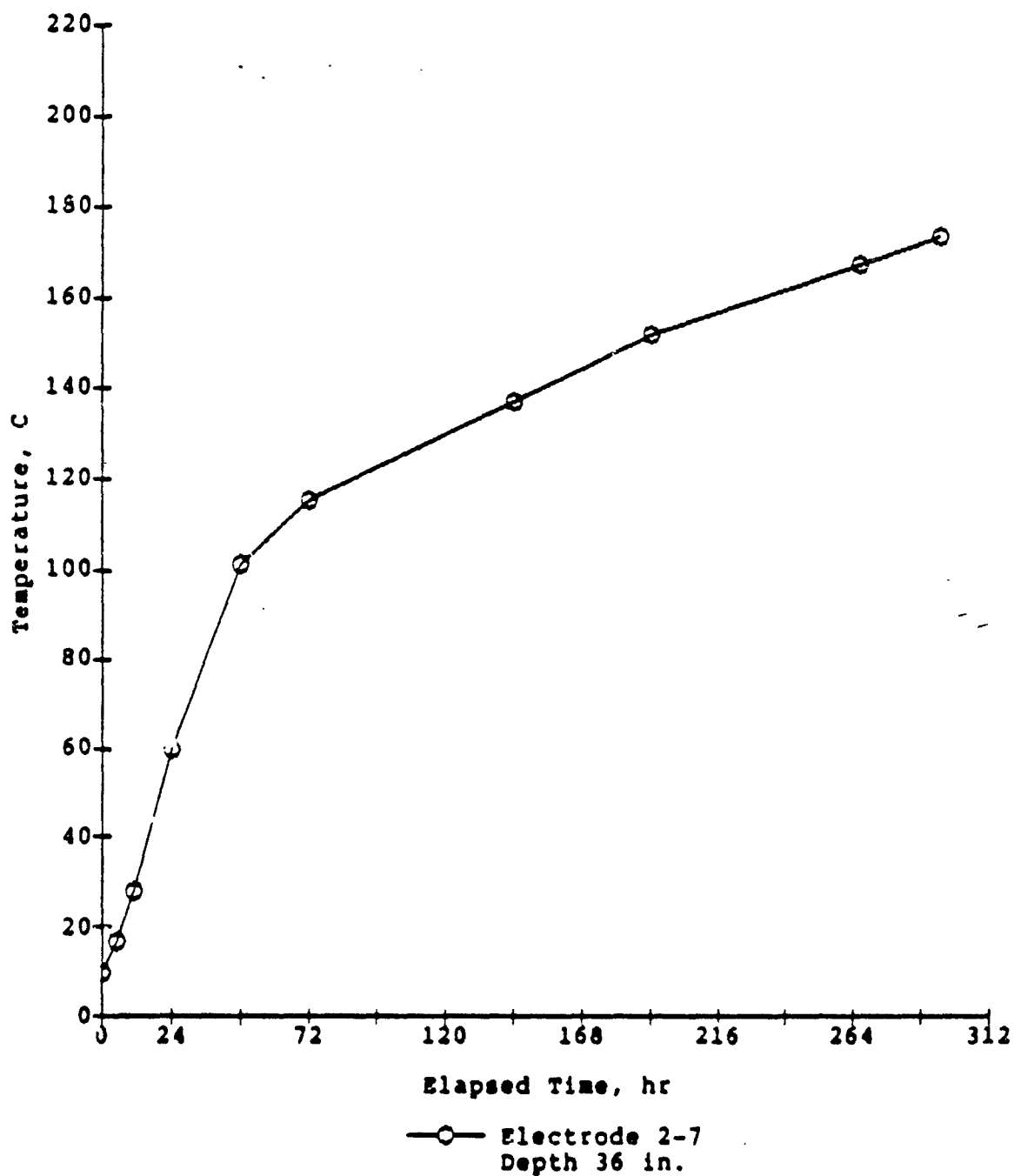


Figure E-9. Rate of Temperature Rise at the Center of the Heated Volume.