AD-A246 345

(2)

CETHA-TS-CR-91053

US Army Corps of Engineers

Toxic and Hazardous Materials Agency

FINAL REPORT

OPTIMIZATION OF COMPOSTING FOR EXPLOSIVES CONTAMINATED SOIL

Prepared For:

U.S. Army Toxic and Hazardous Materials Agency
(USATHAMA)
Contract DAAA15-88-D-0010
Task Order Number 10
Work Order 2281-08-10

November 1991

Prepared By:

ROY F. WESTON, INC.
1 Weston Way
West Chester, Pennsylvania 19380

THAMA Form 45, 1 Jul 90

The views, opinions, and/or findings contained in this report should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

The use of trade names in this report does not constitute an official endorsement or approval of the use of such commercial products. This report may not be cited for purposes of advertisement.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

REPORT SECURITY CLASSIFICATION
Unclassified

1b. RESTRICTIVE MARKINGS

SECURITY CLASSIFICATION AUTHORITY

3. DISTRIBUTION / AVAILABILITY OF REPORT
Unlimited

DECLASSIFICATION / DOWNGRADING SCHEDULE

PERFORMING ORGANIZATION REPORT NUMBER(S)

5. MONITORING ORGANIZATION REPORT NUMBER(S)
CETHA-TS-CR-91053

NAME OF PERFORMING ORGANIZATION
Roy F. Weston, Inc.

6b. OFFICE SYMBOL *(If applicable)*
5-1

7a. NAME OF MONITORING ORGANIZATION
U.S. Army Toxic and Hazardous Materials Agency (USATHAMA)

ADDRESS *(City, State, and ZIP Code)*
One Weston Way
West Chester, PA 19380

7b. ADDRESS *(City, State, and ZIP Code)*
ATTN: CETHA-TS-D
Aberdeen Proving Ground, MD 21010-540

NAME OF FUNDING / SPONSORING ORGANIZATION
USATHAMA

8b. OFFICE SYMBOL *(If applicable)*
CETH-TS-D

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
Contract DAAA15-88-D-0010 Task 10

ADDRESS *(City, State, and ZIP Code)*
TTN: CETHA-TS-D
berdeen Proving Ground,MD 21010-5401

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
			T.O. 10

TITLE *(Include Security Classification)*
Optimization of Composting for Explosives Contaminated Soil

PERSONAL AUTHOR(S)
R.T. Williams and P.J. Marks

TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM 9/89 TO 9/91	30 September 1991	

SUPPLEMENTARY NOTATION

COSATI CODES		
FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*
Composting, Bioremediation, Biodegradation, Contaminated Soil, Explosives, TNT, RDX, HMX

ABSTRACT *(Continue on reverse if necessary and identify by block number)*
composting optimization field study for explosives-contaminated soil was onducted at the Umatilla Army Depot Activity (UMDA) in order to maximize he quantity of soil treated per unit time. Different amendments and soil oading levels were investigated in mechanically agitated and static pile eactors. In addition to the removal of TNT, RDX, and HMX, compost oisture, temperature, and pH were monitored. Toxicity reduction was onitored, using the same samples analyzed by WESTON, at Oak Ridge National aboratory. All three explosives were significantly degraded. Degradation ropped as soil loading increased from 30 to 40 volume percent in each eactor type. More effective degradation was achieved in the mechanically gitated reactor than in the static pile reactors. Amendment composition ffected degradation rate and extent, as well as operating parameters such s pH.

DISTRIBUTION / AVAILABILITY OF ABSTRACT
☐ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS

21. ABSTRACT SECURITY CLASSIFICATION
Unclassified

NAME OF RESPONSIBLE INDIVIDUAL
Wayne Sisk and Capt. Kevin Keehan

22b. TELEPHONE *(Include Area Code)*
(410) 671-2054

22c. OFFICE SYMBOL
CETHA-TS-D

CETHA-TS-CR-91053

FINAL REPORT

OPTIMIZATION OF COMPOSTING FOR EXPLOSIVES CONTAMINATED SOIL

Prepared For:

U.S. Army Toxic and Hazardous Materials Agency
(USATHAMA)
Contract DAAA15-88-D-0010
Task Order Number 10
Work Order 2281-08-10

Accesion For	
NTIS CRA&I	☑
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution /	
Availability Codes	
Dist	Avail and / or Special
A-1	

November 1991

Prepared By:

ROY F. WESTON, INC.
1 Weston Way
West Chester, Pennsylvania 19380

WESTON

TABLE OF CONTENTS

TABLE OF CONTENTS
(continued)

TABLE OF CONTENTS
(continued)

LIST OF FIGURES

LIST OF FIGURES
(continued)

LIST OF FIGURES
(continued)

LIST OF FIGURES
(continued)

LIST OF FIGURES
(continued)

LIST OF TABLES

WESTON

LIST OF TABLES
(continued)

LIST OF ACRONYMS

ABL	Allegany Ballistics Laboratory
ADA	ammunition demolition activity
Battelle	Battelle Pacific Northwest Laboratory
CFUs	colony forming units
Comp B	Composition B (60% RDX, 40% TNT)
CRREL	Cold Regions Research and Engineering Laboratory
EC	effective concentration
EPA	Environmental Protection Agency
EPIC	Environmental Photographic Interpretation Center
HDPE	high-density polyethylene
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HPLC	High-Performance Liquid Chromatograph
HSL	Hazardous Substance List
LAAP	Louisiana Army Ammunition Plant
LANL	Los Alamos National Laboratory
MAIV	mechanically agitated in-vessel
mA	milliamp
mL	milliliter
NPL	National Priorities List
ORNL	Oak Ridge National Laboratory
RCRA	Resource Conservation and Recovery Act
RDX	hexahydro-1,3,5-trinitro-1,3,4-triazine
RFA	Resource Facility Assessment
SP	static pile
SWMU	solid waste management units
TNT	2,4,6-trinitrotoluene
UMDA	Umatilla Army Depot Activity
USABRDL	U.S. Army Biomedical Research and Development Laboratory
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
μm	micron
XAD	Amberlite nonionic polymeric absorbent

SECTION 1

EXECUTIVE SUMMARY

A composting optimization field study for explosives-contaminated soil was conducted at the Umatilla Army Depot Activity (UMDA), Hermiston, Oregon. The goal of this UMDA composting optimization field study was to increase the quantity of explosives contaminated soil processed in a composting treatment system per unit time. In order to achieve this goal, either a higher percentage of soil must be incorporated into the mixture to be composted and/or the contaminants must be destroyed at a higher rate. To increase the rates of degradation either more effective catalysts (microorganisms) must be utilized or the physical/chemical/biological environment must be improved or better matched to the microorganisms such that the explosives are metabolized more rapidly.

Two levels of composting technology were investigated: a mechanically agitated in-vessel (MAIV) system and an aerated static pile (SP) system. Eight static pile and four mechanically agitated tests were completed. The key variables investigated were soil loading percentage and overall amendment composition. In addition, a bioaugmented investigation, using a microbial inoculum developed by Dr. Pat Unkefer of Los Alamos National Laboratory (LANL), was conducted in a static pile reactor. The UMDA field program was designed to conduct the tests necessary to obtain the data required for implementation of composting as a cost-effective alternative to incineration.

All three explosives present at UMDA demonstrated significant degradation during treatment by composting. Explosives reduction data were collected to obtain rate and operating parameter information over a wide range of conditions for development of full scale designs. Kinetic rate of destruction information for MAIV versus SP technology, as well as the effect of soil loading on kinetic rate, was crucial. The half-lives in the SP reactors varied from a low of 6.4 days for TNT with 10% soil to a high of 24.9 days for TNT with 40% soil. The half-life for TNT in both 10% soil MAIV tests were low (5.2 and 5.1

days). The half-lives for TNT in the MAIV system with 25 and 40% soil were 6.4 and 14.9 days, respectively.

The rate of degradation and the extent of degradation of all three explosives dropped markedly as soil loading was increased from 30 to 40 volume percent in each reactor type. The optimum soil loading rate for full scale implementation of composting must be determined based on a cost/engineering analysis. However, based on this study, the optimal soil loading rate is likely to not be much higher than approximately 30% soil. Two amendment mixtures resulted in excellent degradation of explosives. The mixture used in a previous field demonstration at Louisiana Army Ammunition Plant and duplicated at UMDA was superior, but both were effective. These results indicate that amendment composition is crucial for effective degradation, but that acceptable mixtures likely can be developed depending on the local availability of waste materials.

SECTION 2

BACKGROUND INFORMATION

2.1 NATURE OF THE PROBLEM

Manufacturing and handling of explosives and propellants at Army industrial facilities have resulted in the contamination of soils and sediments. Contamination has often resulted from disposal practices that were common and acceptable at the time of discharge.

Because of the potential for groundwater contamination, and the subsequent migration of hazardous substances, treatment of the contaminated source is necessary to protect the environment and avoid costly actions in the future. Incineration is currently the only demonstrated technology for the remediation of explosives contaminated soils. Incineration is publicly undesirable and essentially economically unfeasible for the remediation of small sites due to the large expenditures required for the mobilization and demobilization of the incineration systems. For small sites, a more economical treatment technology needs to be developed even if treatment requires a longer duration. A candidate for the latter type is composting.

2.2 COMPOSTING

Composting is a process by which organic materials are biodegraded by microorganisms, resulting in the production of organic and/or inorganic byproducts and energy in the form of heat. This heat is trapped within the compost matrix, leading to the self-heating that is characteristic of composting. Composting for bioremediation is initiated by mixing biodegradable organic contaminants (explosives in soil in the present study) with organic carbon sources.

The environment in compost is substantially different from that within aerobic soils in that the matrix to be composted has a much higher concentration of organic matter. This organic-rich environment leads to intense microbial metabolic activity and the production of heat. The production of metabolic heat and the insulative properties of the compost matrix create a self- heating environment that serves to further stimulate microbial activity. If left unchecked, temperatures may exceed 70°C, a temperature that inhibits most microorganisms and leads to a decline in metabolic activity.

The efficiency of the composting process is affected by temperature, moisture content, pH, chemical and biological characteristics, as well as the concentrations of the organic substrates, the concentrations of inorganic nutrients such as nitrogen and phosphorus, heat production and retention characteristics of the compost, and the partial pressure of oxygen within the composting material.

Composting may be implemented at one of three general levels of technology. These levels differ in the degree of manipulation required and process control attained. Equipment and operating costs typically increase at higher technological levels. At the lowest level, the material to be composted is simply shaped into the form of a pile and allowed to self-heat. Water and/or nutrients may be added. However, air exchange is poor, and temperatures may fluctuate widely within the composting material. Periodically turning the material increases aeration, but process control remains negligible unless the piles are turned with a frequency based on operating parameters such as temperature. This level of technology is often referred to as a "windrow" system, because of the long rows of narrow compost piles typically employed.

At the next technological level, an aeration/heat removal system is utilized to increase process control over the composting system. The aeration/heat removal system typically takes the form of a network of perforated pipe underlying the compost pile. The pipe is attached to a mechanical blower, and air is periodically drawn or forced through the compost to provide aeration and heat removal. This level of technology is often referred to as a "static pile." Static pile technology can be implemented inside structures such as tanks or bins, as well as with piles of various shapes.

At the highest technological level, a system of enclosed composting vessels and automated materials handling equipment is used (in addition to an aeration/heat removal system) to produce a semi-continuous or continuous treatment process. This type of system is often referred to as "in-vessel" composting.

While these generalities regarding technology levels are useful rules of thumb, they must be used with caution. For example, static pile composting can be conducted in vessels or in windrows. Furthermore, windrows without instrument and blower controlled temperature regulation may be maintained within potentially acceptable (depending on the application) operating ranges for various parameters by the use of frequent turning based upon process monitoring.

2.3 BIOREMEDIATION USING COMPOSTING

Composting is widely used to stabilize wastewater sludges and municipal refuse in the United States and Europe (Biocycle Special Report, 1987). The primary objectives of refuse/sludge composting are to:

- Reduce the volume of waste or sludge.
- Reduce the moisture content of the composting material.
- Destroy potentially odorous nitrogen- and sulfur-containing organic compounds.
- Destroy pathogenic microorganisms.
- Stabilize the compost material for ultimate disposal.

In contrast, the primary objective of hazardous materials composting is to convert hazardous organic substances into innocuous products for ultimate disposal (Williams and Myler, 1990). Rapid processing is desirable, but remains secondary to successful treatment of the contaminants. While hazardous materials composting systems share many of the characteristics of sludge and refuse composting systems, operating parameters may differ according to the primary objective of the process and the nature of the waste being treated.

2.4 APPLICABILITY OF COMPOSTING FOR EXPLOSIVES

Previous studies have demonstrated the susceptibility of explosives and propellants to microbial degradation. These studies have been reviewed by Williams et al. (1988) and Woodward (1990). Routes of bioconversion, intermediate and final products, and analytical methods to assess the results have been determined. Successful composting of the explosives TNT and RDX in soil has been conducted and biodegradation mechanisms are known for some of their manufacturing byproducts as well as for the nitrate ester propellants. Composting of these energetic compounds has been done on a pilot scale in reactor vessels sufficiently large enough to simulate field conditions.

Field demonstrations of composting explosives-contaminated (TNT, HMX, RDX) and propellant-contaminated (nitrocellulose) soils (Williams et al., 1988; 1989 and "in press") were successful in terms of reducing explosive and propellant concentrations through biodegradation. The objective of these previous tests was to demonstrate the efficacy of composting under actual field conditions.

2.5 UMATILLA ARMY DEPOT ACTIVITY

Umatilla Army Depot Activity (UMDA) was selected by USATHAMA as the site of the composting optimization field study. UMDA is an active Army facility located on nearly 20,000 acres (approximately 23 square miles) in Hermiston, Oregon (Figure 2-1). UMDA was originally purchased by the U.S. Army in 1940 and was established as an ordnance depot for storing chemical and conventional munitions. The functions of the depot were extended to include ammunition demolition (1945), renovation (1947), and maintenance (1955). In 1962, the storage of chemical munitions began at UMDA. In August 1973, the installation was redesignated as an Activity by the U.S. Army Material Command.

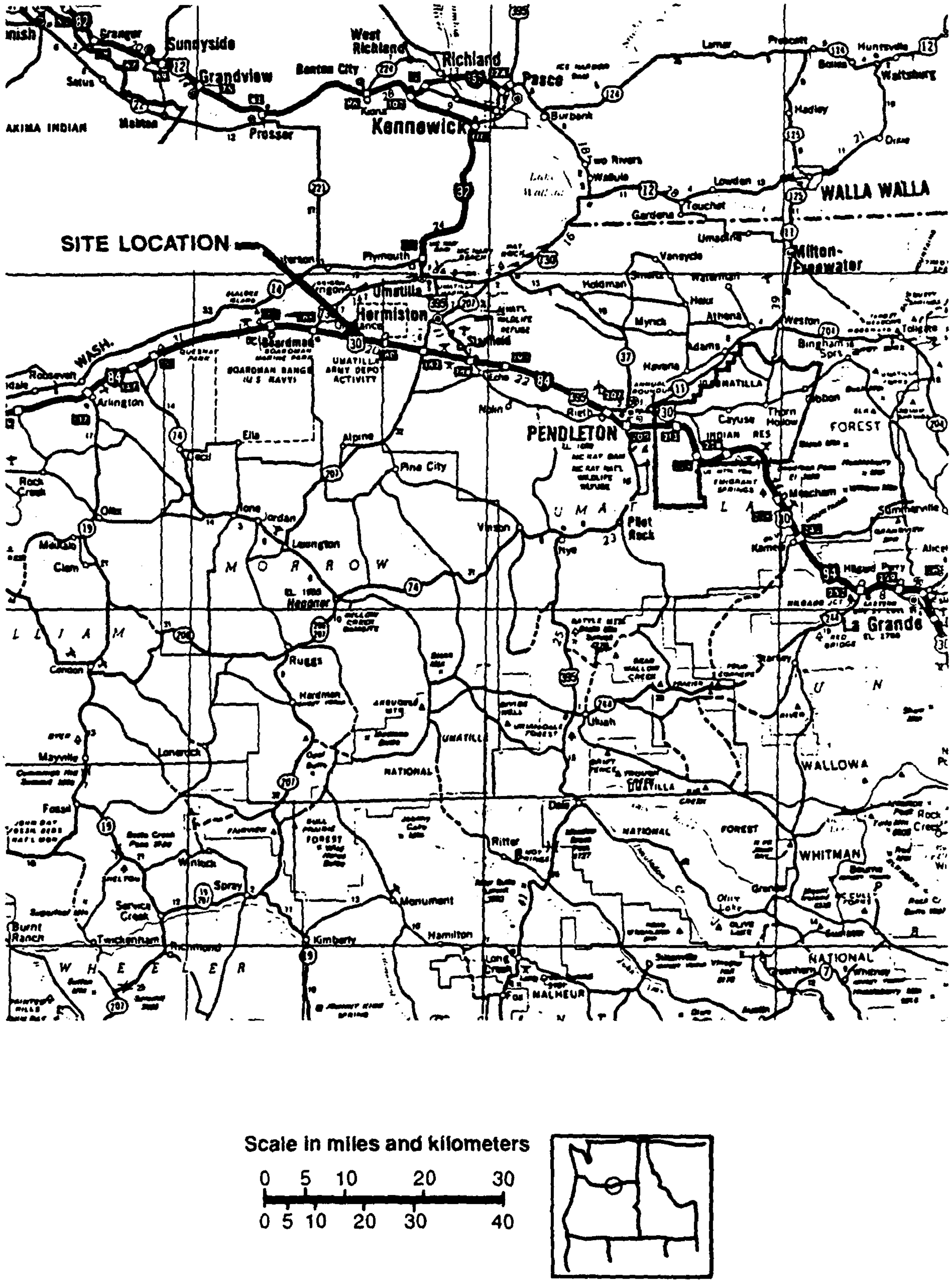


Figure 2-1. UMDA geologic location map.

UMDA continues to be used to store chemical and conventional munitions in igloos on-site (Figure 2-2). Chemical munitions include nerve and blister agents, white phosphorus projectiles, missiles, and propellants. Munitions rework and demilitarization of conventional munitions are still being performed, with defective and/or expired lots of demilitarized powder burned regularly.

In 1986, the U.S. Environmental Protection Agency (EPA) (Region X) conducted a Resource Conservation and Recovery Act (RCRA) Resource Facility Assessment (RFA) to identify releases or potential releases from various solid waste management units (SWMU) or spill sites at UMDA. Based on this assessment, EPA advised USATHAMA to collect additional information so that proper corrective measures could be formulated for selected SWMUs. Meanwhile, a SWMU, known as the explosives washout lagoons area (Figures 2-3 and 2-4), had been placed on the National Priorities List (NPL) because of the presence of explosive compounds in the water table aquifer. USATHAMA, through a previous investigation at the explosives washout lagoons area, had identified the presence of the explosive compounds in the water table aquifer.

2.6 SITE DESCRIPTION AND HISTORY

UMDA is located in a semi-arid environment (approximate annual precipitation and evaporation of 9 and 32 inches, respectively) in northeastern Oregon's Umatilla and Morrow counties. Primary population centers within a 6-mile radius of UMDA include Hermiston (population 9,870), Umatilla (population 3,120), and Irrigon (population 865).

The explosives washout operations, formerly conducted in Building 489, involved the removal of explosives from munitions, bombs, and projectiles by means of water and/or steam-cleaning techniques. Some of the munitions demilitarized at this location included 500- and 750-pound Composition B (60% RDX, 40% TNT) bombs and 90-mm projectiles. The washout operations included sizable amounts of Composition B and TNT. During the life of the washout plant, sludges built up in the prerinse and rinse tanks. These sludges were removed as necessary and placed in the washout tank. Sludges that accumulated in

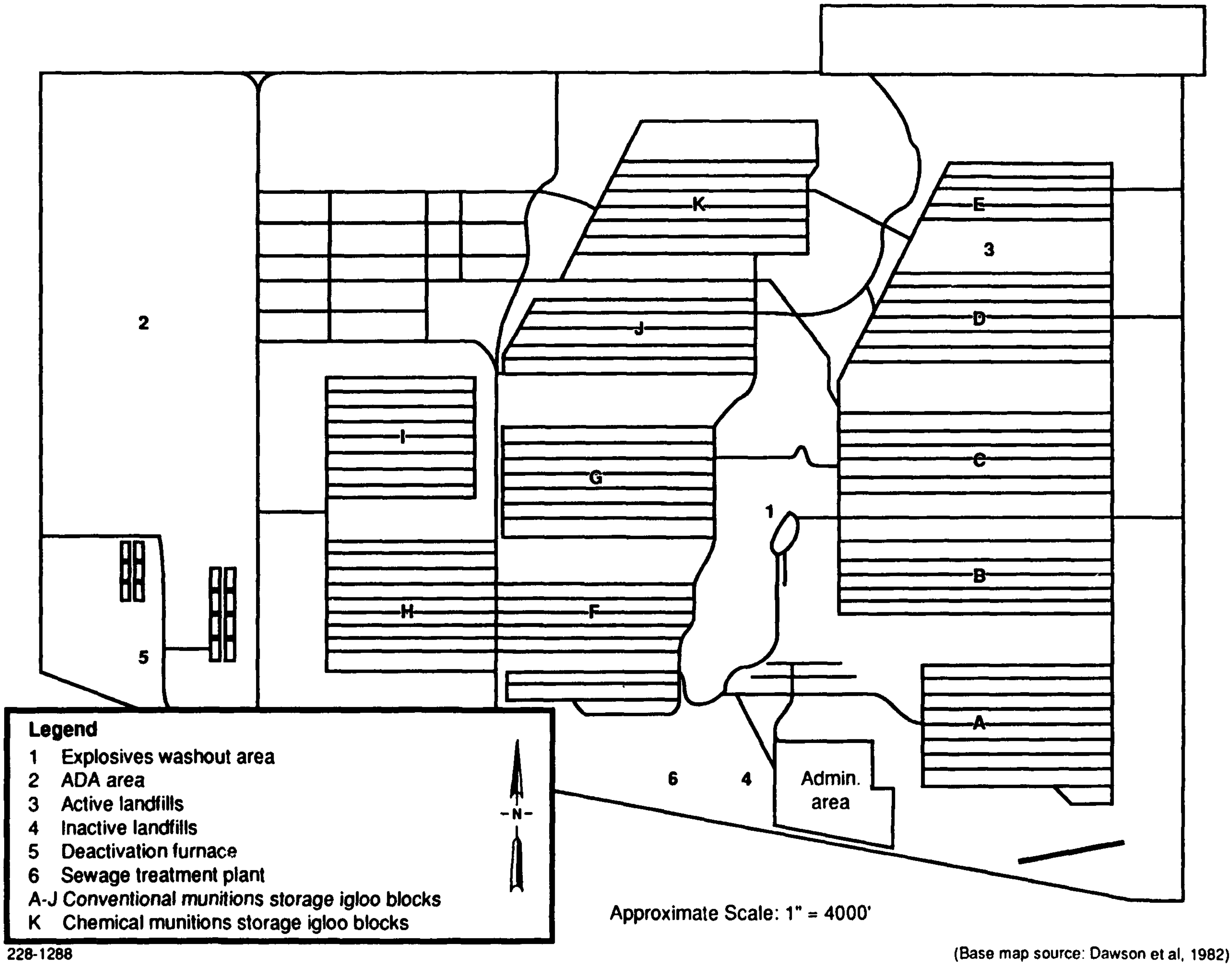


Figure 2-2. Overview of UMDA layout.

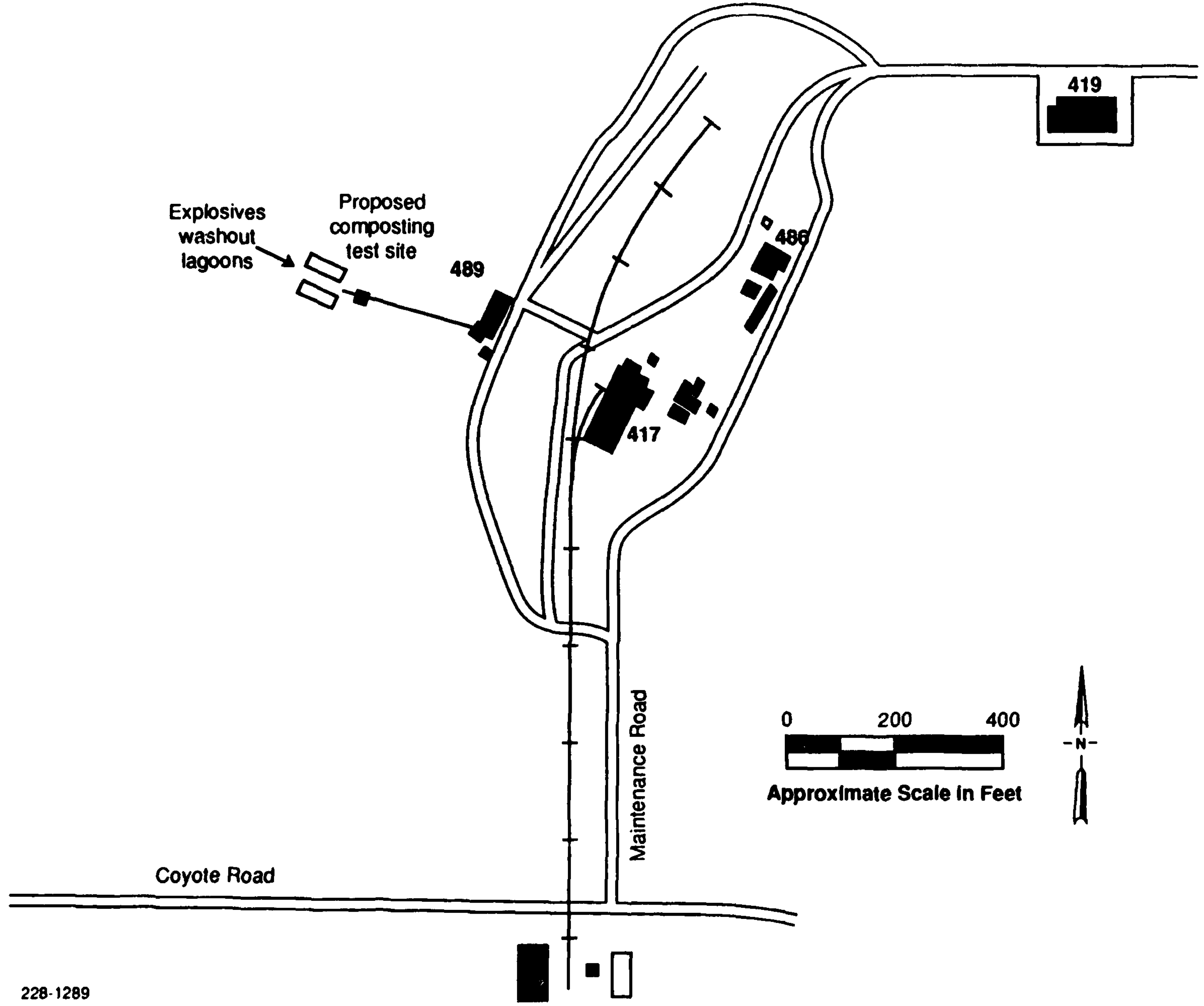


Figure 2-3. Washout lagoon site.

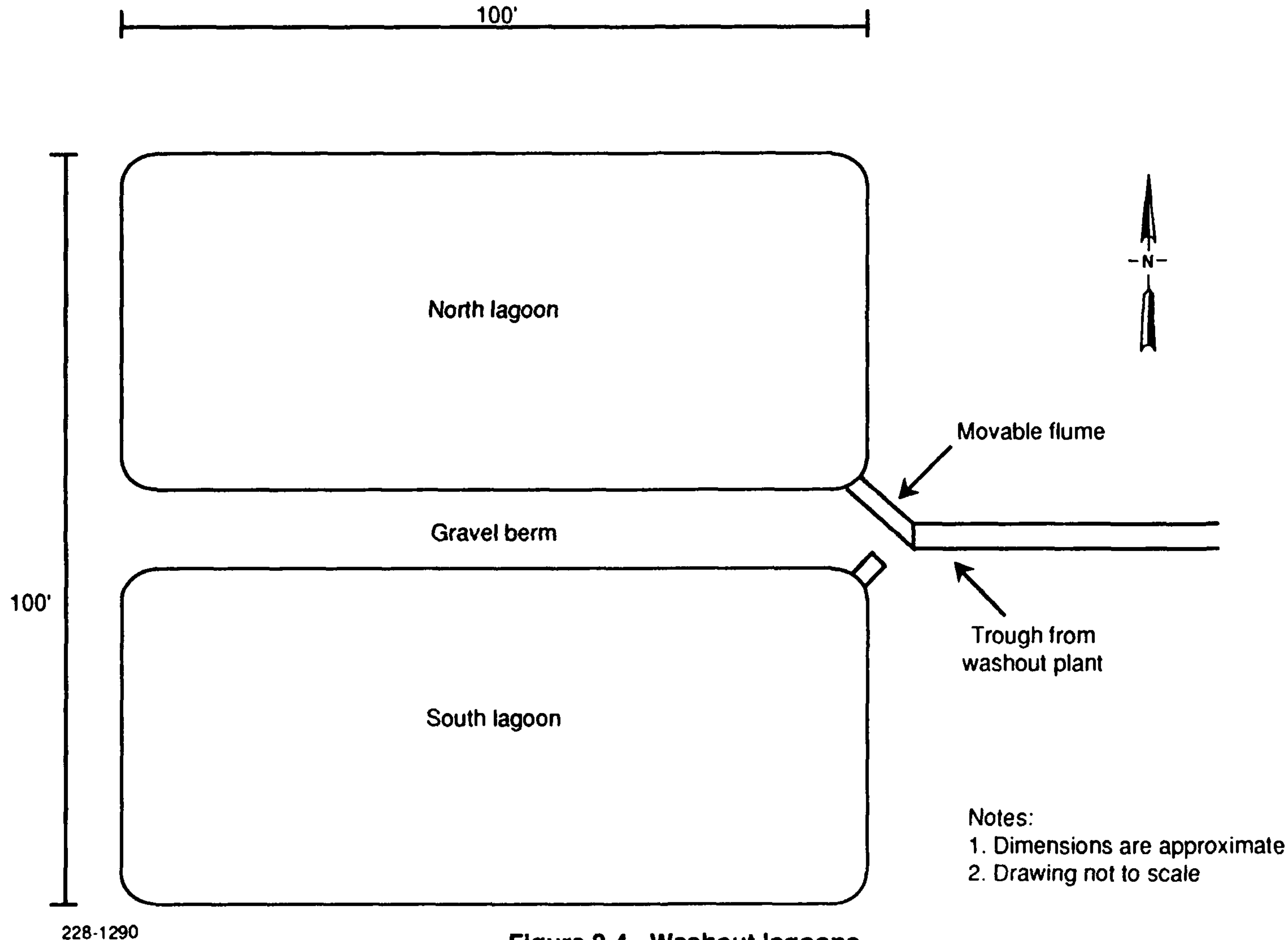


Figure 2-4. Washout lagoons.

the washout tank were pumped to the reclaiming operation. Explosives in the washout tank sludges were separated from the water, concentrated, dried, formed into pellets, and packaged for resale. Liquors from the reclaiming operation were returned to the washout tank.

A concrete sump, located midway between the washout plant, Building 489, and the washout lagoons, served to settle out explosives particles/solids prior to discharge of process water to the lagoons. During washout operations, the concrete sump containing washwater (pink water)/solids was pumped 2 to 3 times per week into a 500-gallon tank. This tank was then transported to the ammunition demolition activity (ADA) area, where the contents were discharged into the northernmost burn trench.

Excess wastewaters generated from the sump were conveyed via gravity flow in a trough to two infiltration lagoons located in Coyote Coulee. The trough is a steel, open top, three-sided drainage channel designed to minimize spills and leaks. Former UMDA washout building employees have indicated to USATHAMA that overflow from the trough occurred very infrequently and usually was due to plugs of explosives accumulated in the trough. The entire explosives washout system was drained, flushed, and cleaned approximately once every week. The infiltration lagoons received all of the approximate 150,000 gallons of wastes generated during the weekly turnarounds.

The two infiltration lagoons were operated in an alternating manner. Washout wastes, also known as pink water due to their characteristic color, were accumulated in one of the lagoons, while the wastes in the other lagoon were allowed to dry. Wastewaters were accumulated in a given lagoon to a depth of approximately 3.5 feet and/or until the rate of infiltration was substantially reduced by the accumulation of solids. The washout wastewaters were then directed to the other lagoon by a movable flume at the discharge end of the rectangular chute. After drying, the residual solids were transported to the ADA area for open burning.

Both of the infiltration lagoons are gravel-lined and occupy a total area of approximately 10,000 square feet. The lagoons were operated from the mid-1950s until 1965, and it is estimated that a total of up to 85 million gallons of pink water may have been discharged to the lagoons. Former UMDA employees have indicated that both lagoons have been rebuilt over the years.

The Installation Assessment performed in December 1978 identified the explosives washout lagoons as one of two major contaminated areas on-post. The other major contaminated area identified was the ADA area. The major contaminants were identified as explosive wastes, and a preliminary environmental survey was recommended.

In 1980, the Environmental Photographic Interpretation Center (EPIC) listed the explosives washout lagoons area as a potentially hazardous site. Aerial photographs from 1958 and 1970 were compared, and it was determined that significant impacts or changes to the environment had occurred during this period. In 1981, Battelle Pacific Northwest Laboratory (Battelle) performed an environmental survey at UMDA. Battelle installed nine monitor wells and collected soil samples to a depth of 7.5 feet below grade in the explosives washout lagoons area.

During the Battelle environmental survey, explosives were detected in the surface soil of the explosives washout lagoons (Table 2-1). Soil samples from both of the lagoons revealed detectable concentrations of 2,4,6-trinitrotoluene (2,4,6-TNT) and hexahydro-1,3,5-trinitro-1,3,4-triazine (RDX). The northern lagoon displayed 2,4,6-TNT concentrations up to 38 $\mu g/g$ of soil (dry weight basis) and RDX concentrations of 350 $\mu g/g$. The southern lagoon had 2,4,6-TNT, RDX, DNT (total), and tetryl concentrations of 2,800, < 8.9, 9.7, and 12 $\mu g/g$, respectively (see Table 2-1).

2,4,6-TNT and RDX (38 and 43 $\mu g/g$, respectively) were detected in the subsurface soil below the washout lagoons to depths of 7.5 feet (the lowest depth sampled). The concentrations of 2,4,6-TNT and RDX at a depth of 2.5 feet below the surface were 180 and

Table 2-1

Summary of Surface Soil Explosives Data from the Battelle Environmental Survey for Explosives Washout Lagoons

Explosive	Surface Soil Concentrations ($\mu g/g$*) North Lagoon	South Lagoon
2,4,6-TNT	38	2,800
RDX	350	< 8.9
2,4-DNT	ND	4.3
2,6-DNT	ND	5.4
Tetryl	ND	12

ND - Not Detected.
*Dry weight

260 $\mu g/g$, respectively. The concentrations of both explosives compounds decreased with increasing depth.

2.7 SOIL CHARACTERIZATION

Data available on the explosives content of the washout lagoon soils were deemed insufficient for the purposes of the composting optimization study. WESTON and USATHAMA were both concerned that the explosives content of the soils might be too low to conduct an evaluation of composting. In addition, concern existed regarding the possible presence of unexpected organics and/or heavy metals.

In order to confirm the concentration of explosives in the washout lagoons, surface samples were collected on 5 October 1989. An experimental field analysis kit for TNT was used to guide the collection of samples. Data from this analysis are not reported. Surface samples (0 to 4 inches) were then collected from four locations in each lagoon and submitted to WESTON's Lionville PA analytical laboratory for explosives analysis.

A composite sample was prepared from the four samples collected in the south lagoon. This sample was submitted for a HSL list organics and metals analysis. With the exception of elevated levels of nitroaromatics, no substantial concentrations of other contaminants were detected. Data from the explosives analysis are reported in Table 2-2, and the data from the hazardous substance list survey are presented in Appendix A.

The 5 October 1989 survey reinforced our concerns regarding the quantity of explosives in the soils. Ideally, the homogenized soil used to prepare the mixtures to be composted should contain a minimum of 10,000 mg/kg of total explosives. With the exception of two locations in the south lagoon, the quantity of explosives detected in the soils was significantly less than 10,000 mg/kg.

Table 2-2

Explosives Content of Washout Lagoon Soils from the 5 October 1989 WESTON Survey (in mg/kg*)

Lagoon	TNT	HMX	RDX
<u>South Lagoon</u>			
• End of spillway	45,580	<127	< 98
• Center of lagoon	318	< 1	< 1
• West end, center	618	< 1	2
• Southeast corner, sidewall	87,620	485	731
<u>North Lagoon</u>			
• End of spillway	14	15	5
• Center of lagoon	< 1.9	2.3	2.0
• West end, center	1,618	58	246
• Northwest corner, sidewall	4.4	< 1.3	< 1

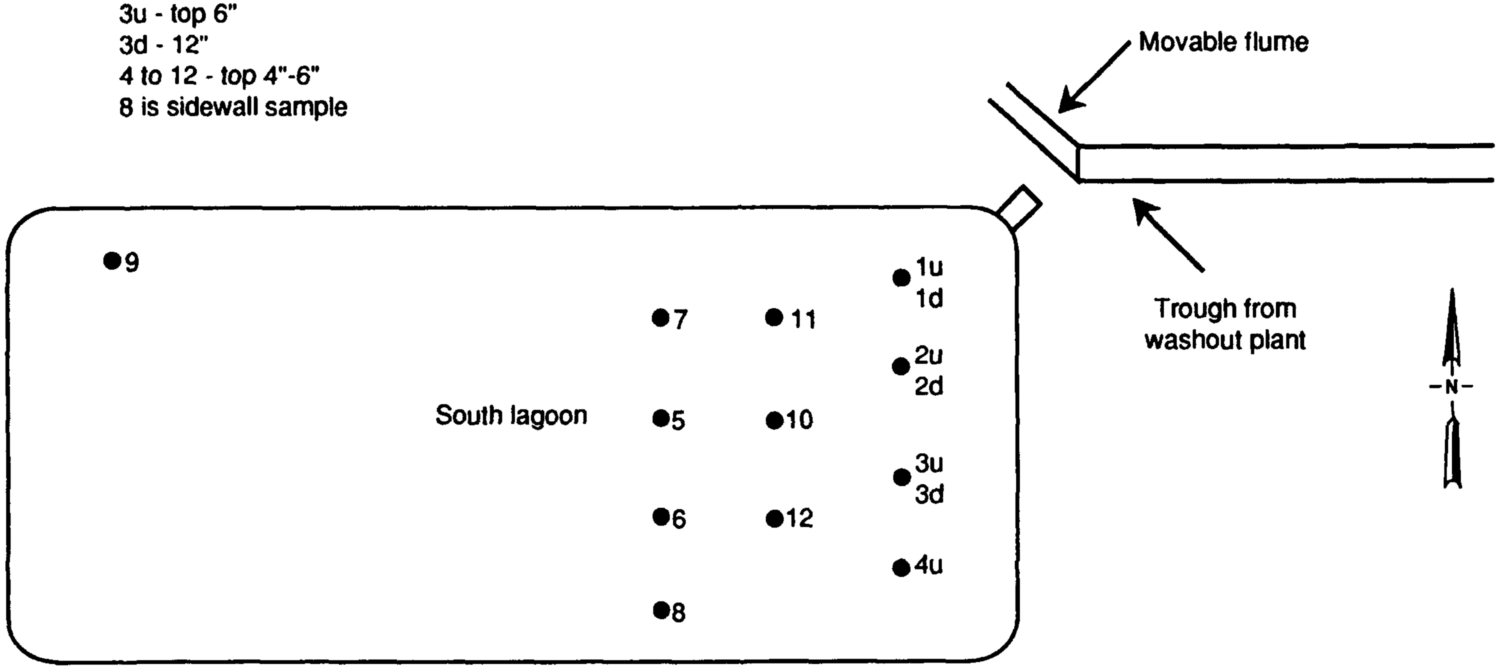
*Field moisture not determined

A more extensive survey of the south lagoon was conducted on 30 January 1990 to better identify the areas of higher explosives content required for this study. Sixteen samples were collected from the sampling points shown in Figure 2-5. Samples were analyzed by Dr. Tom Jenkins at the Cold Regions Research and Engineering Laboratory using a new improved field test method he developed and verified by HPLC analysis. Results from the HPLC analysis are presented in Table 2-3.

The results indicated that the highest content of explosives was present in the top 3 to 5 inches of soil, and that the highest concentrations were present at the drainage channel end of the lagoon and in the sidewalls. The presence of the heaviest concentrations of explosives in the top 3 to 5 inches of soil was readily apparent by the deep red coloration of the soils within this region of the core samples.

Numerous core samples examined from various locations within the south lagoon confirmed this observation. Based upon the data in Table 2-3, a determination was made that adequate soil could be obtained from the south lagoon at UMDA. However, special precautions were deemed necessary for excavating adequate quantities of appropriately contaminated soil (see Section 3). Excavation was done by hand for the top 6 inches of soil in selected regions of the lagoons. Fifteen cubic yards of soil was excavated using this method.

Notes: 1u - top 6"
1d - 18"
2u - top 4"
2d - 4"-8"
3u - top 6"
3d - 12"
4 to 12 - top 4"-6"
8 is sidewall sample



228-1290A

Figure 2-5. Sampling points during 30 January 1990 survey.

Table 2-3

Explosives Content of South Washout Lagoon Soils from the 30 January 1990 WESTON Survey

Sample South Lagoon	TNT (mg/kg*)
1 u	15,500
1 d	2,250
2 u	7,430
2 d	8,350
3 u	4,020
3 d	1,170
4 u	8,510
5	3,980
6	130
7	1,150
8	38,600
9	7,680
10	1,290
11	240
12	180

*Dry weight
u, upper
d, deep

WESTON

SECTION 3

TEST OBJECTIVES/APPROACH

3.1 OBJECTIVE

The specific objective of the composting optimization field study was to evaluate key parameters that have the potential to increase the quantity of soil processed in a compost treatment system per unit time. Soil throughput may be increased by either increasing the soil fraction present in the mixture to be composted and/or increasing the rate of transformation of the explosives to innocuous end products. The overall objective was to develop the database needed to implement composting as a less expensive treatment process than incineration for explosives-contaminated soil.

3.2 TECHNICAL ISSUES REQUIRING INVESTIGATION

The goal of this UMDA composting optimization field study was to increase the quantity of soil processed in a composting treatment system per unit time. In order to achieve this goal, either a higher percentage of contaminated soil must be incorporated into the mixture to be composted and/or the contaminants must be degraded at a higher rate. To increase the rates of degradation either more effective catalysts (microorganisms) must be utilized or the physical/chemical/biological environment must be improved or better matched to the microorganisms such that the explosives are metabolized more rapidly.

In practice, higher degradation rates may be achieved by bio-augmentation (provided microorganisms with more effective metabolic pathways/enzymes are available and will function well in a compost matrix), optimizing the composition of the amendment mixture (within the constraints of what is geographically available and economically feasible), optimizing the environmental conditions (moisture, oxygen, pH, nutrients, electron acceptors, surface contact, etc.) in the composting matrix, and/or by increasing the bio-availability of the contaminants should desorption of the explosives from soil particles be a limiting constraint.

The key technical issues for investigation in this composting optimization field study focused on the amendments used to prepare the mixture to be composted, control of the environmental conditions, the amount of soil included in the mixture to be composted, and the incorporation of a TNT degrading microorganism. In addition, the toxicity of the compost and the final fate of the explosives was extensively evaluated by Dr. Wayne Griest (Griest et al., 1991) at Oak Ridge National Laboratory (ORNL). Toxicity tests at ORNL were conducted using Ceriodaphnia dubia and Ames assays.

The criteria for selecting operating conditions to test were driven by the overall economics of the treatment process. This was not necessarily a straightforward evaluation. For example, a higher cost amendment may prove more economical to use than a less effective but cheaper amendment, since soil throughput may be higher with the more costly amendment. A similar situation exists with soil percentage. Contaminants may be transformed at a higher rate at a lower soil percentage, but system throughput may be higher using a higher soil percentage and accepting lower rates of transformation. In addition, it is advantageous to minimize the amount of material that must be handled and disposed.

A cost analysis (Lowe et al., 1989) has indicated that the soil volume fraction plays a greater role in controlling the overall economics of composting than the transformation kinetics. The overall goal for composting is to achieve a 28% soil fraction for SP technology while keeping the cost of amendments to less than $50 per ton. This would result in a treatment cost of approximately $100 (1990 dollars) per ton of soil treated. To maintain a similar treatment cost with MAIV technology, the soil fraction required is approximately 40%.

In addition to process performance issues, two key areas of concern remain from the Louisiana Army Ammunition Plant composting field demonstration (Williams et al., 1988): the final fate of the explosives and the toxicity of the compost residue.

Overall, the key technical issues examined in the present study were:

- Selection of optimum carbon sources and bulking agents, as well as a mixing strategy for achieving homogeneity.
- Determining the highest percentage of soil usable within the mixture to be composted for SP and MAIV technology.
- Testing the performance of mechanically agitated versus static pile composting.
- Evaluating whether bio-augmentation or microbial population selection by controlling operating parameters would enhance transformation rates.
- Maintaining environmental parameters (i.e., moisture, temperature, pH, and oxygen) such that transformation of contaminants was optimized.
- Evaluating the final fate of the explosives.
- Monitoring the reduction in toxicity of the mixture being composted.

3.3 APPROACH

Two types of pilot studies were conducted to investigate the issues of concern. The first type was an investigation of in-vessel, mechanically agitated (MAIV) composting using a specially fabricated pilot unit (Fairfield Engineering). Two variables were investigated in this unit: soil/amendment mixture ratio and amendment mixture composition. Four tests were conducted. The first two tests investigated differing amendment compositions using 10% soil by volume. The final two tests utilized an optimum amendment composition within a mixture containing either 25 or 40% soil by volume.

The second type of pilot study investigation utilized was aerated static piles (SP). Eight static pile tests were completed. Six of these investigated the soil/amendment mixture ratio as a test variable. Five separate concentrations of contaminated soil (7, 10, 20, 30, and 40% soil by volume) were investigated. In addition, one control compost was run using noncontaminated soil at 10% by volume. This test was included to provide a control compost for the toxicity analysis conducted by ORNL. The seventh test was an investigation where soil was augmented with a microbial inoculum developed by Dr. Pat Unkefer of Los Alamos National Laboratory (LANL). This bioaugmented LANL test was conducted in a

SP reactor to facilitate monitoring of test performance. The eighth test used 10% contaminated soil, the same amendment mixture used in the 25% soil MAIV test, and contained a mesh bag filled with 200 g of compost spiked with ^{14}C-TNT.

The first seven aerated static pile studies were conducted concurrently, and were initiated at the same time as the first in-vessel pilot study. The four MAIV investigations were conducted consecutively.

The toxicity of the compost and the possible presence of transformation products were evaluated in an independent program headed by Dr. Wayne Griest of ORNL.

An extensive Test Plan and Safety Plan were developed and approved for the operating procedures and methods employed in the conduct of this field study.

WESTON

SECTION 4

MATERIALS AND METHODS

4.1 SITE LAYOUT

The site layout is presented in Figure 4-1. Prior to placing equipment on site, a bulldozer was used to level the ground where the pilot units were to be placed. A single greenhouse covered all seven static pile pilot units. A separate greenhouse covered the mechanically agitated composting unit. Both of these greenhouses were semicircular in cross-section and constructed to be of a temporary nature. The monitoring equipment and analytical instrumentation were housed in a field trailer along with other supplies. The temperature in the trailer was controlled at approximately room temperature.

4.2 MECHANICALLY AGITATED COMPOSTING UNIT

A Fairfield system pilot unit was selected for use. The composting reactor consisted of a 9-foot diameter tank capable of maintaining a 4-foot deep bed of material. The capacity was approximately 7 cubic yards. Air piping was imbedded in gravel in the bottom of the reactor to aerate the material to be composted. The top of the reactor was a rotating cover with a feeding hopper and six 9-inch diameter agitator augers. A water seal was constructed around the tank cover to maintain a relatively gas tight seal between the rotating top cover and tank sides. Underneath the tank was a rubber belt discharge conveyor that was used to remove material through a rectangular opening approximately 12 by 18 inches in the center of the reactor floor. A schematic of a full scale Fairfield unit is shown in Figure 4-2.

An explosive safety hazard analysis was conducted by Allegany Ballistics Laboratory (ABL) on an existing Fairfield pilot unit. A new pilot unit was ordered for use at UMDA. Results of the ABL hazard analysis were used to guide the engineering design prior to construction of this new unit.

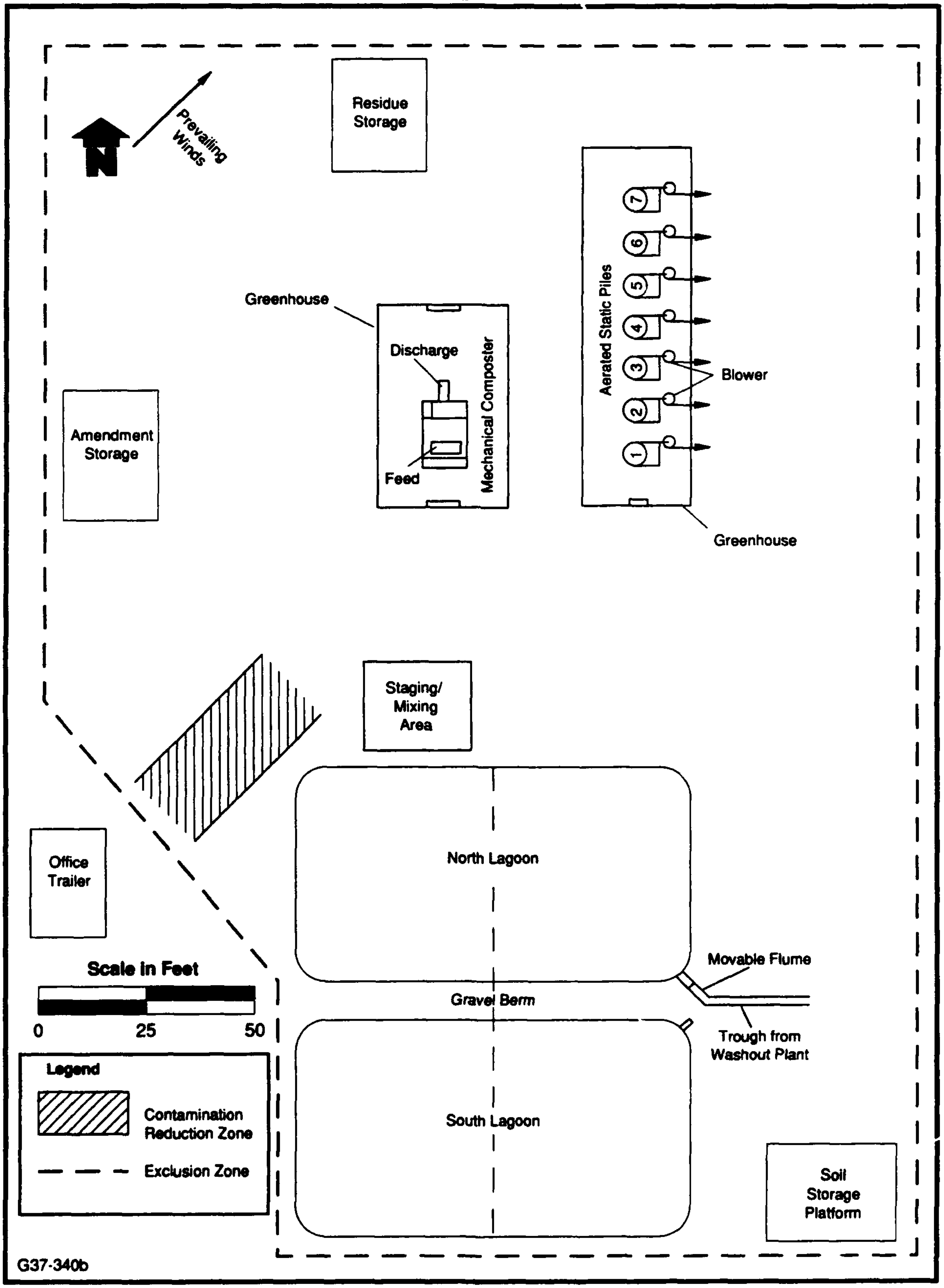


Figure 4-1. Conceptual site layout.

FAIRFIELD SYSTEM

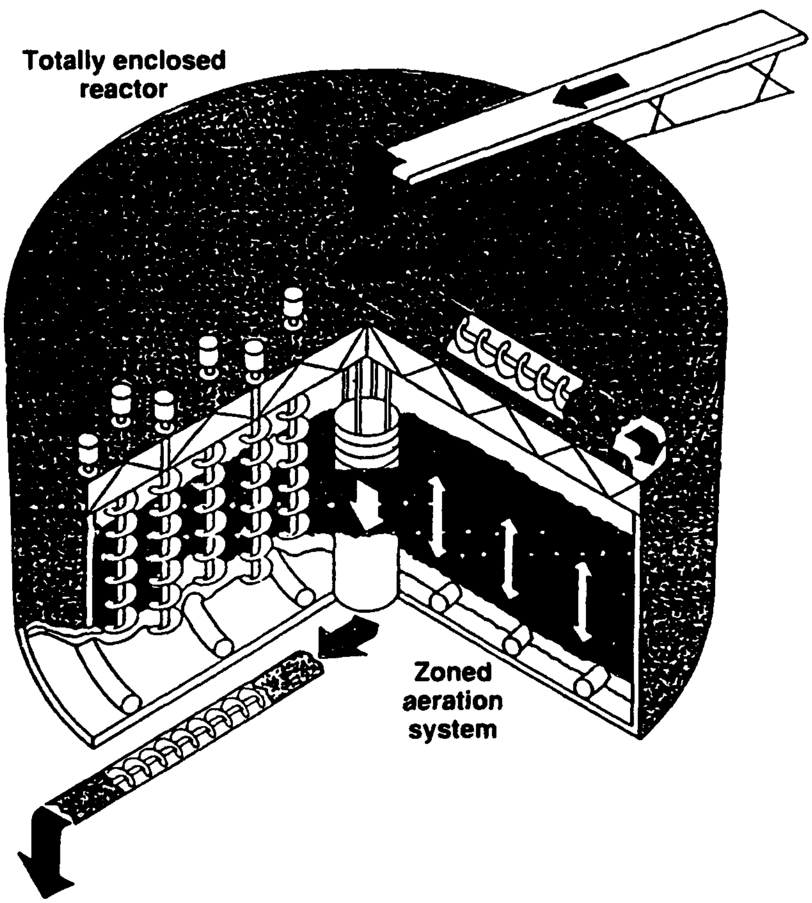


Figure 4-2 Fairfield Full Scale Unit.

The pilot unit was placed on 8-foot, 6-inch by 6-inch ties at UMDA. The unit weighs approximately 15,500 pounds. Loading of the unit was conducted using a front-end loader. A dirt ramp was constructed at one end of the pilot unit to enable the front-end loader to access the loading bin. The pilot unit was covered by a greenhouse following loading with the first in-vessel test mixture.

4.3 STATIC PILE COMPOSTING VESSELS

The design of the static pile pilot reactors assembled at UMDA is illustrated in Figure 4-3. The tanks were of 500-gallon capacity and were made of fiberglass. They had two air inlet ports to help distribute air evenly. A perforated wooden platform was placed on wooden blocks 6 inches from the bottom of each composter. Wood chips were placed in the bottom of the tank beneath the wooden platform. A port was placed in the side of the tank near the top to allow moisture, oxygen, and temperature probes to be inserted. In an effort to prevent moisture loss, influent air was forced through a tank of water prior to entering the bottom of the pilot unit.

Each of the pilot units was insulated (two inches) to prevent heat loss. The pilot units were covered by a greenhouse following loading of the pilot units with the first test mixtures. Each pilot unit functioned independently of the other pilot units.

4.4 MATERIALS HANDLING

A front-end loader, equipped with a 3/4-cubic yard bucket, and a wheelbarrow were used to transport materials. Smaller scale materials handling activities were performed with hand tools. The mechanically agitated pilot unit was unloaded directly into a front-end loader bucket. The static pile tanks were unloaded by hand into a front-end loader bucket.

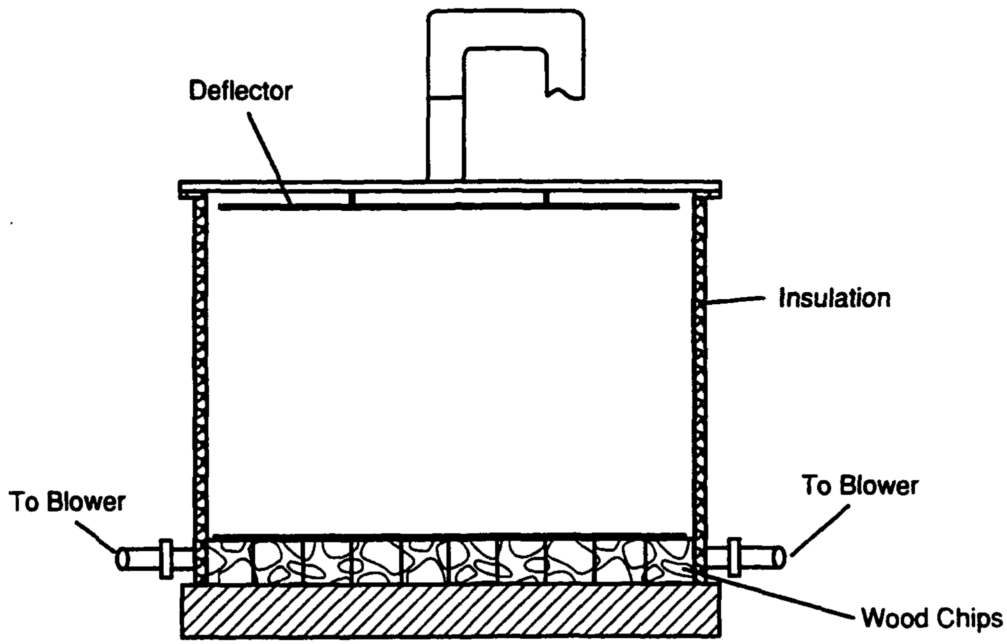


Figure 4-3. Schematic of Aerated Static Pile Reactor.

4.5 TEST SOIL/SEDIMENT

Soil samples were subjected to an explosives and HSL scan (see Section 2.7). Based upon this analysis, the soils in the south lagoon (Figure 2-4) were selected for use.

Approximately 14 cubic yards of test soil were required for the test program. The top layer (0 to 6 inches) of soil contained the highest concentration of explosives. In order to achieve adequate quantities of appropriately contaminated soil for the test, this contaminated surface layer was carefully removed from the most contaminated areas of the lagoon without mixing in less contaminated soil. These higher contamination areas, in general, were in the drainage channel end of the lagoon and along the sidewalks.

The bottom of the lagoon had a number of stones in the 0.5- to 4-inch diameter size range scattered throughout the sandy soils. The sidewalls, however, had a fairly solid layer of these stones as a covering. These stones complicated excavation and had the potential to jam the mechanically agitated pilot unit. Consequently, the soil was screened during excavation.

Excavation was accomplished by hand using suitable shovels. The depth of excavation was guided by the color of the soils and previous analytical data. Excavated soil was sieved through a screen (0.5 inch) as it was placed into a wheelbarrow. The screened soil was then dumped into a 3-sided temporary storage bin constructed of plywood with a liner underneath. The bin was approximately 18 feet by 8 feet with 3-foot sides and was located adjacent to the south lagoon. Soil was mixed using a front-end loader bucket following excavation and was tarped and stored in this storage bin.

Following excavation, five samples of the soil were collected and analyzed. The results obtained are presented in Table 4-1.

Uncontaminated soil (approximately 1 cubic yard) was obtained from an undeveloped area near the lagoon site.

Table 4-1

Explosives Content of Excavated Soil
(mg/kg*)

	TNT	RDX	HMX
Sample 1	14,300	1,400	293
Sample 2	13,800	798	242
Sample 3	10,600	697	209
Sample 4	15,700	898	255
Sample 5	12,500	1,560	364
Mean	13,380	1,071	273

*Dry weight

4.6 AMENDMENT SELECTION

For the purposes of this study, amendments were any materials included in the mixture to be composted in addition to the soil. Parameters evaluated in the amendment selection process for the carbon sources included: pH, C:N ratio, moisture content, homogeneity, availability, cost, total metabolic energy content, rate at which the carbon substrates were utilized, texture, form, porosity, bulk density, and inorganic nutrient content. The target C:N ratio was 30:1.

An evaluation of materials available in the UMDA area was conducted. A wide variety of materials were found to be available in the agriculturally rich states of Oregon and Washington. These materials included animal waste (cattle, poultry, buffalo, horse), field crops (wheat, barley, corn, hay, potatoes, alfalfa, etc.), fruit, vegetable, and nut field waste, as well as processing waste from potato plants, canneries, dairies, wineries, and fisheries. A major concern was the seasonal availability of these materials, since it was desirable to have each amendment be consistent from test to test.

A strategy for selecting amendments was developed by WESTON and Woods End Research Laboratory. Once regionally available materials were identified, an analysis of a sample of each was conducted. Parameters evaluated included density, solids, pH, and total nitrogen and carbon. In addition, the respiratory potential of each amendment was determined. Using this information, test mixtures were developed. These mixtures were subjected to additional respiratory potential studies. The three optimal mixtures identified in these studies were tested in adiabatic composting trials (Figure 4-4). Based upon these results, an amendment mixture (mix 2 in Figure 4-4, mix A in Table 4-2) was selected for use in the first 10% soil MAIV test (MAIV-1). This amendment mix also was utilized for the static pile tests (with the exception of the bioaugmented test [SP-4] and the ^{14}C-TNT mesh bag SP test [SP-8]). These were tests SP-1 to SP-3 and SP-5 to SP-7. The ingredients and ratio (by volume) of the non-soil amendments used were: sawdust (30%), apple pomace (15%), chicken manure (20%), and chopped potato waste (35%). These amendments and the others used in the UMDA composting program are summarized in Table 4-2.

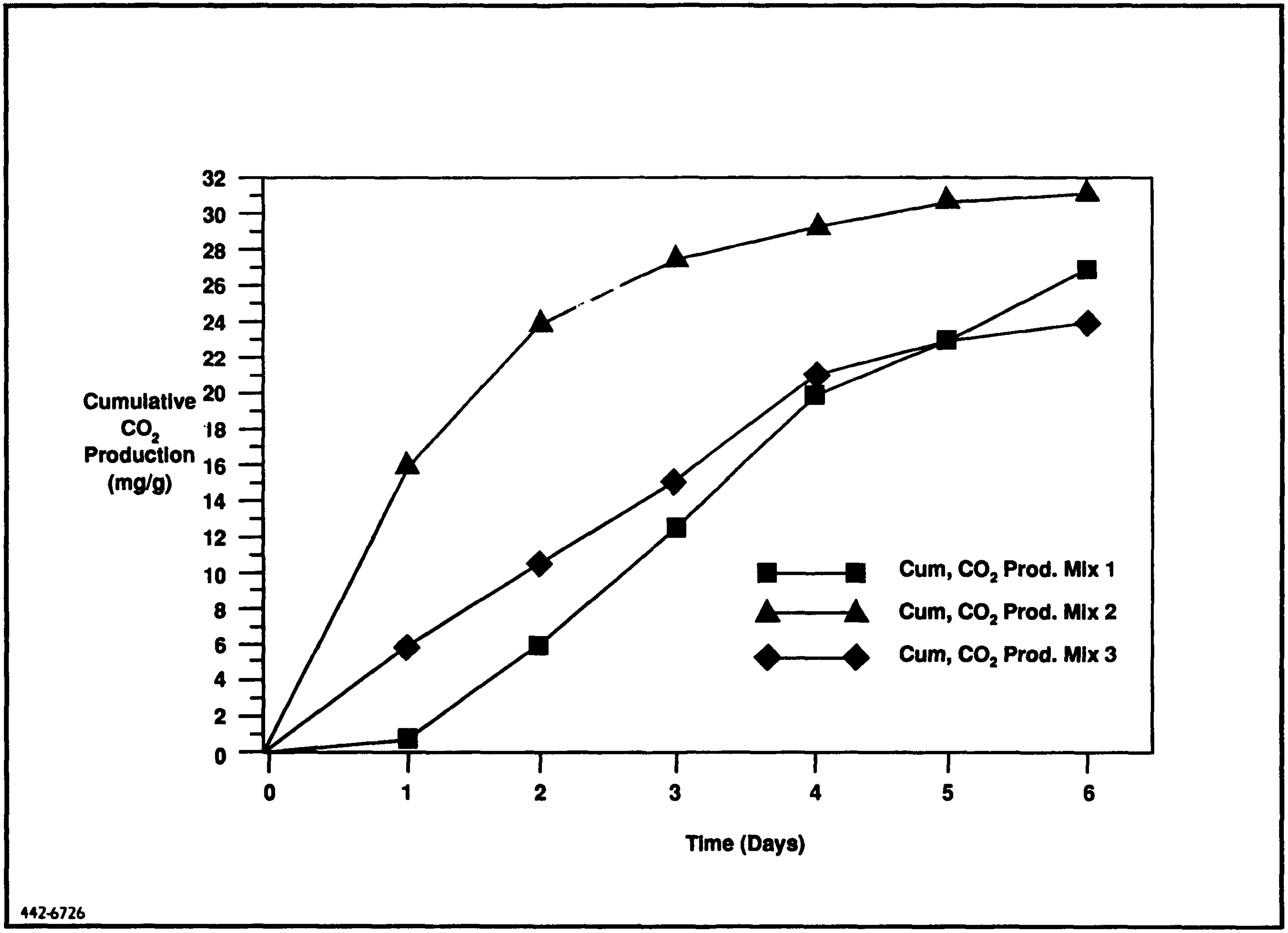


Figure 4.4. Cumulative Respiratory Potential for Amendment Mixtures in Adiabatic Tests

Table 4-2

Non-Soil Amendments Used in UMDA Composting Tests

	Amendment Mix		
	A	B	C
Test	SP-1	MAIV-2	MAIV-3
Test	SP-2		MAIV-4
Test	SP-3		SP-8
Test	SP-5		
Test	SP-6		
Test	SP-7		
Test	MAIV-1		
Amendments			
Sawdust	30%		22%
Apple pomace	15%		6%
Chicken manure	20%		
Chopped potato waste	35%		17%
Horse manure/straw		50%	
Buffalo manure		10%	
Alfalfa		32%	22%
Horse feed		8%	
Cow manure			33%

The selection strategy for the second 10% soil MAIV test (MAIV-2) was based upon duplicating the mixture utilized in the Louisiana Army Ammunition Plant investigation (Williams et al., 1988). The objective was to compare the effectiveness of the composting process for two very different soil types. The ingredients and ratio (by volume) of the non-soil amendments used was: horse manure/straw (50%), buffalo manure (10%), alfalfa (32%), and horse feed (8%). Buffalo manure was used to supplement the horse manure because the horse manure available in the local area did not meet all the desired criteria and because the buffalo manure had suitable characteristics.

The ingredients and ratio (by volume) of the nonsoil amendments used in the 25% soil and 40% soil MAIV tests (MAIV-3 and MAIV-4, respectively) as well as in the ^{14}C-TNT containing SP test (SP-8) were sawdust/alfalfa (44%), cow manure (33%), apple waste (6%), and potato waste (17%). The same batches of amendments were used for the 25% soil MAIV test (MAIV-3) and the ^{14}C-TNT containing SP test (SP-8) since they were established at the same time. New batches of each ingredient were obtained for the 40% soil MAIV test.

A microbial inoculum developed by Dr. Pat Unkefer was investigated in one of the static pile tanks (test SP-4) to facilitate process monitoring. The amendments and soil percentage used in this investigation were determined by Dr. Unkefer based on the specific requirements of the added microorganisms. The amendment/soil mixture consisted of 1 cubic yard of contaminated soil, 3/10 yards of sawdust, 40 lb of ammonia sulfide (21:0:0), 10 gallons of sodium acetate (solution prepared by Dr. Unkefer, concentration not determined), and 11 kg of L-arginine. Water was added (approximately 10 gallons) to reach a moisture content of approximately 50% at the direction of Dr. Unkefer. The test matrix was fed at day 77, at the request of Dr. Unkefer, with 28 lb of dry sodium acetate, which was mixed directly into the test matrix. Following this addition, 12 lb of sodium acetate, dissolved in 12 gallons of water, was added to the surface of the matrix.

4.7 AMENDMENT/SOIL MIX PREPARATION

The amendments and soil to be included in each mixture to be composted were measured volumetrically using a calibrated front-end loader bucket. The weight of all components used in the mixture also was determined. The bulk density of each component was measured prior to mixture preparation. At the direction of USATHAMA, volume was used as the key measurement for individual ingredients.

The components to be included in the mixture to be composted were placed in a 3-sided mixing bin where a front-end loader was used to mix the components. The soil mixtures for the first set of SP tests were individually prepared starting with the uncontaminated mixture and then going from the lowest contaminated soil percentage in order to the highest percentage.

4.8 TEMPERATURE MONITORING

Each of the seven static pile units contained five thermocouples in the compost mixture. These probes were located in the compost mixture at the top of the compost mixture/tank center, middle/center, bottom/center, and upper and lower side. In addition, a sixth thermocouple was located in the gas headspace above the compost and just in front of the exhaust port.

A temperature probe was placed into the central region of the compost mixture through the sidewall of the Fairfield composter. This probe had to be removed when the rotating cover was in operation. In addition, a temperature probe was located in the exhaust line of the Fairfield composter.

Data from all thermocouples were automatically logged directly into a computer, as described in Section 5. All the composters (seven aerated piles and the mechanical composter) fed output data to a single data acquisition/process control computer system, which cycled among the piles automatically. In addition to these automatic readings, manual readings were periodically taken using a hand-held probe and meter, particularly in the Fairfield composter.

4.9 TEMPERATURE CONTROL

In the Fairfield pilot unit, two thermocouples provided feedback control over airflow to the reactor. Airflow and, consequently, ventilative heat removal was controlled by means of two valves. In addition, manual control could be initiated by means of a blower timer and manual adjustment of the air control valves.

The optimum range for temperature was determined to be 50 to 55°C. Temperature was controlled by temperature feedback and ventilative heat removal. At preselected intervals, the readings from the five temperature monitoring thermocouples in the compost of the static pile reactors were summed and averaged automatically by the temperature control software. This value was then compared with a set point. Average temperatures above this set point caused the blower to be turned on. The blower remained on until subsequent average readings were at or below a second setpoint.

In the event that the average temperature in a pile did not trigger blower operation for heat removal, a backup system actuated the blower on a timed basis to oxygenate the compost. A schematic of the entire monitoring/controlling system is shown in Figures 4-5 and 4-6.

4.10 OXYGEN MONITORING

Lines from the exhaust outlet of each static pile composter and the Fairfield pilot unit carried exit gas to a manifold that sent exit gas to an oxygen analyzer present in a metal sampling cabinet within the static pile greenhouse (Figure 4-5). The valve for the vessel to be sampled was opened and a vacuum pump was used to draw gas from the vessel through the oxygen analyzer. A sufficient volume of gas to purge the sample line was drawn prior to analyzing the oxygen content. Data from each sample were transmitted directly to the computer in the trailer. Once the oxygen analysis was completed, the operator proceeded

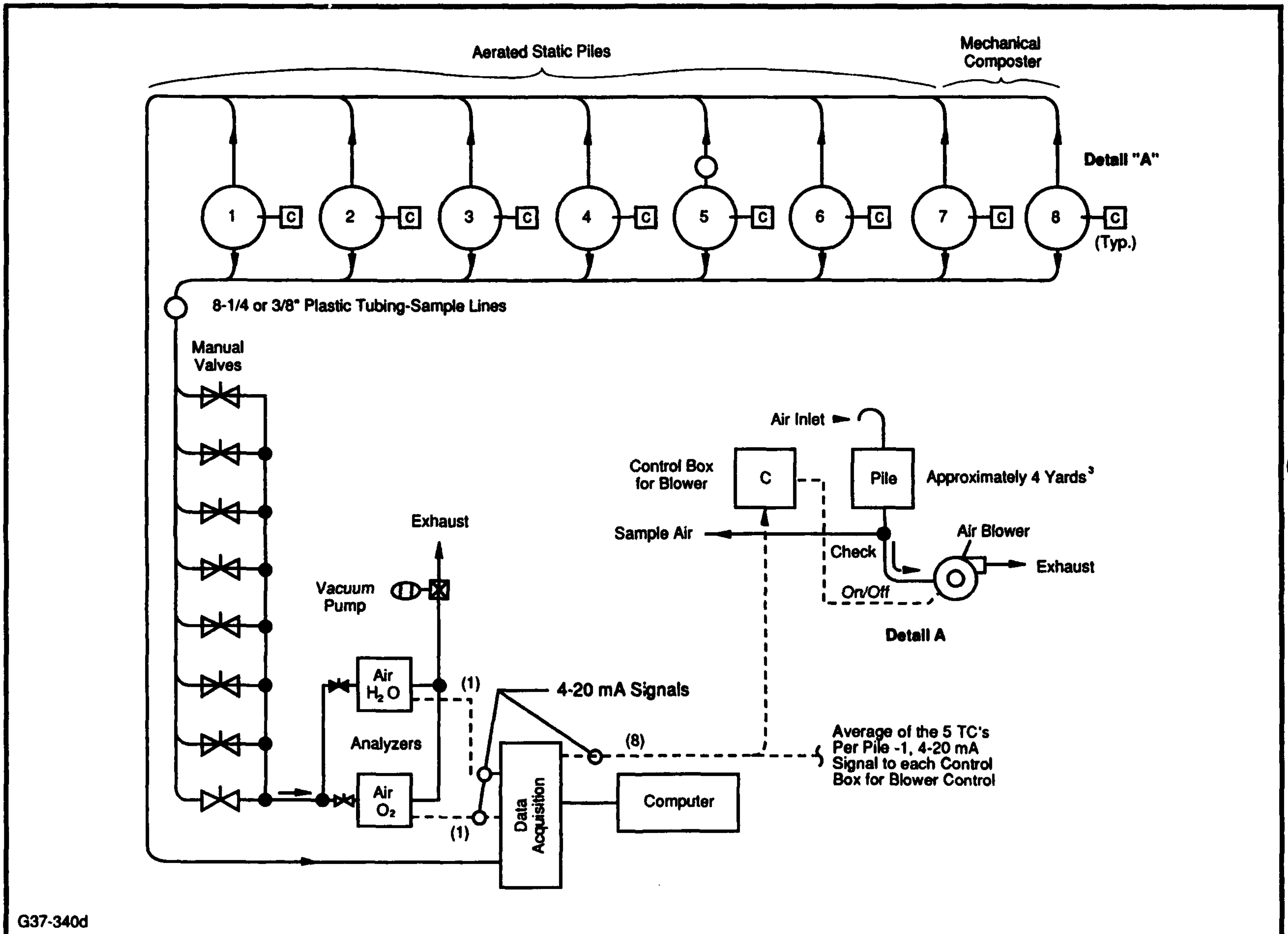
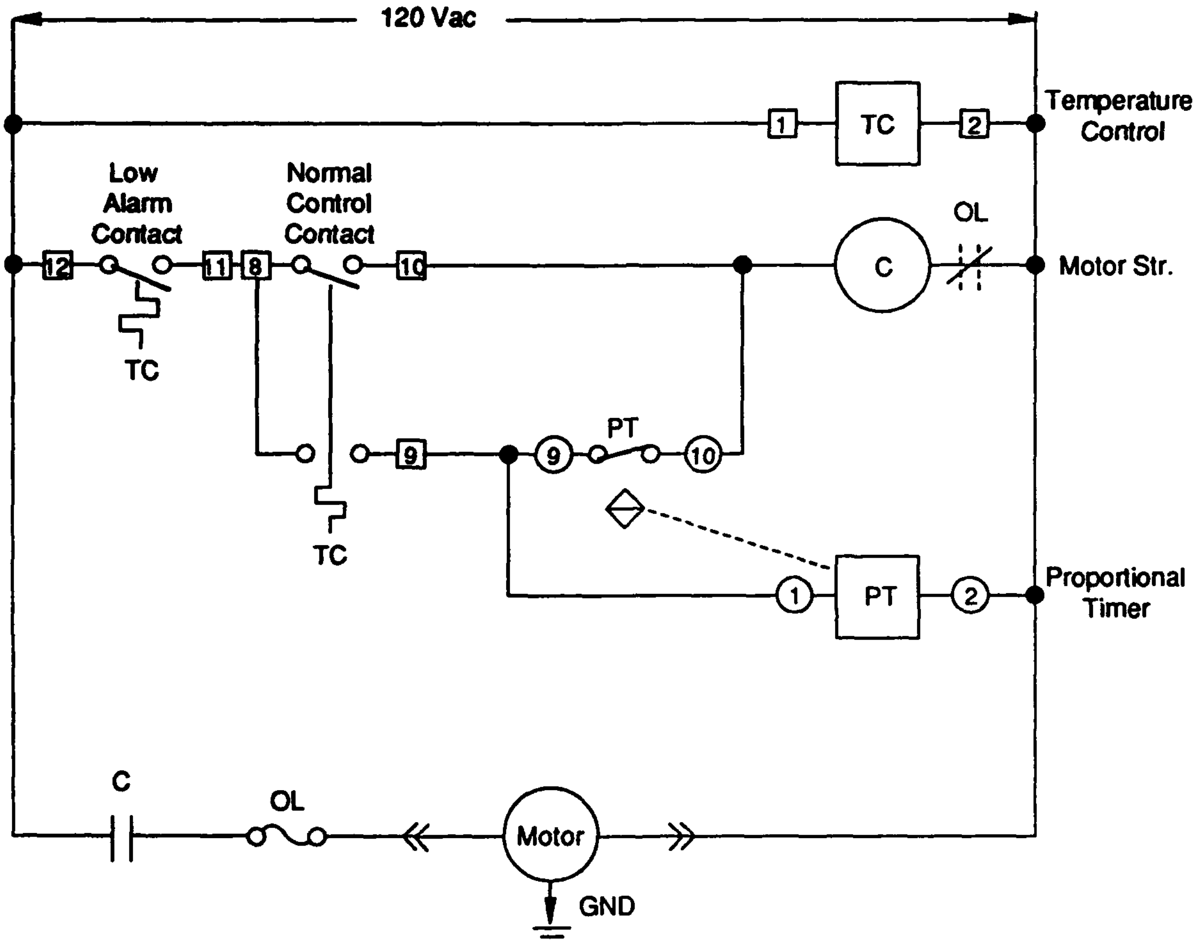


Figure 4-5. Process control/monitoring schematic.



Estimated Settings for Operation-
1. Temperature Controller-
   High = 55°C
   Low = 50°C
   Dead Bank = 50 to 55°C

2 Proportional Timer-
   Maximum Time = 4 min.
   Interval = 1 min.

442-6724

Figure 4-6. Temperature control schematic.

with the moisture analysis. Once the analysis for each vessel was accomplished, the operator closed the sample line valve and analyzed the next vessel.

In addition to the exit gas analysis system, a manual probe and hand-held meter was used to monitor the oxygen content within the compost matrix. A manual pump was used to draw interstitial air into the sampling tube and past an oxygen cell.

4.11 OXYGEN CONTROL

It has been shown (Finstein et al., 1986) that approximately five to seven times more airflow is required to remove heat than to provide oxygen. Consequently, oxygen was provided in the course of controlling temperature. No attempt was made to regulate oxygen content at a precise level. During the warm-up phase of operation, the blowers operated on a timer cycle to ensure that adequate oxygen was present.

4.12 MOISTURE MONITORING

Moisture content in the mixtures in each of the pilot units was monitored using grab samples three times per week at different depths. Three grab samples were taken and moisture content (water holding capacity and percent moisture) determined by weight loss following drying.

In addition, moisture was monitored in the exit gas in a manner analogous to that used for oxygen. The lines carrying the exit gas to the single analyzer were temperature controlled (heated) to avoid errors caused by temperature fluctuations and condensation.

4.13 MOISTURE CONTROL

Water was added directly to a composting mixture when moisture fell below the desired range. This range was approximately 45 to 55%, but varied somewhat depending upon the nature of the amendments and soil percentage. Moisture addition was accomplished using a hose. Application of water was conducted such that the total volume of water added could be calculated.

4.14 pH MONITORING

pH was monitored three times per week initially at different depths using three grab samples from each pilot unit. pH was determined by placing 20 grams of compost in a 100-milliliter (mL) beaker with 20 mL of deionized water, stirring the suspension every 10 minutes for 30 minutes, allowing the suspension to settle for 1 hour, and measuring the pH of the liquid by pH meter.

4.15 pH CONTROL

pH control was discussed with USATHAMA as part of the amendment selection process. One of the initial amendment selection criteria included choosing a mixture with as low a tendency as possible to produce pH extremes. Accomplishing pH control once an SP test was initiated was considered to be excessively disruptive to the test since mixing a pH control agent into the compost would be required. A decision was made not to actively attempt to control basic pH levels until a pH of 9.5 or greater was reached. Acidic pH values below 5.5 were countered by increasing oxygenation to avoid anaerobic conditions and acid formation.

4.16 SAMPLING

Samples for chemical characterization were taken from the compost mixture by hand and also using a soil auger (Forestry Suppliers, Inc.). The area where the sample was to be taken was first exposed. The auger was then inserted into the appropriate area of the compost pile and a core sample removed. Sample locations used were recorded in a data logbook. Samples were packed in labeled amber bottles and shipped by overnight freight using chain-of-custody procedures. The auger was cleaned between each individual sampling.

The sample locations for each time point for the static piles were from the same plane within the composting vessel. Two samples were taken from the top of the compost mixture approximately 6 inches in from the sidewall. Two samples were taken from the bottom of the compost mixture directly below where the top samples were taken. The fifth sample was taken from the pile center. This center location was approximately where diagonal lines connecting the top and bottom samples from opposite sides of the tank would cross. For subsequent samplings, the sampling plane was rotated. The sample plane was flagged for each sampling.

4.17 SAMPLE PREPARATION

Samples collected at UMDA were required to be split so that both WESTON and ORNL had the same representative sample to analyze. In order to accomplish this uniformity, a sample preparation strategy was developed by the United States Geological Survey Office in Denver, Colorado, USATHAMA, and WESTON.

Samples were individually air dried in a storage shed at UMDA. This required approximately 6 to 8 days, after which the samples were sent by overnight carrier to WESTON. When the samples were received by WESTON, each of the individual samples was processed through a Wiley mill. Each sample was then split into an "A" and "B" sample using a riffle-type splitter. The "A" sample was submitted to WESTON Analytics for TNT, HMX, and RDX analysis. The "B" samples were combined into one sample using the splitter and sent to ORNL.

In order to test the ability to satisfactorily clean the mill and splitter, as well as to achieve identical split samples, a system test was performed. Two contaminated soil compost mixtures and two uncontaminated soil compost mixtures were processed using the procedure diagrammed in Figure 4-7. The samples produced were analyzed for TNT. The results presented in Figure 4-7 demonstrate that excellent sample homogenization and splitting were achieved, and that the equipment was satisfactorily cleaned between samples.

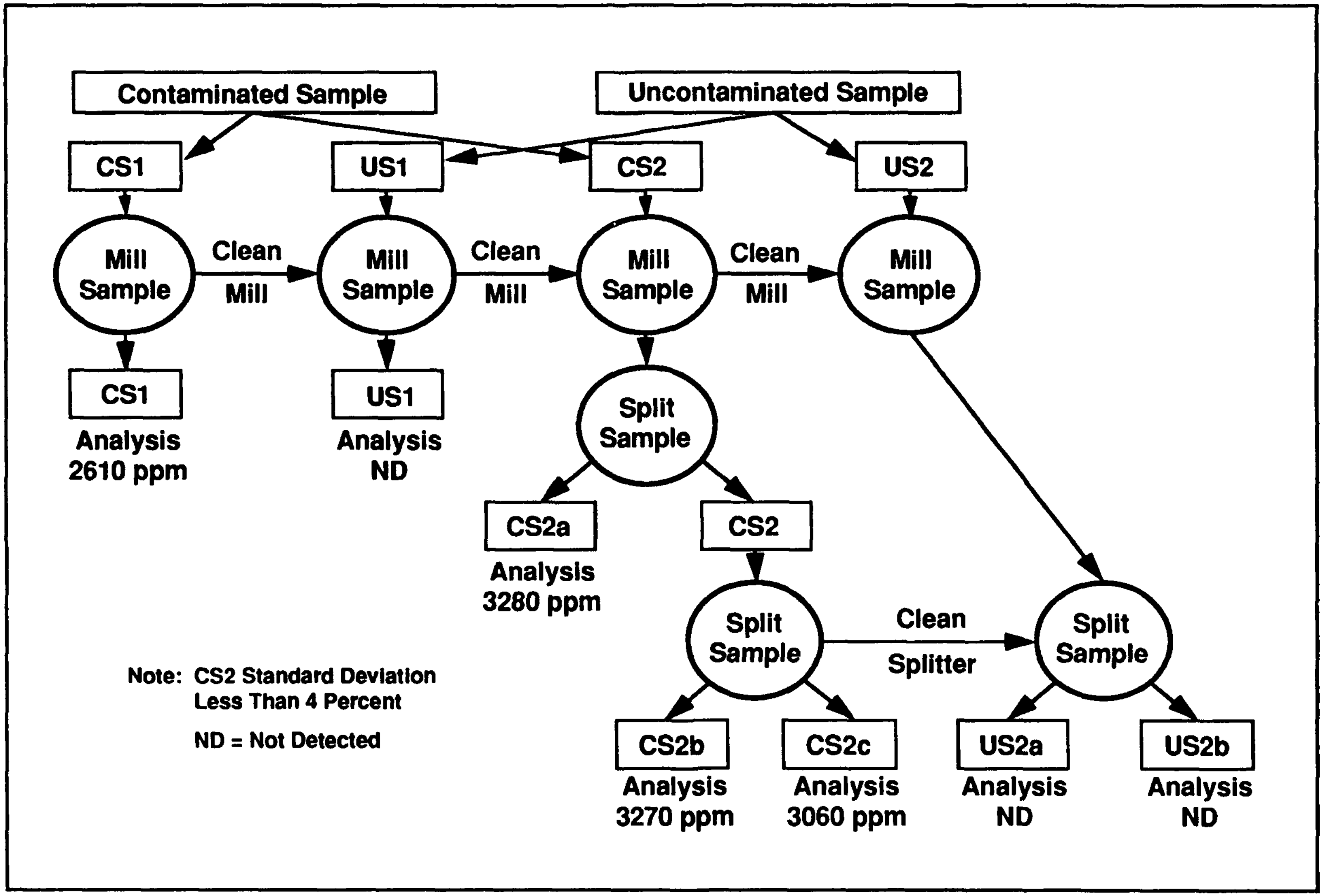


Figure 4-7. Sample Homogenization/Splitting Study

957

4.18 ANALYTICAL

Compost samples were analyzed for TNT, RDX, and HMX by USATHAMA Method LW02 (see Appendix A), modified for the extraction and analysis of compost. A less detailed description of the method is provided in the following discussion, including modifications made for the analysis of UMDA compost.

All samples from tests SP-1 to SP-7 and MAIV-1 to MAIV-3 were analyzed by WESTON. Samples from SP-8 and MAIV-4 were analyzed for explosives at ORNL due to financial constraints in WESTON's contract. Samples from SP-8 and MAIV-4 were prepared by WESTON using the methods described in Section 4.17 prior to shipment to ORNL.

Sample Extraction and Preparation

- Approximately 1.0 gram of dried and milled compost was accurately weighed into a 5-mL serum vial with a Teflon-lined crimp cap.
- Acetonitrile (4.0 mL) was added.
- The jar was shaken by hand for 1 minute.
- The sample was allowed to settle for 15 minutes.
- Approximately 2 mL of extract was filtered with a 0.2-micron (μm) Teflon filter and retained in a 4-mL autosampler vial.
- At the time of analysis, 200 μL of the extract was diluted with 600 microliter (μL) of 2:1 water/methanol.

Percent Solids Determination

Approximately 5 grams of dried and milled compost was accurately weighed into an aluminum weighing pan and dried overnight at 105°C. The sample was reweighed the following day, and percent solids in the sample determined by weight loss.

WESTON

High-Performance Liquid Chromatograph (HPLC) Analysis

The following instrument conditions were used to analyze UMDA compost samples:

- Column: Zorbax C-8, 250 mm x 4.6 mm, 5 micron.
- Detector: UV absorbance at 250 nm.
- Mobile phase: 52% methanol/48% water.
- Flow rate: 1.5 mL/min.
- Injection volume: 50 µL.

The HPLC was calibrated by analysis of the following series of standards. The standard concentrations shown are in units of mg/kg in compost (assume 10-gram sample, no dilution). For example, a compost sample that contained 5.08 mg/kg of HMX would produce an extract with the sample response of the 2x standard. Linear regression was performed for each analyte and used to quantify sample response.

Standard	HMX	RDX	2,4,6-TNT
0.5x	1.27	0.98	1.92
1x	2.54	1.96	3.84
2x	5.08	3.92	7.68
5x	12.7	9.80	19.2
10x	25.4	19.6	38.4
20x	50.8	39.2	76.8
50x	127	98.0	192
100x	254	196	284

The daily protocol for sample analysis consisted of the following steps:

- Full calibration curve and linear regression for all analytes.
- QA/QC samples.
- Sample extracts.
- Final 2x and 10x calibration standards.

Samples were diluted with mobile phase as necessary to bring target analytes into the calibration range. Final quantification of explosives was determined by the following formula:

sample response (ppm) x D x 1/W = mg/kg analyte

Where:

D = dilution of extract
W = dry weight of sample

QA/QC Samples

The following QA/QC samples were analyzed with each batch of compost samples:

- Method blank.
- 2x standard spike (at 1x calibration level, page 4-19).
- 10x standard spike (at 5x calibration level, page 4-19).
- 10x standard spike duplicate.

Samples were prepared as described for compost samples, with the following exceptions. USATHAMA-standard soil was used as the sample matrix. One gram of soil was weighed into a 5-mL serum bottle, and 2.0 mL of acetonitrile was used for extraction.

Detection Limits

The following detection limits were determined for analysis of TNT, RDX, and HMX in compost:

- TNT 3.84 mg/kg
- HMX 2.54 mg/kg
- RDX 1.96 mg/kg

4.19 MICROBIAL ENUMERATION

The population density of heterotrophic microorganisms was determined for selected compost samples. One gram of compost was aseptically transferred into 90 mL of sterile 0.1 M K_2HPO_4 buffer and agitated by hand for 2 minutes. Large particles were allowed to settle after agitation. The compost extract was serially diluted into sterile phosphate buffer (1 mL extract into 9 mL buffer) to a dilution of 10^{-9}. Each dilution was either spread-plated or pour-plated onto nutrient agar plates (Difco Laboratories). Plates were incubated at 55°C for 5 days. Total colony counts were made after days 2 and 5 of incubation. The total number of microbial (bacterial and fungal) colonies on each plate were used to calculate the number of colony-forming units (cfu) per gram of dry compost.

4.20 MICROTOX

A subsample was taken from selected compost samples and the initial soil samples sent to WESTON's analytical laboratory for TNT, HMX, and RDX analysis. An aqueous extract of those samples was prepared by diluting 5 grams of milled compost in 50 mL of distilled water. Solutions were mixed for 1 hour on a wrist-action shaker. Solids were allowed to settle and the liquid extract was filtered and collected in glass vials. Subsequent dilutions were made depending on the degree of toxicity of each extract. This sample was then analyzed using the Microtox (Microbics Corporation) analysis for toxicity. Raw data were used to calculate the EC_{50} for each sample at test intervals of 5 and 15 minutes.

4.21 AIR SAMPLING

At selected times, approximately day 5, 15, 30, and 60, exhaust air from a 10% contaminated soil static pile (SP-8) was analyzed for the presence of nitroaromatics. Air was drawn through a XAD resin trap downstream from the vacuum pump (see Subsections 4.10 and 4.12) for a period of approximately 4 hours to capture nitroaromatics. This XAD resin was extracted and the extract analyzed for nitroaromatics.

4.22 SAFETY

Specific safety requirements were addressed in the UMDA Safety Plan prepared for this project by WESTON. Soil excavation and materials handling up until the point when compost was placed into the compost vessels were conducted in OSHA level C. Other operations were conducted in OSHA level D. All appropriate safety equipment was maintained on-site. A telephone was placed in the site trailer to serve as a direct link with the base and surrounding communities should an emergency occur.

4.23 COMPOST RESIDUE DISPOSAL

At the end of each pilot test, the compost residue produced was disposed of in a basin excavated specifically for this purpose. The basin was lined with high-density polyethylene (HDPE). After each batch of compost residue was placed in this basin, HDPE was placed over the basin to prevent the entry of water or wind dispersal of the residue. The cover edges were folded with the edges of the basin liner and weighted with 8-foot 6x6's to prevent movement of the cover.

This HDPE-lined basin constituted interim disposal for the compost residues, with the ultimate disposal method to be determined as part of the remedial action plan for the UMDA NPL site.

4.24 MICROBIAL INOCULATION

Dr. Pat Unkefer of LANL developed a microbial inoculum with proven effectiveness at mineralizing TNT in laboratory tests. This microbial inoculum was evaluated in a static pile reactor (Test SP-4). Dr. Unkefer and USATHAMA developed the test design based on data developed at LANL. WESTON established the field test under the on-site guidance of Dr. Unkefer.

4.25 INTEGRATION WITH ORNL - TOXICITY, FATE, AND ^{14}C-TNT STUDIES

ORNL was contracted by the U.S. Army Biomedical Research and Development Laboratory (USABRDL) for toxicological testing and chemical characterization of the composting residues generated during this optimization study. In order to support this study, WESTON prepared and provided samples to ORNL as previously described in Subsection 4.17.

In addition, a composted radiolabeled TNT was prepared by placing one mesh bag containing 200 g of compost into the 10% contaminated soil static pile. Prior to distributing compost into this bag, 4.0 x 10^8 dpm (~ 0.2 mCi) of ^{14}C-TNT (shipped to WESTON by ORNL) was mixed with 400 g of compost. One-half of the total ^{14}C was mixed with each 200 g batch of compost. This ^{14}C addition was done at WESTON's Fate and Effect Laboratory using compost shipped overnight from UMDA. One 200 g portion was shipped directly to ORNL for analysis, and the second to UMDA for placement in the compost matrix above a pyrex pan. The bag enclosed in the compost pile was removed and shipped directly to ORNL at day 90. Prior to conducting this part of the ORNL program, approval was obtained from the Department of Defense. Monitoring of 10 samples of the surrounding compost for ^{14}C was conducted at day 90. No ^{14}C was detected in these samples. The compost residue from this test was then placed in the lined storage basin.

4.26 TEST SCHEDULE

The starting and ending dates for each of the tests conducted are presented in Table 4-3.

4.27 CALCULATION OF EXPLOSIVE HALF-LIVES

There are several ways one might evaluate the kinetic rate of destruction of TNT, RDX, and HMX during the test period. The rate constants for the disappearance of each explosive could be calculated. This would require the degradation kinetics to be pseudo first order and the decay curve to be logarithmic (exponential) with time. However, reaction rates are very sensitive to changes in temperature, which varied considerably during the test period.

Table 4-3

Soil Loading and Starting and Ending Dates for Each Test Conducted

Test	% Soil/ Amendment Mix*	Day 0	Day 44	Day 90
SP-1	7/A	9/21/90	----	12/20/90
SP-2	10/A	9/21/90	----	12/20/90
SP-3	20/A	9/21/90	----	12/20/90
SP-4	80/ inoculated	11/2/90	----	1/30/91
SP-5	30/A	9/21/90	----	12/20/90
SP-6	40/A	9/21/90	----	12/20/90
SP-7	10 UC/A	9/21/90	----	12/20/90
SP-8	10/C	2/6/91	----	5/7/91
MAIV-1	10/A	9/19/90	11/2/90	----
MAIV-2	10/B	11/8/90	12/21/90	----
MAIV-3	25/C	2/6/91	3/22/91	----
MAIV-4	40/C	4/3/91	5/18/91	----

*See Table 4-2

There were both seasonal changes and daily temperature changes. In general, reaction rate constants double with a temperature increase of 10°C (18°F). Therefore, a meaningful rate equation and rate constant would be difficult to determine.

Normally, half lives would be calculated using the following equation:

$$t_{1/2} = \ln(2)/k = 0.693/k$$

where k = rate constant

But, for the reasons cited above, the rate constants could not be accurately determined using this approach. Therefore, half-lives were determined by linear interpolation between the two data points that bracket the concentration, which is half of the original concentration ($C_{1/2}$). Half lives were only calculated for tests where the concentration actually fell to below one-half the original concentration during the course of the test. This value was calculated using the following equation:

$$t_{1/2} = (t_2 - t_1) * \left[\frac{((C_o/2) - C_1)}{(C_2 - C_1)} \right] - t_1$$

Where: C_o = the initial concentration. ($C_o/2$ is one-half the initial concentration).

C_1 = concentration at t_1

C_2 = concentration at t_2

$t_{1/2}$ = the half-life, when the concentration is one-half the initial concentration.

SECTION 5

RESULTS

5.1 EXPLOSIVES DEGRADATION

5.1.1 Static Pile Tests

Explosive degradation in each static pile varied depending upon soil content and other factors. Overall, TNT exhibited the highest percent degradation (79 to 99%), followed by RDX (0 to 93%), and HMX (2 to 80%). Percent reduction data are summarized in Table 5-1. Kinetic rate of destruction is the key measurement of destruction for the purposes of this test. Half-lives for the various tests are presented in Table 5-2.

TNT degraded in the initial static pile composting tests (Figure 5-1) regardless of the initial TNT concentration. The majority of TNT reduction occurred within the first 44 days of the study; all decreases during this test period were statistically significant ($P < 0.05$; Appendix C). TNT concentrations on Day 90 of the study were not significantly different from those observed on Day 44 ($P > 0.05$; Appendix C). While the greatest percent degradation was observed in the 30% contaminated soil pile, a significant ($P < 0.05$; Appendix C) decline in TNT was also observed in the 40% contaminated soil pile (Tables 5-1, 5-3).

Declines in RDX concentrations varied from pile to pile (Table 5-3, Figure 5-2). The greatest RDX degradation in the initial static pile composting tests was observed in the 7% contaminated soil pile (SP-1), while no significant reduction was observed in the 40% contaminated soil pile (SP-6) ($P > 0.05$; Appendix C) or the 20% contaminated soil pile (SP-3) ($P > 0.05$).

HMX degradation in the initial static pile composting tests also varied widely. The greatest HMX degradation in these initial tests, 37% of the initial concentration, was observed in the 7% contaminated soil pile. No significant ($P > 0.05$) degradation was observed in either the 20% or 40% contaminated soil piles (SP-3 and SP-6, respectively) (Table 5-3; Figure 5-

3). The greatest degradation occurred in the first 20 days of the study (corresponding to the active composting phase) in piles where significant degradation was actually observed.

Explosives degradation profiles for each static pile are shown in Figures 5-4 through 5-9. Figure 5-9 presents the explosives reduction data for the static pile test that contained the ^{14}C-TNT test (see Subsection 4.25). This study was conducted after the initial static pile tests using a different amendment mix. In addition, operating parameters such as temperature were not affected by instrument failures. The bulk mixture samples for SP-8 were analyzed and the data provided (Table 5-3) by Oak Ridge National Laboratory. Test SP-8 contained the same amendment mixture as the 25% and 40% soil MAIV tests (MAIV-3 and MAIV-4). This mixture composted well in all three tests (Figures 5-30, 5-35, 5-36). More rapid and extensive degradation of all three explosives was observed in SP-8 than in any other SP test. This was particularly true of HMX (80% reduction) and RDX (93% reduction). This demonstrates the importance of amendments and achieving and maintaining active composting.

5.1.2 Mechanically Agitated Tests

Four mechanically agitated in-vessel (MAIV) pilot tests were completed. The first two MAIV tests were amendment selection tests using 10% contaminated soil. The second two tests were soil loading tests conducted with 25% and 40% soil. Different amendments were used in MAIV-1 and MAIV-2 (Table 4-2). These two mixtures differed from the amendment mixture used in both MAIV-3 and MAIV-4. The first 44-day test reduced TNT concentration by 97%, RDX by 90%, and HMX by 29% (Table 5-1; Figure 5-10). In the second 44-day test, TNT was reduced by 99%, RDX by 99%, and HMX by 95% (Table 5-1; Figure 5-11). The majority of degradation in both tests occurred within the first 10 days of the study (Table 5-3; Figures 5-10, 5-11).

In the first soil loading test (MAIV-3), TNT was reduced by 99%, RDX by 97%, and HMX by 68% (Table 5-1, Figure 5-12). In the 40% soil loading test (MAIV-4), reduction was significant for TNT, but not for either HMX or RDX. TNT was reduced by 97%, RDX by

18%, and HMX by 0% (Table 5-1, Figure 5-13). Once again, the majority of the degradation that did occur, occurred in the first 20 days of the study (Table 5-3, Figures 5-12, 5-13).

Half-lives for all explosives in the MAIV tests are presented in Table 5-2. These data indicated that MAIV-2 exhibited the best explosives degradation.

5.1.3 Los Alamos National Laboratory Bioaugmented Test

Explosives in a static pile test inoculated with microbes developed at the Los Alamos National Laboratory demonstrated no statistically significant ($P > 0.05$) degradation (Tables 5-1 and 5-3; Figure 5-14).

Table 5-1

Percent Reduction of Explosives in UMDA Compost Experiments

Test	% Soil/ Amendment Mix*	Percent Reduction HMX	RDX	TNT
SP-1	7/A	39	73	91
SP-2	10/A	21	46	96
SP-3	20/A	5	16	94
SP-4	80/inoculated	2	4	6
SP-5	30/A	11	22	98
SP-6	40/A	2	0	79
SP-7	10 UC/A	n/a	n/a	n/a
SP-8	10/C	80	93	99
MAIV-1	10/A	29	90	97
MAIV-2	10/B	95	99	99
MAIV-3	25/C	68	97	99
MAIV-4	40/C	0	18	97

n/a - Uncontaminated soil pilot unit, no explosives present.
* - See Table 4-2

Table 5-2

Half-Lives for TNT, RDX, and HMX

Test	% Soil/ Amendment Mix**	Half-Life (Days) TNT	RDX	HMX
SP-1	7/A	6.6	27.7	*
SP-2	10/A	6.4	*	*
SP-3	20/A	14.8	*	*
SP-4	80/inoculated	*	*	*
SP-5	30/A	16.1	*	*
SP-6	40/A	24.9	*	*
SP-7	10 UC/A	*	*	*
SP-8	10/C	6.9	12.8	*
MAIV-1	10/A	5.2	15.4	*
MAIV-2	10/B	5.1	5.3	*
MAIV-3	25/C	6.4	4.4	*
MAIV-4	40/C	14.9	*	*

* - Data do not permit calculation of half-life.
** - See Table 4-2

Table 5-3

Average Concentrations and Standard Deviations of Explosives Data From UMDA Composting Pilot Studies

Reactor		Average			Standard Deviation		
		HMX mg/kg	RDX mg/kg	2,4,6-TNT mg/kg	HMX Std Dev	RDX Std Dev	2,4,6-TNT Std Dev
Static 1	Day 0	120	776	1,144	5.9	79	106
7% Soil	Day 10	124	647	270	22	150	63
	Day 20	70	428	271	16	85	84
	Day 44	84	324	173	27	131	157
	Day 90	73	213	107	14	125	90
Static 2	Day 0	180	1,008	4,984	11	58	551
10% Soil	Day 10	125	973	1,114	12	205	249
	Day 20	127	723	719	15	85	378
	Day 44	181	939	241	27	167	147
	Day 90	142	542	200	20	186	115

Table 5-3
(continued)

Reactor		Average HMX mg/kg	Average RDX mg/kg	Average 2,4,6-TNT mg/kg	Standard Deviation HMX Std Dev	Standard Deviation RDX Std Dev	Standard Deviation 2,4,6-TNT Std Dev
Static 3	Day 0	194	1,076	5,716	9.6	38	491
20% Soil	Day 10	175	1,178	3,322	33	198	372
	Day 20	174	961	2,370	23	125	534
	Day 44	195	964	517	27	199	58
	Day 90	184	902	331	33	222	238
Static 4	Day 0	243	1,234	11,320	16	64	808
microbes	Day 2	255	1,258	11,580	30	130	909
added	Day 4	250	1,222	11,700	17	97	387
80% Soil	Day 8	247	1,236	11,880	12	69	327
	Day 16	274	1,246	11,740	35	165	1,494
	Day 20	265	1,184	10,900	18	68	791
	Day 90	238	1,180	10,640	32	199	978

Table 5-3
(continued)

Reactor		Average			Standard Deviation		
		HMX mg/kg	RDX mg/kg	2,4,6-TNT mg/kg	HMX Std Dev	RDX Std Dev	2,4,6-TNT Std Dev
Static 5	Day 0	236	1,178	7,908	14	53	336
30% Soil	Day 10	216	1,278	5,058	41	192	901
	Day 20	175	1,003	3,242	45	183	695
	Day 44	230	741	526	19	209	337
	Day 90	210	924	174	33	122	117
Static 6	Day 0	310	1,572	9,858	29	136	1,014
40% Soil	Day 10	372	1,974	9,440	24	119	596
	Day 20	290	1,556	5,956	57	258	1,313
	Day 44	310	1,192	1,736	29	508	416
	Day 90	305	1,674	2,086	30	174	442

Table 5-3
(continued)

Reactor		Average			Standard Deviation		
		HMX mg/kg	RDX mg/kg	2,4,6-TNT mg/kg	HMX Std Dev	RDX Std Dev	2,4,6-TNT Std Dev
Static 7	Day 0	2.54	1.96	3.84	0.00	0.00	0.00
10% UC	Day 10	4.96	16.5	17.7	4.96	28.8	28.1
	Day 20	2.54	1.96	3.84	0.00	0.00	0.00
	Day 44	2.54	2.00	3.84	0.00	0.09	0.00
	Day 90	2.54	1.96	5.82	0.00	0.00	0.42
Static 8*	Day 0	307	618	3,850	67	100	650
10% Soil	Day 10	203	386	1,078	52	96	536
	Day 20	92	112	117	50	54	104
	Day 44	55	43	39	26	32	30
	Day 90	61	46	41	26	15	31

* Analyses and data reduction done by Oak Ridge National Laboratory.

Table 5-3
(continued)

Reactor		Average HMX mg/kg	Average RDX mg/kg	Average 2,4,6-TNT mg/kg	Standard Deviation HMX Std Dev	Standard Deviation RDX Std Dev	Standard Deviation 2,4,6-TNT Std Dev
MAIV-1	Day 0	169	1,011	3,452	11	61	255
10% Soil	Day 10	140	1,009	165	13	96	73
	Day 20	71	74	63	15	33	70
	Day 44	120	104	90	22	18	119
MAIV-2	Day 0	119	575	3,126	5	37	257
10% Soil	Day 10	34	33	61	8	13	18
	Day 20	6.5	6.3	16	1	4	9.1
	Day 44	6.1	3.8	5.6	2	2.6	3.0
MAIV-3	Day 0	161	597	5,208	47.6	190	1872
25% Soil	Day 10	133	464	1,145	4.9	30	424
	Day 20	81	89	27	8.6	29	35
	Day 44	51	18	14	13.1	11	11

Table 5-3
(continued)

Reactor		Average			Standard Deviation		
		HMX mg/kg	RDX mg/kg	2,4,6-TNT mg/kg	HMX Std Dev	RDX Std Dev	2,4,6-TNT Std Dev
MAIV-4*	Day 0	456	754	6,950	20	44	190
40% Soil	Day 10	522	843	5,100	48	58	760
	Day 20	627	840	1,785	37	148	536
	Day 44	601	621	209	79	114	188

* Analyses and data reduction done by Oak Ridge National Laboratory.

Note: All explosives concentrations in Table 5-3 are expressed on a dry weight basis.

FIGURE 5-1 SUMMARY OF TNT CONCENTRATION OVER TIME IN INITIAL (9/21/90 START DATE) UMDA STATIC PILE TESTS

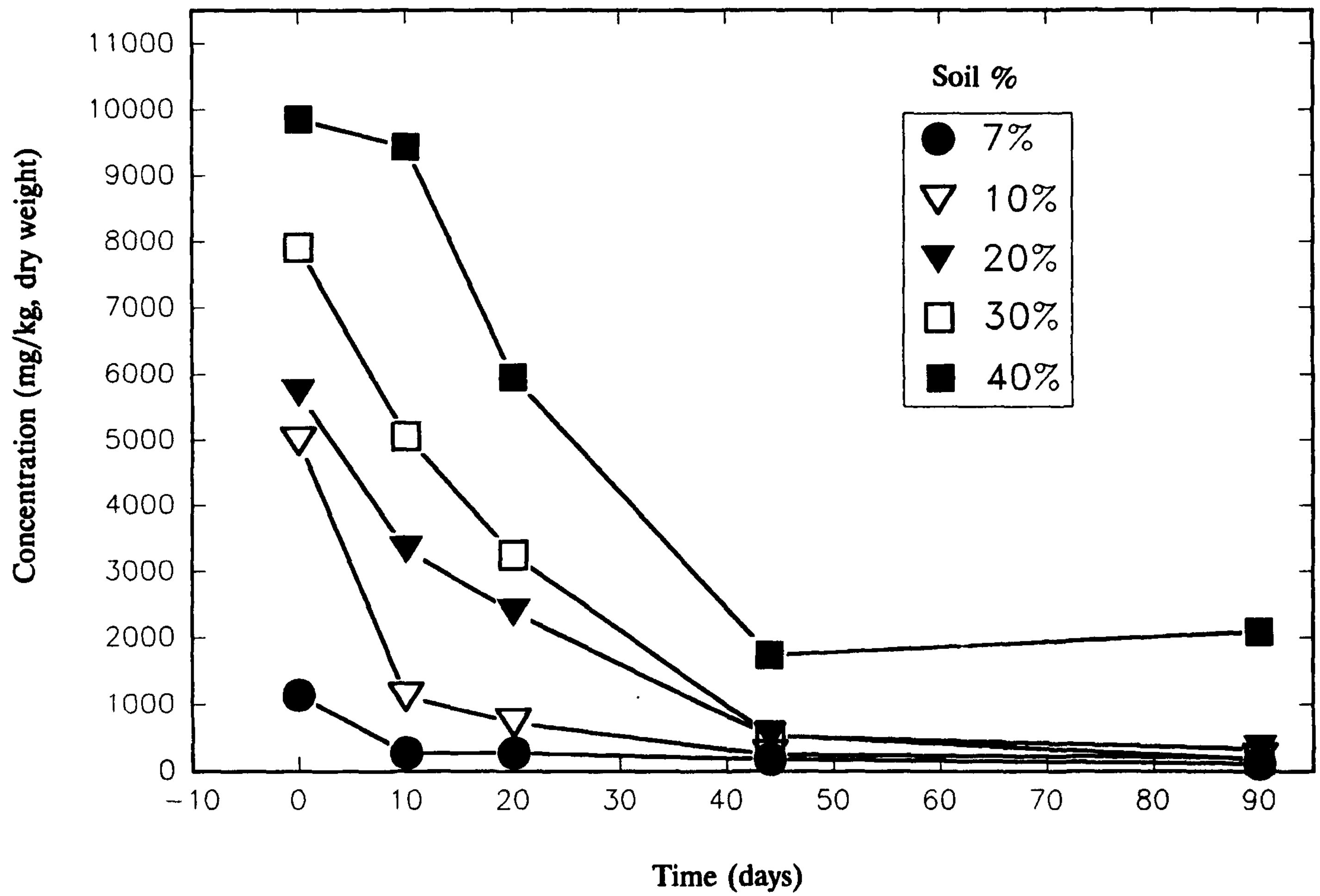


FIGURE 5-2 SUMMARY OF RDX CONCENTRATION OVER TIME IN INITIAL (9/21/90 START DATE) UMDA STATIC PILE TESTS

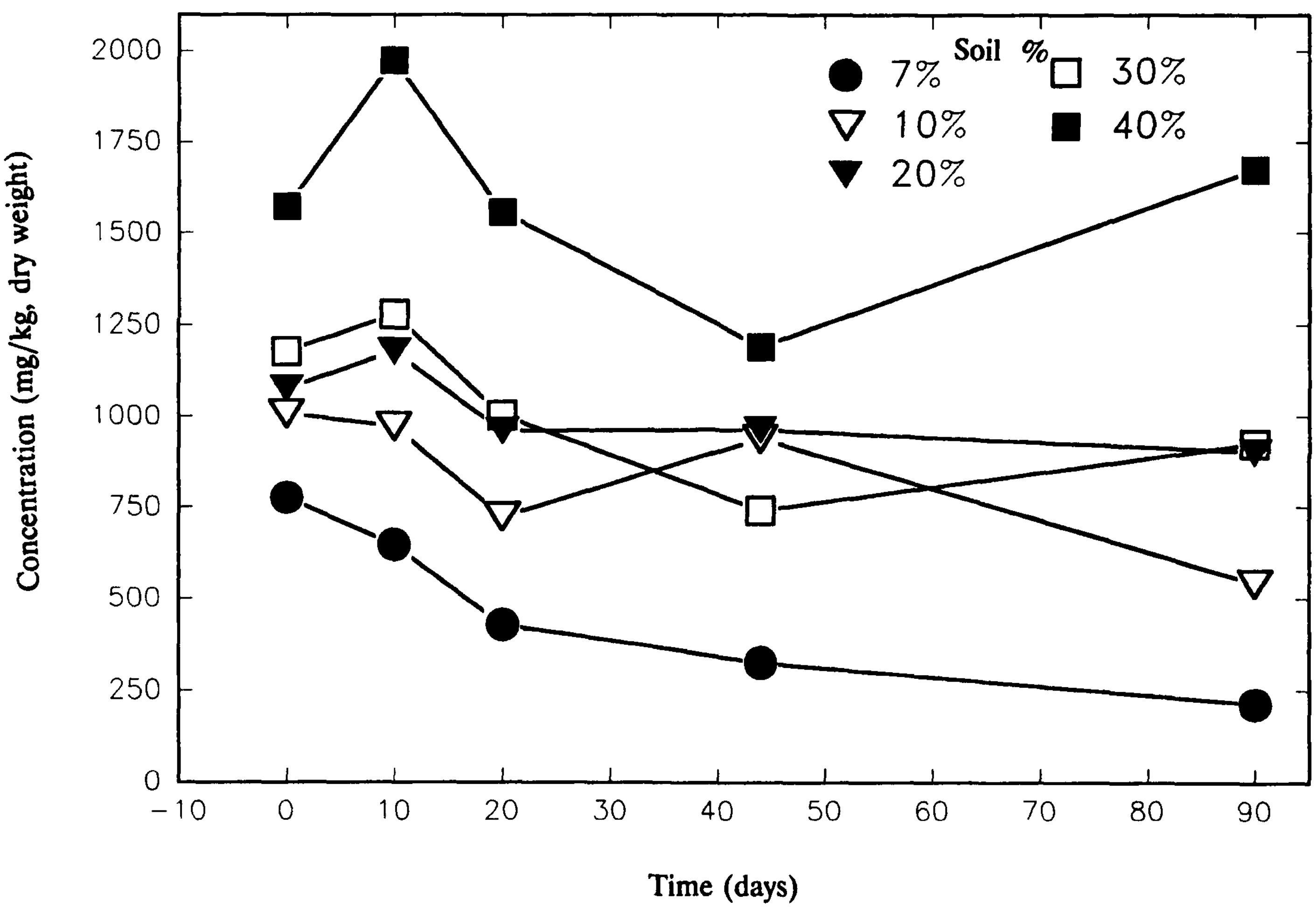


FIGURE 5-3 SUMMARY OF HMX CONCENTRATION OVER TIME IN INITIAL (9/21/90 START DATE) UMDA STATIC PILE TESTS (SP-1, SP-2, SP-3, SP-5, SP-6)

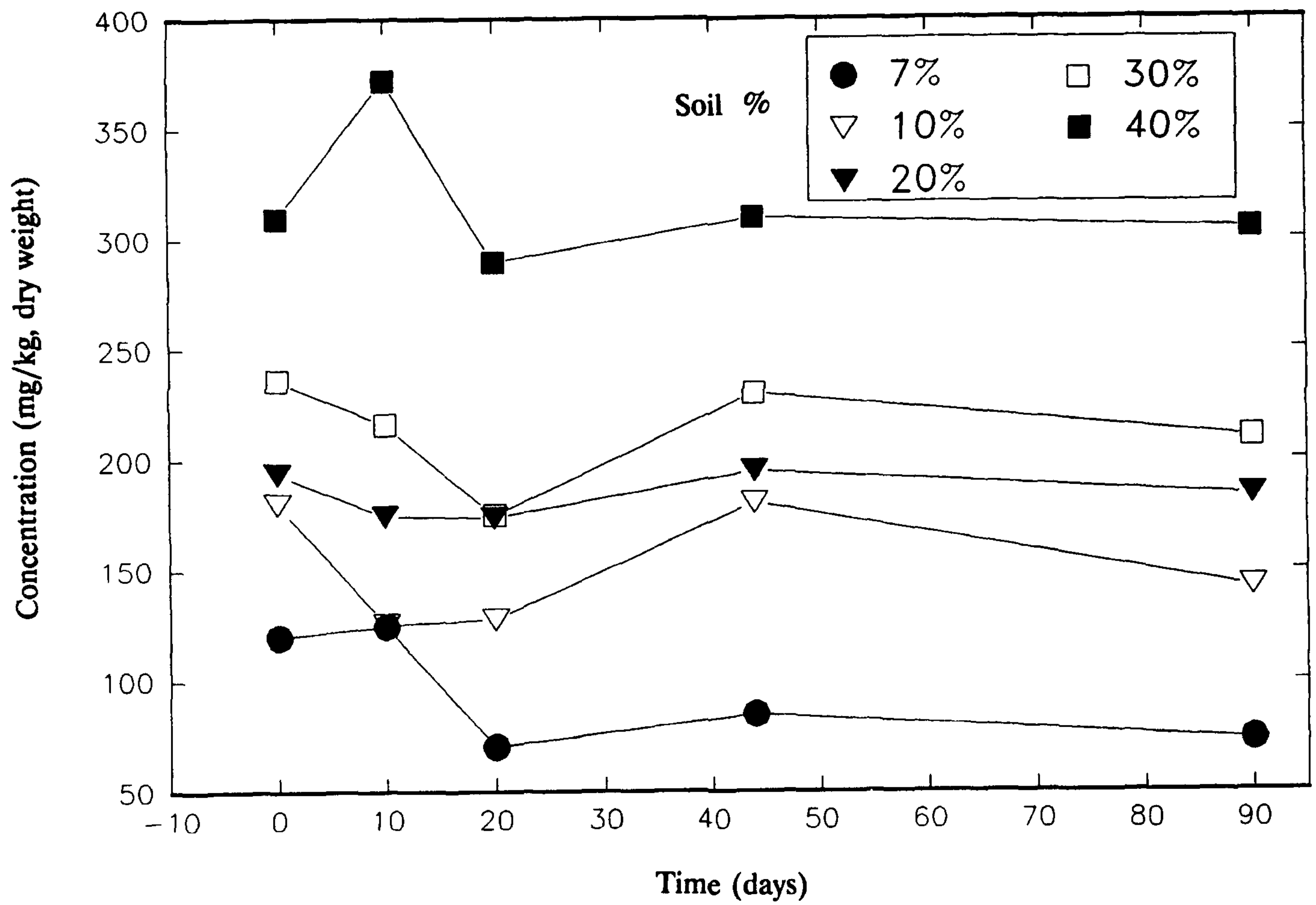


FIGURE 5-4 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 7% CONTAMINATED SOIL (SP-1)

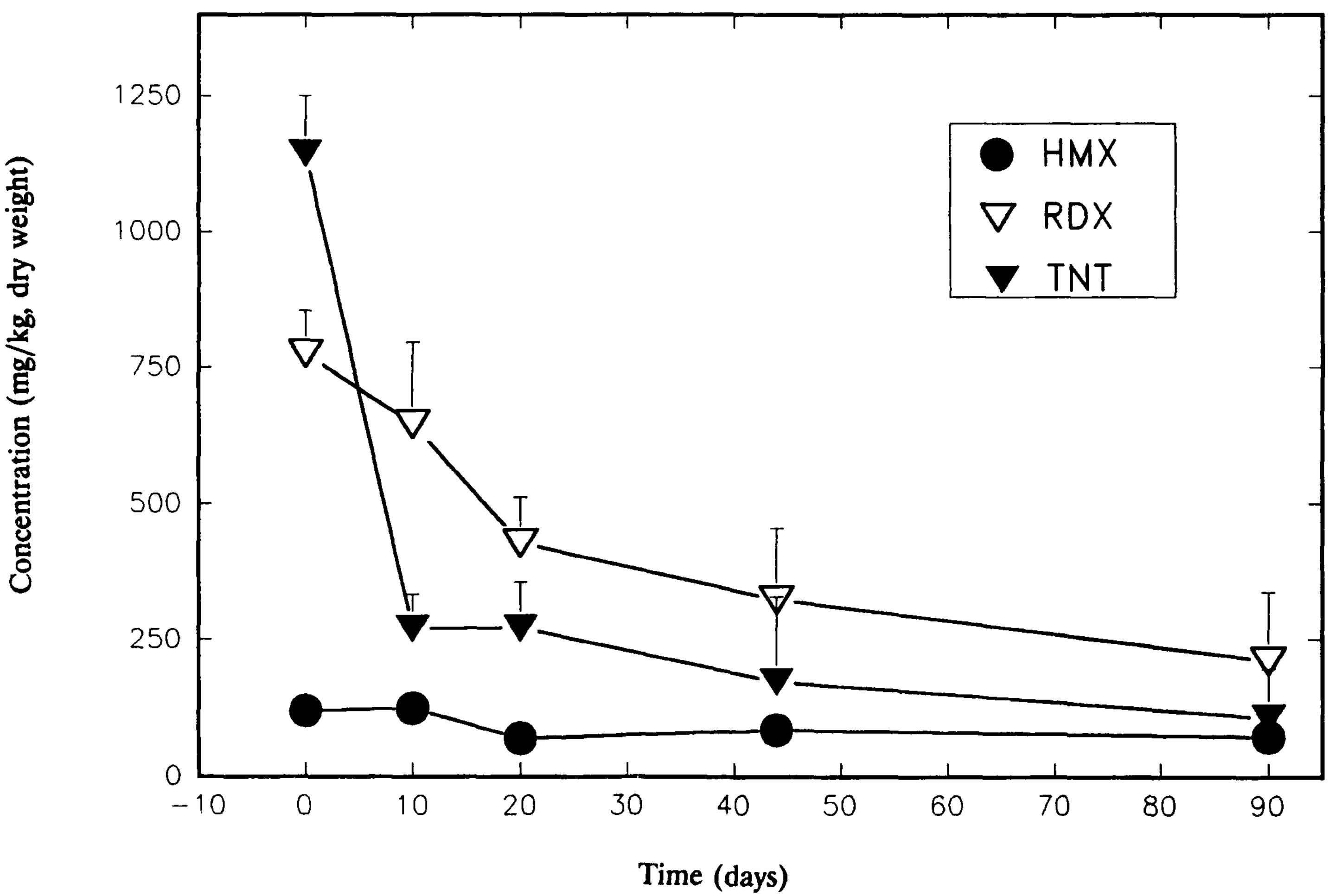


FIGURE 5-5 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 10% CONTAMINATED SOIL (SP-2)

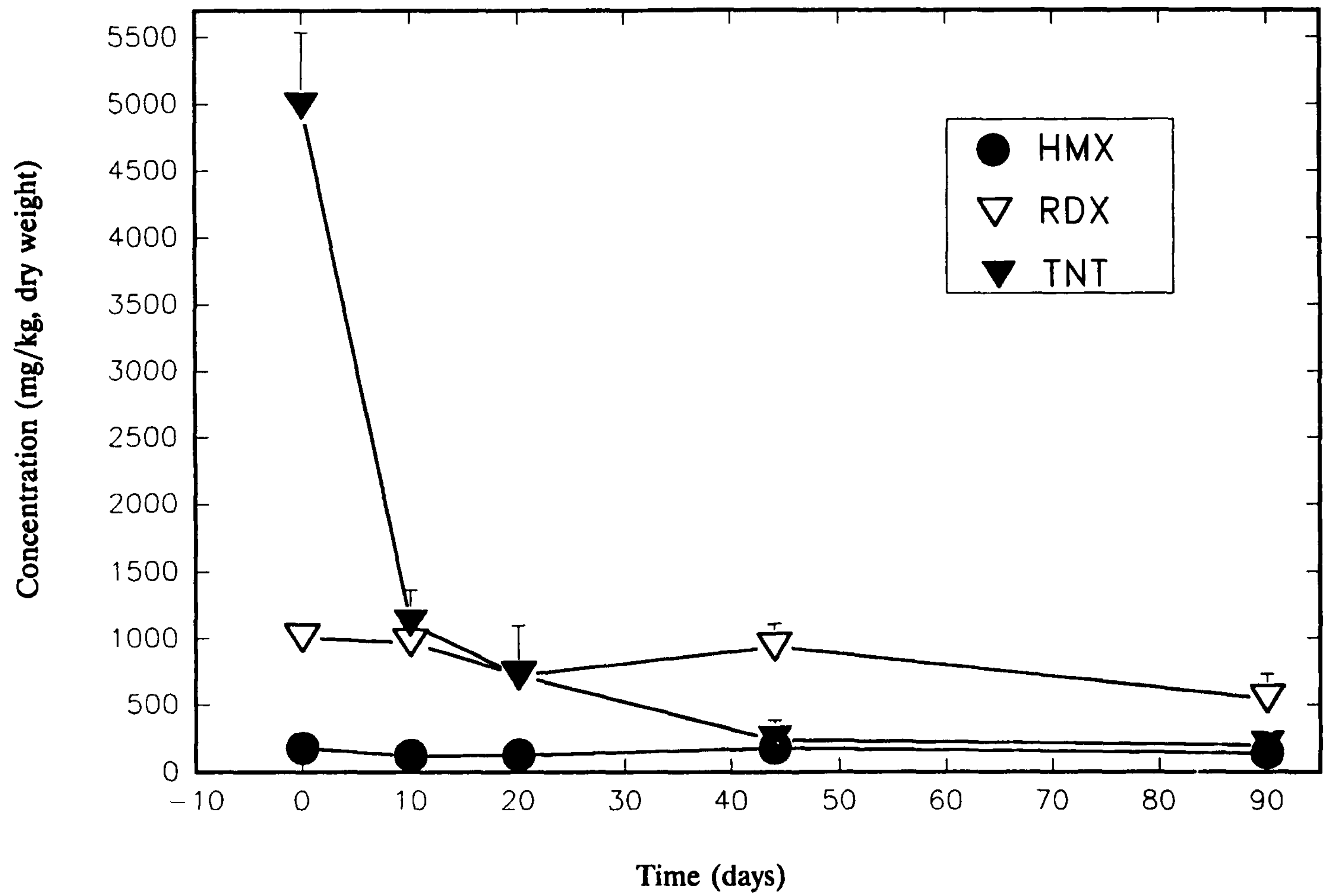


FIGURE 5-6 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 20% CONTAMINATED SOIL (SP-3)

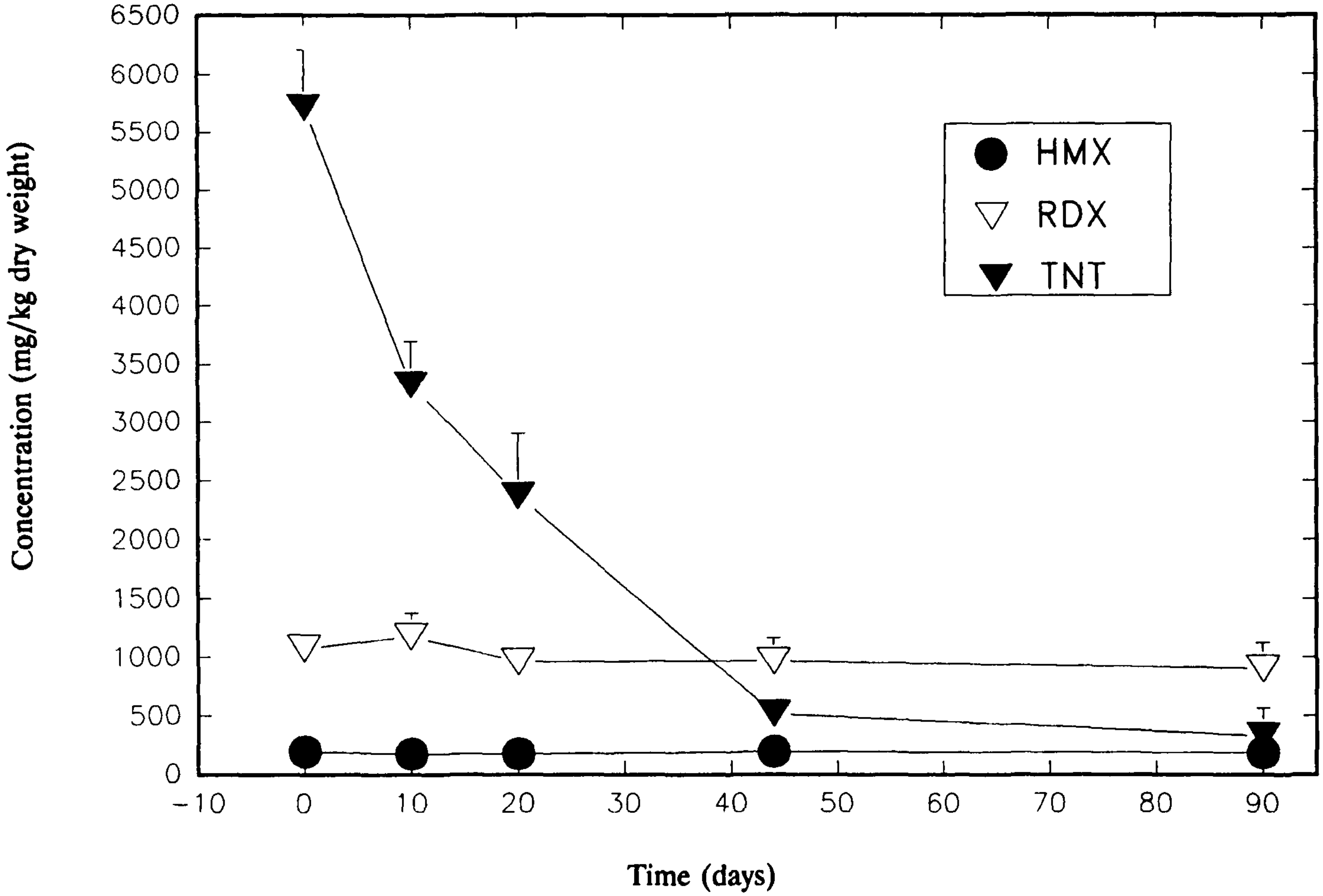


FIGURE 5-7 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 30% CONTAMINATED SOIL (SP-5)

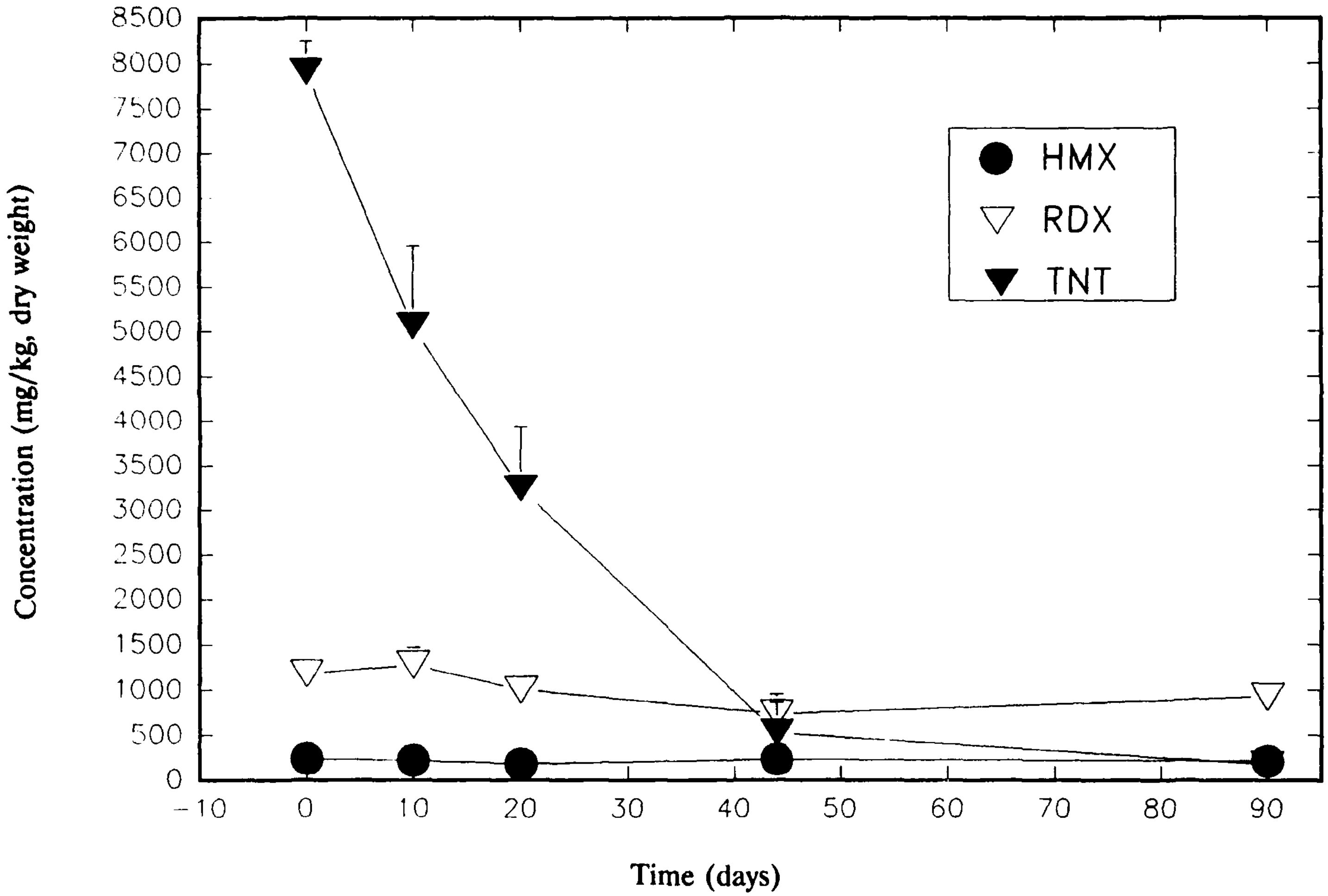


FIGURE 5-8 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 40% CONTAMINATED SOIL (SP-6)

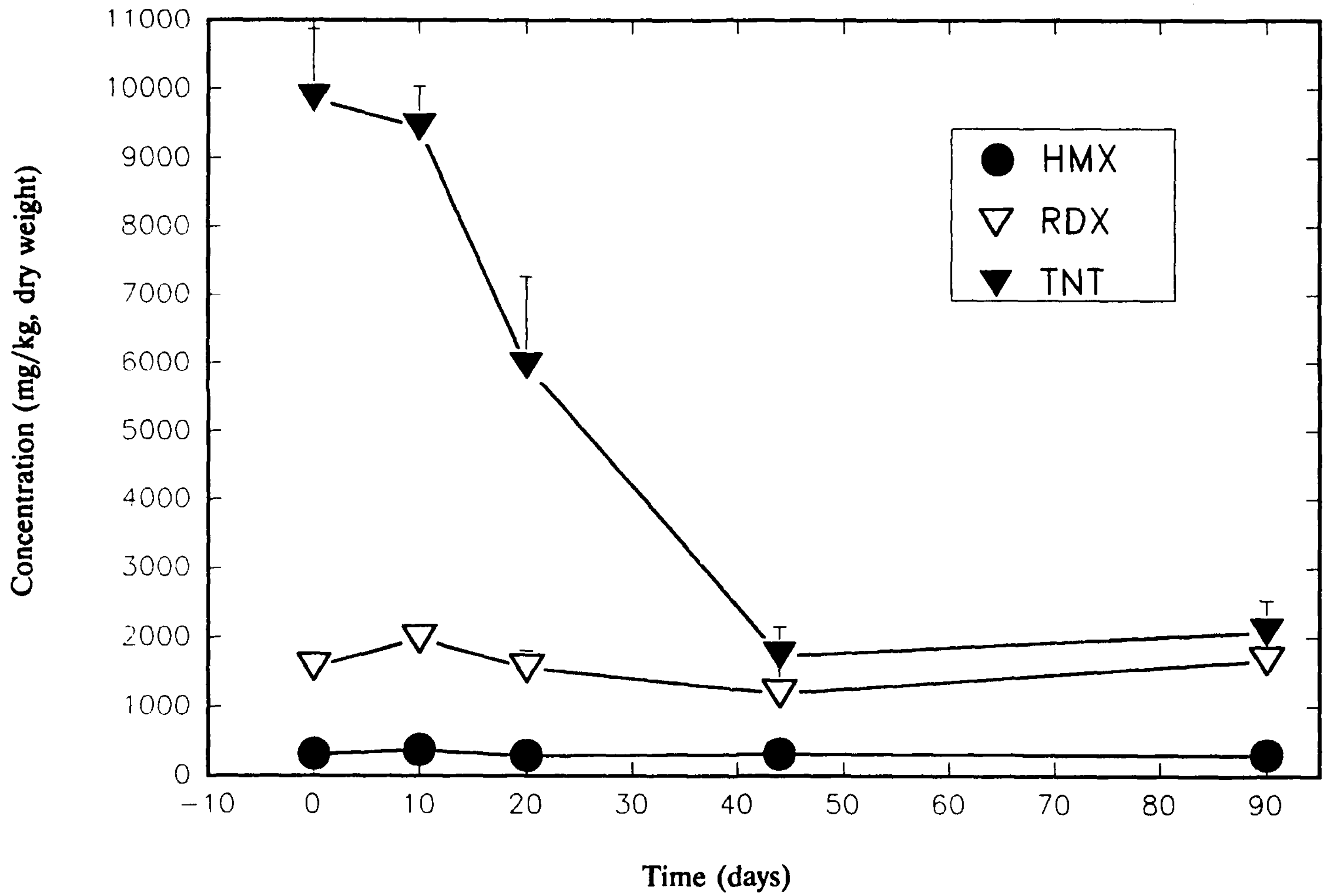


FIGURE 5-9 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN STATIC PILE REACTOR WITH 10% CONTAMINATED SOIL (SP-8)

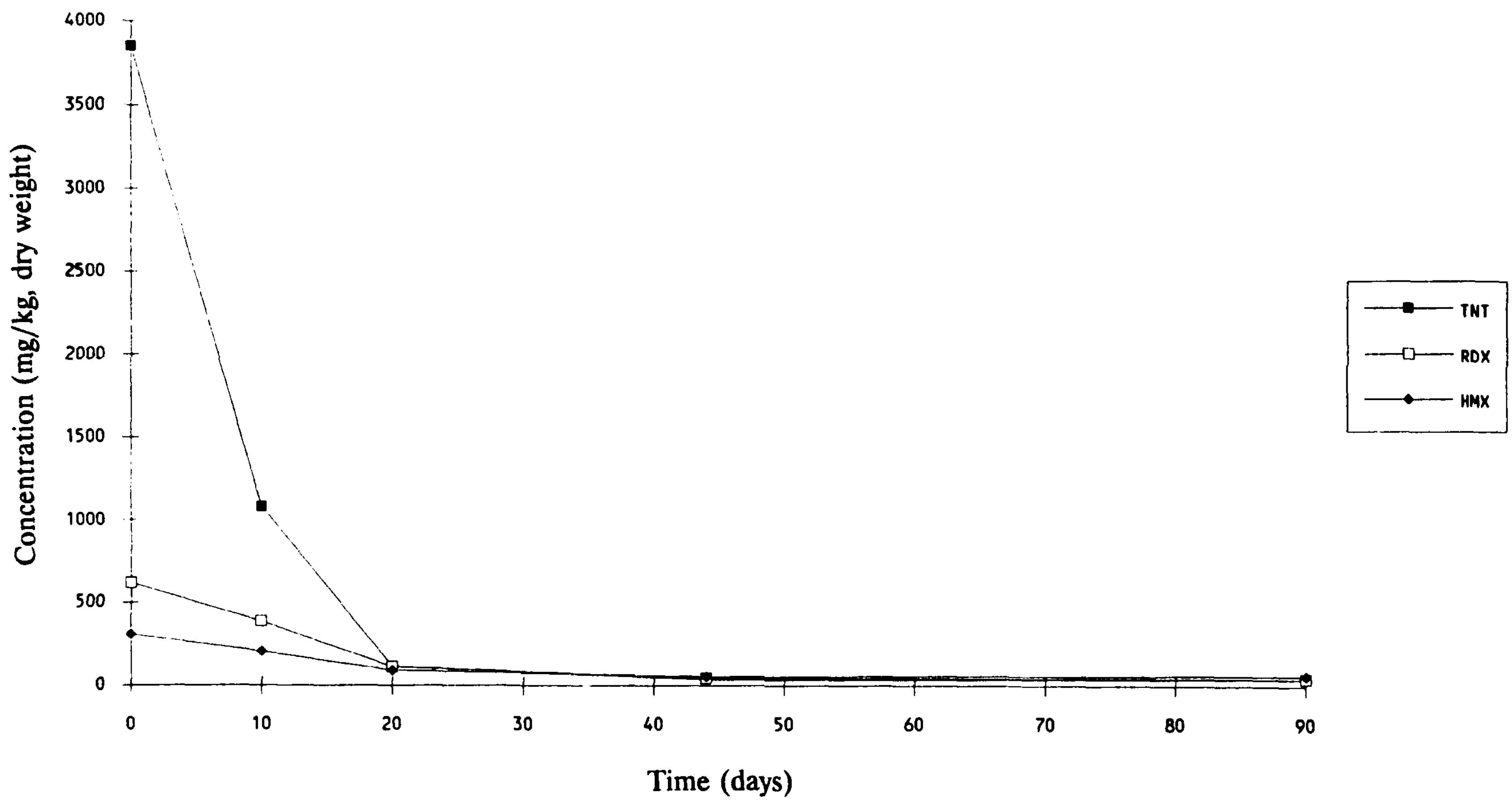


FIGURE 5-10 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN MAIV TEST 1 (10% SOIL)

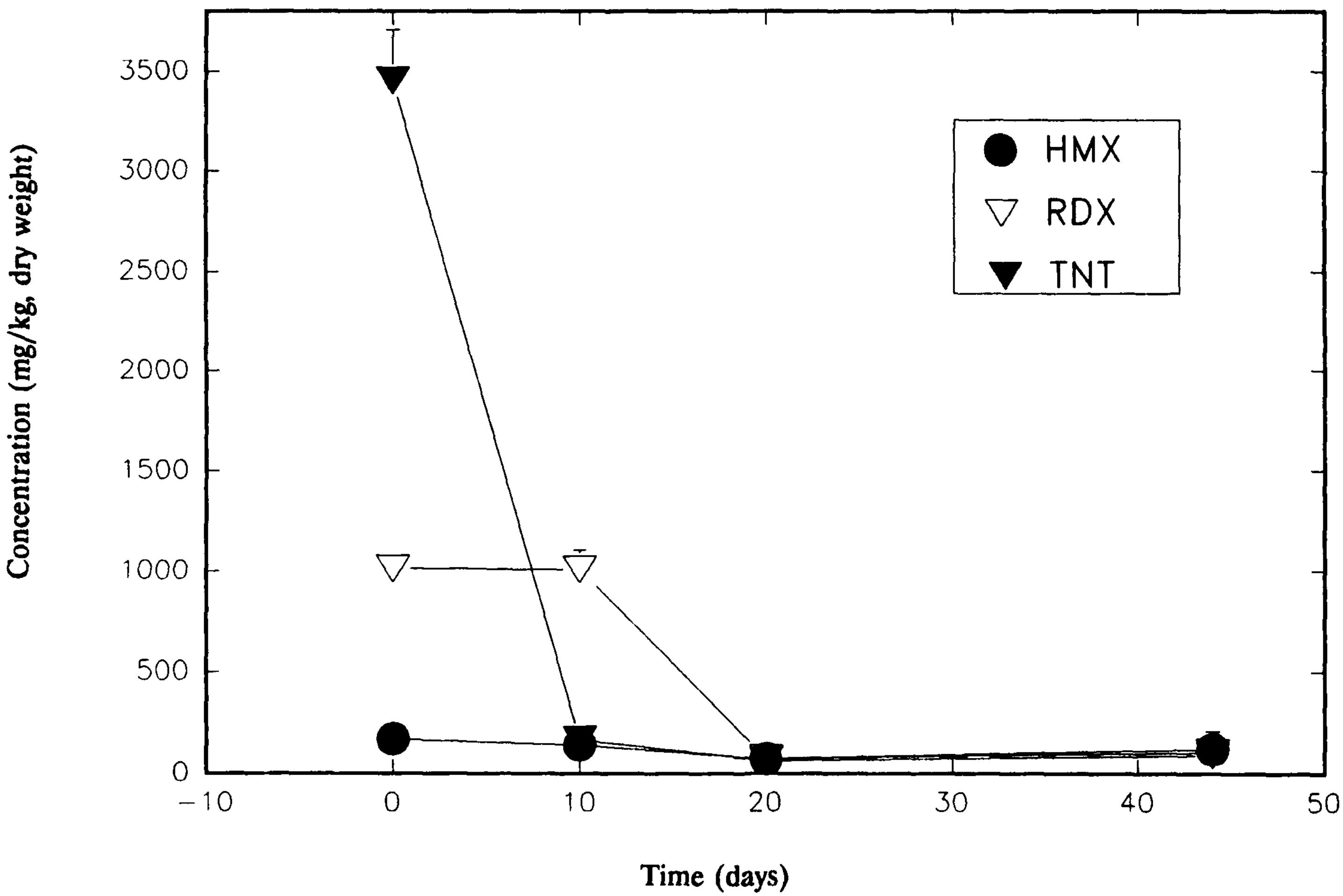


FIGURE 5-11 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN MAIV TEST 2 (10% SOIL)

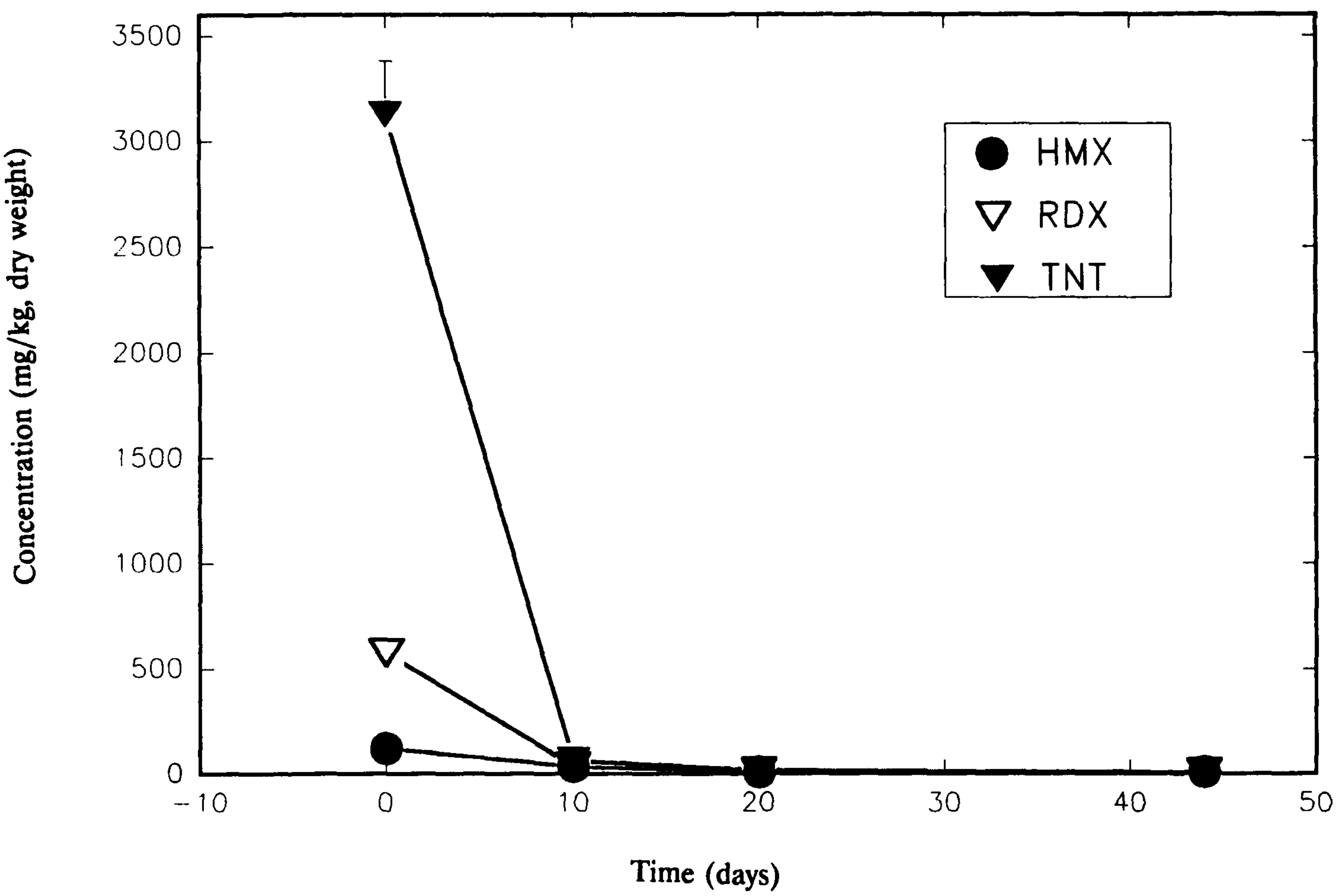


FIGURE 5-12 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN MAIV TEST 3 (25% SOIL)

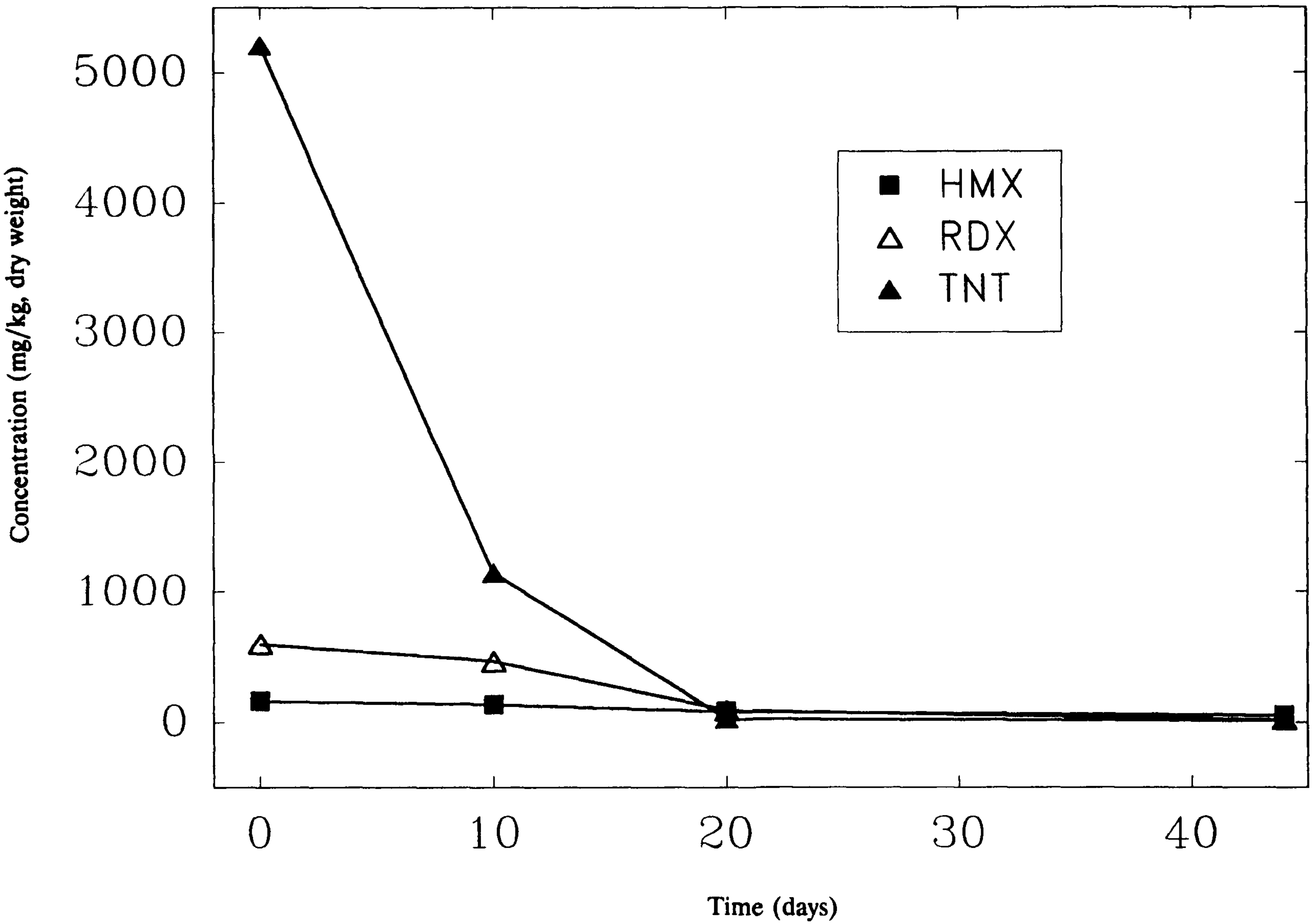


FIGURE 5-13 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN MAIV TEST 4 (40% SOIL)

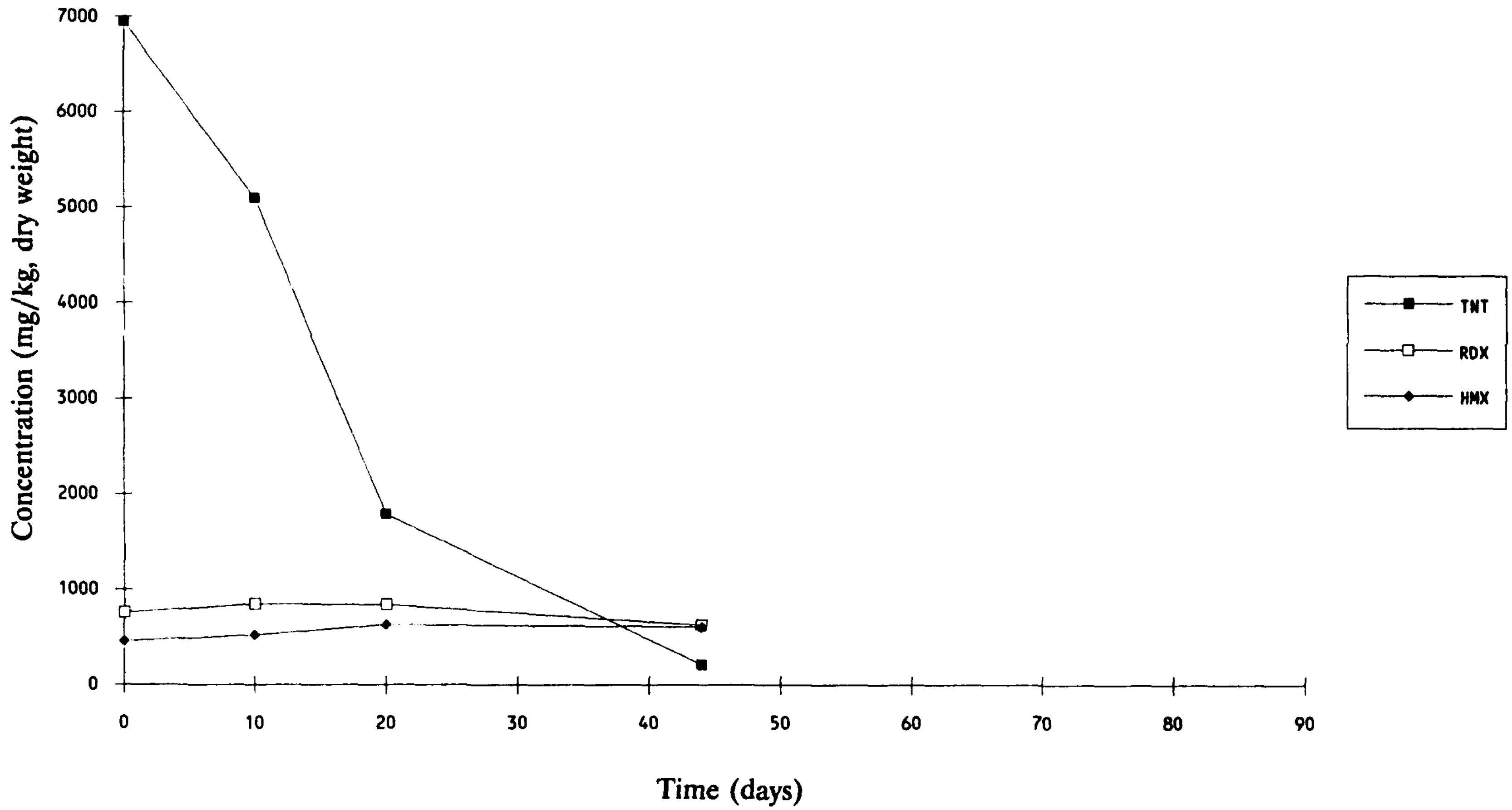
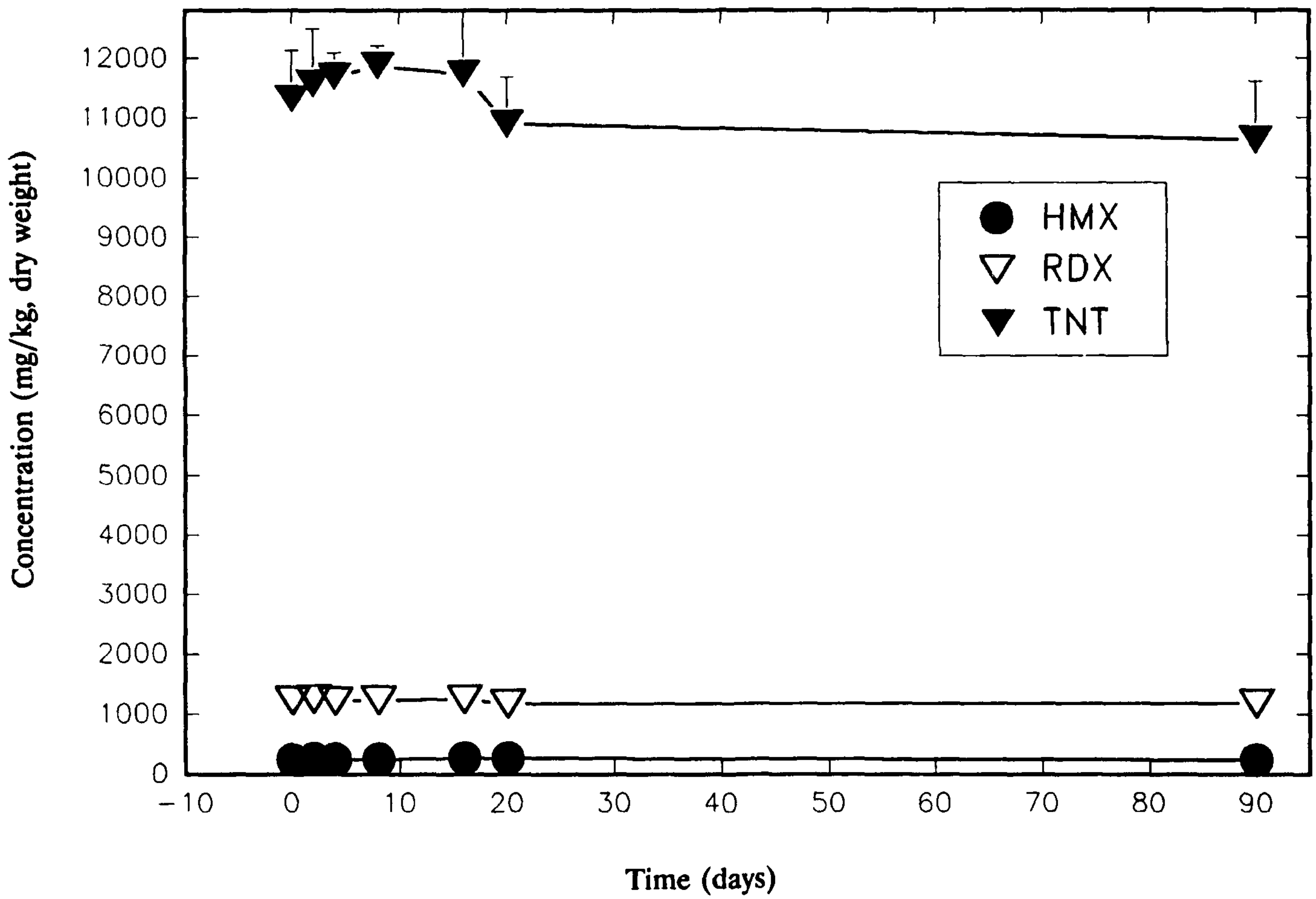


FIGURE 5-14 CONCENTRATION OF TNT, HMX, AND RDX OVER TIME IN BIOAUGMENTED TEST (SP-4)



5.2 TEMPERATURE

5.2.1 Ambient Temperature

Ambient temperature is reported in Figure 5-15 for the entire 241 days during which testing occurred, and for each individual test period in Figures 5-16 to 5-22. Daily high and low temperatures are reported for each test period.

5.2.2 Static Piles

Temperature profiles for the static pile pilot tests are shown in Figures 5-23 through 5-30.

5.2.3 Mechanically Agitated System

Electronic temperature data from the Fairfield mechanical composter for the first two MAIV tests are shown in Figure 5-31 and 5-32. Since only one probe was present in the pile, and this probe needed to be removed during agitation, the temperature record was not as extensive as desired. In addition, difficulties were encountered with the electronic apparatus used to collect these data. Manual temperature readings were taken frequently from several locations within the pile. These data are presented in Figures 5-33 to 5-36.

5.3 MOISTURE

Moisture is a key composting parameter. Percent moisture and water holding capacity were determined, and the percent of water holding capacity calculated. The percent of water holding capacity is the key factor, since water holding capacity (and therefore the percent moisture required to saturate the mixture) varies significantly depending on the ratio of inorganic to organic matter.

For each static pile test, samples were taken for moisture analysis from the top, middle, and bottom of the pile for each sampling day. Three replicate samples were taken from the MAIV tests on each sampling day. The results of these determinations are presented in

Tables 5-4 to 5-11 for the static pile tests and Tables 5-12 to 5-15 for the MAIV tests. The three samples from the MAIV tests were almost identical for each of the three parameters determined (percent moisture, water holding capacity, percent of water holding capacity). No consistent differences were observed between the top, middle, and bottom data for any of the three parameters in any of the static pile tests.

The relationship between percent moisture, water holding capacity, and percent of water holding capacity is significant. These values are plotted for the average of the top, middle, and bottom samples for the static piles in Figures 5-37 to 5-44. The average percent moisture, water holding capacity, and percent of water holding capacity for the three samples taken at each time point for the MAIV tests are plotted in Figures 5-45 to 5-48.

5.4 OXYGEN

Oxygen data from the manual probe and electronic analyzer analyses consistently showed oxygen to be above 17%. In one case an electronic failure resulted in a blower being off for a period of approximately 4 hours in SP-2. Oxygen readings (by manual probe) fell to approximately 3% during this time. Within one-half hour of the blower being reactivated, all oxygen readings were once again above 17%.

5.5 pH

pH data for all static pile tests are listed in Tables 5-7 (SP-4), 5-11 (SP-8), and 5-16 (SP 1,2,3,5,6, and 7, and for the MAIV tests in Tables 5-16 (MAIV-1), 5-13 (MAIV-2), 5-14 (MAIV-3), and 5-15 (MAIV-4). Data for the top, middle, and bottom for each static pile test are plotted in Figures 5-49 to 5-56. The average pH values for the three samples collected in the MAIV tests at each time point are plotted in Figures 5-57 to 5-60.

5.6 AIR MONITORING

No TNT, RDX, or HMX were detected in any of the four exhaust gas samples analyzed from SP-8.

5.7 MICROTOX

The Microtox toxicity assay uses living bioluminescent marine bacteria as the test organisms. These bacteria (*Photobacterium phosphoreum*) are grown under optimal conditions, harvested, and then freeze-dried for purchase as test organisms. The freeze-dried bacteria are rehydrated with a reconstitution solution at the time of testing.

The Microtox instrument measures the light output of the bacteria before and after they are exposed to a test chemical. The degree of light loss is indicative of the metabolic inhibition of the test organism. This inhibition has been shown to be related to the toxicity of the test chemical.

Exposure time of the bacteria to the test chemical is an important bioassay parameter. Chemicals vary in their dose-response. Some chemicals may produce effects within a 5 minute exposure period and others, such as bivalent metals, may require 15 minutes or more to complete their effect. The Microtox bioassay determines the dose-response of a test chemical to the test organism. An effective concentration (EC) can then be calculated. An EC_{50} is the concentration of the test chemical which causes a 50% reduction is light output as compared with the control.

The basic Microtox bioassay procedure includes four serial dilutions of the test chemical and a reagent blank. The reagent blank is used to normalize the responses of the four serial dilutions during data reduction.

The objective of this study was to evaluate the utility of the Microtox bioassay in monitoring toxicity reduction within compost containing contaminated soil. The results are shown in Tables 5-17 and 5-18.

Toxicity gradually decreased through time for the composts containing contaminated soil in both the 5-minute and 15-minute bioassays. The control treatment results (SP-7) indicated toxicity on day 0, but the toxicity significantly decreased by day 10 of the study. Toxicity values for contaminated soil mixtures were always higher than for the uncontaminated soil mixture.

5.8 MICROBIAL ENUMERATION

Analysis of microbial numbers was limited by financial constraints. Day 0, 20, and 90 samples were analyzed for tests SP-2, SP-3, and SP-7. Day 0 and 20 samples were analyzed for tests MAIV-1 and MAIV-2. The number of colony forming units (CFUs) did not vary significantly from one sample date/reactor to another. CFU values were in the range of 10^4 to 10^5 for all samples.

Table 5-4

Moisture Data for Static Pile Test SP-1, 7% Soil

				ST - 1 DATA								
				START DATE 9/21/90 7% soil								
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
10	43.04		62.73	50.60	33.02	45.50				64.38	60.80	63.00
13	40.74	64.75	64.13	43.79	38.43	40.00	67.54	63.24	63.48	61.60	64.80	66.00
16	42.83	66.80		41.50	43.20	43.80	67.50	66.60	66.30			
17			55.10							52.10	57.50	55.70
20	34.80	63.03	60.57	32.60	37.70	34.10	62.50	65.50	61.10	60.10	60.20	61.40
23	38.45	63.43		37.40	38.55	39.40	62.30	63.90	64.10			
24			51.87							47.50	53.10	55.00
27	31.87	60.20	52.47	27.40	33.90	34.30	57.60	63.90	59.10	43.80	57.20	56.40
30	32.70	61.80		26.40	37.40	34.30	60.10	65.30	60.00			
34			42.00							44.10	37.00	44.90
37	25.33	60.17		27.50	21.30	27.20	62.10	57.80	60.60			
38			50.27							54.70	48.50	47.60
41	32.00	63.47		36.30	31.00	28.70	66.20	63.80	60.40			
66			64.63							66.30	61.60	66.00
69	40.23	62.27	64.10	41.20	38.00	41.50	62.10	61.70	63.00	68.70	62.90	60.70
72	39.53	61.67		43.00	38.40	37.20	62.70	61.00	61.30			

WESTON

Table 5-5

Moisture Data for Static Pile Test SP-2, 10% Soil

				ST - 2 DATA								
				START DATE 9/21/90		10% SOIL						
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			67.45							76.76	60.70	64.90
10		60.24	62.63				63.70	60.47	56.56	64.30	63.10	60.50
11	39.73			48.90	36.70	33.60						
13	40.12	64.00	64.53	41.80	40.36	38.20	65.00	64.00	63.00	65.10	62.40	66.10
16	41.00	62.40		44.20	37.10	41.70	64.60	59.40	63.20			
17			56.83							56.20	58.20	56.10
20	34.57	60.37	61.03	34.80	34.60	34.30	60.50	59.40	61.20	57.40	59.10	66.60
23	36.97	60.43		34.50	34.90	41.50	60.00	59.10	62.20			
25			52.00							42.70	59.30	54.00
27			55.27							53.50	58.50	53.80
28	30.03	57.67		24.20	34.30	31.60	56.70	57.80	58.50			
30	32.73	59.17		31.40	34.70	32.10	58.60	59.30	59.60			
34			33.87							14.40	42.00	45.20
37	19.57	57.57		8.20	24.30	26.20	57.20	57.50	58.00			
38			44.90							43.30	42.20	49.20
41		56.27					57.50	54.70	56.60			
69	25.50		51.03	25.50	23.10	27.90				65.10	44.20	43.80
72	27.87	54.30		37.10	23.20	25.30	57.10	52.60	53.20			

WESTON

Table 5-6

Moisture Data for Static Pile Test SP-3, 20% Soil

				ST - 3 DATA								
				START DATE 9/21/90 20% SOIL								
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			60.92							59.85	62.80	60.10
10		52.91	62.57				51.82	53.96	52.95	61.00	62.80	63.90
11	32.23			31.00	33.90	31.80						
13	35.44	56.60	57.63	34.02	35.60	36.70	55.70	56.70	57.40	54.40	58.80	59.70
16	29.73	52.37		26.05	31.07	32.06	48.80	53.80	54.50			
17			56.60							57.20	54.80	57.80
20	29.40	54.40	56.50	31.50	27.50	29.20	55.10	50.30	57.80	55.70	56.50	57.30
23	29.63	52.27		29.60	29.40	29.90	53.10	52.10	51.60			
25			58.27							58.10	59.50	57.20
27		51.03	56.17				50.60	51.50	51.00	49.00	61.40	58.10
28	29.77			29.40	30.70	29.20						
30	29.87	49.77		24.10	35.10	30.40	40.10	56.90	52.30			
34			43.77							21.70	51.90	57.70
37	21.63	48.47		9.60	26.60	28.70	44.10	51.30	50.00			
38			56.70							55.80	56.50	57.80
41	29.13	51.33		28.30	28.30	30.80	50.70	50.10	53.20			
66			59.46							66.40	56.97	55.00
69	30.23	50.67	54.10	34.10	28.90	27.70	50.90	50.70	50.40	56.50	50.20	55.60
72	26.37	48.77		27.20	24.90	27.00	48.20	49.50	48.60			

Table 5-7

Moisture and pH Data for Bioaugmented Static Pile Test SP-4, 80% Soil

	ST-4 DATA DAY 0 = 11/2/90											
	CONTAMINATED SOIL 59.7% SAWDUST 29.8%											
	SODIUM ACETATE 3..7% L-ARGININE 1.5% AMMONIUM SULFIDE 5.2%											
DAY	PER CENT WATER				WHC			%WHC			pH	
	T	M	B	T	M	B	T	M	B	T	M	B
4	9.4	9.8	9.4	24.4	23.7	23.4	38.3	41.9	40.2	7.6	7.6	7.6
8	7.9	7.6	8.2	22.9	22	24.7	34.6	34.5	33.1	7.1	7.2	7.1
12	10	9.7	9.7	24.8	24.9	24.3	40.4	39.1	40.2			
16	9.7	9.6	9.7	23.4	23.5	23.8	41.5	41	40.8	7.6	7.7	7.7
18	9.8	9.4	9.5	24.6	22.5	23.2	39.9	41.8	40.9	7.6	7.5	7.3
24		10.1	10.6		23.5	23.7		43.2	44.8	7.6	7.5	7.4
27	9.5	9.3	9.9	23.6	22.6	23.2	40.1	41.3	42.7	7.7	7.6	7.3
31	11	10	12.1	22.9	22.6	22.9	48.1	44.1	52.8	7.6	7.4	7.5
35	10.1	10.7	12.5	22.7	23.5	22.6	51.8	45.5	55.44			
40	10.6	10.8	11.3	22.7	22.3	21.7	46.5	48.2	51.9	7.5	7.2	7
45	10.5	10.9	14.2	21.9	22.7	23.6	48.2	48.2	60.2	7.6	7.3	7.2
48	9.4	10.4	10.6	22	21.9	20.1	42.8	47.5	53.2	7.6	7.4	7.4
76	9.7	9.9	11.3	22.5	22.6	22.9	43.2	44	49	7.2	7.3	7.4
77	15	16	14.7	21.3	21.7	22.4	70.5	73.2	65.4	7.5	7.5	7.5
81	13.6	13.4	15.6	22.2	23.1	22.2	61.5	58.2	70.3	7.3	7.4	7.4
90	10.8	11.5	12.6	22.7	22.7	22.3	47.4	50.4	56.7	7.2	7.2	7.2

WESTON MANAGERS DESIGNERS/CONSULTANTS

Table 5-8

Moisture Data for Static Pile Test SP-5, 30% Soil

				ST - 5 DATA								
				START DATE 9/21/90 30% SOIL								
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			54.04							58.06	54.77	49.30
10	26.07	48.21	59.27	27.10	27.70	23.40	46.67	50.50	47.46	56.70	58.40	62.70
13	28.53	48.20	53.17	28.30	28.10	29.20	49.90	48.10	46.60	53.00	53.60	52.90
16	24.50	45.73		23.60	25.40	24.50	44.60	46.50	46.10			
17			56.10							54.80	56.70	56.80
20	25.13	44.87		24.30	25.10	26.00	44.40	44.30	45.90			
23		44.93					43.90	44.50	46.40			
25	26.07		55.80	24.30	25.10	28.80				55.50	55.40	56.50
28	24.47	44.17	57.57	23.60	23.90	25.90	43.50	43.10	45.90	58.70	55.40	58.60
30		44.80					44.70	45.70	44.00			
31	25.80			26.30	25.30	25.80						
34			53.23							47.40	53.80	58.50
38	23.83	44.73	56.23	20.30	24.30	26.90	42.90	45.20	46.10	55.90	58.10	54.70
41	26.43	46.70		25.90	29.20	24.20	46.20	49.70	44.20			
66			58.03							63.50	57.10	53.50
69	29.67	42.07	58.27	26.20	39.90	22.90	41.20	42.30	42.70	64.80	57.90	52.10
72	24.40	41.97		26.70	24.40	22.10	41.30	42.10	42.50			
90												

WESTON

Table 5-9

Moisture Data for Static Pile Test SP-6, 40% Soil

				ST - 6 DATA								
				START DATE 9/21/90		40% SOIL						
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			56.16							52.35	61.33	54.80
10	20.10	37.95	51.07	19.80	19.80	20.70	37.82	38.28	37.75	56.30	50.80	46.10
13	25.87	43.20	50.17	23.40	21.30	32.90	41.60	41.90	46.10	49.00	52.40	49.10
16	20.63	41.00		19.70	22.40	19.80	40.30	42.60	40.10			
17			51.80							53.30	59.30	42.80
20	21.30	40.60		21.60	26.40	15.90	40.40	44.10	37.30			
23		40.50					40.90	40.80	39.80			
24	21.03			20.30	22.30	20.50						
25			51.90							52.20	50.60	52.90
27		39.87					39.30	39.10	41.20			
28	20.47		47.73	19.80	19.80	21.80				49.70	40.90	52.60
30		40.13					38.40	40.90	41.10			
31	20.63			19.10	21.20	21.60						
35			50.67							48.90	48.00	55.10
38	50.67	40.40	52.00	48.90	48.00	55.10	40.90	39.40	40.90	51.20	51.80	53.00
41	21.33	41.07		20.70	21.50	21.80	40.50	41.50	41.20			
66			55.63							56.50	56.00	54.40
69	22.40	40.27	57.47	22.30	22.30	22.60	39.40	39.80	41.60	57.80	57.90	56.70
72	22.57	39.27		22.50	23.30	21.90	39.00	40.20	38.60			
90												

WESTON

Table 5-10

Moisture Data for Static Pile Test SP-7, 10% Uncontaminated Soil

				ST -7 DATA								
				START DATE 9/21/90 10% UC SOIL								
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			59.88							56.15	62.80	60.70
10	35.73		45.80	35.40	37.10	34.70				49.60	38.80	49.00
11		59.00					63.05	59.10	54.86			
13	27.27	59.63	47.57	30.10	23.70	28.00	60.70	61.10	57.10	41.40	48.60	52.70
16	27.20	56.93		22.90	27.40	31.30	55.20	56.50	59.10			
17			51.23							43.10	54.40	56.20
20	30.70	59.67		24.70	33.60	33.80	57.10	61.80	60.10			
23		58.10					58.90	58.00	57.40			
24	28.87		38.67	28.70	30.20	27.70				43.50	37.60	34.90
27	20.10	51.80	47.90	22.40	19.90	18.00	51.10	52.90	51.40	46.10	49.90	47.70
30	26.13	54.50		21.90	29.80	26.70	47.60	59.70	56.20			
34			45.90							44.30	47.20	46.20
37	25.97	56.57		24.50	27.20	26.20	55.20	57.70	56.80			
38			50.70							53.40	48.90	49.80
41	29.47	58.37		33.30	28.90	26.20	62.20	59.10	53.80			
66			60.13							64.00	65.80	50.60
69	35.97	59.40	59.17	37.20	32.00	38.70	58.20	58.70	61.30	56.20	60.20	61.10
72	33.43	56.60		31.40	34.20	34.70	55.90	57.10	56.80			
90												

Table 5-11

Moisture and pH Data for Static Pile Test SP-8, 10% Soil

	ST-8 DATA DAY 0 = 2/6/91											
	CONTAMINATED SOIL 10% SAW/ALFALFA 37%											
	APPLE POMACE 9% POTATO 16%											
DAY	PER CENT WATER				WHC			%WHC			pH	
	T	M	B	T	M	B	T	M	B	T	M	B
	48.5			66.2			73.3			6.2		
5	53.5	46.2	43.2	67.3	62.7	61.6	79.7	73.7	70.7	7.7	7	6
7	42.1	45.7	45.3	60.6	61.5	62.8	69.4	74.4	71.7	6.1	6.1	5.5
10	45.4	38.3	42.7	62.3	58.7	60.1	42.7	60.1	71.1	6.2	5.2	6.3
13	35.7	37.6	39.2	60.3	62.4	62.5	59.2	60.1	62.7	8.6	7.9	8.3
15	50.9	36.3	38	64.6	59.2	58.9	78.7	61.3	64.5	6.9	7.6	8
20	25.3	30.7	30.3	56.4	56	56.1	44.9	54.8	54.8	8.9	9	8.3
22	43	38.5	37.7	61.4	60.9	57.8	70	63.3	65.3	8.3	8	8.2
26	41.6	32.5	30.6	62.3	56	56.4	66.7	58	54.2	8.6	8.5	8.5
28	37.8	33.7	36.4	56.7	59.5	58.9	66.7	56.2	61.7	8	8.2	8.6
30	43.4	35.6	37.9	61.4	57	60.4	67.8	62.5	62.7	8.2	8.6	8.4
33	38.2	34.7	35	62.5	58.2	59.6	61	59.6	58.8	8.8	8.7	8.5
35	35.1	33.9	32.7	57	57.7	58.7	61.7	58.6	55.6	8.6	8.6	8.6
37	27.6	29.7	33.1	54.7	58.2	60.7	50.5	51.1	54.6	8.8	8.7	8.5
40	42.2	39.4	36.5	59.9	58.7	56	70.4	67	65.2	8.7	8.5	8.6
44	30.2	27.5	35.2	55.1	56.3	56.3	54.8	48.8	62.3	8.5	8.8	8.3
47	35.3	28.7	30.3	60	56	57.1	58.7	51.3	53.1	8.2	8.1	8.4
49	42.9	34.4	37.4	60.5	56	57.4	71	61.4	65.2	5.9	6.6	7.3
51	28.6	25.6	25.7	57.8	55.9	56.2	49.4	46	53	7.1	7.4	7.5
54	36	30.4		58	52.2	54.8	62.1	55	64.9	7.2	7.3	7.5
58	44	38.5	40.1	58.6	56.7	59.4	75.1	68	67.6	6.2	6.9	7.1
61	41	37.3	35.1	58.1	56.8	54.7	70.1	65.5	64.2	6.9	6.9	7.1
63	35.5	27.7	35.8	58.5	57.1	58.5	60.6	48.6	61.3	6.2	6.7	7
65	41.1	31.5	35.1	59.9	54.4	56.3	68.6	57.8	62.3	6.2	6.6	6.7
68	41.8	30.2	29.7	61.5	55.8	55.5	67.9	54.3	53.4		6.5	6.7
70	40.3	36.4	38.1	57.9	58.6	57.4	69.7	62	66.5	7	7.4	7.4
72	41.4	42.5	41.1	57.9	58.8	57.7	71.5	72.3	71.2	6.3	6.5	6.8
75	41.6	36.1	38.8	57.5	55.1	58.9	72.2	65.5	65.9	6.6	6.7	6.3
77	40.9	43.2	43.8	57.6	58	59.5	71.1	74.5	73.5	6.2	6.7	6.9
79	38.1	35	37	57.2	56	53.9	66.7	62.4	69.6	6.1	6.3	6.3
82	39.7	35.6	37.4	57.7	56.2	56	69.1	63.3	66.8	7.3	7.3	7.5
84	39.7	33.9	33	58.2	57.1	53.2	68.1	59.5	61.8	6.7	6.7	6.5
86	36.8	30.7	23.7	56.2	55.3	54.9	65.5	55.6	44.7	6.6	6.7	6.5
90	37.9	35.8	40.1	57.3	54.9	55.4	66.2	65.1	72.4	5.5	6	6.5

Table 5-12

Moisture Data for MAIV Test 1, 10% Soil

				MC-1 DATA								
				START DATE 9/19/90 10% SOIL								
DAY	AVG	AVG	AVG	PER CENT WATER				WHC			%WHC	
	% WATER	WHC	%WHC	T	M	B	T	M	B	T	M	B
8			55.51							58.35	51.17	57.00
10			59.20							57.90	62.00	57.70
11	33.57	60.58		33.30	32.60	34.80	57.05	63.70	61.00			
13	36.10	60.83	50.37	35.00	37.50	35.80	60.50	60.00	62.00	51.00	51.20	48.90
16	28.80	57.20		28.30	29.30	28.80	55.50	57.20	58.90			
20	28.77	56.73		28.00	28.70	29.60	55.90	57.30	57.00			
24	28.20	54.13		27.80	27.60	29.20	54.60	53.80	54.00			
27			55.20								55.20	
28	28.40	55.00		28.00	27.00	30.20	53.80	54.60	56.60			
30	30.87	55.80		30.10	31.00	31.50	55.30	55.30	56.80			
34			55.90							56.20	54.60	56.90
37	32.67	58.43		32.80	31.90	33.30	58.40	58.40	58.50			
38			64.43							62.50	64.10	66.70
41	37.27	57.87		36.80	36.00	39.00	58.90	56.20	58.50			
66			44.80							39.40	45.60	49.40
69	23.73	53.03	54.23	20.60	25.50	25.10	52.40	55.90	50.80	52.30	58.00	52.40
72	30.30	55.87		28.40	32.30	30.20	54.20	55.70	57.70			
90												

WESTON

Table 5-13

Moisture and pH Data for MAIV Test 2, 10% Soil

	MC-#2 DATA DAY 0 = 11/8/90											
	CONTAMINATED SOIL - 9.7% - HORSE BEDDING 44.7%											
	ALFALFA HAY - 28.8% BUFFALO MANURE 8.8% HORSE FEED 8.0%											
DAY	PER CENT WATER				WHC			%WHC			pH	
	T	M	B	T	M	B	T	M	B	REP 1	REP 2	REP 3
0	45.3			65.8			68.9			7.2		
1	32.6	36	35.3	55.7	59.9	60.7	58.4	60.1	58.1	6.7	6.5	6.4
6	36.8	36.2	31.4	60.7	63.2	60.4	60.6	57.3	52	8.2	8.3	
10	28.9	26.5	24.8	59.9	61.2	60.3	48.1	43.3	41.1	9	9.1	9.2
12										8.9	8.9	8.9
18	20.6	25.5	25.1	52.4	55.9	50.8	39.4	45.6	49.3	9	9	8.4
20	28.4	33.9	30.2	54.2	55.7	57.6	52.3	60.9	52.4	9	9.2	9.1
25	33.3	31.4	31.7	57.6	55	54.5	57.7	57.1	58.2	8.8	8.8	8.6
29	30.2	32.1	31.5	50.5	50	51.6	59.8	64.2	61			
34	34.1	29.9	33.5	49.9	46.5	51.5	68.2	64.5	65.1	8.4	8.1	8.5
39	32.9	33.9	33.8	50.3	52.2	51.9	65.5	64.1	65.1	7.8	8.3	8.7
44	31.9	32.9	30.9	50.2	50	46.5	63.5	65.7	66.3	8.9	8.9	8.7

WESTON MANAGERS DESIGNERS/CONSULTANTS

Table 5-14

Moisture and pH Data for MAIV Test 3, 25% Soil

	MC-#3 DATA DAY 0 = 2/6/91											
	CONTAMINATED SOIL - 25.8%			SAW/ALFALFA - 32.0%								
	MANURE (COW) - 25.0%		APPLE WASTE - 4.6%			POTATO - 12.6%						
DAY	PER CENT WATER				WHC			%WHC			pH	
	T	M	B	T	M	B	T	M	B	REP 1	REP 2	REP 3
0	38.5			54.3			70.8			5.7		
5	35.9	36	35.3	52.5	52.8	53.2	68.3	68.2	66.3	4.7	4.7	4.7
7	33.3	34.3	33.8	52.6	53.3	52.8	63.3	64.4	64.1	5.6	5	4.9
10	32.4	33.4	34.6	51.4	51.9	52.6	63.1	63.9	65.5	5	5.9	5.3
13	35.9	34.2	32.7	52.3	53.6	53.3	68.3	63.8	61.3	7.7	7.5	7.2
15	33.8	33.1	33.2	52.2	52	52.8	64.8	63.8	62.9	7	7.1	7.1
20	35.2	31.4	30.2	52.2	51.9	51.2	67.4	60.5	59.1	8.5	8.8	8.9
22	30.5	29.2	31.7	51.2	50.2	52.2	62.8	57.8	60.7	8.4	8.5	8.5
26	36.1	30.8	30.6	50.7	51.6	49	71.3	59.7	62.4	8.8	8.9	8.8
28	37.8	33.7	36.4	56.7	59.5	58.9	66.7	56.2	61.7	8.7	8.9	8.8
30	30.2	31	31.1	48.7	48.1	47.8	61.9	64.4	65.1	8.7	8.8	8.7
33	30.6	32.2	31.4	48.1	49.9	48.4	63.7	65	65	8.4	8	7.9
35	30.7	31.9	32.1	46.4	48.5	47.7	66.2	65.9	67.4	6.3	6.7	7.3
37	33.4	33.6	34	48.9	48.7	48.3	60.3	69.1	70.5	7.2	7.2	7.3
40	31.8	32.8	32.6	45.7	46.2	46.7	69.6	71.1	69.9	7.7	7.5	
44	33.7	33.7	33	49.4	48	47.8	68.2	70.2	69.4	7.4	7.2	7.2

Table 5-15

Moisture and pH Data for MAIV Test 4, 40% Soil

	MC-#4 DATA DAY 0 = 4/4/91											
	CONTAMINATED SOIL - 40.4% SAW/ALFALFA - 26.2%											
	COW MANURE - 19.8% APPLE "POMACE" - 3.7% POTATO - 10.0%											
DAY	PER CENT WATER			WHC			%WHC			pH		
	T	M	B	T	M	B	T	M	B	REP 1	REP 2	REP 3
0	29.3			46.8			62.3			5.4		
4	26.6	25.1	24.9	44	42.6	44.3	60.4	58.9	56.3	5.2	5	5.1
6	25.5	26.3	26.1	42.7	43.3	43.4	59.8	60.7	60.1	6.1	5.7	5.4
8	25.2	24.4	26	42.1	42.4	43.6	60	57.5	59.6	4.9	5	5
10	26.4	23.4	25.4	43.7	43.4	44.4	60.2	54.4	57.2	5.6	6	5.7
12	25.9	25.6	26.4	41.3	41.3	42.3	62.6	62.1	62.4	6.9	6.5	6
14	26	24.2	25.7	43.8	42.7	43.6	59.2	56.8	59	5.6	5.9	6
18	24.7	24.1	23.9	40.9	42.5	42	60.6	56.8	56.9	5.8	6	6
20	25.6	25.3	25	41.2	41.9	42.8	62.3	60.4	58.5	5.3	5.7	5.8
22	27.3	26.5	26.4	42.2	42.9	40.9	64.7	61.8	64.4	5.6	5.7	6
25	23.8	26.1	25.5	41.1	43.1	42.7	57.9	60.5	59.8	7	6.9	6.9
27	26.6	27	27.8	41.7	42.7	42	63.7	63.3	66.1	6.5	6.5	6.6
29	26.5	26.3	26.2	41.6	42.9	42.3	63.7	61.5	61.9	7.5	7.3	7.4
32	26.3	25.9	26.8	43.7	42.5	43.3	60.1	61	62	7.2	7.4	7.4
34	25.5	25.2	25.5	42.4	42.2	41.8	60.1	59.6	60.8	7.1	7.3	7.2
36	22.7	25	24.4	40.5	41.5	39.7	56	60.3	61.4	7.1	7.8	7.9
39	27.1	27	26.7	42.3	42.2	42.1	64.1	62.6	63.4	6.5	6.6	6.9
41	26.7	27.5	25.7	41.3	42.3	42.3	64.6	65.1	60.7	7.4	7.3	7.4
44	26.1	25.7	26.3	42.4	41.4	42.3	61.7	62	62.4	7.3	7.4	7.2
90												

Table 5-16

pH Data for SP-1, SP-2, SP-3, SP-5, SP-6, SP-7, and MAIV-1

												pH TABLE													
DATE	DAY	ST-1			ST-2			ST-3			DAY	ST-5			ST-6			ST-7			DAY		MC-1		
		T	M	B	T	M	B	T	M	B		T	M	B	T	M	B	T	M	B		AVERAGE	1	2	
9/21/90	0	5.00			7.90			6.10			0	6.30			7.10			7.70			0				
9/24/90	3	5.10			5.60			7.30			3	7.50			6.90			5.00			3	6.90	6.90		
9/27/90	6	5.50			5.60			7.30			6	7.00			7.10			5.90			6	5.70	5.70		
9/28/90	7	5.10	4.70	4.70	5.00	4.80	4.90	6.20	6.20	5.60	7	5.80	5.30	5.30	6.30	6.20	5.60	5.10	4.90	4.90	7	6.30	5.70	6.90	6.30
9/29/90	8										8										8	6.23	7.40	5.60	5.70
10/1/90	10	4.70	4.70	4.80	5.60	4.80	5.20	7.20	5.60	5.40	10	7.20	5.30	5.10	5.30	6.50	5.70	7.40	5.00	5.00	10	7.20	7.30	7.40	6.90
10/2/90	11	6.90	5.20	5.10	5.90	5.20	5.60	8.00	5.80	5.60	11	6.50	6.80	5.80	8.10	6.20	6.10	5.30	5.10	5.10	11	8.10	8.10	8.90	7.30
10/5/90	14	4.70	4.70	4.70	6.50	5.20	5.00	8.10	5.60	5.30	14	7.90	6.00		7.90	7.90	6.30	8.50	5.20	4.90	14	8.57	8.60	8.60	8.50
10/10/90	19	5.20	5.60	5.10	5.70	5.40		7.20	5.70	5.50	19	6.30	5.80	5.70	7.90	7.30	6.20	8.60	8.60	8.40	19	9.20	9.20	9.20	9.20
10/12/90	21	5.40	5.30	5.20	5.40	5.50	5.70	5.90	5.70	5.60	21	7.80	5.90	5.70	8.40	6.70	6.60	8.70	8.20	9.00	21	9.37	9.30	9.40	9.40
10/16/90	25	8.00	9.10	9.10	7.80	5.60	7.70	7.50	5.60	5.50	25	6.90	5.90	5.60	8.40	6.50	6.10	8.90	9.20	9.10	25	9.30	9.30	9.30	9.30
10/18/90	27	8.40	4.60	5.70	5.50	6.30	7.60	6.00	4.80	4.80	27	7.10	4.90	5.40	8.20	7.30	6.00	8.70	8.90	8.80	27	9.03	9.10	9.00	9.00
10/23/90	32	9.40	9.20	9.10	9.30	9.10	9.10	8.30	8.70	8.80	32	8.60	7.60	5.90	8.90	7.00	6.80	9.20	9.20	9.00	32	9.33	9.40	9.30	9.30
10/25/90	34	8.90	9.00	8.80	9.00	9.20	9.00	9.10	7.10	8.20	34	8.70	7.80	8.50	8.80	8.50	8.70	9.50	9.20	9.30	34	9.43	9.30	9.50	9.50
10/29/90	38	7.90	8.50	8.80	8.30	8.70	8.60	6.00	8.40	9.10	38	6.60	5.50	5.30	8.10	7.40	7.40	8.90	9.20	9.10	38	9.17	9.20	9.10	9.20
11/1/90	41	8.50	8.60	8.40	9.00	8.90	9.00	8.10	8.50	8.90	41	7.70	7.40	7.50	8.50	8.40	8.70	8.90	9.30	9.20	41	9.30	9.30	9.30	
11/6/90	46	8.90	8.80	9.10	9.20	8.50	9.20	8.90	8.90	9.00	46	8.70	9.00	8.50	8.80	8.70	8.50	9.20	9.30	9.20	90				
11/9/90	49	8.60	8.70	8.60	8.40	8.30	8.10	7.90	8.50	8.60	49	8.50	8.40	7.90	8.10	8.20	8.30	8.60	8.60	8.40					
11/14/90	54	8.60	8.70	8.60	8.80	8.80	8.80	8.20	8.50	8.50	54	8.80	8.80	8.30	8.50	8.40	8.50	8.80	8.90	8.80					
11/17/90	57	8.80	9.00	9.20	8.90	8.90	9.00	9.10	8.90	8.80	57	9.10	9.10	9.00	8.70	8.50	8.70	9.00	8.90	8.90					
11/19/90	59	9.10	8.90	9.00	9.10	9.00	9.00	9.10	9.10	9.00	59	9.00	9.10	9.10	8.70	8.80	8.90	9.20	9.20	8.90					
11/26/90	66	8.90	9.10	8.90	9.00	8.90	8.90	9.20	8.90	8.90	66	9.00	9.10	8.60	8.90	8.80	8.70	9.00	9.00	8.90					
/29/90	69	9.10	9.20	9.20	9.20	9.20		9.10	9.10	9.00	69	9.20	9.20	8.80	8.90	8.80	8.80	9.00	9.10	9.10					
12/3/90	73	8.90	8.70	8.80	8.80	8.80	8.60	8.90	8.80	8.60	73	8.70	8.80	8.90	8.70	8.60	8.70	8.90	8.90	8.80					
12/13/90	83	8.90	8.90	8.90	8.90	8.90	8.90	8.70	8.60	8.70	83	8.80	8.80	8.90	8.70	8.70	8.70	8.90	8.70	8.80					
12/17/90	87	8.90	9.00	8.80	8.90	8.90	8.90	8.90	8.80	8.70	87	8.80	8.90	8.90	8.80	8.70	8.80	9.00	9.00						
12/20/90	90	9.00	8.90	8.90	8.90	8.80	9.00	8.90	8.80	8.80	90	8.80	8.90	8.80	8.80	8.80	8.80	8.90	8.90	8.90					

FIGURE 5-15 AMBIENT HIGH AND LOW TEMPERATURE DURING ENTIRE FIELD TESTING PROGRAM (9/20/90 TO 5/18/91).

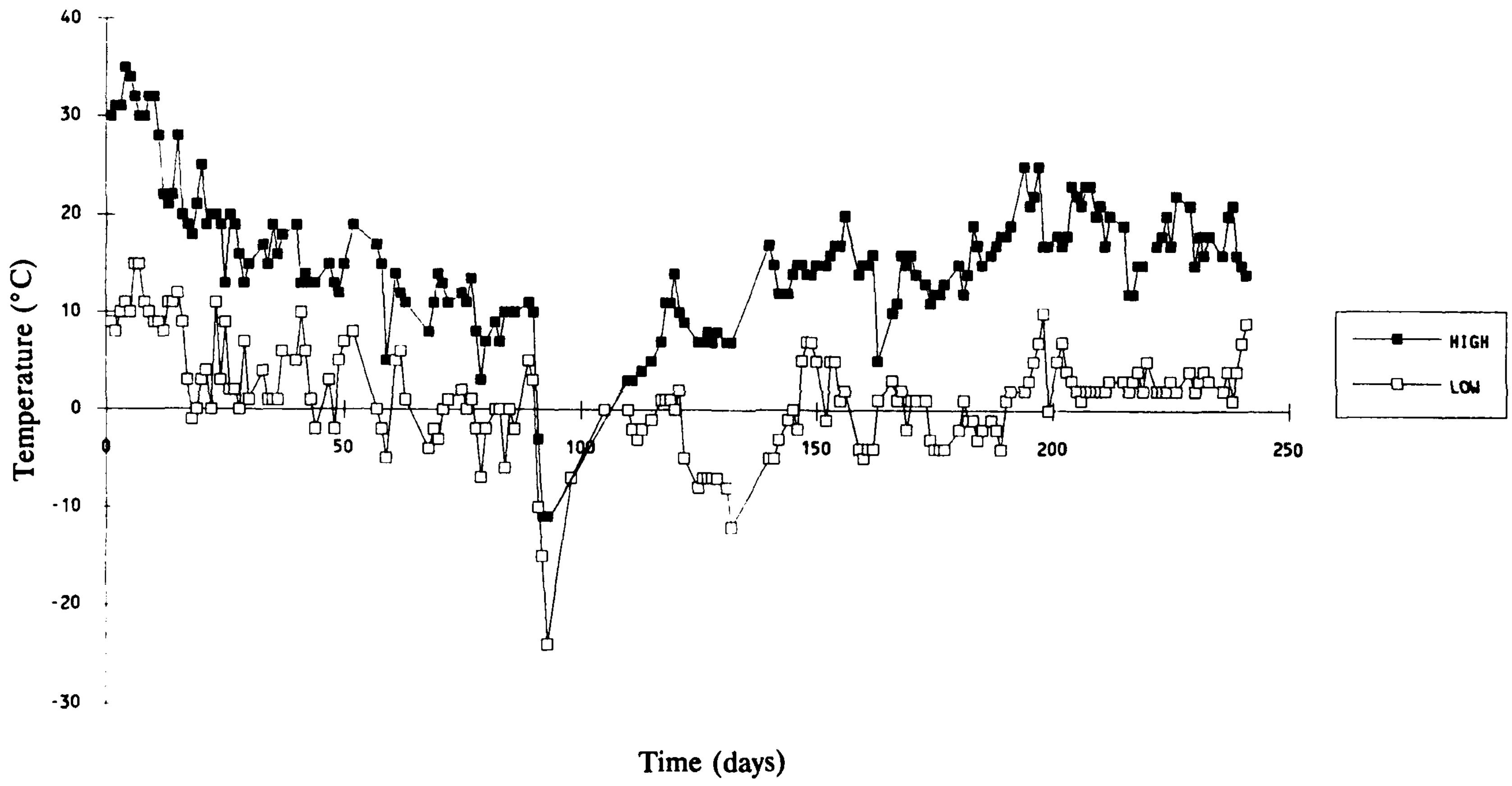


FIGURE 5-16 AMBIENT TEMPERATURE FOR TESTS SP-1, SP-2, SP-3, SP-5, SP-6, AND SP-7 (9/21/90 TO 12/20/90)

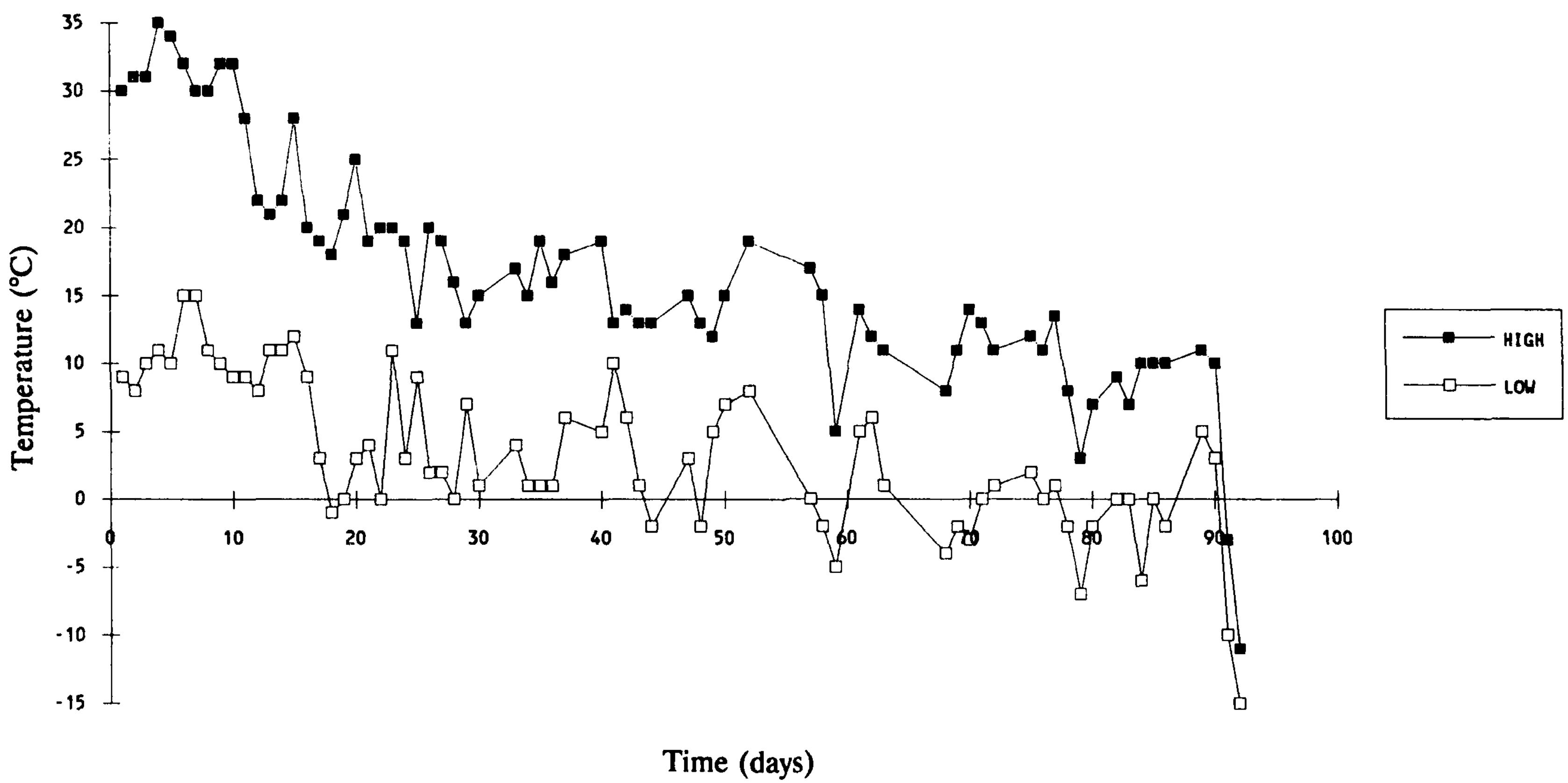


FIGURE 5-17 AMBIENT TEMPERATURE FOR BIOAUGMENTED TEST SP-4 (11/2/90 TO 1/30/91)

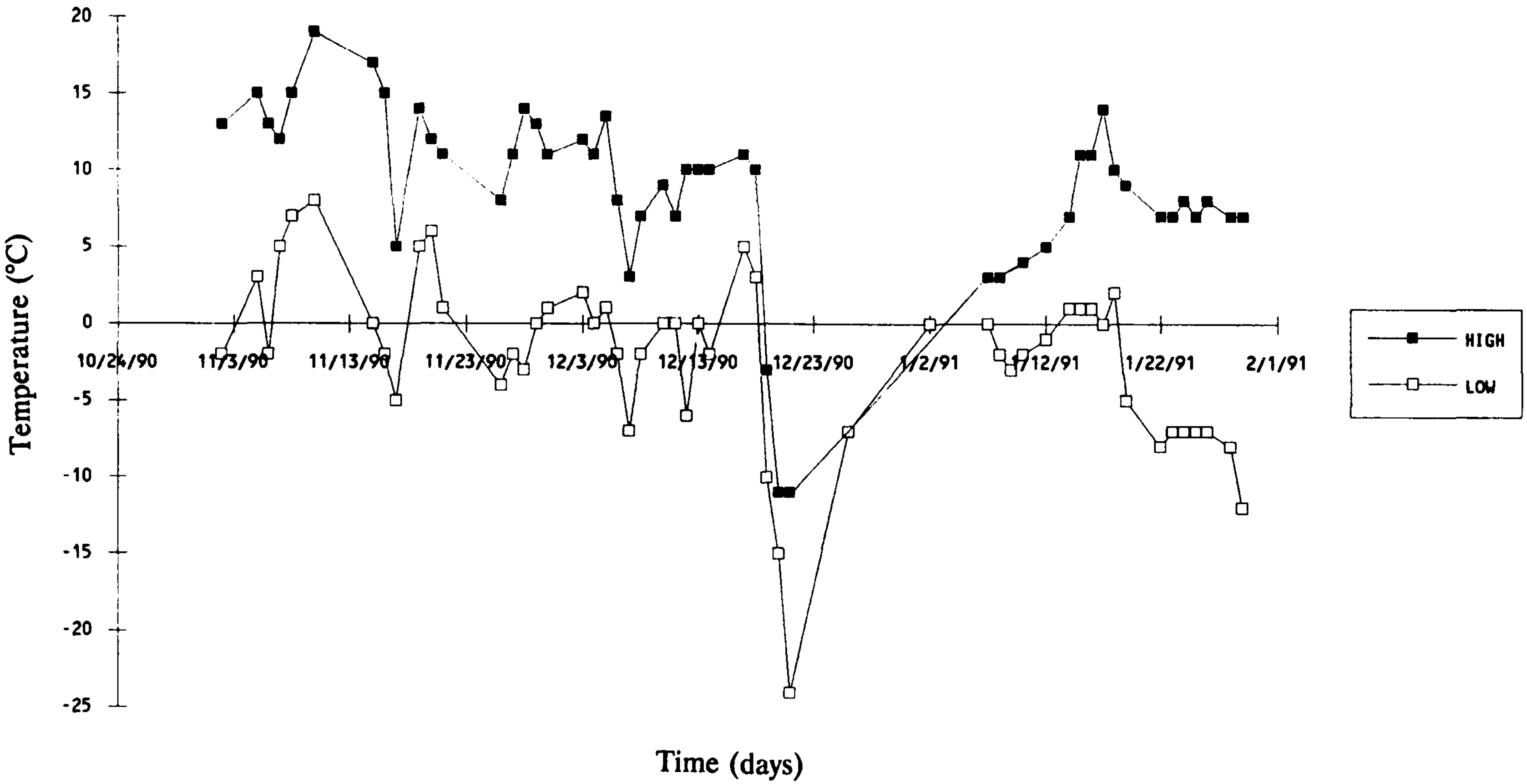


FIGURE 5-18 AMBIENT TEMPERATURE FOR TEST SP-8 (2/6/91 TO 5/7/91)

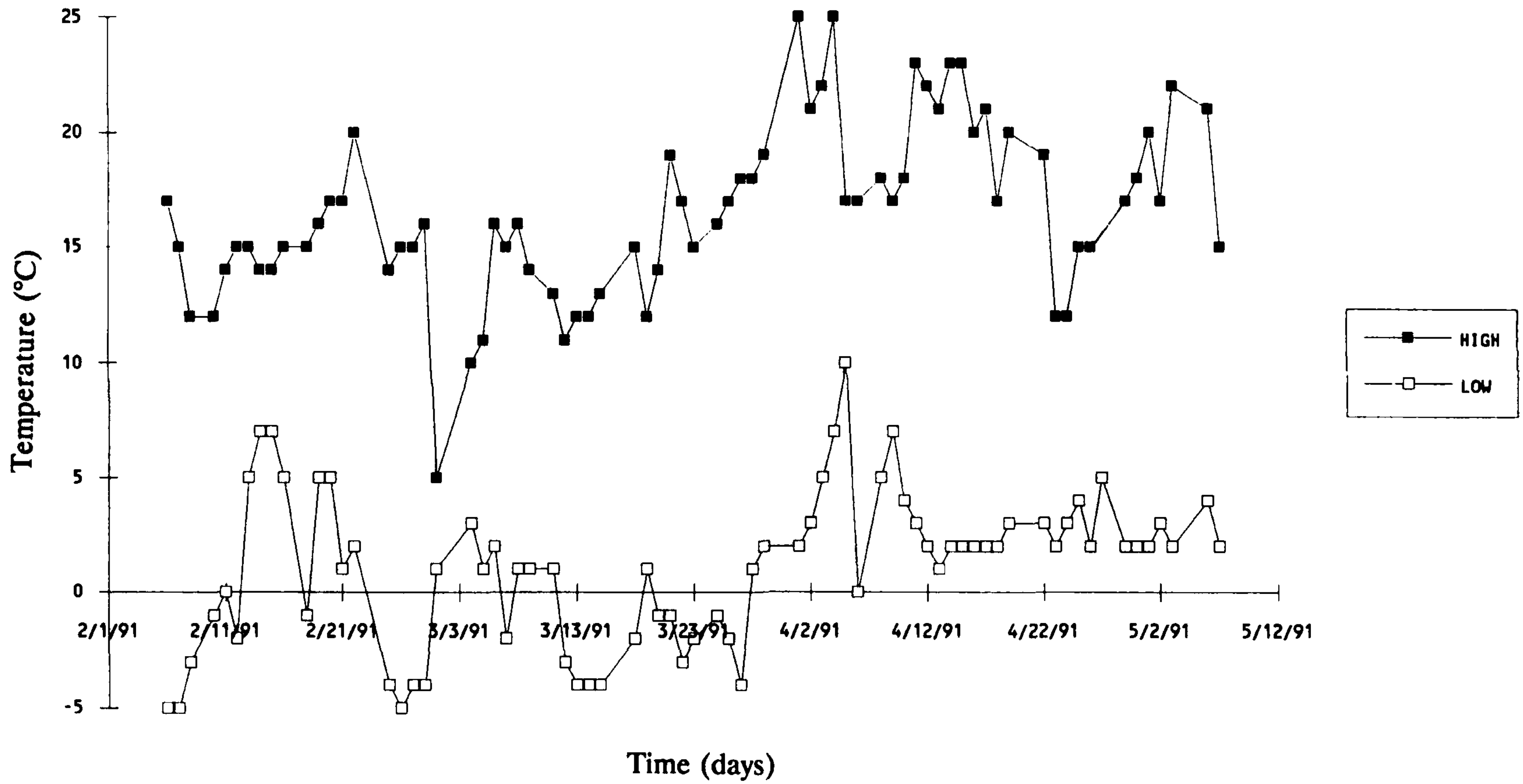


FIGURE 5-19 AMBIENT TEMPERATURE FOR TEST MAIV-1 (9/19/90 TO 11/2/90)

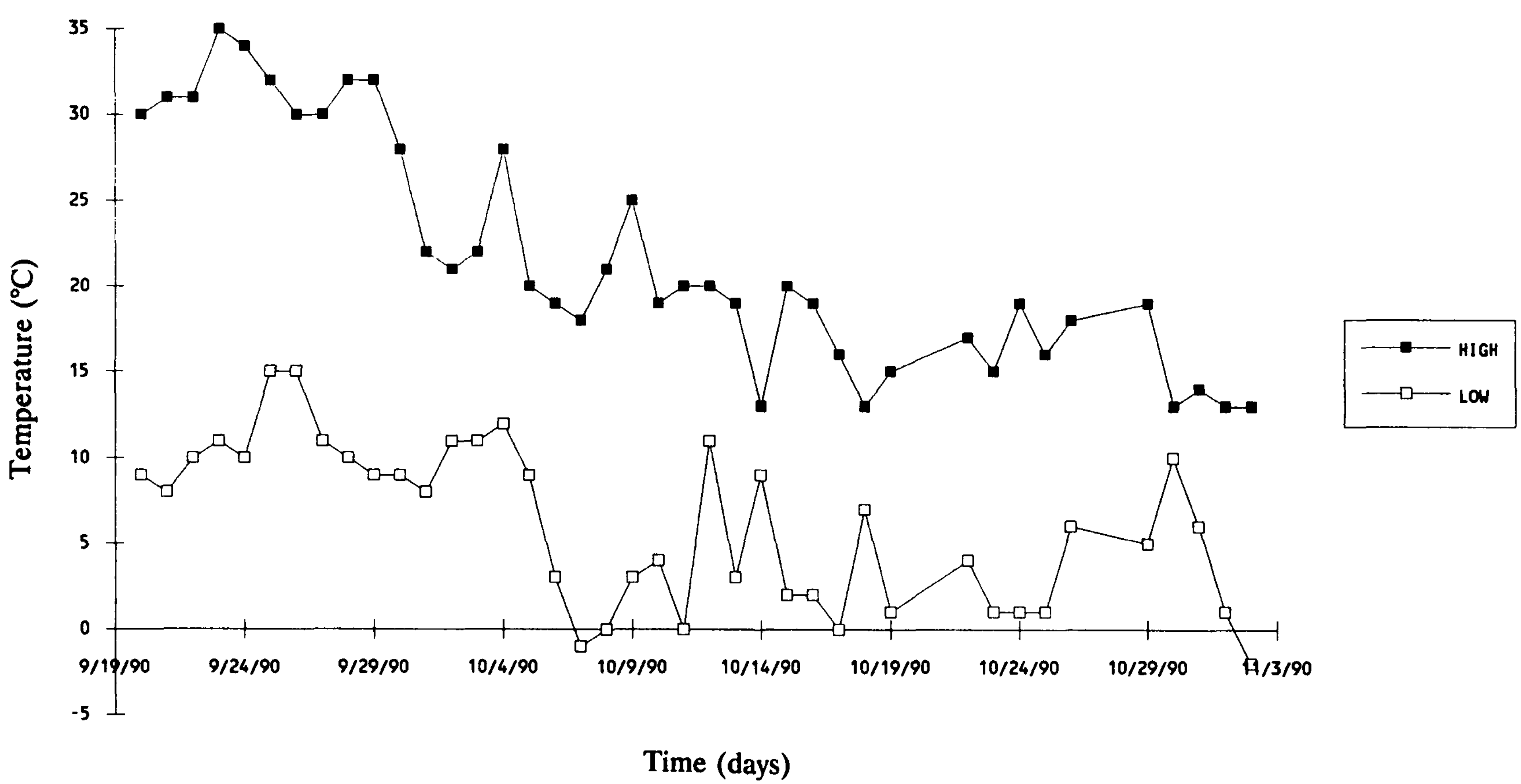


FIGURE 5-20 AMBIENT TEMPERATURE FOR TEST MAIV-2 (11/8/90 TO 12/21/90)

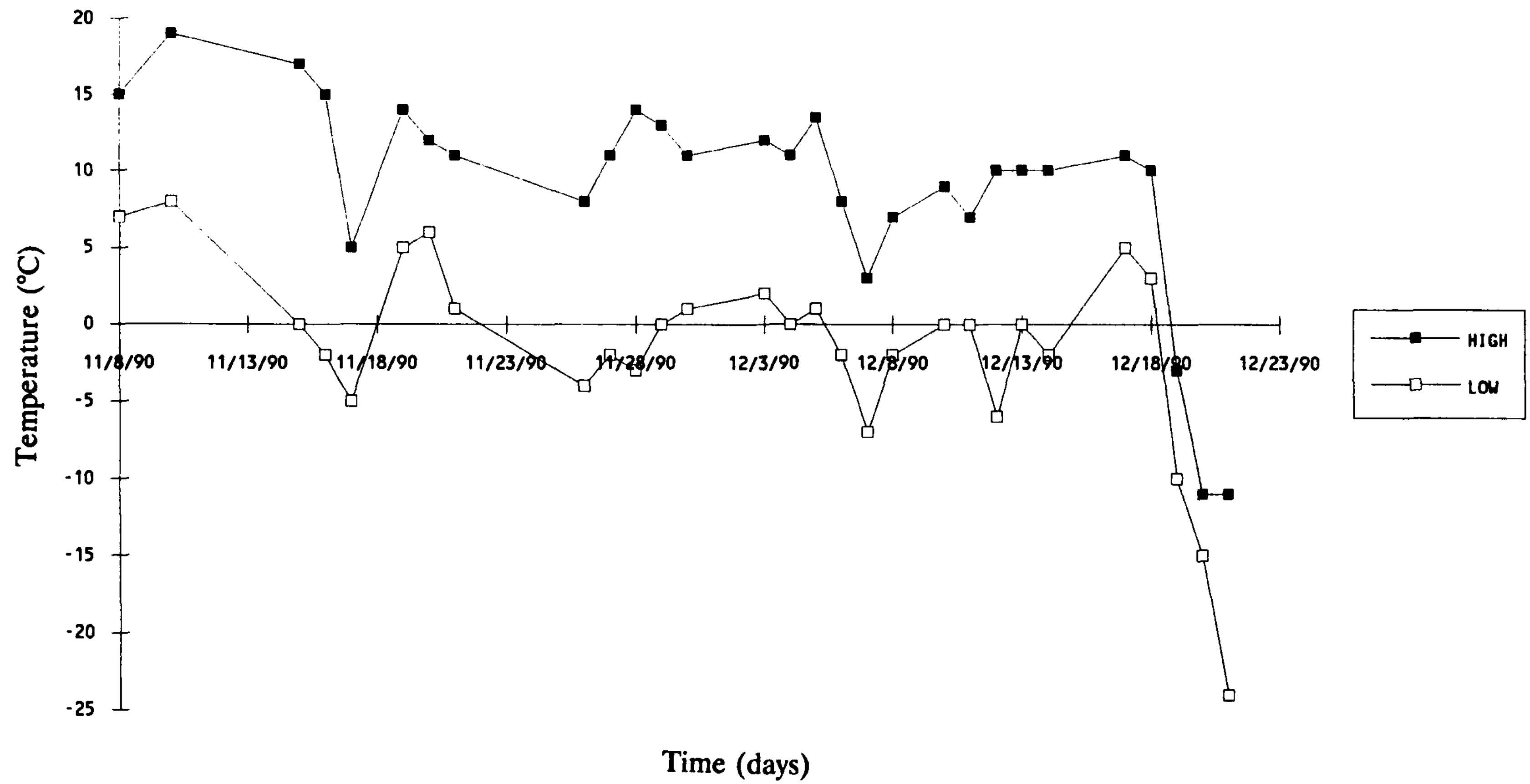


FIGURE 5-21 AMBIENT TEMPERATURE FOR TEST MAIV-3 (2/6/91 TO 3/22/91)

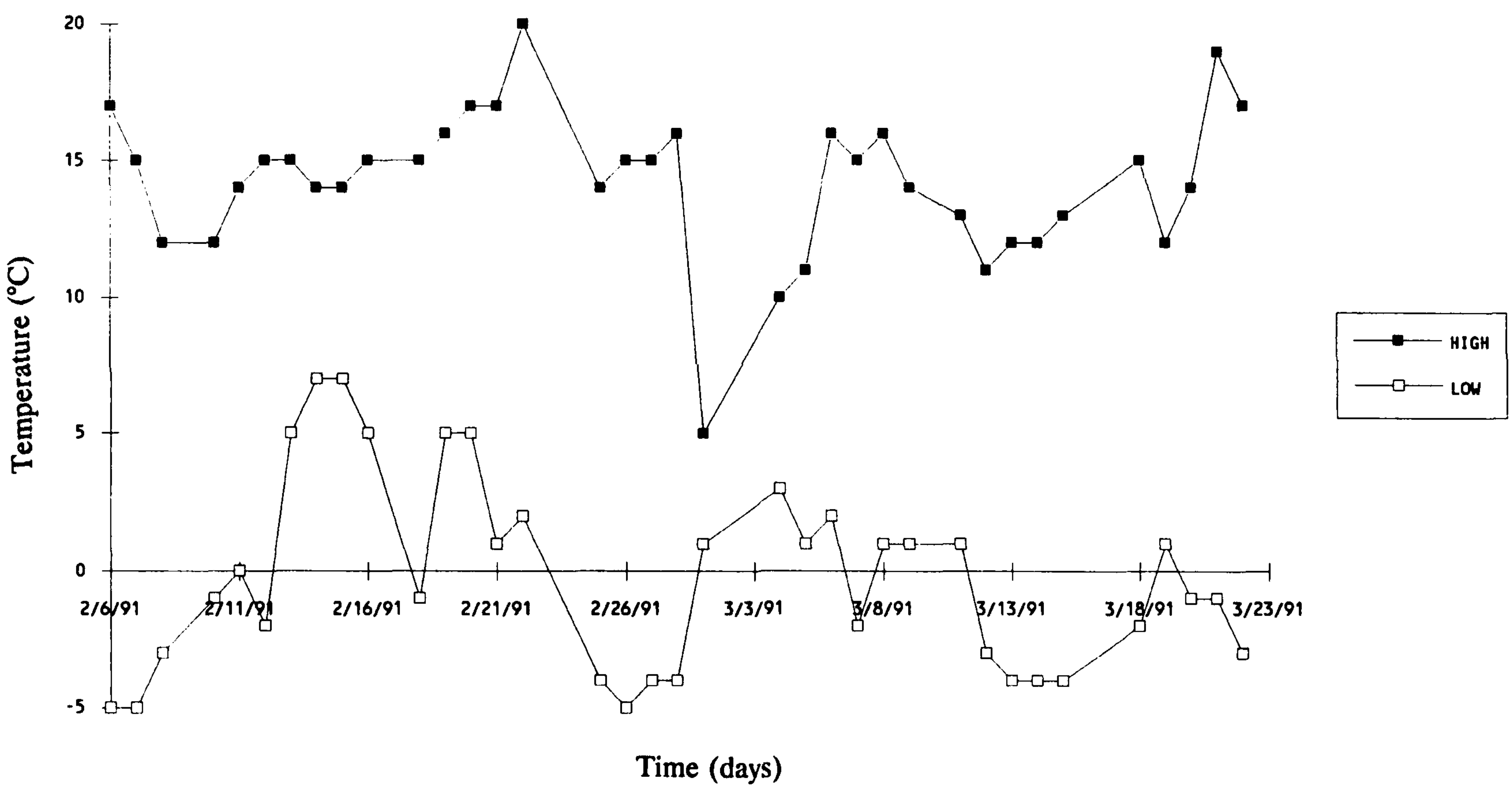


FIGURE 5-22 AMBIENT TEMPERATURE FOR TEST MAIV-4 (4/3/91 TO 5/18/91)

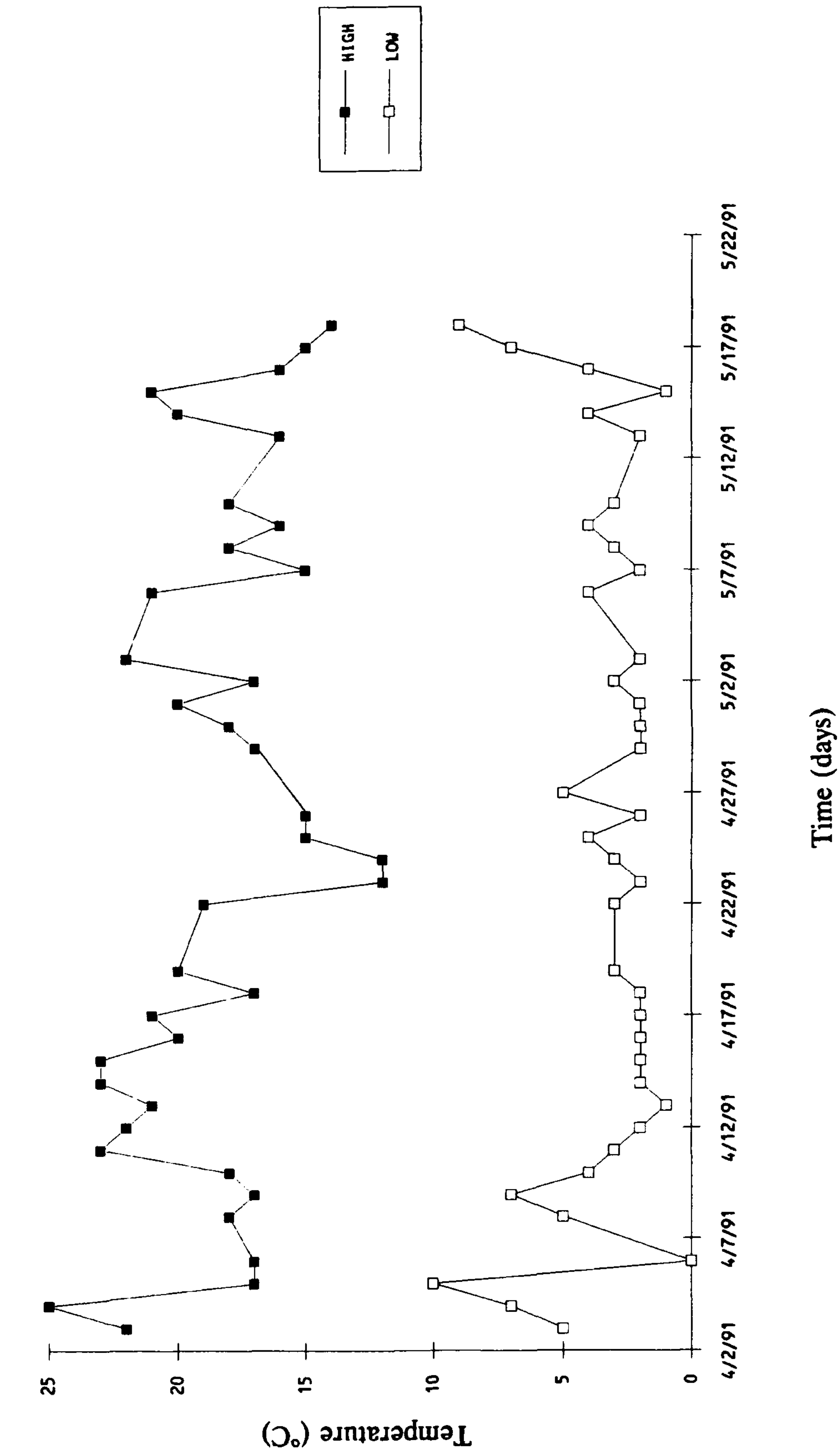


FIGURE 5-23 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-1, 7% SOIL

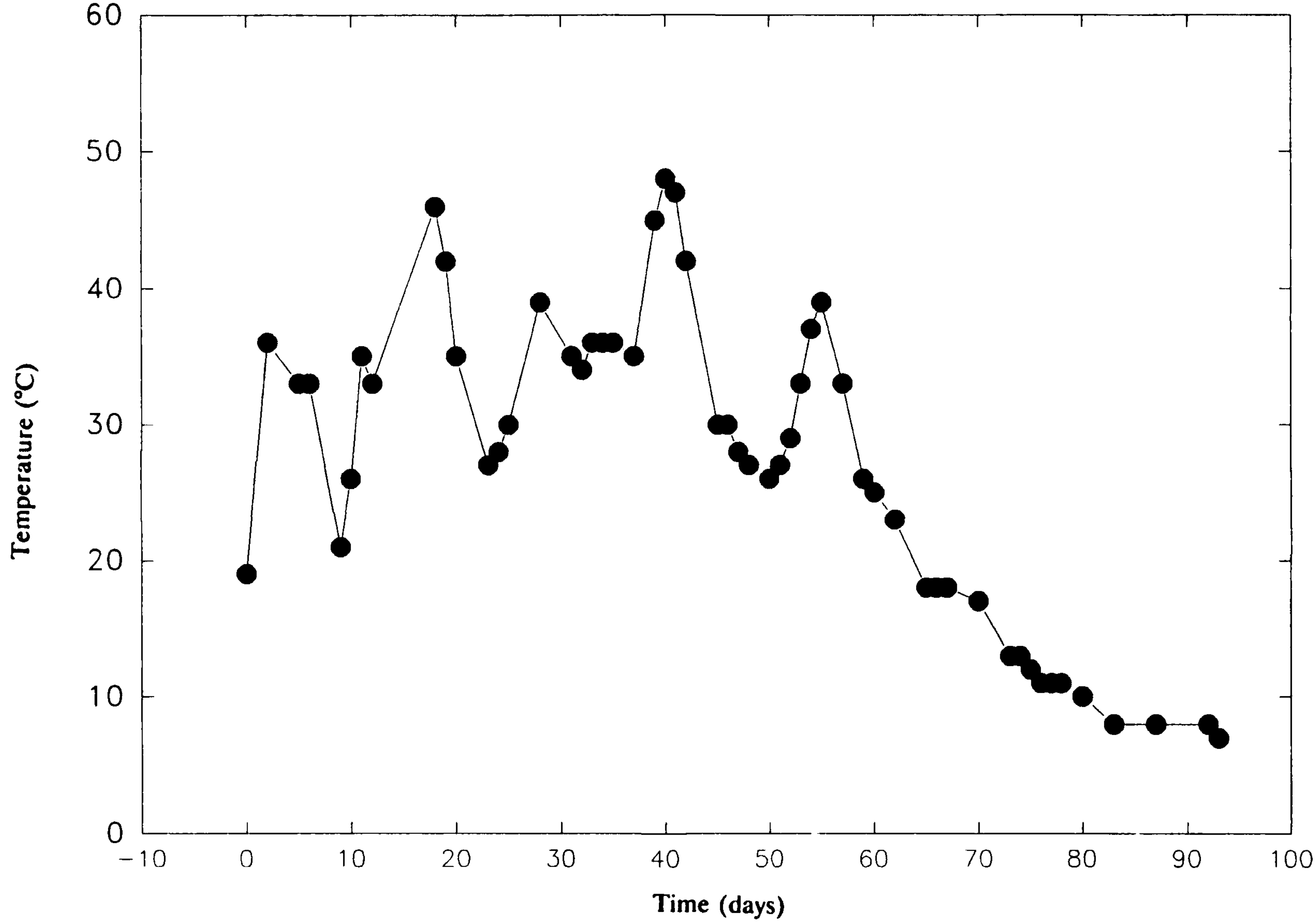


FIGURE 5-24 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-2, 10% SOIL

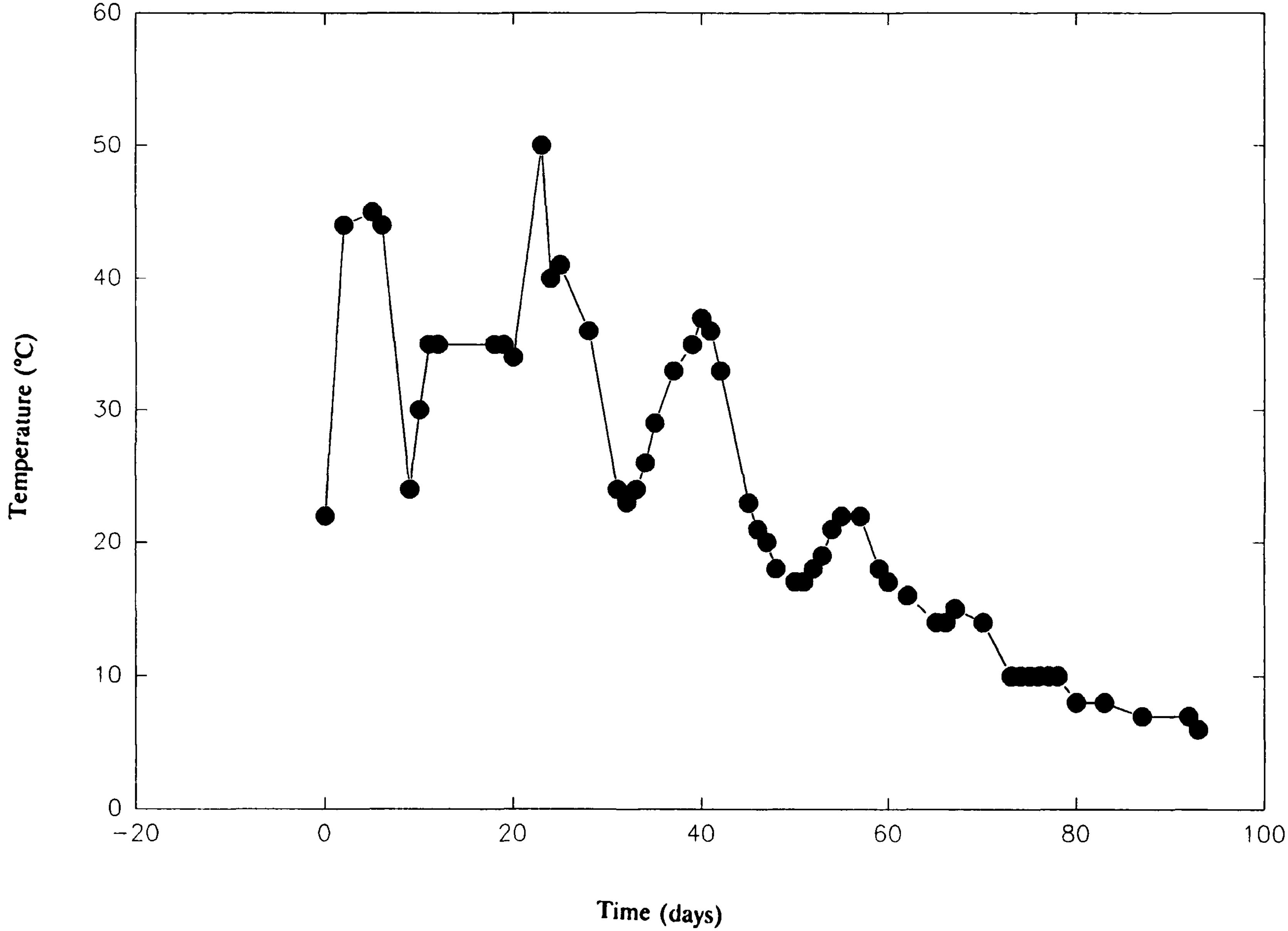


FIGURE 5-25 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-3, 20% SOIL

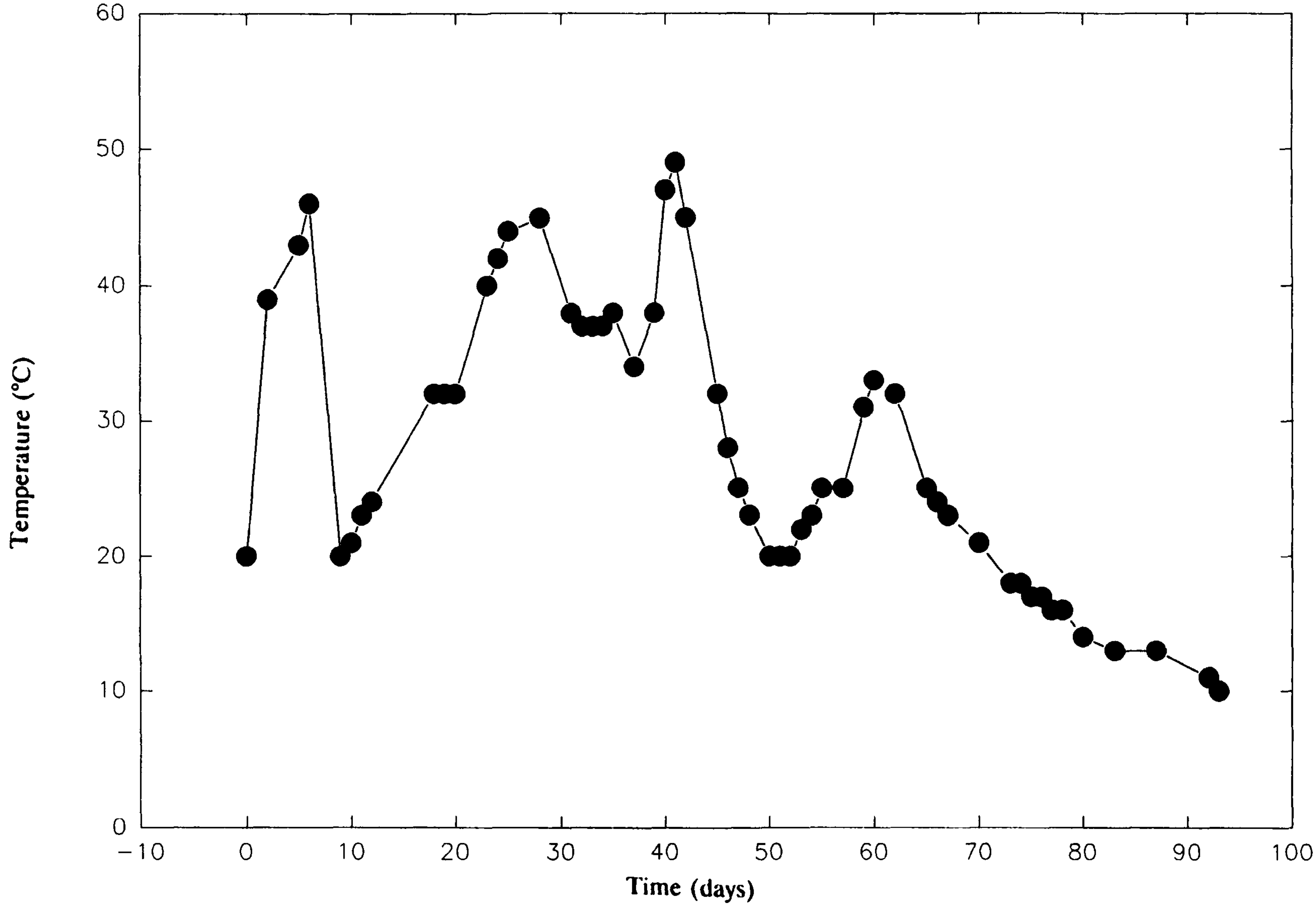


FIGURE 5-26 AVERAGE TEMPERATURE WITHIN BIOAUGMENTED STATIC PILE TEST - SP-4, 80% SOIL

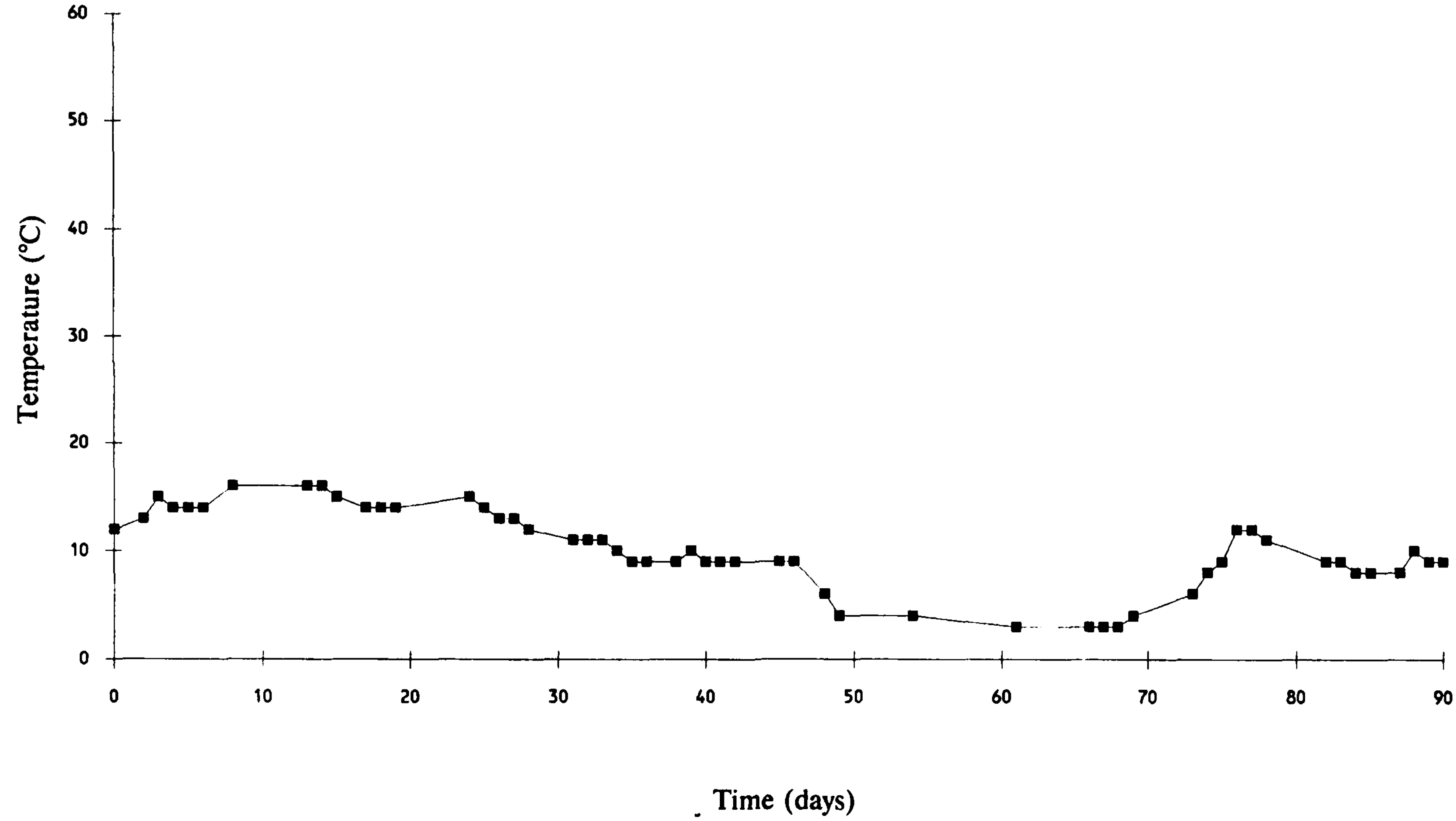


FIGURE 5-27 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-5, 30% SOIL

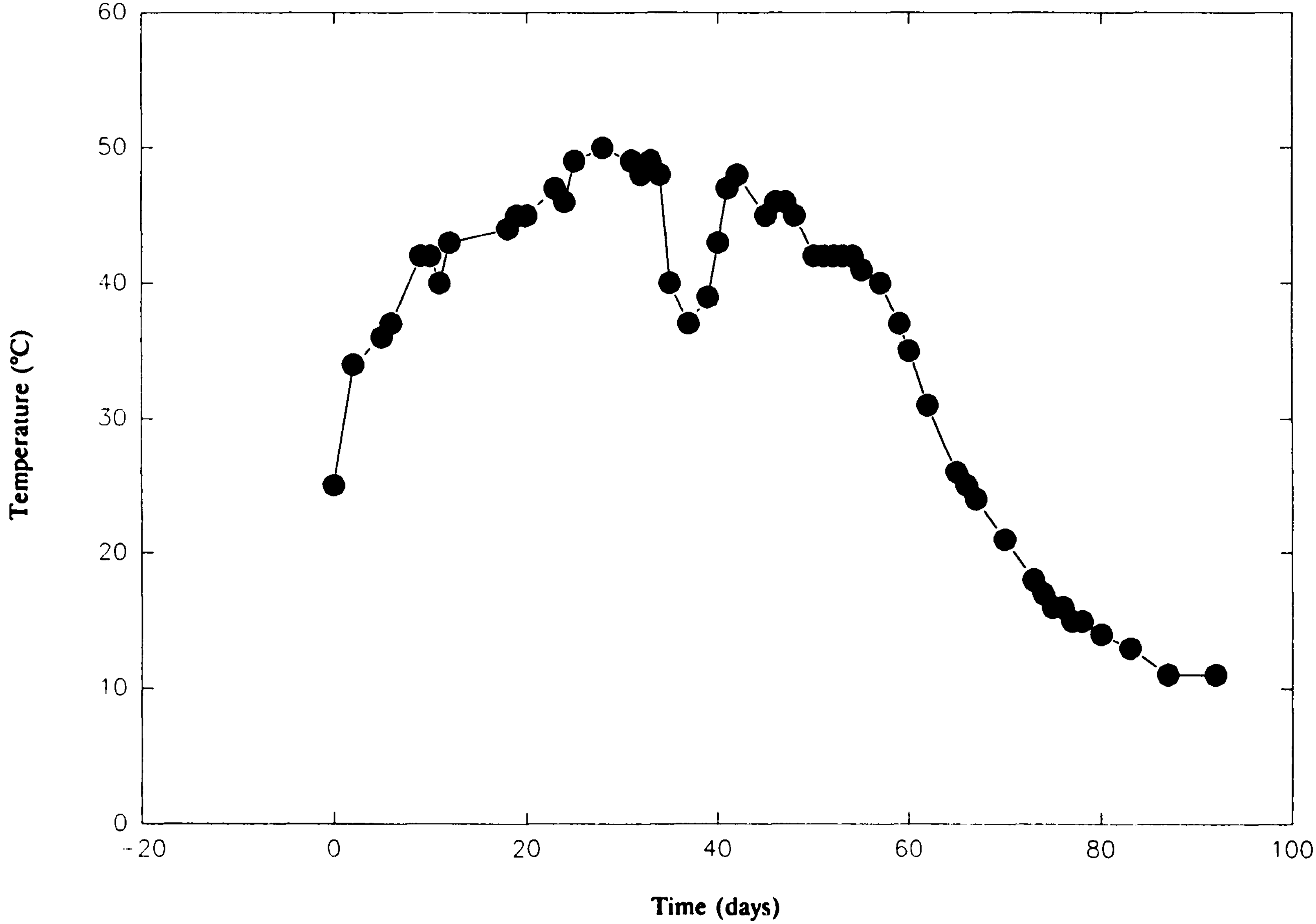


FIGURE 5-28 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-6, 40% SOIL

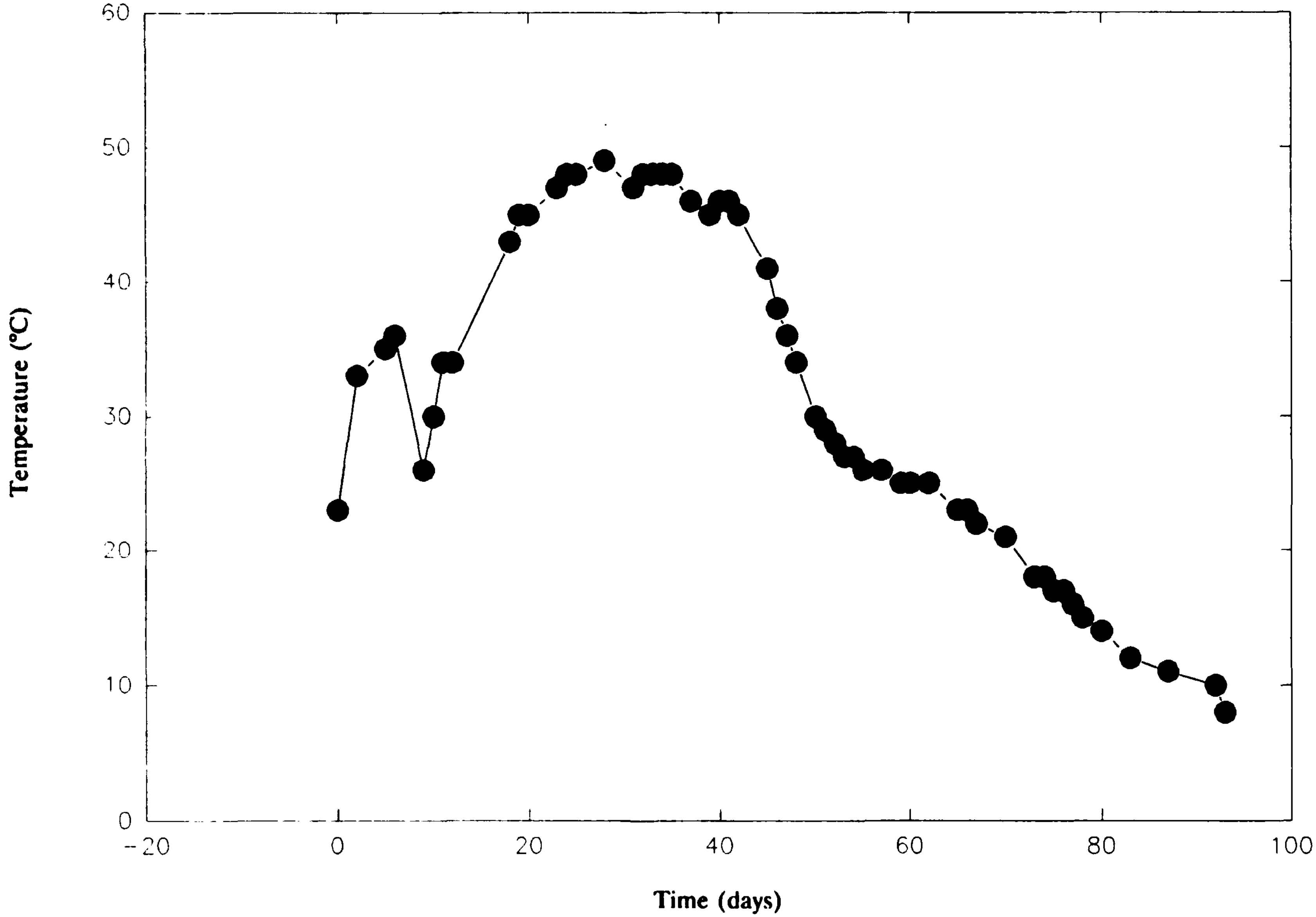


FIGURE 5-29 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-7, 10% SOIL (UNCONTAMINATED)

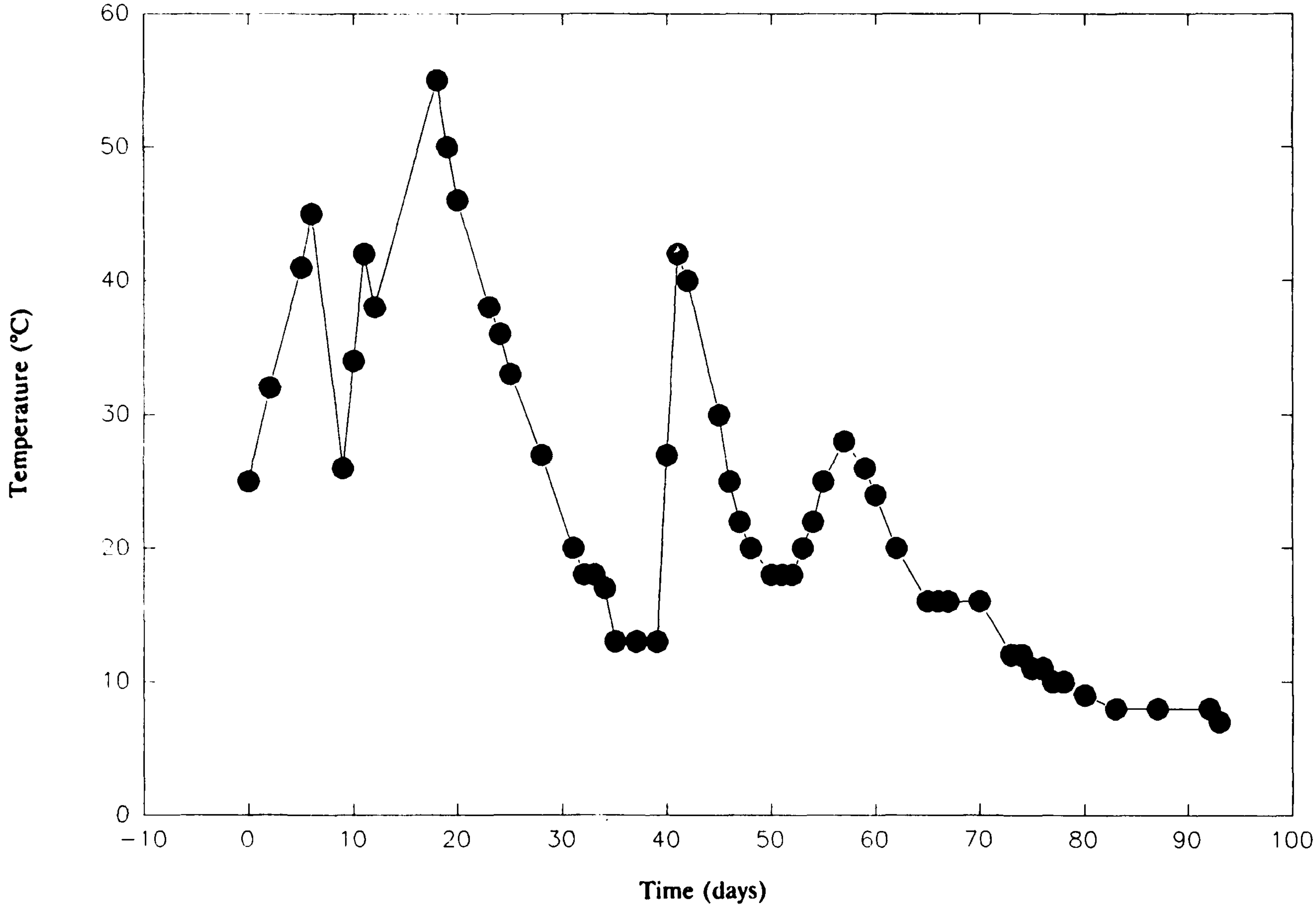


FIGURE 5-30 AVERAGE TEMPERATURE WITHIN STATIC PILE TEST - SP-8, 10% SOIL

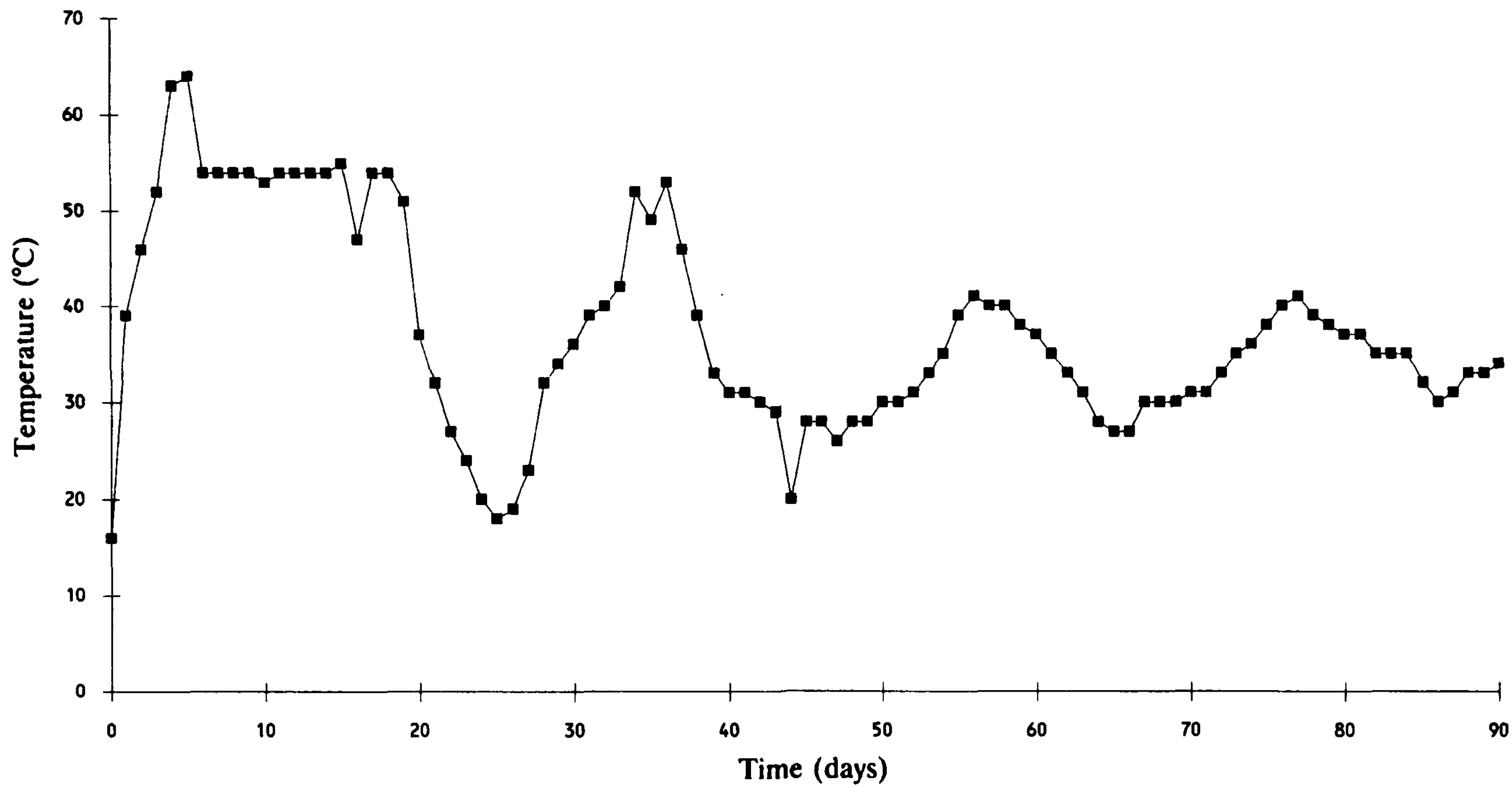


FIGURE 5-31 AVERAGE TEMPERATURE WITHIN MAIV TEST 1, ELECTRONIC DATA

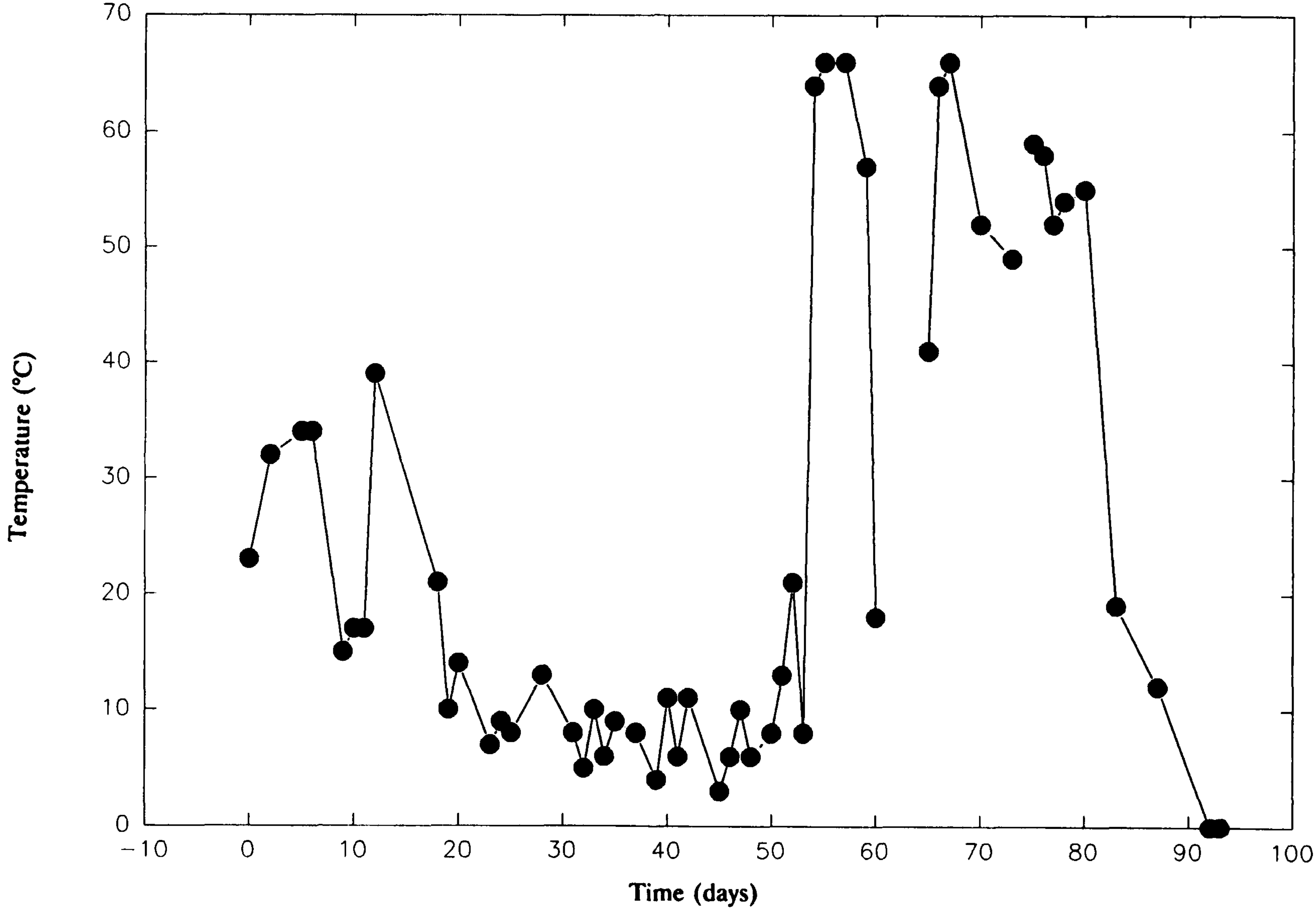


FIGURE 5-32 AVERAGE TEMPERATURE WITHIN MAIV TEST 2, ELECTRONIC DATA

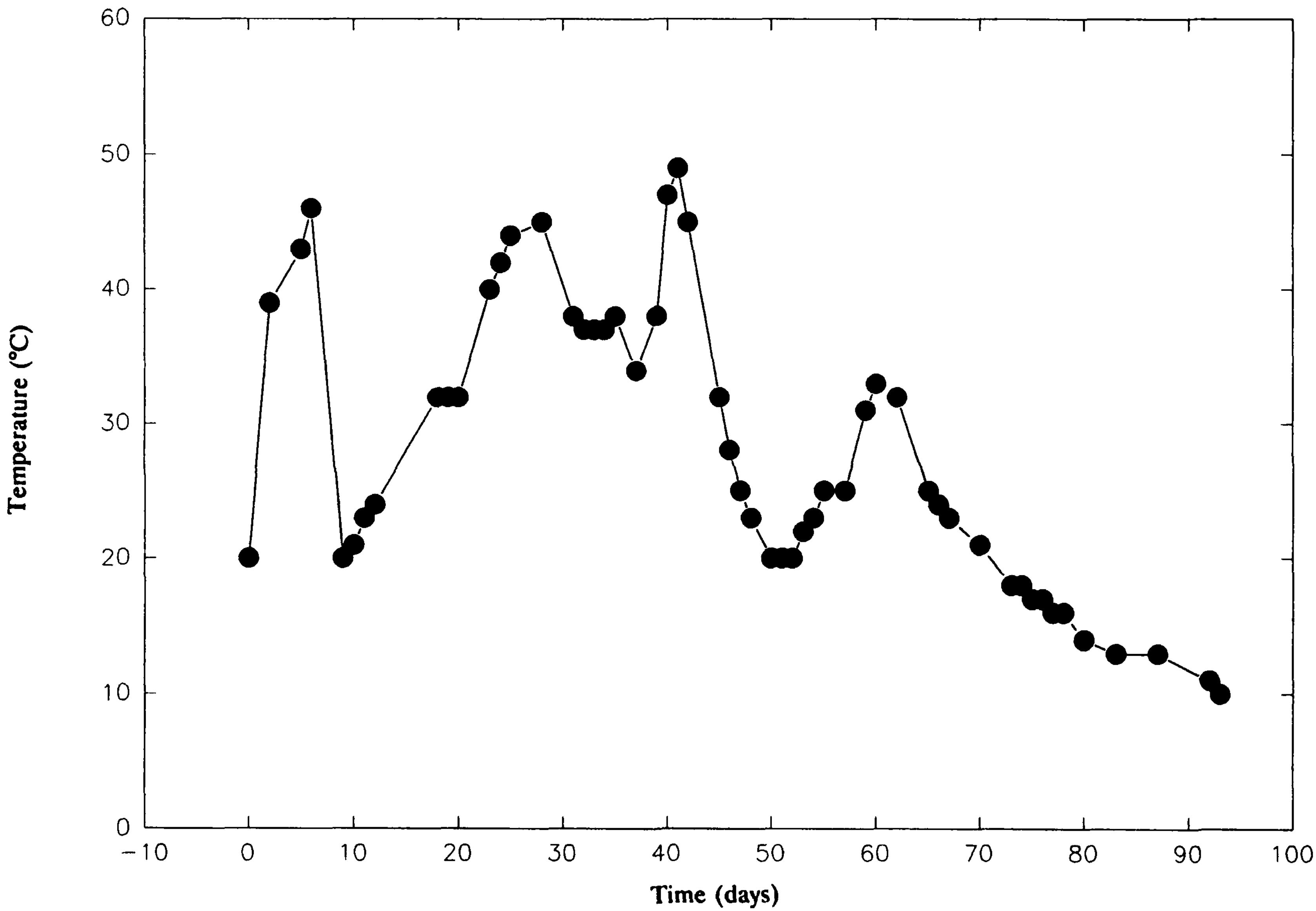


FIGURE 5-33 AVERAGE TEMPERATURE WITHIN MAIV TEST 1, MANUAL DATA

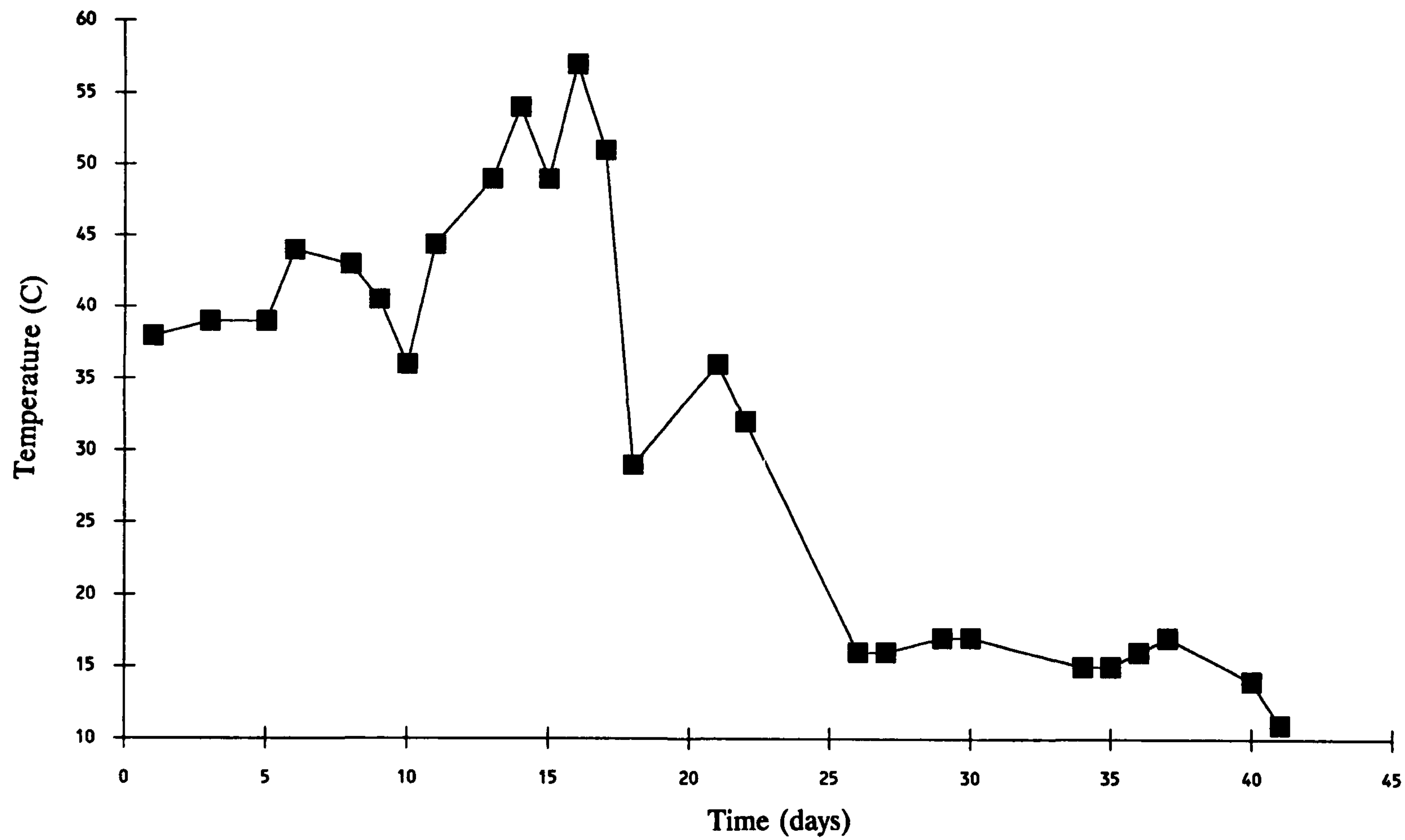


FIGURE 5-34 AVERAGE TEMPERATURE WITHIN MAIV TEST 2, MANUAL DATA

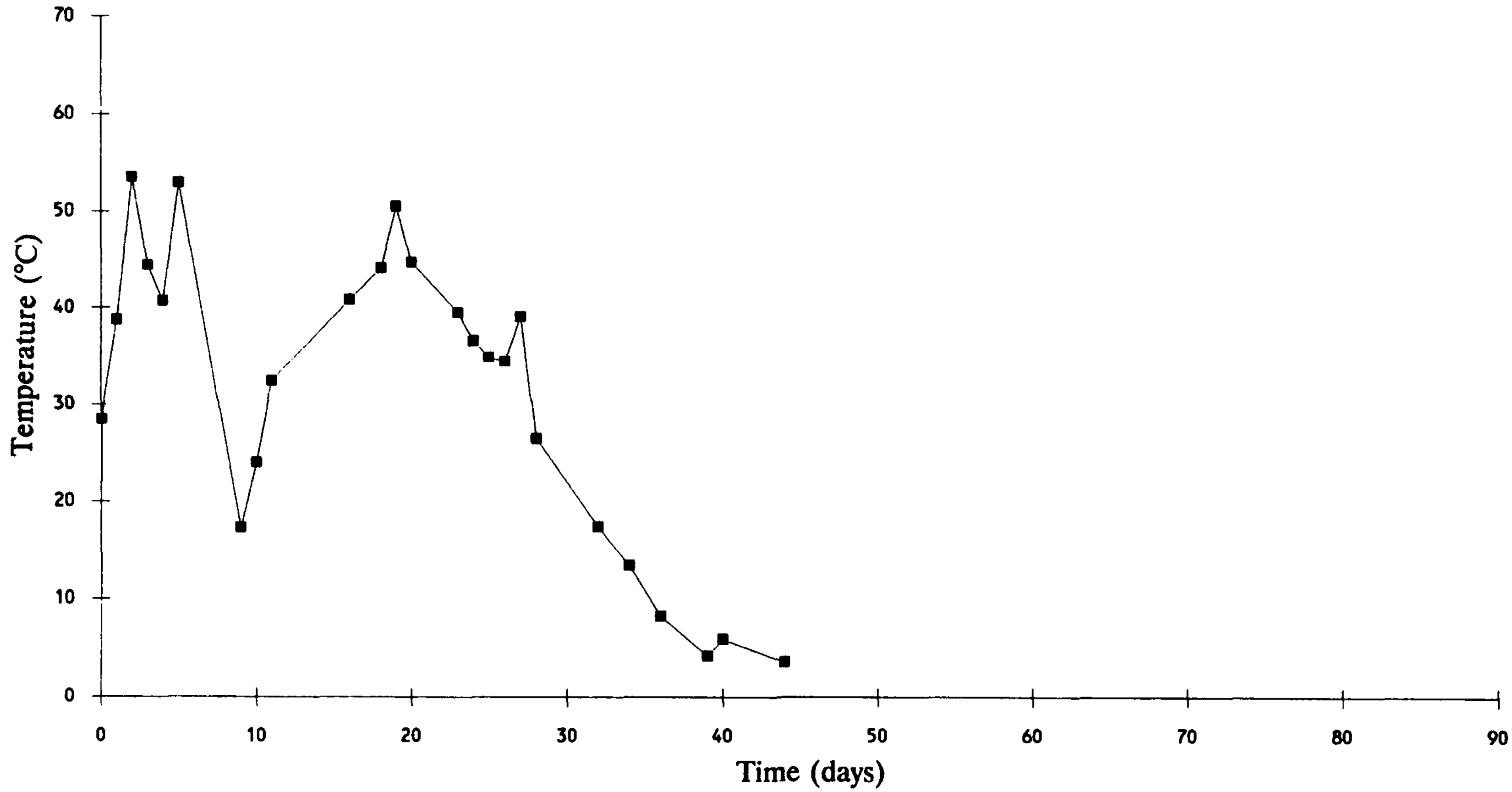


FIGURE 5-35 AVERAGE TEMPERATURE WITHIN MAIV TEST 3, MANUAL DATA

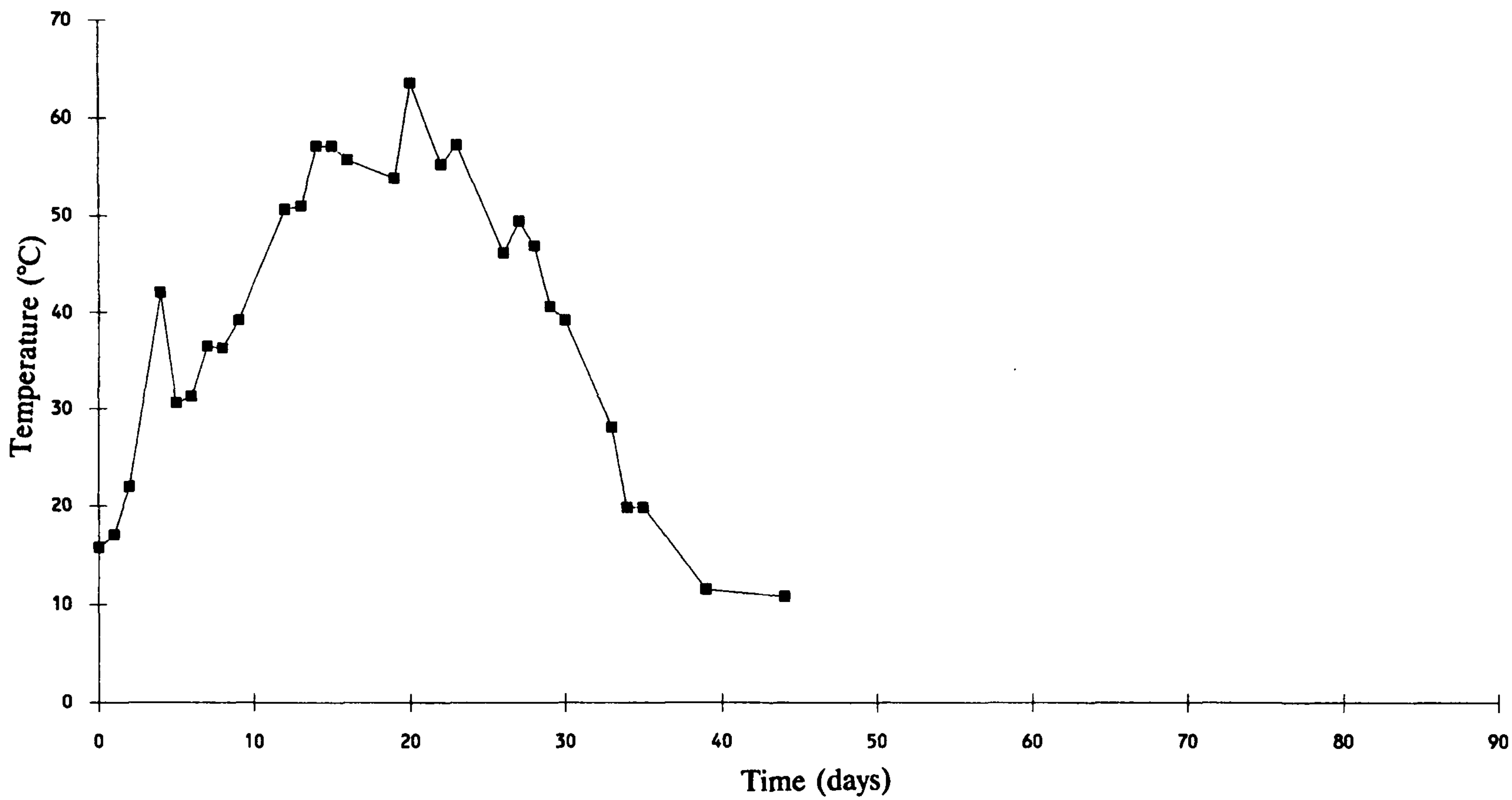


FIGURE 5-36 AVERAGE TEMPERATURE WITHIN MAIV TEST 4, MANUAL DATA

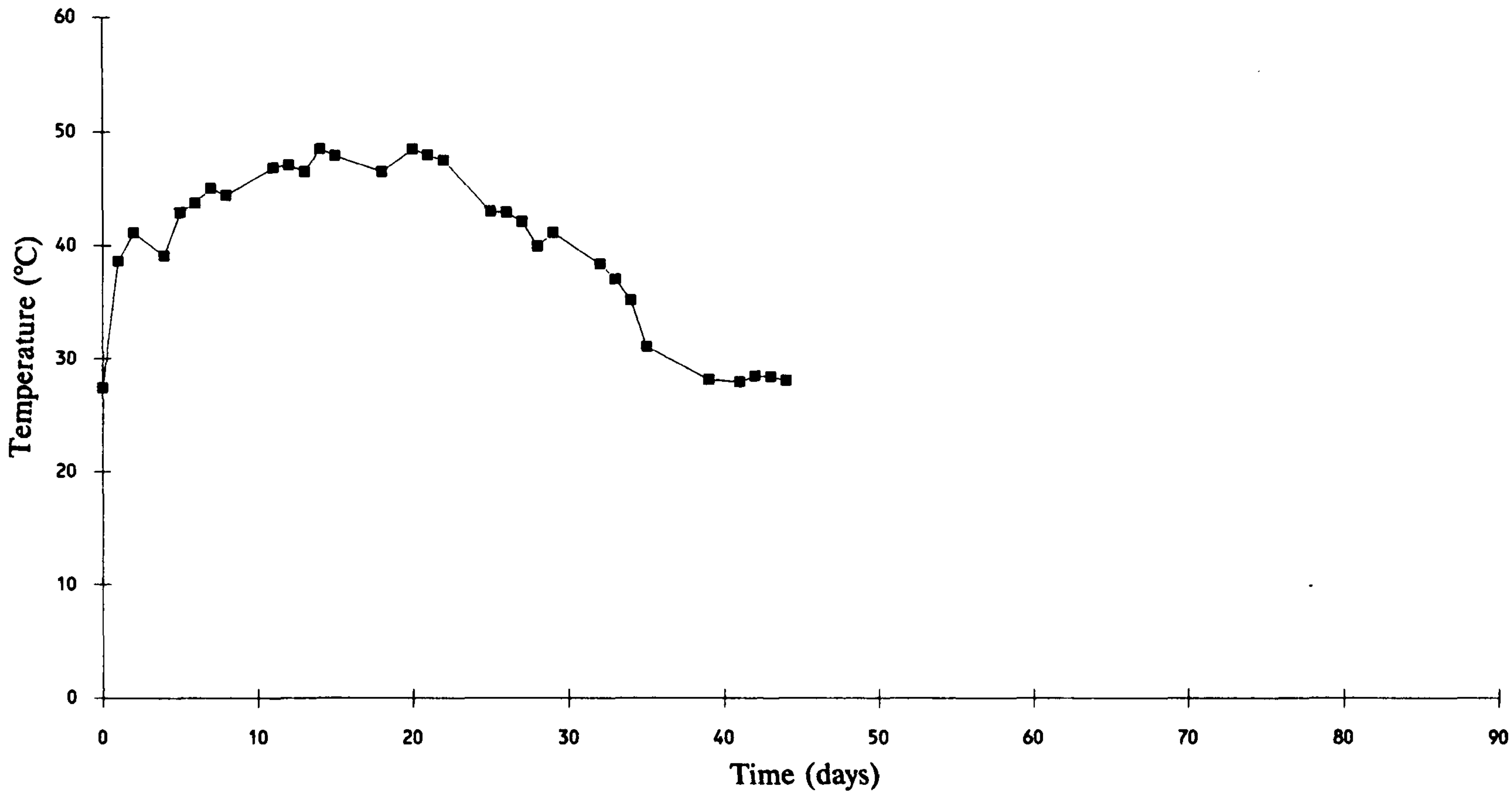


FIGURE 5-37 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-1, 7% SOIL

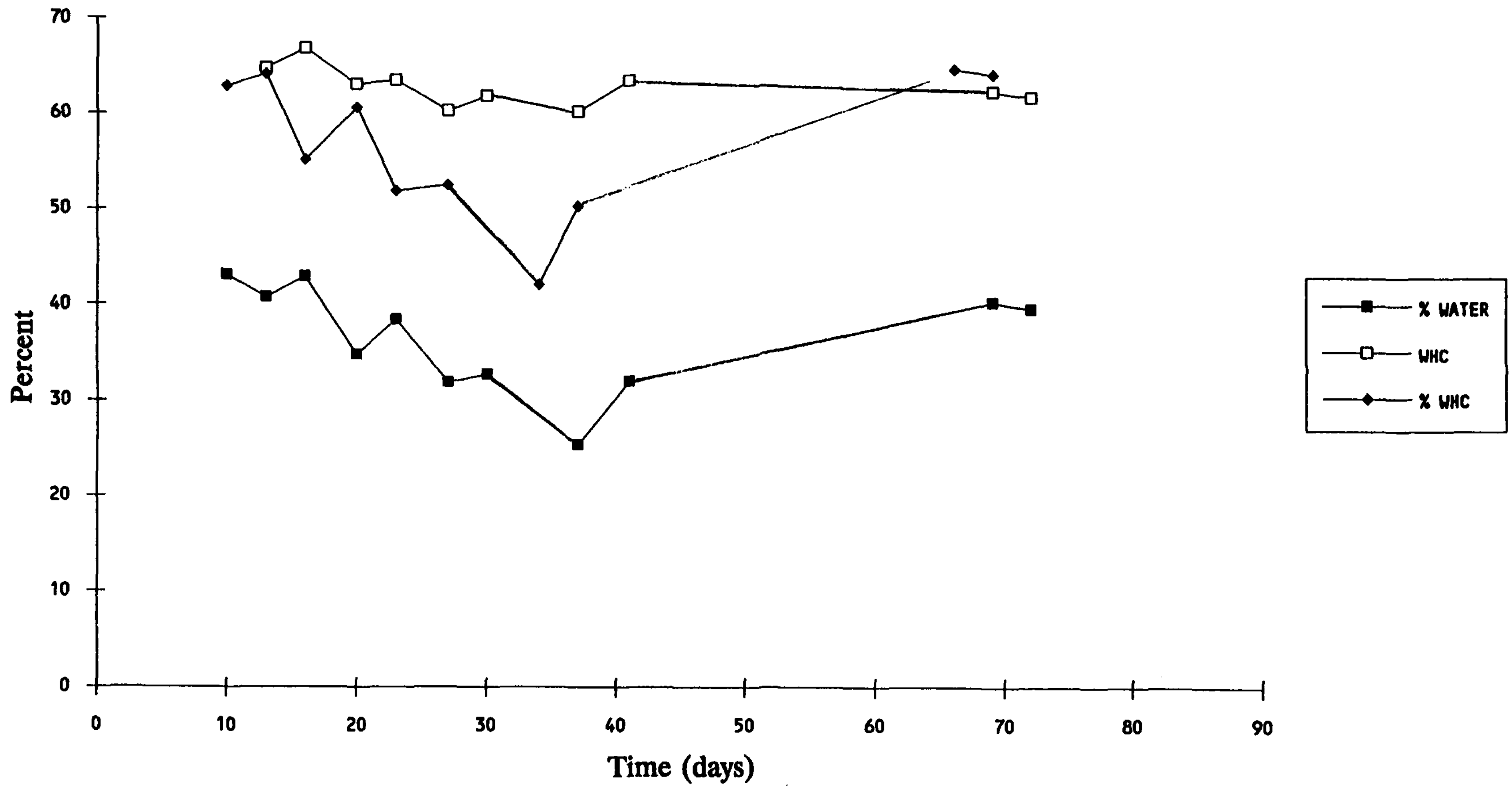


FIGURE 5-38 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-2, 10% SOIL

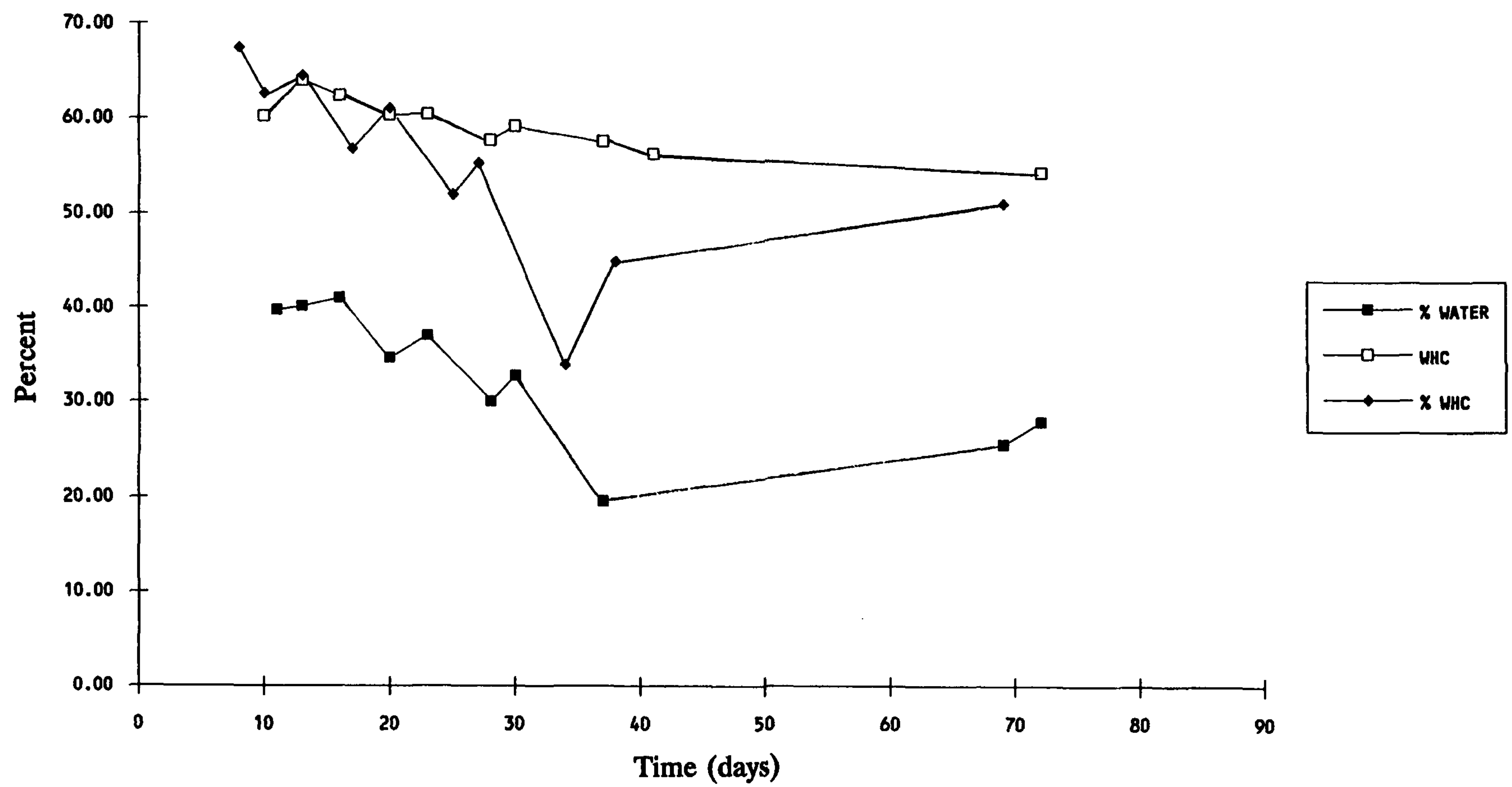


FIGURE 5-39 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-3, 20% SOIL

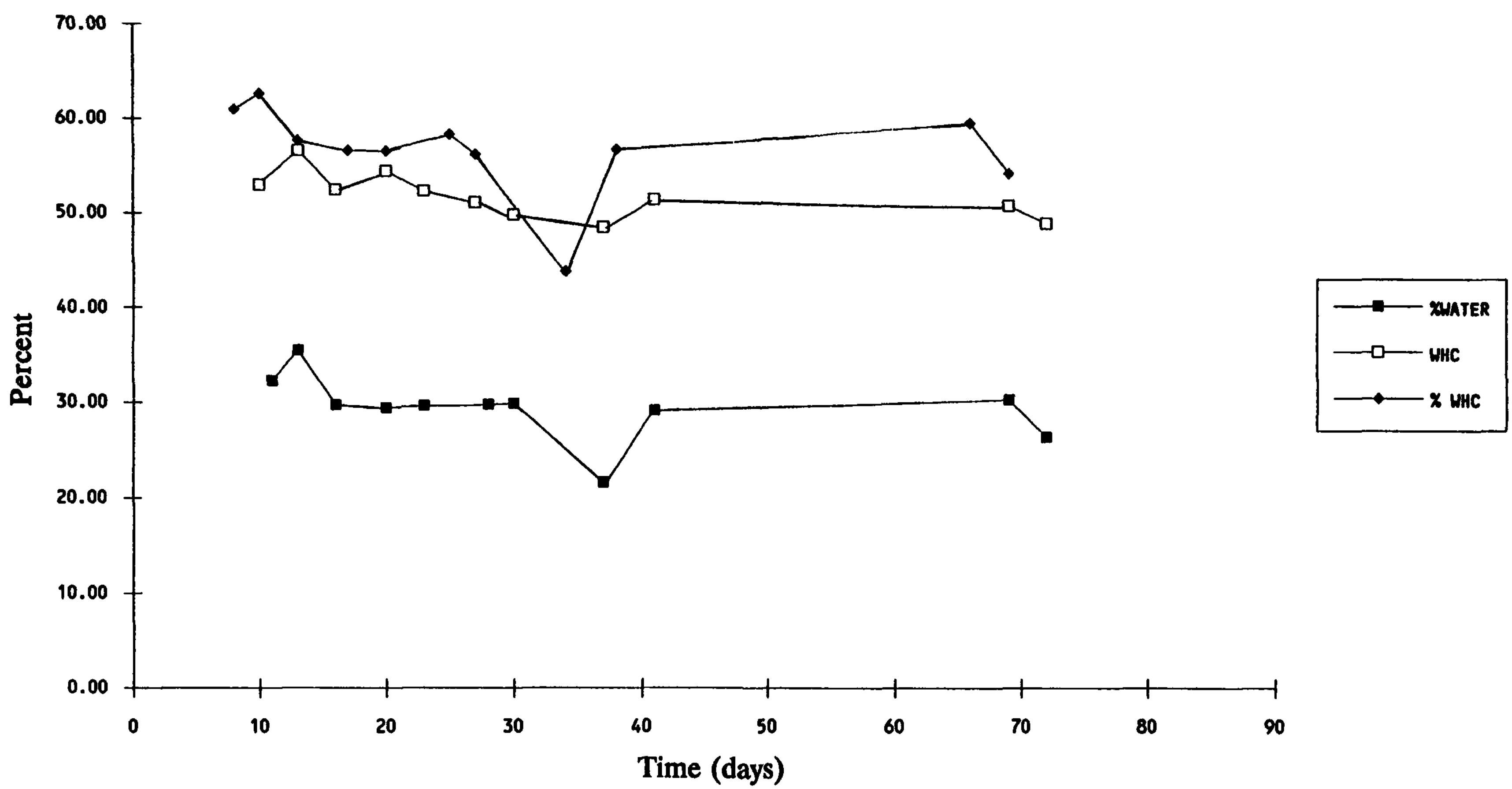


FIGURE 5-40 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR BIOAUGMENTED STATIC PILE TEST SP-4, 80% SOIL

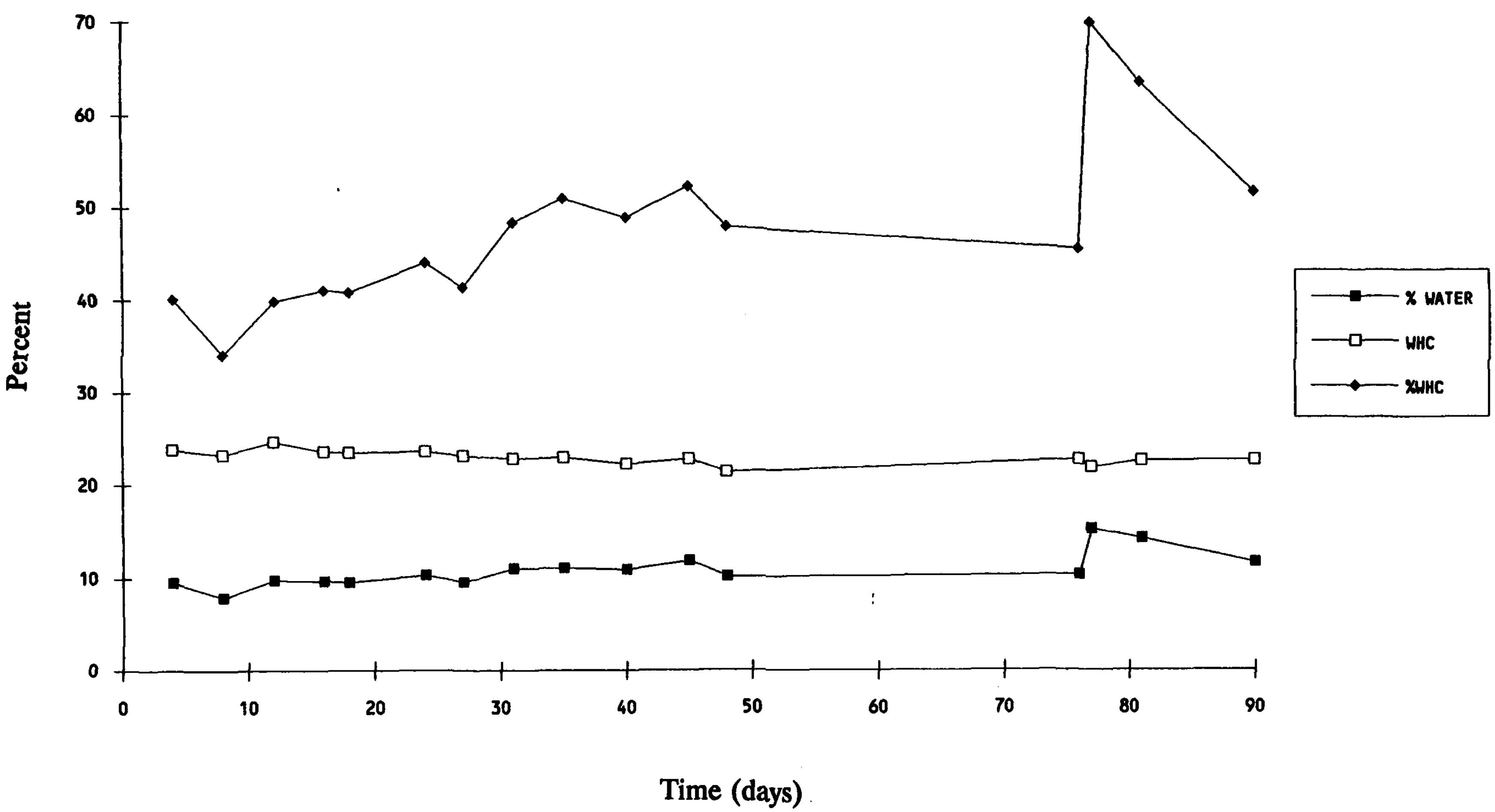


FIGURE 5-41 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-5, 30% SOIL

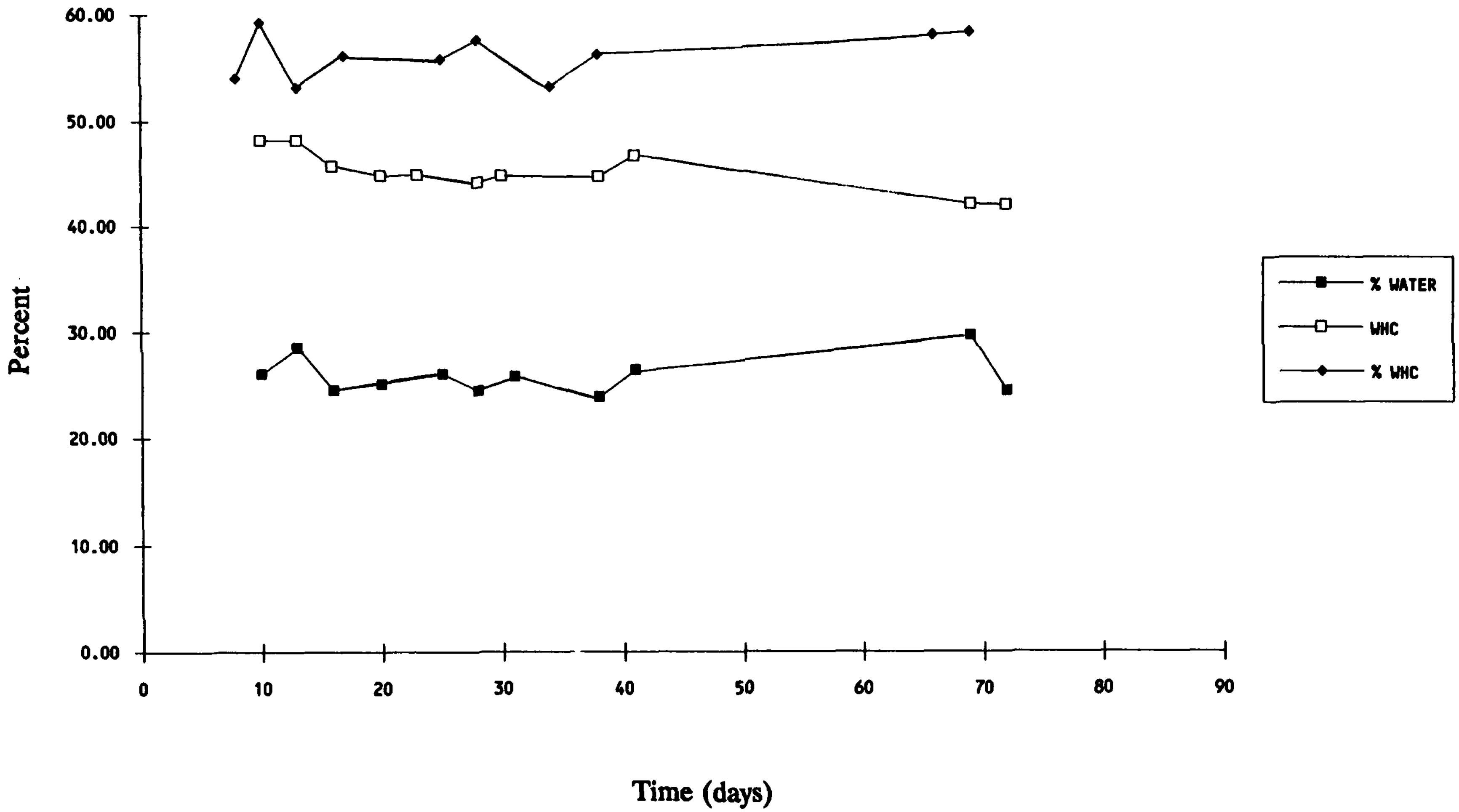


FIGURE 5-42 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-6, 40% SOIL

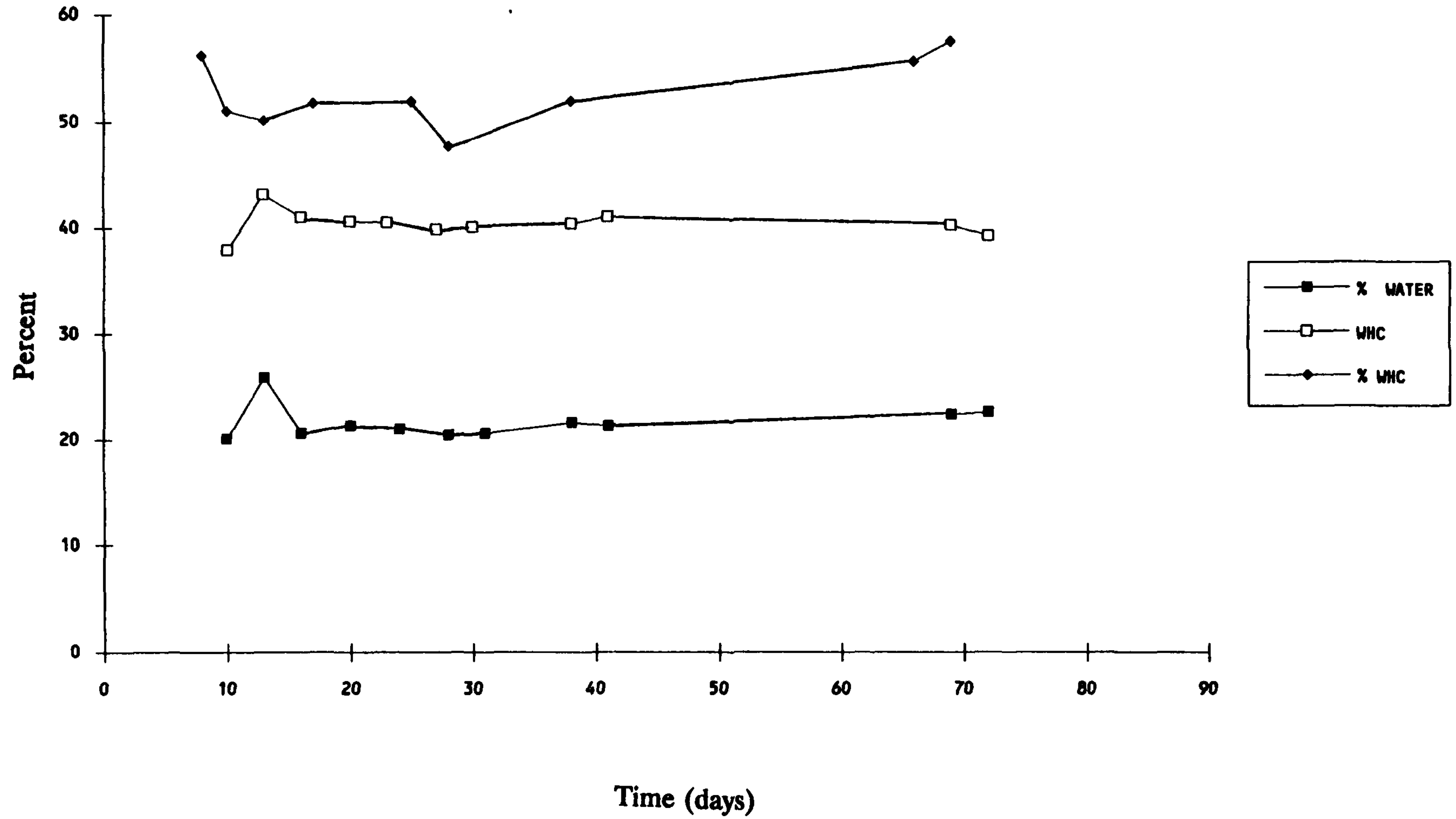


FIGURE 5-43 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-7, 10% UNCONTAMINATED SOIL

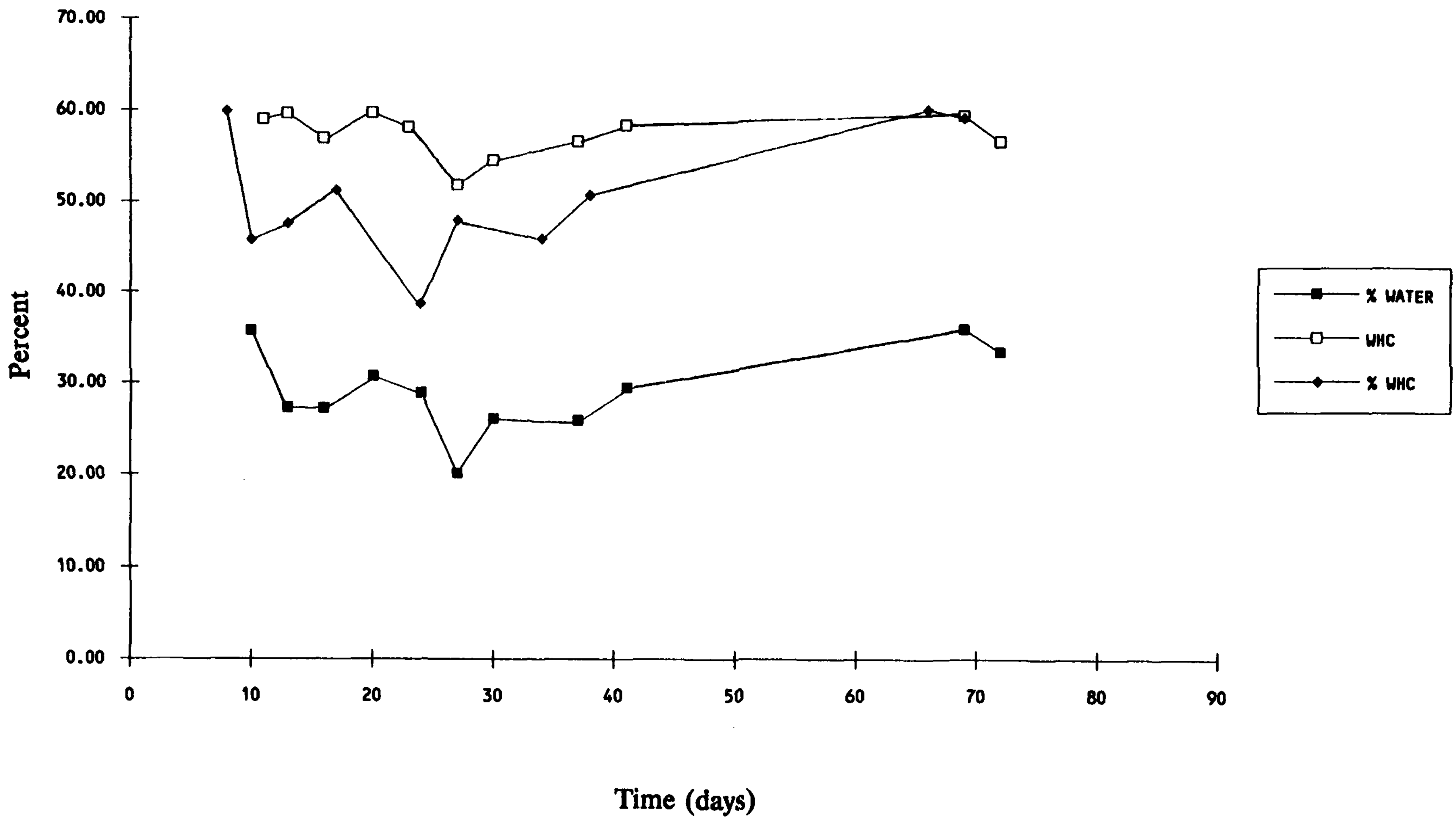


FIGURE 5-44 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR STATIC PILE TEST SP-8, 10% SOIL

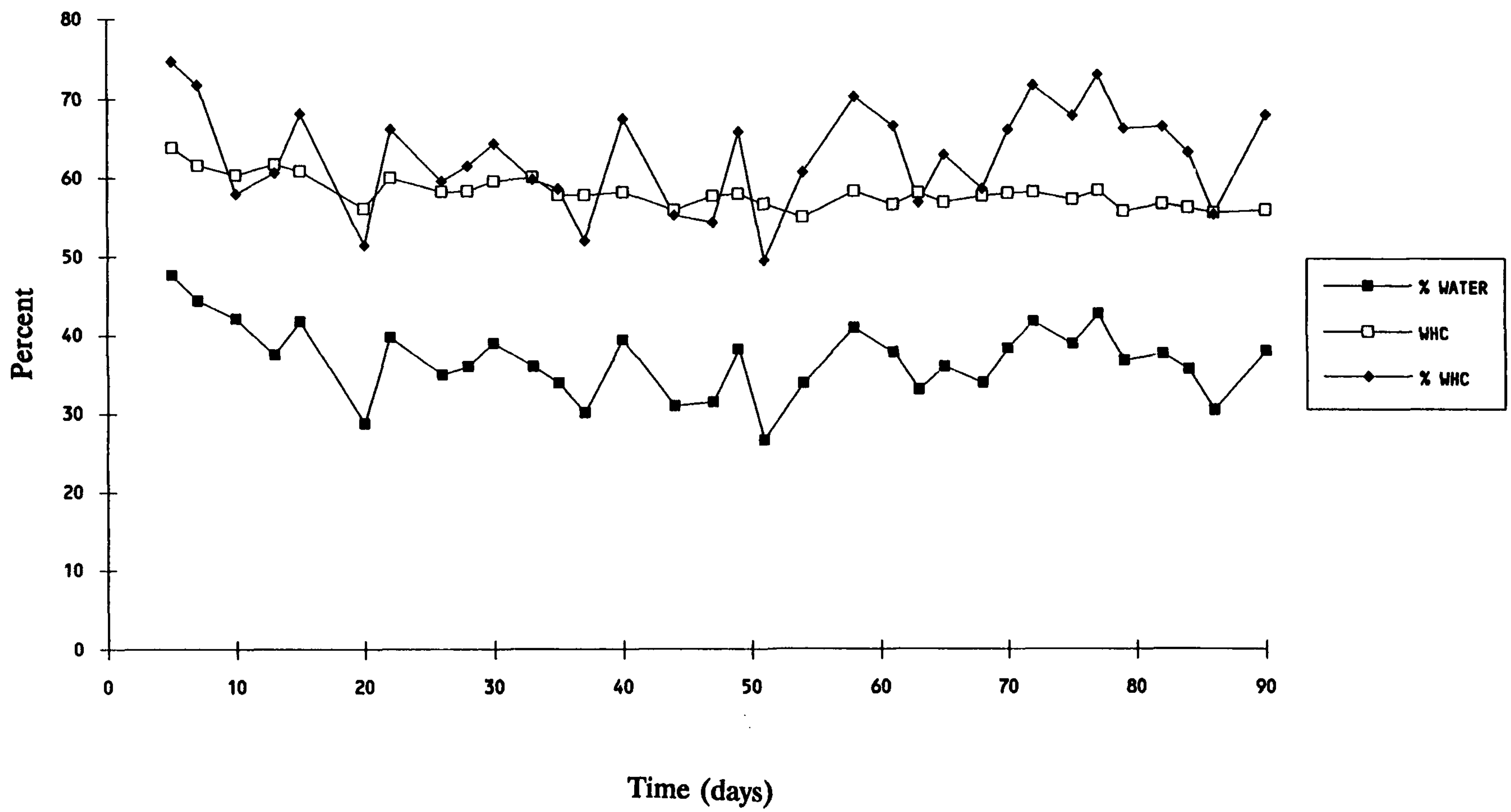


FIGURE 5-45 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR MAIV TEST 1, 10% SOIL

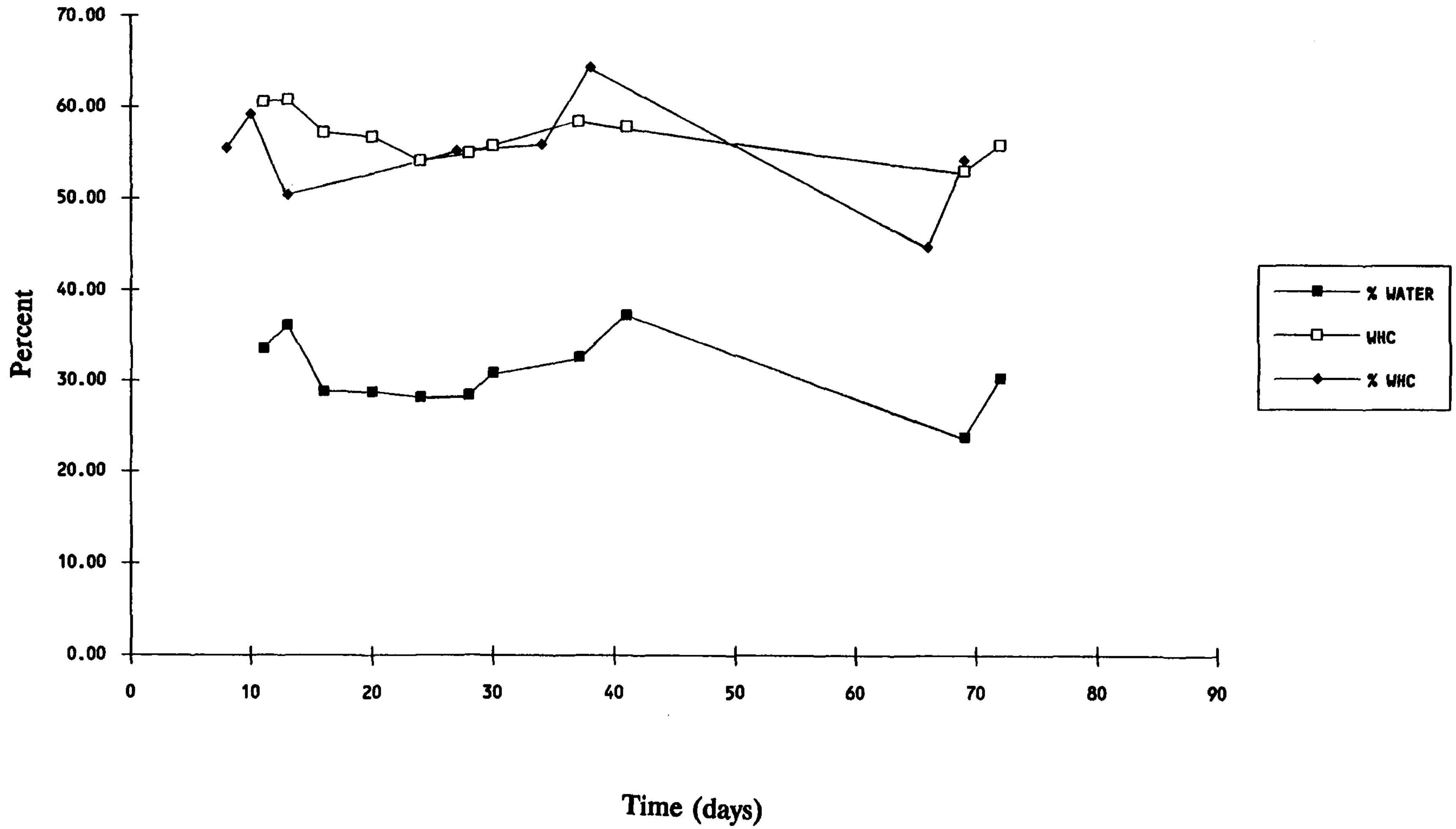


FIGURE 5-46 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR MAIV TEST 2, 10% SOIL

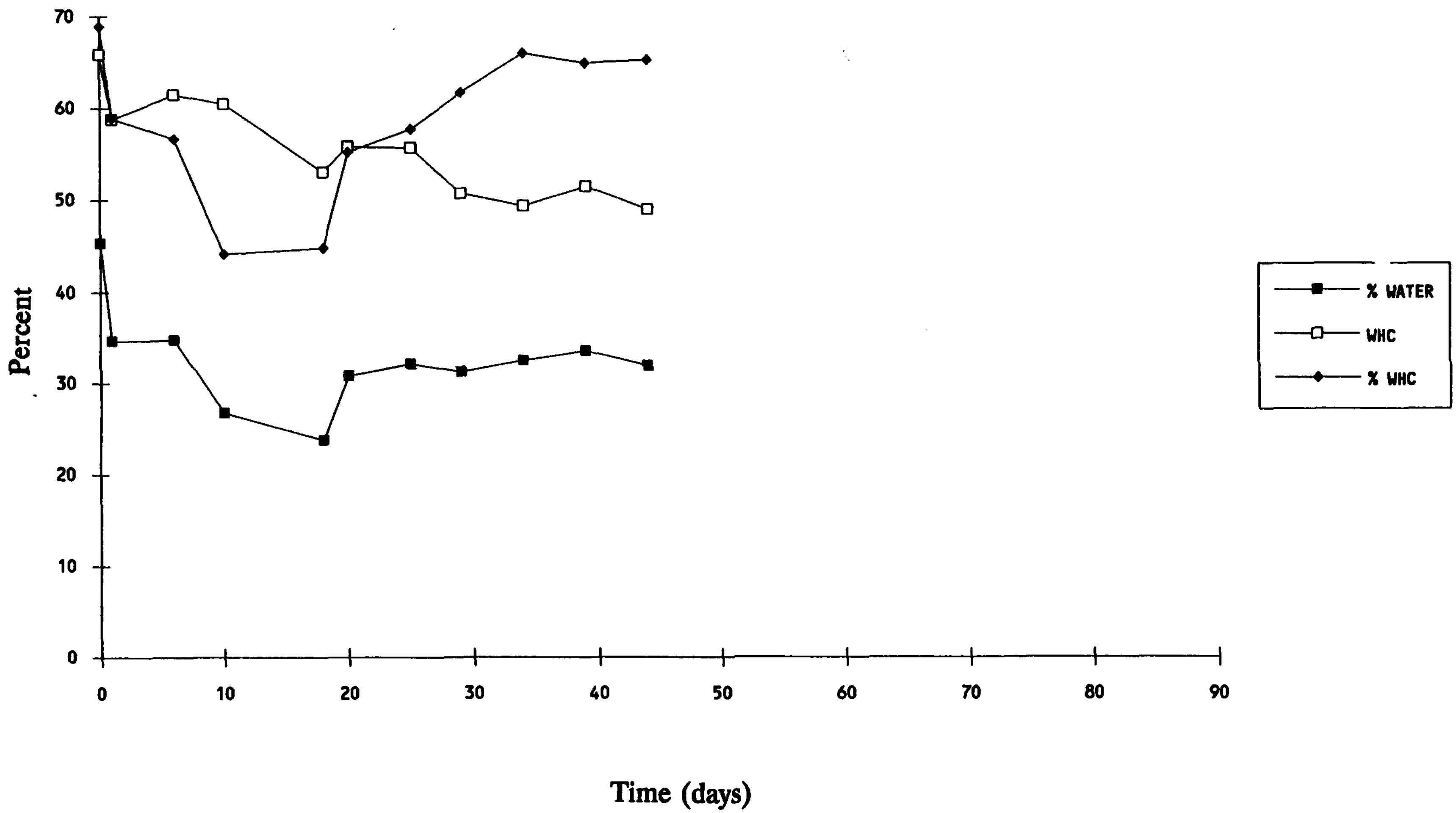


FIGURE 5-47 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR MAIV TEST 3, 25% SOIL

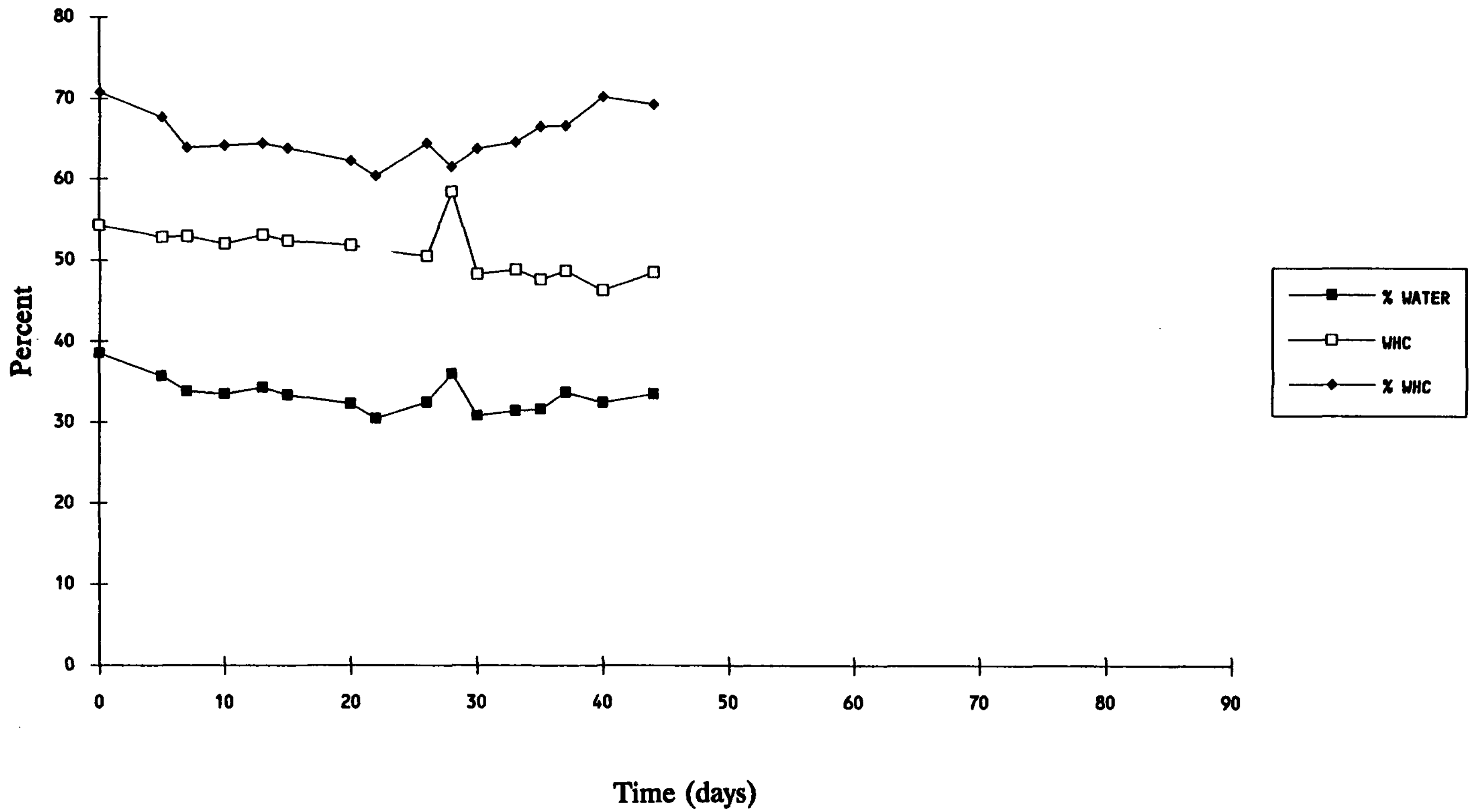


FIGURE 5-48 PERCENT MOISTURE, WATER HOLDING CAPACITY, AND PERCENT OF WATER HOLDING CAPACITY FOR MAIV TEST 4, 40% SOIL

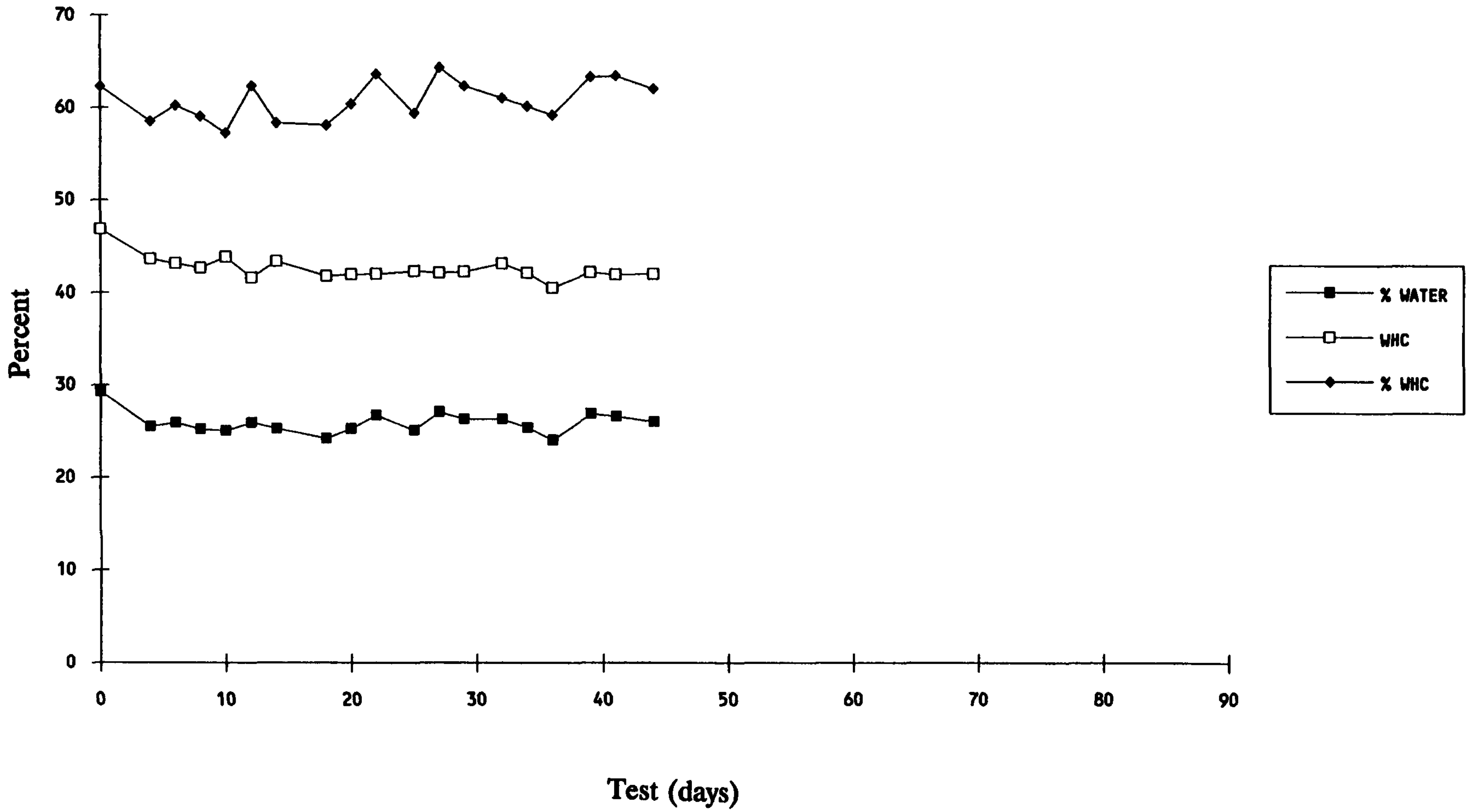


FIGURE 5-49 pH FOR STATIC PILE TEST SP-1, 7% SOIL

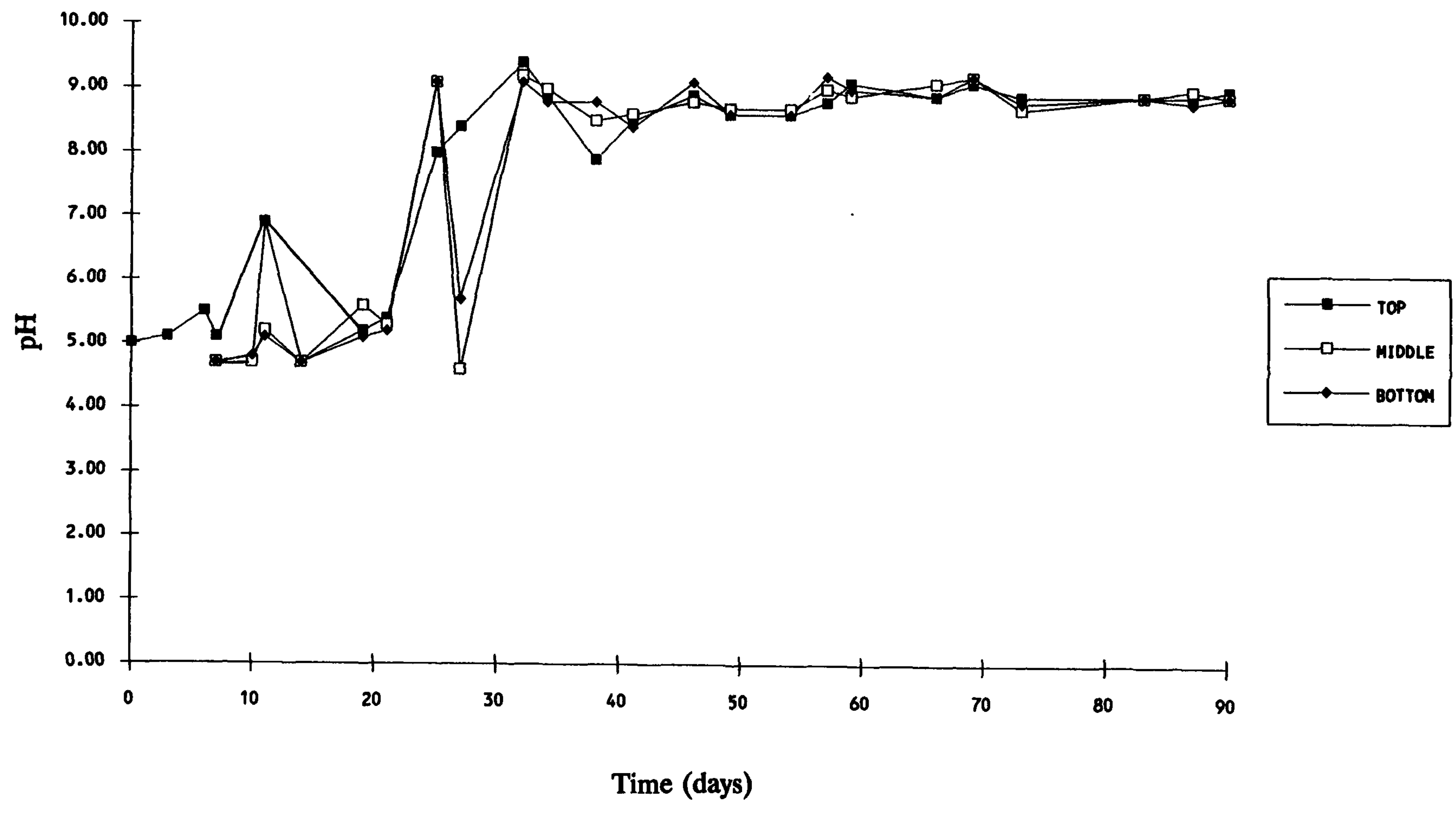


FIGURE 5-50 pH FOR STATIC PILE TEST SP-2, 10% SOIL

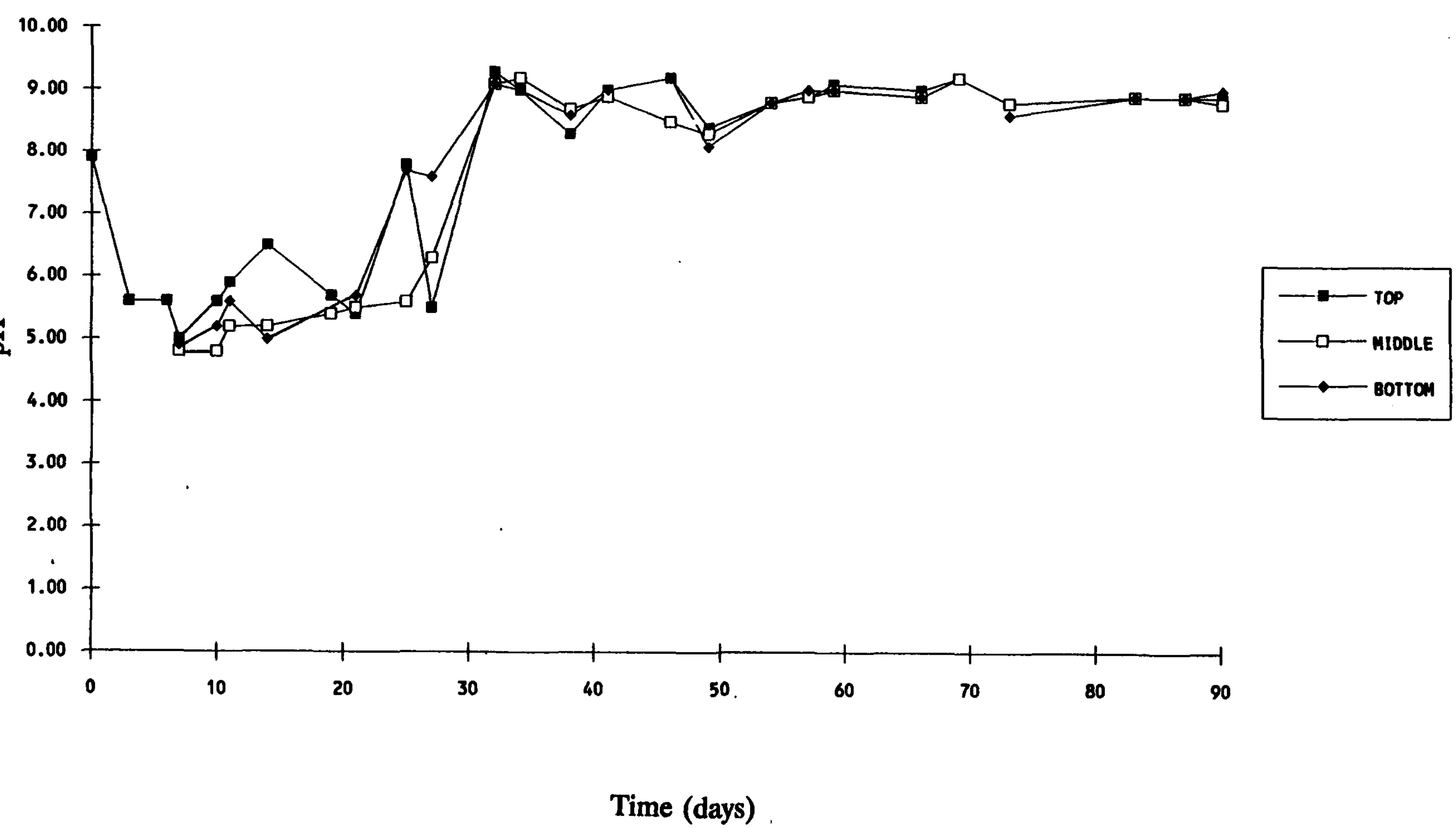


FIGURE 5-51 pH FOR STATIC PILE TEST SP-3, 20% SOIL

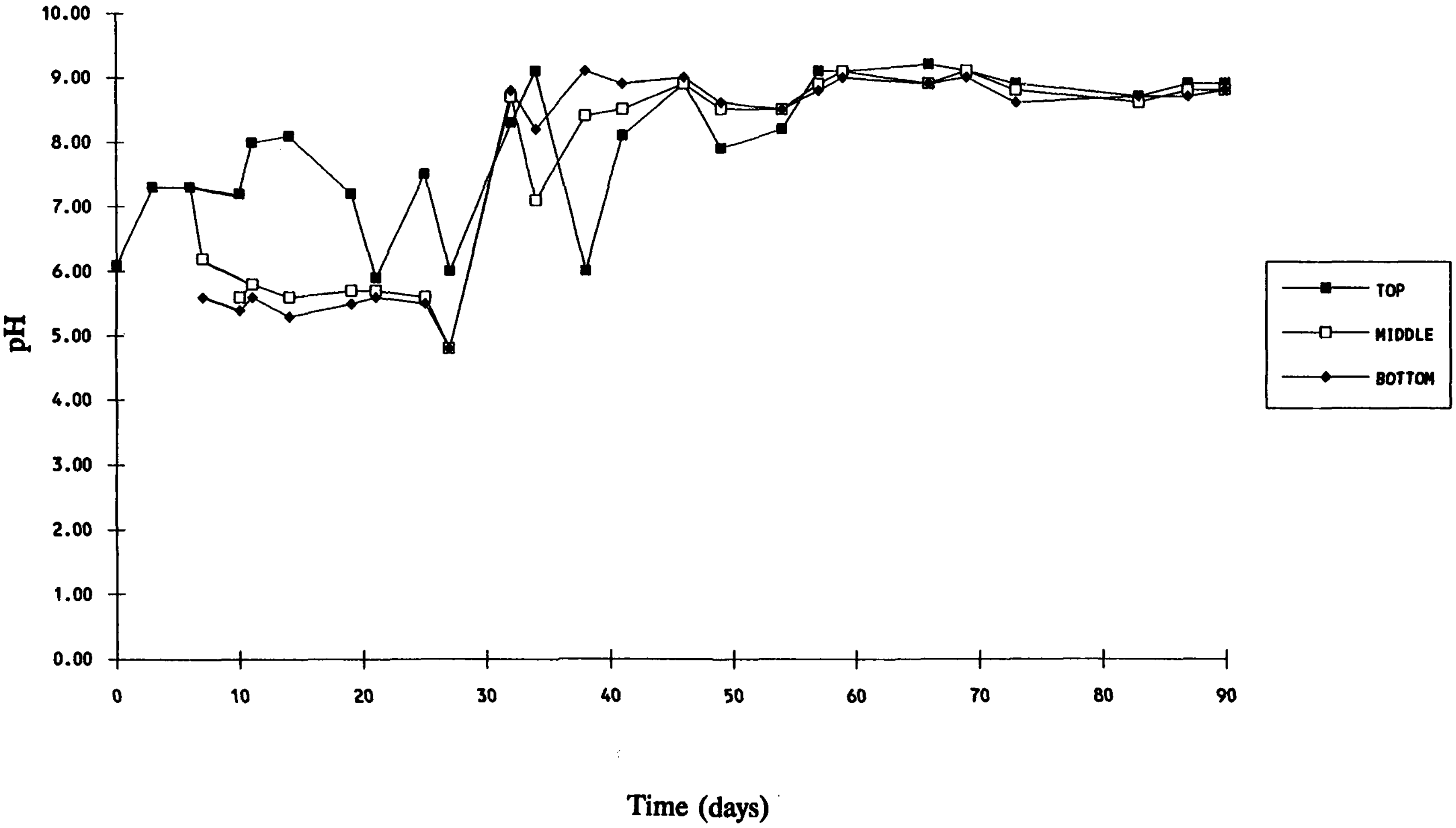


FIGURE 5-52 pH FOR BIOAUGMENTED STATIC PILE TEST SP-4, 80% SOIL

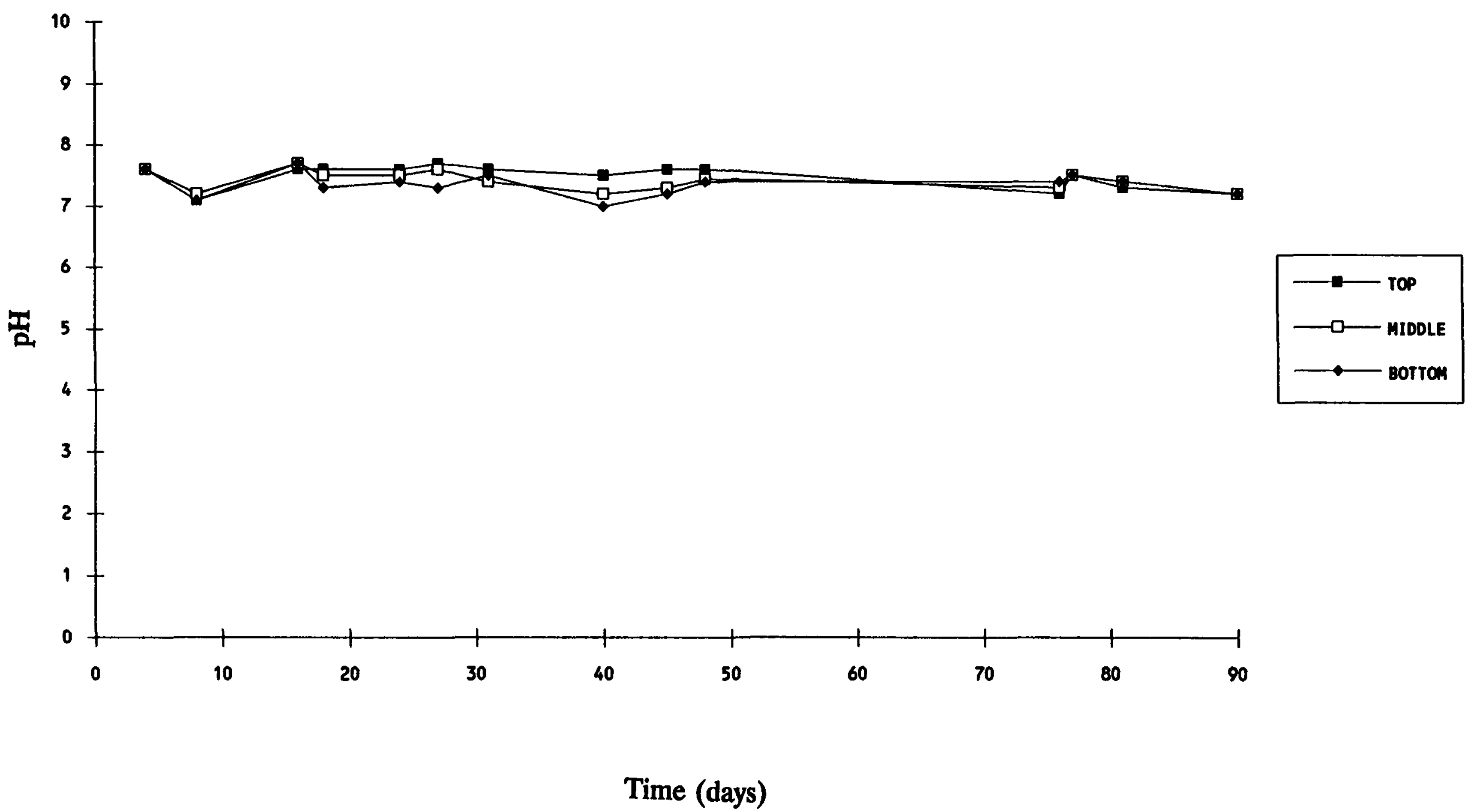


FIGURE 5-53 pH FOR STATIC PILE TEST SP-5, 30% SOIL

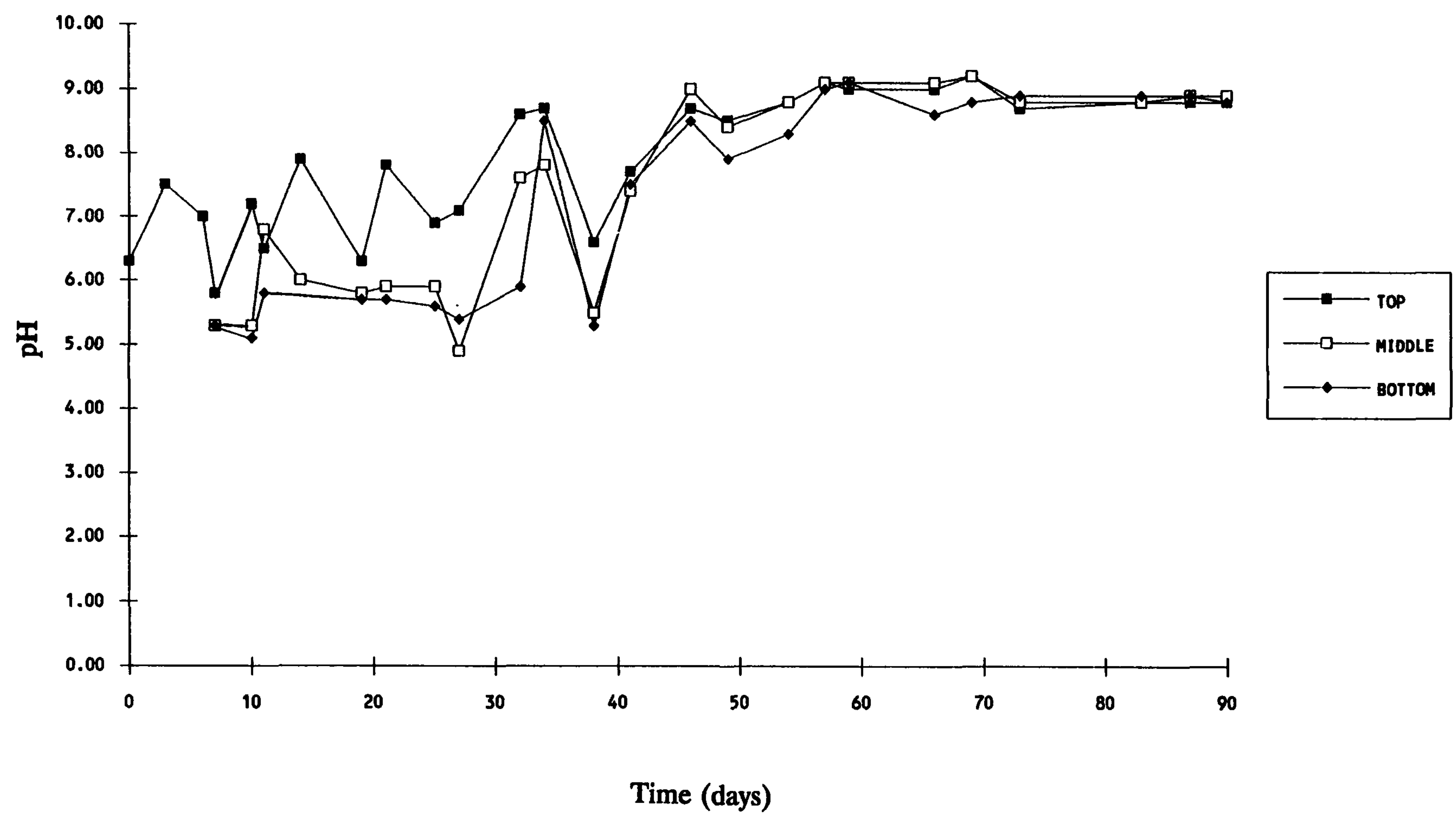


FIGURE 5-54 pH FOR STATIC PILE TEST SP-6, 40% SOIL

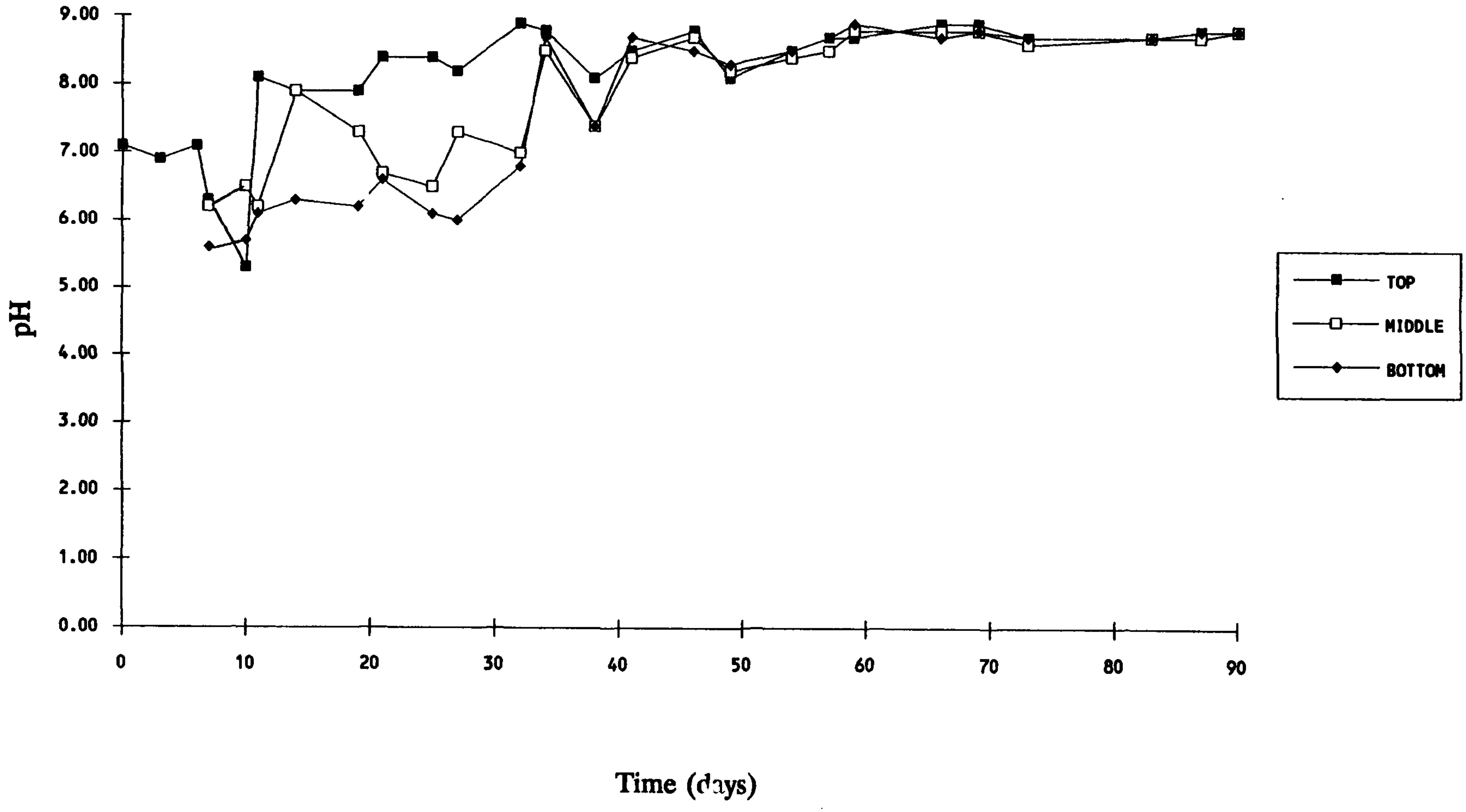


FIGURE 5-55 pH FOR STATIC PILE TEST SP-7, 10% UNCONTAMINATED SOIL

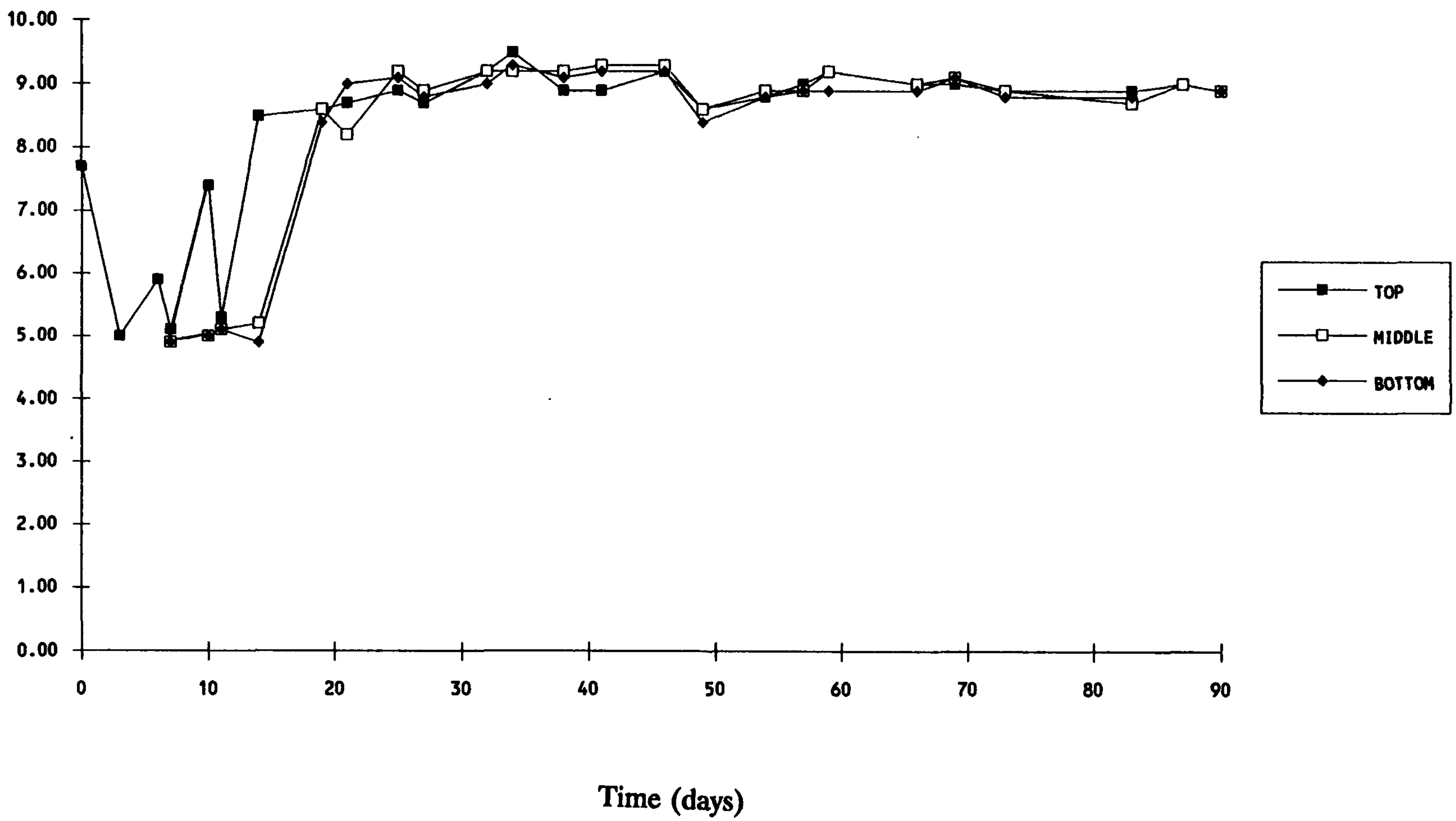


FIGURE 5-56 pH FOR STATIC PILE TEST SP-8, 10% SOIL

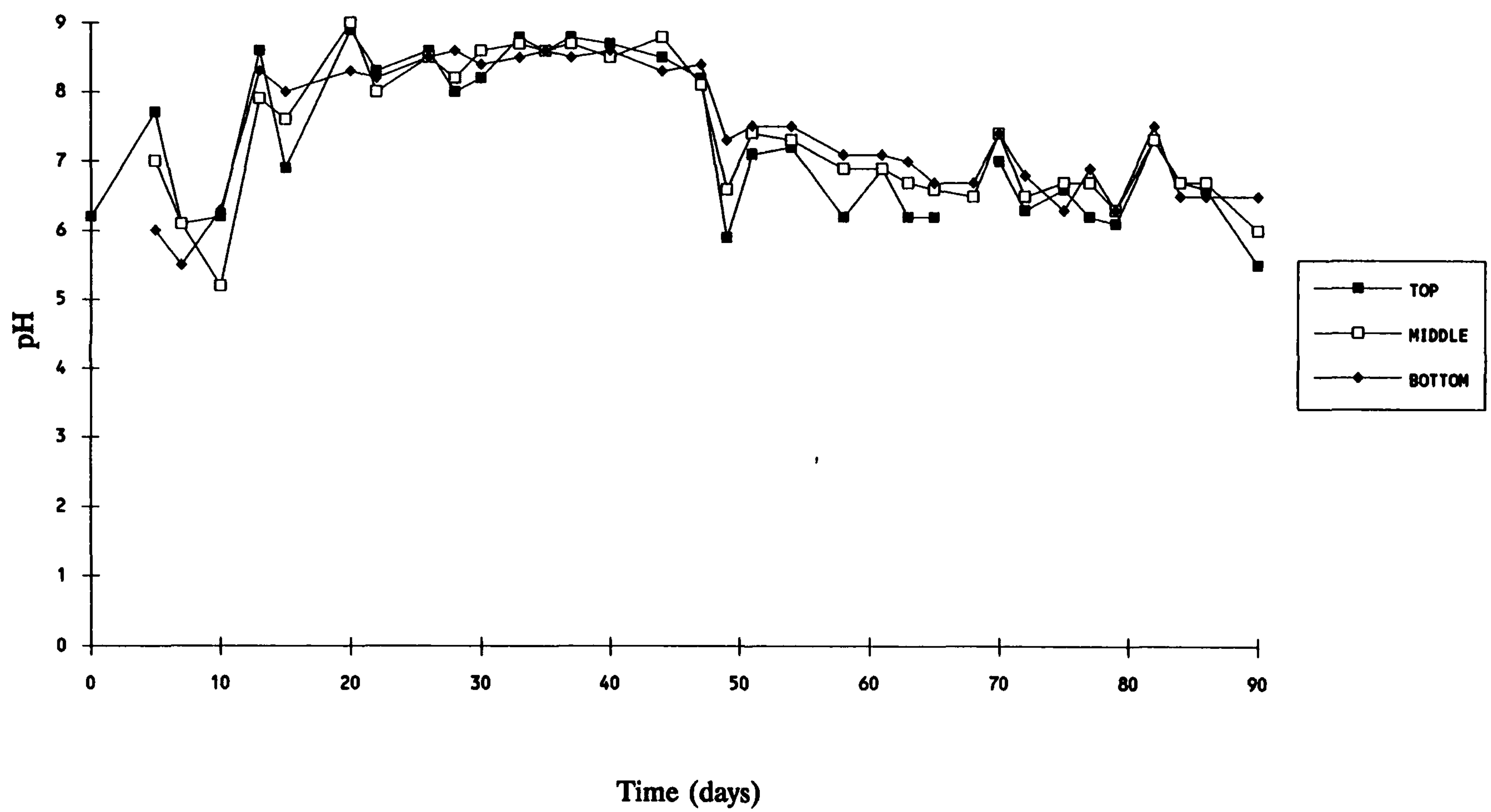
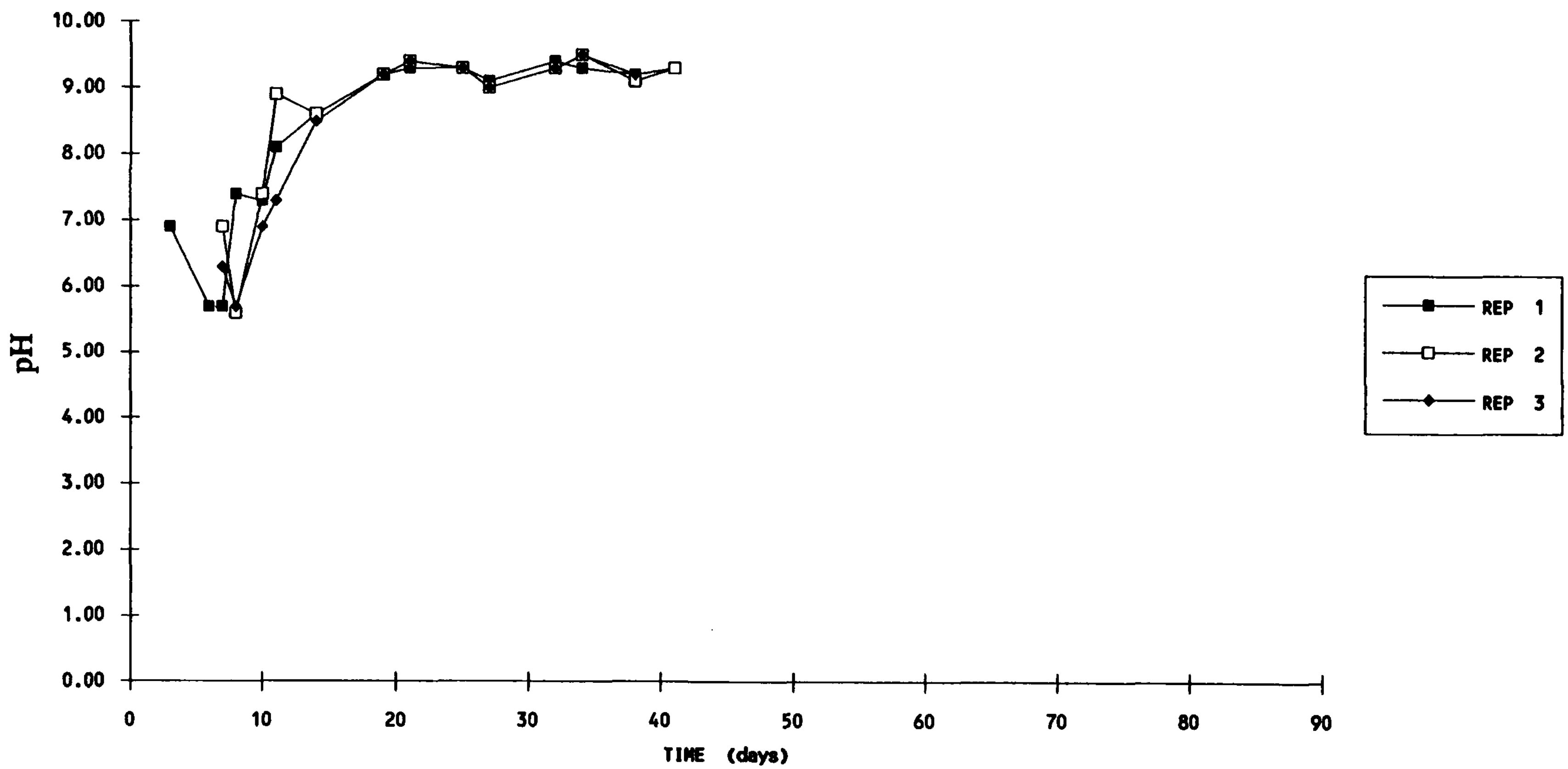


FIGURE 5-57 pH FOR MAIV TEST 1, 10% SOIL



Time (days)

FIGURE 5-58 pH FOR MAIV TEST 2, 10% SOIL

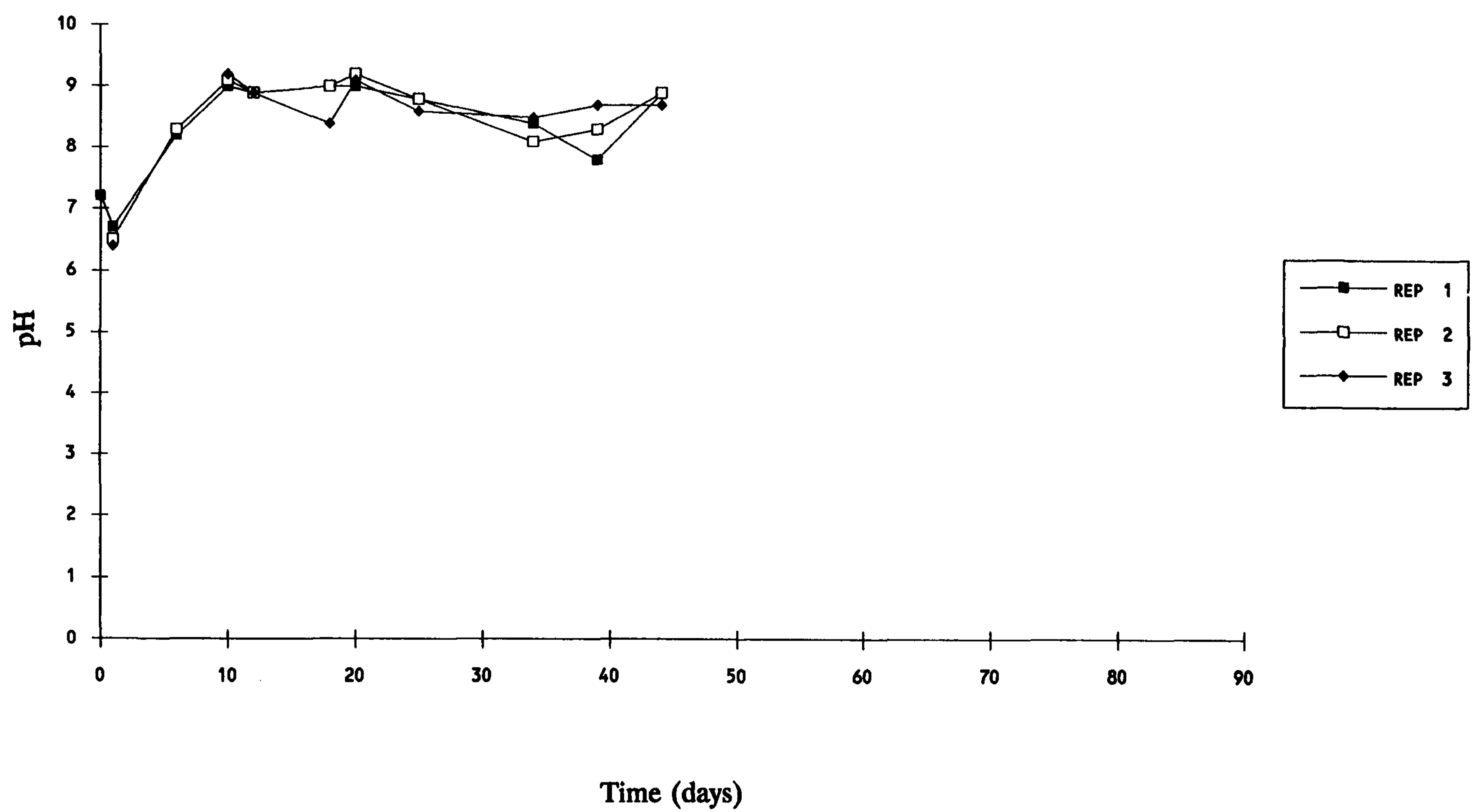


FIGURE 5-59 pH FOR MAIV TEST 3, 25% SOIL

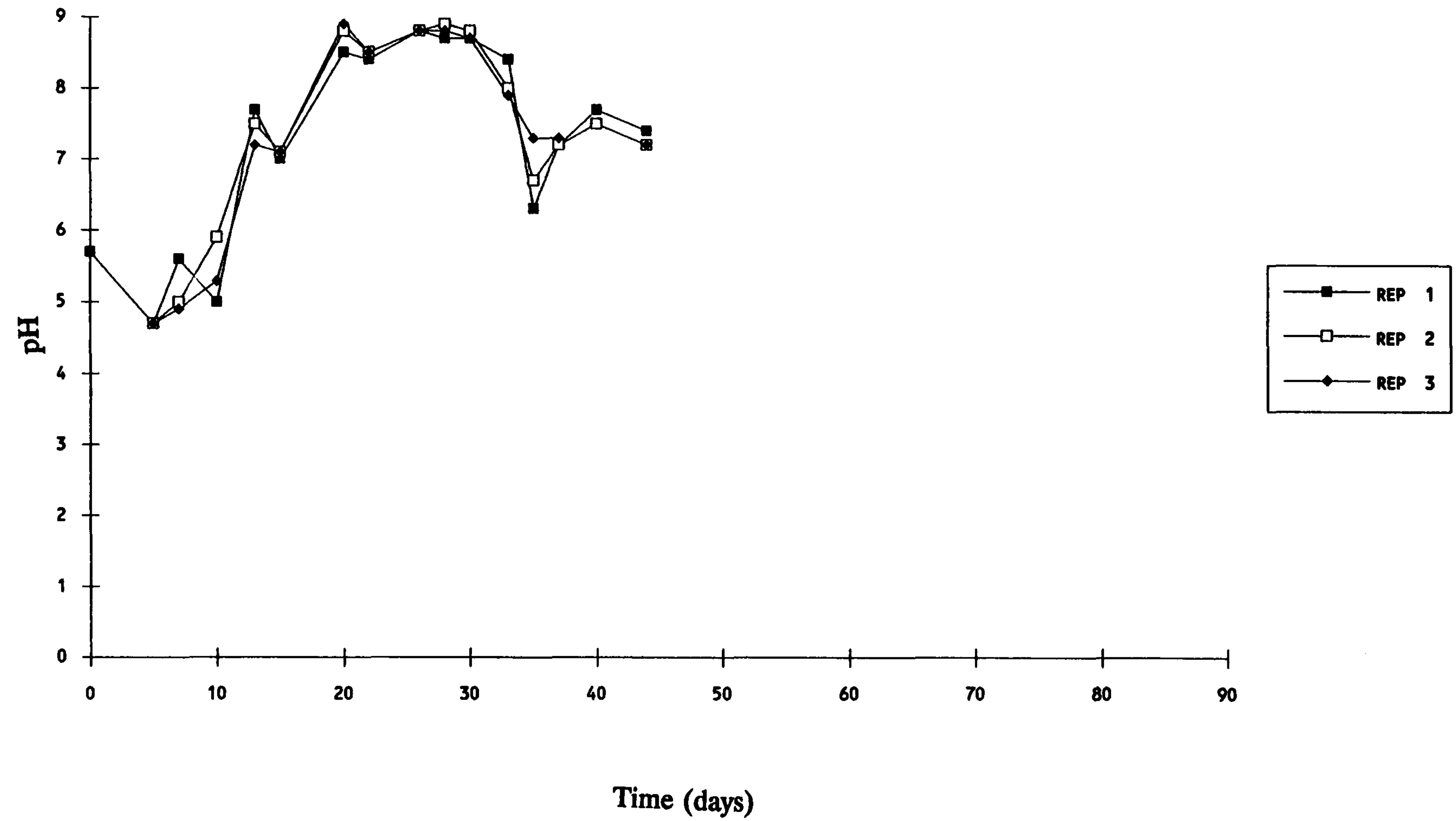


FIGURE 5-60 pH FOR MAIV TEST 4, 40% SOIL

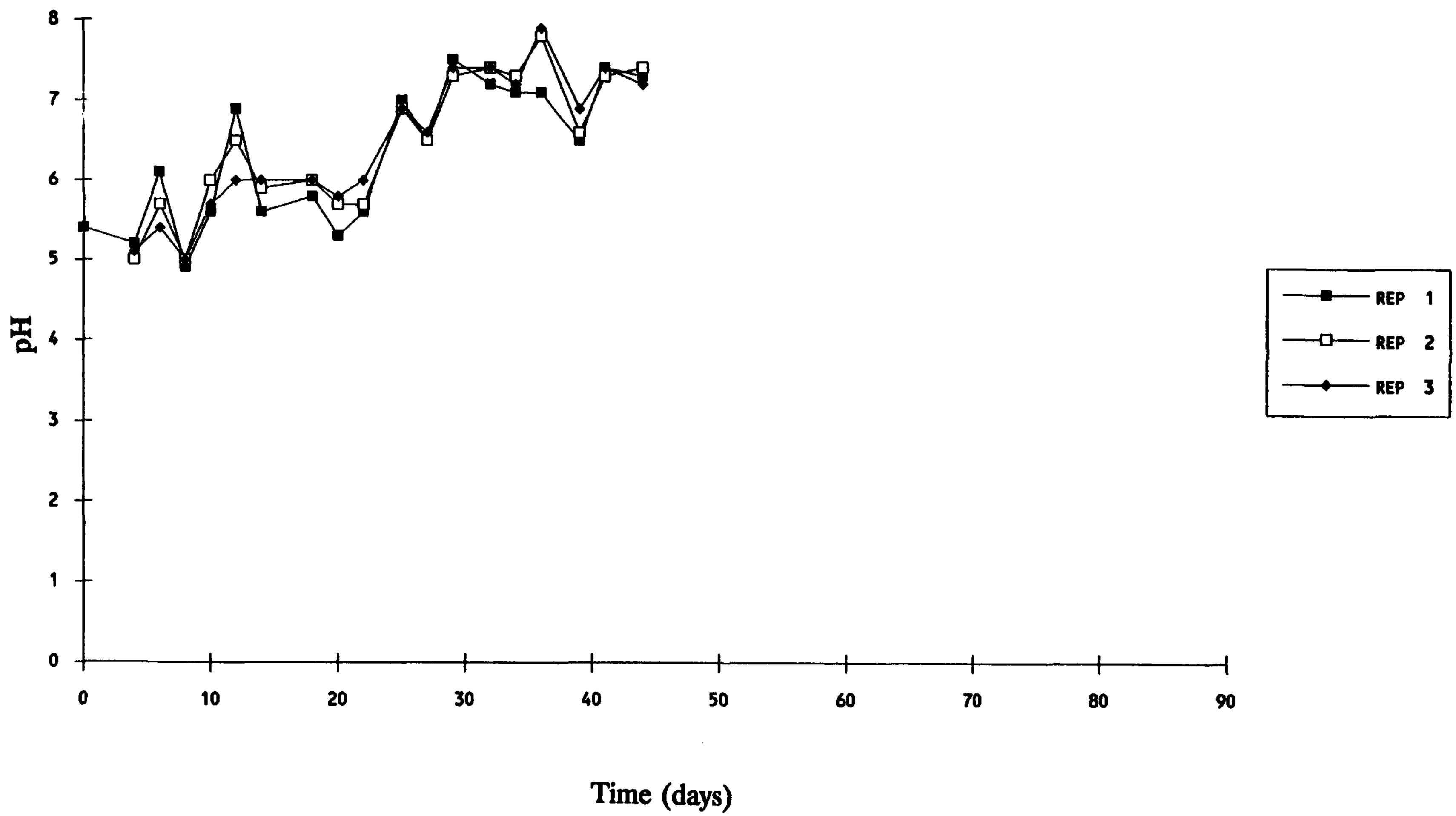


Table 5-17

5-Minute Microtox EC_{50} Data for SP-2, SP-3, SP-7, and MAIV-1

Test	% Soil	Day	Extract Percent Causing 50% Light Output Reduction (EC_{50})
SP-2	10	0	5
		10	8
		20	9
		44	13
SP-3	20	0	5
		10	10
		20	16
		44	24
SP-7	10 (UC)	0	22
		10	75
		20	62
		44	87
MAIV-1	10	0	5
		10	15
		20	21
		44	24
MAIV-2	10	0	18
		10	20

Table 5-18

15-Minute Microtox EC_{50} Data for SP-2, SP-3, SP-7, MAIV-1, and MAIV-2

Test	% Soil	Day	Extract Percent Causing 50% Light Output Reduction (EC_{50})
SP-2	10	0 10 20 44	3 7 11 23
SP-3	20	0 10 20 44	2 10 12 17
SP-7	10 (UC)	0 10 20 44	34 82 -- 82
MAIV-1	10	0 10 20 44	3 18 24 29
MAIV-2	10	0 10	16 23

SECTION 6

DISCUSSION

6.1 DISCUSSION OF INDIVIDUAL PARAMETERS

6.1.1 Explosives Degradation

All three explosives present at UMDA were significantly degraded by composting. In terms of removal percentage, TNT was consistently degraded to the greatest extent. RDX was the next most degraded explosive, except in test SP-6, where HMX degradation exceeded RDX degradation. HMX was degraded to a much lesser extent than either TNT or RDX, again with the exception of SP-6. As discussed in the following subsections, degradation was much more rapid and extensive in the MAIV tests than in the first seven SP tests. No change occurred in explosives concentration in the test inoculated with the LANL microorganisms.

6.1.1.1 Static Pile Tests

This series of tests was designed to determine the effect of soil loading (contaminated soil at 7, 10, 20, 30, and 40%) on degradation kinetics. In addition to the above tests, three other SP tests were conducted. SP-7 (10% uncontaminated soil) was conducted as a background control for the toxicity studies initiated at ORNL. SP-4 was conducted as a test to investigate the potential of an inoculum developed at LANL to degrade TNT, HMX, and RDX. SP-8 (10% contaminated soil) was conducted to examine the fate of ^{14}C-TNT in compost enclosed in a small mesh bag. The bulk compost mixture in SP-8 was prepared using a different amendment mix than the previous SP tests.

As shown in Table 5-1, the degradation percentage generally decreased for HMX and RDX with increased soil loading. The degradation percentage was relatively constant for TNT for soil percentages of 7, 10, 20, and 30%, although the best performance in the first seven tests was achieved at 30% soil. As discussed in other sections, the 30% soil test was not affected by instrument problems to the same extent as the 7, 10, and 20% SP tests. The removal percentage for TNT decreased markedly when soil loading increased from 30 to 40%. The TNT half-life increased steadily with increasing soil loading (from 6.6 days with 7% soil to 24.9 days with 40% soil). Although these trends in removal percentage could be interpreted as a decrease in microbial effectiveness at higher soil loadings, this interpretation is complicated by the increased quantity of explosives present in the mixture as the soil percentage was increased.

The vigorousness of the composting (as determined by compost temperature) in all the SP tests (with the exception of SP-8) was affected by malfunctions in the blower instrumentation system, especially during the first four weeks of the test period. The tests using 7, 10, and 20% contaminated soil were particularly disrupted, as illustrated by the graphs of compost temperature (Figures 5-23, 5-24, and 5-25) for those tests. The temperature profiles for the tests using 30 and 40% soil show much less temperature fluctuation (Figure 5-27 and 5-28). As soil percentage was increased from 30 to 40%, the half-life and reduction percentage data showed decreasing contaminant destruction effectiveness. These data most likely reflect the inability to achieve vigorous microbial activity and thermophilic self-heating at soil loadings equal to or greater than 40%.

The final TNT concentration was not statistically different for 7, 10 (SP-2), 20, and 30% soil tests. The average final concentrations ranged from 107 to 331 mg/kg. For the 40% soil static pile, however, the final TNT concentration achieved was significantly higher (2,086 mg/kg). In SP-8, the final TNT concentration (46 mg/kg) was significantly lower than all the other SP tests except SP-1 (where they were equivalent). For RDX, the final concentration achieved in the 7% soil pile (213 mg/kg) was significantly less than that achieved in the 10 (SP-2), 20, and 30% piles. The final RDX concentration in SP-8 was significantly less than that achieved in the 7% soil pile (SP-1). The final RDX concentrations in the 10 (SP-2), 20, and 30% soil piles did not differ significantly from each other, although they were significantly lower than the final RDX value in the 40% soil pile. The significant differences in the final HMX concentration followed a pattern similar to that of RDX.

The majority of the explosives degradation occurred during the most active composting phase. This generally corresponded to the first 6 weeks of the test.

6.1.1.2 MAIV Tests

The first two MAIV tests were designed to examine the effect of different amendments on explosives removal. Overall, the mix for MAIV-2, which was approximately the same mix used in the LAAP field demonstration (Williams et al., 1988), performed best in terms of contaminant destruction. The differences in reduction percentage and half-life for TNT for the two mixes are insignificant. However, the final TNT concentration (5.6 mg/kg) achieved using the mix for MAIV-2 was much lower than that achieved for the mix used in MAIV-1 (90 mg/kg). For RDX and HMX, the differences in reduction percentage, half-life (which could not be calculated for HMX), and final concentration all demonstrated that the mixture used in test MAIV-2 was superior.

The half-life, reduction percentage, and final concentrations all illustrate that contaminant degradation in both 10% soil MAIV tests was superior to that achieved in the 10% soil SP-2 test. SP-8 was also conducted with 10% soil. However, SP-8 composted much more effectively than SP-2 (compare Figures 5-24 and 5-30), primarily because of instrumentation difficulties during the SP-2 test. Comparing the explosives reduction data for SP-8 with that from MAIV-1 and MAIV-2 illustrates the importance of amendments. SP-8 performed slightly better than MAIV-1, but not as effectively as MAIV-2.

In spite of the use of 25% soil instead of 10% soil, the reduction percentage, half-lives, and final concentration achieved in MAIV-3 were generally significantly better than, or at least comparable to, the results achieved in MAIV-1. The destruction achieved with the mixture used in the 25% soil MAIV test was comparable to that achieved in MAIV-2 (LAAP mixture) with the exception of HMX, which was reduced to a much greater extent with the mixture used in MAIV-2. The differences in explosives removal between 10 and 25% soil in the MAIV system appear to be more a function of amendment composition than of soil loading.

MAIV-4 (40% soil) used the same amendments in the same ratio as those used in the mixtures tested in SP-8 and MAIV-3. The percent reduction of TNT in MAIV-3 (99%) and MAIV-4 (97%) were comparable. The final TNT concentration in MAIV-3 (14 mg/kg), however, was much lower than that achieved in MAIV-4 (209 mg/kg). HMX and RDX concentrations were essentially unchanged in MAIV-4, whereas they were significantly and extensively destroyed in MAIV-3. This illustrates that despite the use of an effective amendment mixture, high soil loadings have the potential to inhibit explosives degradation.

6.1.1.3 Los Alamos National Laboratory Bioaugmented Test

No significant changes occurred in TNT, RDX, or HMX concentration in this test. A variety of factors may have been responsible for this failure. The data necessary to identify the specific reason that no degradation was observed, however, does not appear to exist.

The reasons for failure of the explosives to degrade in this test could fall into a number of categories, including: (1) ecological factors, such as the inability of the added microorganisms to compete with, or survive in the presence of, the native microflora; (2) biochemical factors, such as the lack of enzymes required to metabolize the explosives; (3) toxicity, caused either by the explosives themselves or by other factors present in the UMDA soils; (4) chemical factors, such as inappropriate pH, oxygen tension, moisture, nutrients, pH, temperature, etc.; and (5) substrate (explosive) inaccessibility.

No background information was available to evaluate the possible effects of ecological factors on the survival and metabolism of the inoculum in UMDA soils prior to the test. Samples from SP-4 were sent to LANL regularly after day 10 of the test period for enumeration of the inoculum. WESTON has not seen these data, but has been told that inoculum organisms were present at the end of the test period. This indicates that the inoculum organisms were able to survive, but does not indicate how active they were.

Biochemical factors cannot be dismissed as a possible explanation. The inoculum has been demonstrated to mineralize TNT in laboratory studies conducted at LANL. However, the rate and extent of explosives mineralization achievable at the concentrations present in UMDA soil have not been determined.

Toxicity from either the explosives or various soil factors present in the UMDA soil (including background UMDA soil) does not appear to have been investigated. Consequently, the impact of this factor cannot be evaluated.

Chemical factors are a likely explanation for failure. However, insufficient data exist to pinpoint a specific cause. The optimum conditions for the inoculum apparently have not been determined. Even if optimal pH, moisture, oxygen, etc. were known and could be created in the UMDA soil, it is still not clear that the inoculum could survive and metabolize in UMDA soil. Although temperature was not at the mesophilic optimum during the test, temperature is unlikely to be the sole explanation for no observable degradation. Some degradation should have occurred at the test temperature, although at a lower rate than would have been observed at a higher temperature.

Finally, it is not clear whether the inoculum had the capability to degrade "aged" explosives (explosives present in soils for a prolonged period of time) compared with the TNT used as an added substrate in LANL laboratory experiments. Interaction with soil fractions may have rendered the explosives in the UMDA soil unavailable to the inoculum. All previous investigations appear to have been based on ^{14}C-TNT added to laboratory experiments.

6.1.2 Temperature

6.1.2.1 Ambient

The UMDA winter of 1990/91 was one of the coldest in the last 20 years, with temperatures reaching -22°F at one point. At the other extreme, temperatures exceeded 110°F during the summer. These temperature extremes disrupted the normal operation of the temperature control instrumentation. In addition, extreme temperatures occasionally affected compost temperature directly.

6.1.2.2 Static Pile Tests

The temperatures achieved and maintained in the first set of static piles (SP-1, SP-2, SP-3, SP-5, SP-6, and SP-7) were affected by malfunctions of the control instrumentation. The system did not meet the specifications requested of the supplier when installed in the field and required troubleshooting during the first set of static piles. Temperatures rose quickly in 7, 10, and 20% soil mixtures prior to the occurrence of problems in the control system. These problems caused excessive blower operation on some occasions and inadequate blower operation on others. The 30 and 40% soil mixtures heated more gradually but also more steadily and were affected less by the instrumentation problems.

The temperatures within this first set of piles never maintained the desired 50°C-plus thermophilic temperatures. Although this could be attributed to the amendment mixture, it was more likely caused by malfunctions leading to excessive operation of the temperature control system (blowers). Although ambient temperatures steadily declined during the test period for SP-1, SP-2, SP-3, SP-5, SP-6, and SP-7, it is unlikely that this exerted a controlling influence over the temperature within the static pile reactors. The heating and temperature maintained within the 30 and 40% soil reactors is particularly impressive considering the high percentage of soil.

SP-4, which contained 80% soil and the LANL inoculum, maintained temperatures roughly comparable to ambient conditions. Because of the large mass of inorganic material in SP-4, the temperature did not shift rapidly or reach the extremes of the ambient temperature. There was no expectation that SP-4 would self-heat in a manner similar to that of the other composting systems. The ratio of readily biodegradable biomass to inorganic mass (which represented a large heat sink) was far too low for significant self-heating.

The temperature in SP-8 rose quickly and maintained good thermophilic composting for approximately 3 weeks. The success of the composting process in this test, as compared with the results of the first set of static tests, appears to be primarily a function of the temperature control system, which operated without malfunction during the SP-8 test. In addition, the amendment mix used in SP-8 (as well as MAIV-3 and -4) composted quite well. The temperature drop in SP-8 following day 21 correlates well with a drop in ambient temperature. It is likely that by day 21 a significant amount of the organic material in the composting mixture had been degraded, and consequently, the intensity of the microbial activity after this period was not sufficient to maintain thermophilic temperatures against decreasing ambient conditions. When ambient temperatures increased, the temperature in SP-8 did as well. The SP reactors held approximately 3 yd^3 of material. It would not be surprising for this small quantity of material to be influenced by ambient temperatures following the depletion of most of the readily utilizable organic matter.

6.1.2.3 MAIV Tests

The temperature in MAIV-1 rose gradually over the first 12 days of the study until temperatures in excess of 50°C were achieved. These thermophilic temperatures were maintained for only a few days before temperature declined rapidly to ambient. Part of the problem in the initial stages of the MAIV-1 test can be attributed to fine-tuning the instrumentation in the MAIV unit. This test served as the initial run in the MAIV reactor. Ambient temperatures for this test were high during the initial stages of the test.

MAIV-2 heated rapidly but then cooled at approximately day 10. This cooling correlates with a period of low ambient temperature. However, after day 10, the MAIV temperature climbed back to the 50°C range despite decreasing ambient temperatures as the test progressed into December.

The temperature decline in MAIV-2 following day 20 most likely corresponds to a decrease in the quantity of available carbon, rather than demonstrating an effect of the cool ambient temperatures. The temperature in all four of the MAIV tests began a gradual decline starting at approximately day 20. These first 20 days also correspond to the period of maximum explosive degradation.

Compared with MAIV-2, MAIV-3 heated more gradually to temperatures in excess of 50°C. This probably can be explained by the additional heat required to warm the larger inorganic mass (25% soil) in MAIV-3 compared with MAIV-2 (10% soil). However, MAIV-3 maintained temperatures in excess of 50°C for a much longer period (approximately 13 days) than any of the other MAIV tests. The longer period of higher temperature in MAIV-3 most likely resulted from continued microbial activity rather than from more gradual cooling of the higher soil mass. The ambient temperatures during MAIV-3 were moderate and probably did not affect the MAIV-3 internal temperatures significantly.

MAIV-4 heated quickly to over 40°C and maintained a temperature in the 42 to 48°C range for a period of approximately 3.5 weeks. It is likely that the large mass of soil significantly impeded the mixture's ability to self-heat to thermophilic conditions or to exhibit significant temperature fluctuations. This conclusion is supported by the fact that the same amendment mixture was used for both MAIV-3 (which achieved higher temperatures) and MAIV-4. The ambient temperatures during the MAIV-4 test were moderate and probably did not affect the test mixture temperatures.

6.1.3 Moisture

The effect of soil on the water-holding capacity of compost mixtures is a key consideration in the preparation of mixtures for composting. Soil has much less water-holding capacity than organic matter. Consequently, the higher the soil fraction, the lower the water-holding capacity. The progressive decrease in water-holding capacity with increasing soil content can be seen in the appropriate figures. At 7% soil, the water-holding capacity at time 0 is approximately 65%, but this decreased to approximately 40% with 40% soil in the mixture. All four of the 10% soil mixtures had roughly the same water-holding capacity despite differences in the amendment mixtures used. The two 40% soil mixtures also had approximately the same water-holding capacity despite differences in the amendment mixture.

In the mixtures with less than 40% soil, a gradual decline in water-holding capacity was observed as the test period progressed. This decrease reflects the increasing ratio of soil to organic matter as the organic matter is metabolized during the test.

Moisture percentage was generally in the 30 to 40% range. This would be considered low if moisture content in these composting tests was based on moisture percentage. However, as explained in Subsection 5.3, moisture percentage cannot be used as a guideline parameter where soil represents a significant percentage of the overall mixture. The percentage of water-holding capacity was used as the key measurement for adjusting water content. The percent of water-holding capacity fluctuated somewhat within each composting test but generally remained within the desired 50 to 65% range.

Initial moisture in the LANL test was adjusted in the field under the direction of Dr. Unkefer. No predetermined operating range for moisture content was provided to WESTON. Consequently, WESTON maintained the initially established conditions of this test as closely as possible.

6.1.4 Oxygen

The data indicate that during periods when the temperature control system was operating properly, all piles received adequate oxygen. During malfunctions in the system, pH data (Subsections 5.5 and 6.5) indicate that inadequate oxygen was present.

Even in well oxygenated compost, the central regions of actively metabolizing particles larger than 0.5 to 1 inch may become oxygen-depleted. Anaerobic metabolism could occur in such regions. In general, the compost mixtures prepared at UMDA had particle sizes of less than 0.5 inch.

6.1.5 pH

The pH in SP-1, SP-2, SP-3, SP-5, SP-6, and SP-7 decreased in the first two to three weeks of the test period to a range of pH 5 to 6. This decrease was more severe in the middle and bottom samples than in the top samples. The severity of this drop is most likely a reflection of the malfunctioning temperature control system. Inadequate oxygenation would result in anaerobic metabolism and the production of organic acids, which would result in lower pH. An analysis of compost from the bottom regions of these tests showed high concentrations of organic acids. After the oxygenation was corrected by fixing the blower/instrumentation system, the pH rebounded to the pH 8-9 range, where it stayed for the remainder of the study. This elevated pH is a function of ammonification within the compost, which results in the release of ammonia.

In SP-8, where no instrument malfunctions occurred and a different amendment mixture was used, the pH dropped initially to a range of pH 5.5 to 6 in the bottom and middle of the pile. The pH rose quickly however, and remained in the range of pH 7 to 7.5. The initial pH drop probably reflects production of organic acids and/or significant quantities of carbon dioxide from degradation. The presence of carbon dioxide would result in the formation of carbonic acid and a slight lowering of pH. The more moderate upper pH in SP-8, as compared with that in the other SP composting tests, most likely reflects differences in ammonification. The latter would be controlled by the properties of the starting amendment materials.

The pH in SP-4 remained relatively constant. This would be expected since the quantity and type of amendments added to the soil in this test differed significantly from those added to the composting tests.

In MAIV-1, pH dropped during the initial stages to approximately pH 5.5, but then rose quickly to pH 9 to 9.5. The mixture used in this test was the same one used in the first set of SP tests, which also demonstrated a high pH after the initial stages.

The mixture used, and aeration control achieved, in MAIV-2 resulted in only a slight drop in pH at the beginning of the test and a more moderate pH increase than that observed in MAIV-1. The mixture used in MAIV-3 and -4 was the same as that used in SP-8. The pH did drop initially in these MAIV tests but then increased and remained at a more moderate level than that maintained in the first two MAIV tests. The pH in the latter stages of MAIV-3 and -4 was similar to that in SP-8.

6.1.6 Nonquantitative Observations

6.1.6.1 Odors

In the first set of SP tests in which the pH dropped, the compost had a vinegar-like odor. Once the aeration was improved, the compost developed an ammonia smell. The MAIV-1 mixture developed a very strong ammonia odor, while the MAIV-2 mixture had only a mild ammonia odor.

6.1.6.2 Amendment mixtures

The three amendment mixtures tested at UMDA (Table 4-2) exhibited different composting characteristics. These are reflected most dramatically in the pH and temperature data, as discussed in Subsections 6.2 and 6.5. Problems with the temperature control system during the first set of tests make it difficult to compare the three amendment mixtures in terms of heating characteristics. All three mixtures did compost effectively. The pH values using amendment mixtures B and C were much more constant and moderate than those observed with mixture A. The high pH observed with mixture A appears to be linked to the inclusion of chicken manure. The explosives reduction data indicate that more extensive reduction was achieved with the LAAP duplicating mixture (Amendment mixture B) used in MAIV-2. However, excellent destruction was achieved with amendment mixture C in both MAIV and SP reactors.

A preliminary evaluation of toxicity data by ORNL and USATHAMA has indicated that residual compost toxicity may be correlated with amendment composition. These data were not available for review and evaluation for this report.

6.1.7 Soil Loading

Soil loading is a key parameter affecting the cost of implementing composting for bioremediation. In the SP tests, effective self-heating was inhibited somewhat in the first seven SP tests because of instrumentation failures. The effect of soil loading, however, can still be evaluated. TNT destruction dropped markedly as the soil percentage was increased from 30 to 40% in the SP tests. HMX and RDX destruction was poor in all of the first seven SP tests. In SP-8, good HMX and RDX reduction was achieved, indicating that HMX and RDX can be removed with the proper amendments and system operation.

In the MAIV tests, excellent TNT, HMX, and RDX removal was achieved with 25% soil in the mix. Reasonable TNT destruction was achieved at 40% soil in the MAIV system, but no significant change was observed for RDX or HMX. Self-heating at 40% soil was markedly less than that achieved at 25% soil.

6.1.8 ORNL Toxicity and Chemistry Studies — Summary

Compost samples from the UMDA optimization study were provided to ORNL for toxicological and chemical characterization. EPA Synthetic Precipitation Leaching Procedure leachate and organic solvent leachates were subjected to Ames bacterial mutagenicity tests, acute and chronic toxicity tests using the aquatic crustaceans *Ceriodaphnia dubia*, and rat oral toxicity screening. The leachates were also analyzed for explosives and TNT metabolites.

The main conclusion of the ORNL study was that composting can effectively reduce the concentrations of explosives and bacterial mutagenicity in explosives contaminated soil, and reduce the aquatic toxicity of leachable compounds. Small levels of explosives and metabolites, bacterial mutagenicity, and leachable toxicity remain after composting. (The ORNL report will be published soon by USATHAMA.)

6.1.9 Microtox Tests

Microtox tests were conducted to determine the efficacy of the test in measuring toxicity reduction in composted explosives contaminated soils. Although limited microtox data were obtained, a significant reduction in toxicity was observed. However, the microtox data were not compared with the ORNL data because it was not available at the time this report was prepared.

6.2 DISCUSSION OF INDIVIDUAL TESTS

The following subsections are presented to provide a test-by-test description for each of the studies conducted at UMDA.

6.2.1 SP-1

Key Data:	Soil Loading:	7%
	Amendment Mixture:	A
	Study Dates:	9/21/90 - 12/20/90
	Starting/Final pH:	6.0/9.0
	Days above 50°C:	0
	Starting/Final Moisture (% WHC):	63/64
	Percent TNT Degradation:	91
	Percent HMX Degradation:	39
	Percent RDX Degradation:	73

The effectiveness of the composting in SP-1 (and all of the first set of SP tests) was disrupted by malfunctions in the temperature control/ventilation system. Inadequate aeration in the first few weeks of the program resulted in some anaerobic metabolism and the production of volatile organic acids, especially in the lower regions of the reactors. This problem was greatest in SP-1 and SP-2, as indicated by the lower pH values (4.7 to 4.8) observed for the bottom of these 2 piles. Compost temperatures fluctuated as a function of these problems, and never achieved the desired thermophilic conditions. The pH exhibited a sharp increase after day 25. This delay in pH increase compared with SP-7 is most likely caused by the system malfunctions that resulted in reduced aerobic microbial activity. The best explosives destruction achieved in the first set of static piles was achieved in SP-1.

6.2.2 SP-2

Key Data:	Soil Loading:	10%
	Amendment Mixture:	A
	Study Dates:	9/21/90 - 12/20/90
	Starting/Final pH:	7.9/8.9
	Days above 50°C:	1
	Starting/Final Moisture (% WHC):	60/54
	Percent TNT Degradation:	96
	Percent HMX Degradation:	21
	Percent RDX Degradation:	46

As with SP-1, this test was disrupted by malfunctions in the process control system. Anaerobic metabolism, volatile organic acid production, and declining pH values occurred as a result of these problems. Once again, temperature fluctuated as a result of these problems and active thermophilic composting was not obtained. The pH increase pattern in this test was like that of SP-1. TNT destruction was good in this test, but RDX and HMX reduction were less than that achieved in SP-1.

6.2.3 SP-3

Key Data:	Soil Loading:	20%
	Amendment Mixture:	A
	Study Dates:	9/21/90 - 12/20/90
	Starting/Final pH:	6.1/8.9
	Days above 50°C:	0
	Starting/Final Moisture (% WHC):	61/54
	Percent TNT Degradation:	94
	Percent HMX Degradation:	5

Percent RDX Degradation: 16

This reactor started active composting virtually immediately. The control system for SP-3 was not as affected by the instrumentation problems as those for the other static piles. However, at approximately day 6 a computer default problem resulted in the blower remaining on for an entire evening. This cooled SP-3 to ambient temperature. The temperatures rebounded from this event, but still did not reach the active thermophilic temperatures desired. The pH rose slowly in this test. This slow increase was likely caused, in part, by the inhibition of microbial activity brought about by the described temperature drop. TNT destruction was good in this reactor, but HMX and RDX destruction decreased substantially from that observed in SP-1.

6.2.4 SP-4

Key Data:	Soil Loading:	80%
	Amendment Mixture:	Sawdust, ammonia sulfide, sodium acetate, L-arginine
	Study Dates:	11/2/90 - 1/30/91
	Starting/Final pH:	5.4/7.3
	Days above 50°C:	0
	Starting/Final Moisture (% WHC):	40/52
	Percent TNT Degradation:	6
	Percent HMX Degradation:	2
	Percent RDX Degradation:	4

This test proceeded with very little change in either operating parameters or explosives content. The likely reasons for the failure of this test to degrade explosives are discussed in Subsection 6.1.1.3.

6.2.5 SP-5

Key Data:	Soil Loading:	30%
	Amendment Mixture:	A
	Study Dates:	9/21/90 - 12/20/90
	Starting/Final pH:	6.3/8.8

Days above 50° C:	1
Starting/Final Moisture (% WHC):	54/58
Percent TNT Degradation:	98
Percent HMX Degradation:	11
Percent RDX Degradation:	22

The composting in SP-5 initiated almost immediately and temperatures rose steadily until a plateau in temperature was reached just below 50°C. Considering the high percent inorganic mass in this mixture, the temperature rise achieved was significant. However, it is likely that the performance was dampened to an undefinable extent by instrumentation problems. pH rose slowly in this test. Explosives destruction in SP-5 was, on average, slightly better that in the 20% soil test (SP-3). This indicates the significance of achieving proper operating conditions (SP-5 was not as adversely affected as SP-3).

6.2.6 SP-6

Key Data:	Soil Loading:	40%
	Amendment Mixture:	A
	Study Dates:	9/21/90 - 12/20/90
	Starting/Final pH:	7.1/8.8
	Days above 50° C:	0
	Starting/Final Moisture (% WHC):	56/57
	Percent TNT Degradation:	79
	Percent HMX Degradation:	2
	Percent RDX Degradation:	0

Good self-heating was achieved within this pile despite the high inorganic content. However, temperatures still did not reach the desired thermophilic range. Temperature fluctuations, as with SP-5, were not severe. pH rose much slower in this test than in the lower soil loading tests (7 and 10%) using this same amendment mixture. TNT destruction was significant, but no removal of HMX and RDX occurred.

6.2.7 SP-7

Key Data:

Soil Loading:	10% (Uncontaminated)
Amendment Mixture:	A
Study Dates:	9/21/90 - 12/20/90
Starting/Final pH:	7.7/8.9
Days above 50°C:	2
Starting/Final Moisture (% WHC):	60/59
Percent TNT Degradation:	N/A
Percent HMX Degradation:	N/A
Percent RDX Degradation:	N/A

This test composted quite well. Temperatures quickly rose, and likely would have continued to climb into the thermophilic range had instrumentation problems at approximately day 6 not interrupted the self-heating. The temperature rebound at day 40 was caused by the addition of moisture. As with the other SP tests containing this amendment mixture and a lower soil fraction, pH rose sharply after the first few days of composting.

6.2.8 SP-8

Key Data:

Soil Loading:	10%
Amendment Mixture:	C
Study Dates:	2/6/91 - 5/7/91
Starting/Final pH:	6.2/6.0
Days above 50°C:	22
Starting/Final Moisture (% WHC):	63/67
Percent TNT Degradation:	99
Percent HMX Degradation:	80
Percent RDX Degradation:	93

The amendment mixture used and the proper operation of the control system resulted in excellent composting performance. Thermophilic temperatures were achieved for a prolonged period of time and the pH remained relatively stable. Good destruction of each explosive was achieved.

6.2.9 MAIV-1

Key Data:	Soil Loading:	10%
	Amendment Mixture:	A
	Study Dates:	9/19/90 - 11/2/90
	Starting/Final pH	6.9/9.3
	Days above 50°C:	5
	Starting/Final Moisture (% WHC):	56/54
	Percent TNT Degradation:	97
	Percent HMX Degradation:	29
	Percent RDX Degradation:	90

This test was effected by the control system to some extent, especially in the beginning of the test. The first test run in this new reactor was MAIV-1. Consequently, refinement of the control system operation occurred during this test. Temperatures did reach the thermophilic level, but not as early in the test as likely would have happened had the operating system been fine-tuned prior to this test. The pH rose sharply in this test starting at approximately day 10. This pH rise to high levels was particularly associated with amendment mixture A. TNT and RDX were significantly removed. HMX, however, was removed to a much lower extent than the removal achieved in other MAIV tests.

6.2.10 MAIV-2

Key Data:	Soil Loading:	10%
	Amendment Mixture:	B
	Study Dates:	11/8/90 - 12/21/90
	Starting/Final pH:	7.2/8.9
	Days above 50°C:	3
	Starting/Final Moisture (% WHC):	69/65
	Percent TNT Degradation:	99
	Percent HMX Degradation:	95
	Percent RDX Degradation:	99

This test heated rapidly, but then was effected by low ambient temperatures at approximately day 10. Temperatures rebounded, but a prolonged period of thermophilic conditions was not achieved. The pH rose markedly in MAIV-2. Explosives destruction in MAIV-2 was the best achieved for all three explosives in the UMDA program. This destruction was most likely a function of the amendment mixture since MAIV-3 exhibited better composting (self-heating), but did not equal the explosives destruction achieved in MAIV-2.

6.2.11 MAIV-3

Key Data:		
	Soil Loading:	25%
	Amendment Mixture:	C
	Study Dates:	2/6/91 - 3/22/91
	Starting/Final pH:	5.7/7.4
	Days above 50°C:	14
	Starting/Final Moisture (% WHC):	68/69
	Percent TNT Degradation:	99
	Percent HMX Degradation:	68
	Percent RDX Degradation:	97

This test heated gradually but steadily, and achieved a prolonged period of time above 50°C. The pH rose, but as was characteristic of amendment mixture C, not as sharply or as extensively as with amendment mix A. Explosives destruction was good for all 3 explosives, especially considering the high soil loading level.

6.2.12 MAIV-4

Key Data:		
	Soil Loading:	40%
	Amendment Mixture:	C
	Study Dates:	4/3/91 - 5/18/91
	Starting/Final pH:	5.4/7.3
	Days above 50°C:	0
	Starting/Final Moisture (% WHC):	62/62
	Percent TNT Degradation:	97
	Percent HMX Degradation:	0
	Percent RDX Degradation:	18

Considering the high soil loading, this test composted relatively well. Thermophilic temperatures were not achieved, but the temperature did remain between 45 and 50°C for a prolonged period. The pH rose steadily, but not as extensively as was the case for mixtures containing amendment mix A or B. TNT destruction was good, and was superior to that achieved with 40% soil in a static pile reactor. RDX and HMX destruction, however, were negligible.

SECTION 7

CONCLUSIONS

The UMDA composting optimization study has confirmed previous work conducted at the Louisiana Army Ammunition Plant, which indicated that composting would effectively remove TNT, RDX, and HMX from contaminated matrices. The UMDA study indicated that both static pile and mechanically agitated technological approaches for implementing composting are effective at degrading explosives. The superior performance of the mechanically agitated system, however, indicates that mixing during composting is important for achieving rapid and extensive destruction. The maximum soil loading level for achieving effective degradation appears to be approximately 30 volume percent for both SP and MAIV systems.

Amendment composition is a key parameter controlling explosives degradation. The data indicate that amendments must be carefully selected and combined, but that a variety of acceptable amendment mixtures can be prepared based on the local availability of amendments.

The USATHAMA goal criteria for demonstrating the cost effectiveness of composting were an amendment cost of less than $50/ton and a soil loading level above 20%. These criteria were shown to be achievable in the UMDA program.

REFERENCES

Biocycle Special Report, 1987. Twenty-five years ago in solid waste composting. Biocycle 28: 35-38.

Finstein, M.S., F.C. Miller, and P.F. Strom. 1986. Waste Treatment Composting as a Controlled System. In. BioTechnology, Eds. H.J. Rehm and G. Reed. VCH N.Y., N.Y.

Griest, W.H., R.L. Tyndall, A.J. Stewart, C.H. Ho, D.E. Katon, and W.M. Caldwell. 1991. Characterization of Explosives Processing Wastes Decomposition Due to Compositng (Report Number to be Assigned).

Lowe, W., R.T. Williams, and P.J. Marks. 1989. Composting of Explosives Contaminated Soil Technology. USATHAMA Report No. CETHA-TE-CR-90027.

Williams, R.T. and C.A. Myler. 1990. Bioremediation Using Composting. Biocycle. November 1990.

Williams, R.T., P.S. Ziegenfuss, and P.J. Marks. 1988. Field Demonstration - Composting of Explosives-Contaminated Sediments at the Louisiana Army Ammunition Plant (LAAP). USATHAMA Report No. AMXTH-IR-TE-88242.

Williams, R.T., P.S. Ziegenfuss, and P.J. Marks. 1989. Field Demonstration - Composting of Explosives Contaminated Sediments at the Badger Army Ammunition Plant.

Williams, R.T., P.S. Ziegenfuss, and W.E. Sisk. in press. Composting of Explosives and Propellant Contaminated Soils Under Thermophilic and Mesophilic Conditions. J. of Indust. Microbiol.

Woodward, R.E. 1990. Evaluation of Composting Implementation: A Literature Review. USATHAMA Report No. TCN 89363.

APPENDIX A

DATA FROM HSL SURVEY OF WASHOUT LAGOON SOILS

Roy F. Weston, Inc. - Lionville Laboratory
PEST/PCB ANALYTICAL DATA PACKAGE FOR
USATHAMA-UM

DATE RECEIVED: 11/10/89 RFW LOT # :8911L453

CLIENT ID	RFW #	MTX	PREP #	COLLECTION	EXTR/PREP	ANALYSIS
UMDA LAGOON COMPOSIT	001	S	89LE1135	10/05/89	11/15/89	12/08/89

LAB QC:

PBLK	MB2	S	89LE1135	N/A	11/15/89	12/08/89
PBLK	MB2 BS	S	89LE1135	N/A	11/15/89	12/08/89
PBLK	MB2 BSD	S	89LE1135	N/A	11/15/89	12/08/89

Roy F. Weston, Inc.
Lionville Laboratory

WESTON SM

Client: USATHAMA-UM
RFW#: 8911L453, Pest/PCB
W.O.#: 0010-10-12

Date Received: 11/10/89

The set of samples consisted of 1 soil sample collected on 10/05/89.

The samples were extracted on 11/15/89 and analyzed according to criteria set forth in the Contract Lab Program for Pesticides and PCB's on 12/08/89.

The following is a summary of the QC results accompanying these sample results and a description of any problem encountered during their analysis.

1. All surrogate recoveries are within EPA QC limits
2. All blank spike recoveries are within EPA QC limits.
3. Blank spike recoveries were quantified from the SP2100 (confirmation) column. There was interference with target compound Gamma BHC on the SP2250/2401 (primary) column.
4. Sample "UMDA Lagoon Composite" requi4red a 10-fold dilution because it contained high levels of non-target compounds.

12-15-89

Carter P. Nulton, Ph.D.
Vice President
Lionville Analytical Laboratory

WESTON

GLOSSARY OF PEST/PCB DATA

DATA QUALIFIERS

U - Indicates that the compounds was analyzed for but not detected. The minimum detection limit for the sample (not the method detection limit) is reported with the U (e.g., 10U).

J - Indicates an estimated value. This flag is used in cases where a target analyte is detected at a level less than the lower quantification level. If the limit of quantification is 10 ug/L and a concentration of 3 ug/L is calculated, it is reported as 3J.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination. This flag is also used for a TIC as well as for a positively identified TCL compound.

E - Indicates that the compound was detected beyond the calibration range and was subsequently analyzed at a dilution.

I - Interference.

ABBREVIATIONS

BS - Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

BSD - Indicates blank spike duplicate.

MS - Indicates matrix spike.

MSD - Indicates matrix spike duplicate.

DL - Indicates that recoveries were not obtained because the extract had to be diluted for analysis.

NA - Not applicable.

DF - Dilution factor.

NR - Not required.

Roy F. Weston, Inc. - Lionville Laboratory
Pesticide/PCBs by GC, CLP List

RFW Batch Number: 8911L453 Client: USATHAMA-UM Work Order: 0010-10-12-0000 Report Date: 12/11/89 11:06 Page: 1

Sample Information								
Cust ID:	UMDA LAGOON COMPOSIT		PBLK		PBLK BS		PBLK BSD	
RFW#:	453-001		89LE1135-MB2		89LE1135-MB2		89LE1135-MB2	
Matrix:	SOIL		SOIL		SOIL		SOIL	
D.F.:	10.0		1.00		1.00		1.00	
Units:	ug/wet g		ug/wet g		ug/wet g		ug/wet g	
Surrogate: Di-n-butylchlorendate	77	%	80	%	79	%	78	%
		fl		fl		fl		fl
Alpha-BHC	0.047	U	0.0040	U	0.0040	U	0.0040	U
Beta-BHC	0.047	U	0.0040	U	0.0040	U	0.0040	U
Delta-BHC	0.047	U	0.0040	U	0.0040	U	0.0040	U
gamma-BHC (Lindane)	0.047	U	0.0040	U	51	%	51	%
Heptachlor	0.047	U	0.0040	U	41	%	40	%
Aldrin	0.047	U	0.0040	U	78	%	78	%
Heptachlor epoxide	0.047	U	0.0040	U	0.0040	U	0.0040	U
Endosulfan I	0.047	U	0.0040	U	0.0040	U	0.0040	U
Dieldrin	0.094	U	0.0080	U	54	%	54	%
4,4'-DDE	0.094	U	0.0080	U	0.0080	U	0.0080	U
Endrin	0.094	U	0.0080	U	78	%	74	%
Endosulfan II	0.094	U	0.0080	U	0.0080	U	0.0080	U
4,4'-DDD	0.094	U	0.0080	U	0.0080	U	0.0080	U
Endosulfan sulfate	0.094	U	0.0080	U	0.0080	U	0.0080	U
4,4'-DDT	0.094	U	0.0080	U	106	%	100	%
Methoxychlor	0.47	U	0.040	U	0.040	U	0.040	U
Endrin ketone	0.094	U	0.0080	U	0.0080	U	0.0080	U
alpha-Chlordane	0.47	U	0.040	U	0.040	U	0.040	U
gamma-Chlordane	0.47	U	0.040	U	0.040	U	0.040	U
Toxaphene	0.94	U	0.080	U	0.080	U	0.080	U
Aroclor-1016	0.47	U	0.040	U	0.040	U	0.040	U
Aroclor-1221	0.47	U	0.040	U	0.040	U	0.040	U
Aroclor-1232	0.47	U	0.040	U	0.040	U	0.040	U
Aroclor-1242	0.47	U	0.040	U	0.040	U	0.040	U
Aroclor-1248	0.47	U	0.040	U	0.040	U	0.040	U
Aroclor-1254	0.94	U	0.080	U	0.080	U	0.080	U
Aroclor-1260	0.94	U	0.080	U	0.080	U	0.080	U

U= Analyzed, not detected. J= Present below detection limit. B= Present in blank. NR= Not requested. NS= Not spiked.
%= Percent recovery. D= Diluted out. I= Interference. NA= Not Applicable. *= Outside of EPA CLP QC

Roy F. Weston, Inc. - Lionville Laboratory
BNA ANALYTICAL DATA PACKAGE FOR
USATHAMA-UM

DATE RECEIVED: 11/10/89 RFW LOT # :8911L453

CLIENT ID	RFW #	MTX	PREP #	COLLECTION	EXTR/PREP	ANALYSIS
UMDA LAGOON COMPOSIT	001	S	89LE1135	10/05/89	11/15/89	12/02/89
LAB QC:						
SBLK	MB1	S	89LE1135	N/A	11/15/89	11/30/89
SBLK	MB1 BS	S	89LE1135	N/A	11/15/89	11/30/89
SBLK	MB1 BSD	S	89LE1135	N/A	11/15/89	11/30/89

WESTON

Roy F. Weston, Inc.
Lionville Laboratory

Client: USATHAMA-UM
RFW#: 8911L453
W.O.#: 2284-08-10

Date Received: 11/10/89

The set of samples consisted of 1 soil sample collected on 10/05/89.

The samples were extracted on 11/15/89 and analyzed according to criteria set forth in Method 8270 for TCL Semivolatiles target compounds on 11/30/89 and 12/02/89 (analyzed out of hold as per client request).

The following is a summary of the QC results accompanying these sample results and a description of any problem encountered during their analysis.

1. Non-target compounds were detected in these samples.

2. The extracted sample required ten-fold dilution because it contained high levels of nontarget compounds.

3. Two of 18 obtainable surrogate recoveries are outside of EPA QC limits. However, EPA CLP surrogate recovery criteria are met [ie., no more than one outlier per fraction (acid and base neutral) and no recoveries less than 10%].

4. Ten of 22 blank spike recoveries are outside EPA QC limits.

C. P. Nulton 12-20-89

Carter P. Nulton, Ph.D.
Vice President
Lionville Analytical Laboratory

GLOSSARY OF BNA DATA

DATA QUALIFIERS

U = Compound was analyzed for but not detected. The associated numerical value is the estimated sample quantitation limit which is included and corrected for dilution and percent moisture.

J = Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero; for example, if the limit of detection is 10 ug/L and a concentration of 3 ug/L is calculated, it is reported as 3J.

B = This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination. This flag is also used for a TIC as well as for a positively identified TCL compound.

E = Indicates that the compound was detected beyond the calibration range and was subsequently analyzed at a dilution.

A = This flag indicates that a TIC is a suspected aldol-condensation product.

I = Interference.

X = Additional qualifiers used as required are explained in the case narrative.

ABBREVIATIONS

BS = Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

BSD = Indicates blank spike duplicate.

MS = Indicates matrix spike.

MSD = Indicates matrix spike duplicate.

DL = Indicates that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

NA = Not applicable.

DF = Dilution factor.

NR = Not required.

	Cust ID:	UMDA LAGOON COMPOSIT		SBLK		SBLK BS		SBLK BSD	
Sample	RFW#:	001		89LE1135-MB1		89LE1135-MB1		89LE1135-MB1	
Information	Matrix:	SOIL		SOIL		SOIL		SOIL	
	D.F.:	10.0		1.00		1.00		1.00	
	Units:	ug/wet g		ug/wet g		ug/wet g		ug/wet g	
	Nitrobenzene-d5	D	%	102	%	103	%	100	%
Surrogate	2-Fluorobiphenyl	D	%	69	%	79	%	75	%
Recovery	p-Terphenyl-d14	D	%	106	%	112	%	110	%
	Phenol-d5	D	%	98	%	122 *	%	115 *	%
	2-Fluorophenol	D	%	99	%	103	%	104	%
	2,4,6-Tribromophenol	D	%	61	%	79	%	73	%
Phenol		20	U	0.3	U	109 *	%	102 *	%
bis(2-Chloroethyl)ether		20	U	0.3	U	0.3	U	0.3	U
2-Chlorophenol		20	U	0.3	U	110 *	%	102	%
1,3-Dichlorobenzene		20	U	0.3	U	0.3	U	0.3	U
1,4-Dichlorobenzene		20	U	0.3	U	87	%	85	%
Benzyl alcohol		20	U	0.3	U	0.3	U	0.3	U
1,2-Dichlorobenzene		20	U	0.3	U	0.3	U	0.3	U
2-Methylphenol		20	U	0.3	U	0.3	U	0.3	U
bis(2-Chloroisopropyl)ether		20	U	0.3	U	0.3	U	0.3	U
4-Methylphenol		20	U	0.3	U	0.3	U	0.3	U
N-Nitroso-Di-n-propylamine		20	U	0.3	U	131 *	%	123	%
Hexachloroethane		20	U	0.3	U	0.3	U	0.3	U
Nitrobenzene		20	U	0.3	U	0.3	U	0.3	U
Isophorone		20	U	0.3	U	0.3	U	0.3	U
2-Nitrophenol		20	U	0.3	U	0.3	U	0.3	U
2,4-Dimethylphenol		20	U	0.3	U	0.3	U	0.3	U
Benzoic acid		98	U	2	U	2	U	2	U
bis(2-Chloroethoxy)methane		20	U	0.3	U	0.3	U	0.3	U
2,4-Dichlorophenol		20	U	0.3	U	0.3	U	0.3	U
1,2,4-Trichlorobenzene		20	U	0.3	U	77	%	74	%
Naphthalene		20	U	0.3	U	0.3	U	0.3	U
4-Chloroaniline		20	U	0.3	U	0.3	U	0.3	U
Hexachlorobutadiene		20	U	0.3	U	0.3	U	0.3	U
4-Chloro-3-methylphenol		20	U	0.3	U	136 *	%	128 *	%
2-Methylnaphthalene		20	U	0.3	U	0.3	U	0.3	U
Hexachlorocyclopentadiene		20	U	0.3	U	0.3	U	0.3	U

*= Outside of EPA CLP QC limits.

Cust ID:	UMDA LAGOON COMPOSIT		SBLK		SBLK BS		SBLK BSD	
RFW#:	001		89LE1135-MB1		89LE1135-MB1		89LE1135-MB1	
2,4,6-Trichlorophenol	20	U	0.3	U	0.3	U	0.3	U
2,4,5-Trichlorophenol	98	U	2	U	2	U	2	U
2-Chloronaphthalene	20	U	0.3	U	0.3	U	0.3	U
2-Nitroaniline	98	U	2	U	2	U	2	U
Dimethylphthalate	20	U	0.3	U	0.3	U	0.3	U
Acenaphthylene	20	U	0.3	U	0.3	U	0.3	U
2,6-Dinitrotoluene	20	U	0.3	U	0.3	U	0.3	U
3-Nitroaniline	98	U	2	U	2	U	2	U
Acenaphthene	20	U	0.3	U	104	%	99	%
2,4-Dinitrophenol	98	U	2	U	2	U	2	U
4-Nitrophenol	98	U	2	U	145 *	%	132 *	%
Dibenzofuran	20	U	0.3	U	0.3	U	0.3	U
2,4-Dinitrotoluene	9	J	0.3	U	113 *	%	103 *	%
Diethylphthalate	20	U	0.3	U	0.3	U	0.3	U
4-Chlorophenyl-phenylether	20	U	0.3	U	0.3	U	0.3	U
Fluorene	20	U	0.3	U	0.3	U	0.3	U
4-Nitroaniline	98	U	2	U	2	U	2	U
4,6-Dinitro-2-methylphenol	98	U	2	U	2	U	2	U
N-Nitrosodiphenylamine (1)	20	U	0.3	U	0.3	U	0.3	U
4-Bromophenyl-phenylether	20	U	0.3	U	0.3	U	0.3	U
Hexachlorobenzene	20	U	0.3	U	0.3	U	0.3	U
Pentachlorophenol	98	U	2	U	80	%	67	%
Phenanthrene	20	U	0.3	U	0.3	U	0.3	U
Anthracene	20	U	0.3	U	0.3	U	0.3	U
Di-n-Butylphthalate	20	U	0.3	J	0.4	B	0.4	B
Fluoranthene	20	U	0.3	U	0.3	U	0.3	U
Pyrene	20	U	0.3	U	137	%	136	%
Butylbenzylphthalate	20	U	0.3	U	0.3	U	0.3	U
3,3'-Dichlorobenzidine	39	U	0.7	U	0.7	U	0.7	U
Benzo(a)anthracene	20	U	0.3	U	0.3	U	0.3	U
Chrysene	20	U	0.3	U	0.3	U	0.3	U
bis(2-Ethylhexyl)phthalate	20	U	0.3	U	0.3	U	0.3	U
Di-n-Octyl phthalate	20	U	0.3	U	0.3	U	0.3	U
Benzo(b)fluoranthene	20	U	0.3	U	0.3	U	0.3	U
Benzo(k)fluoranthene	20	U	0.3	U	0.3	U	0.3	U
Benzo(a)pyrene	20	U	0.3	U	0.3	U	0.3	U
Indeno(1,2,3-cd)pyrene	20	U	0.3	U	0.3	U	0.3	U
Dibenzo(a,h)anthracene	20	U	0.3	U	0.3	U	0.3	U
Benzo(g,h,i)perylene	20	U	0.3	U	0.3	U	0.3	U

(1) - Cannot be separated from Diphenylamine. *= Outside of EPA CLP QC limits.

1F
SEMIVOLATILE ORGANICS ANALYSIS SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT SAMPLE NO.

UMDA LAGOON COMPOSIT

Lab Name: Roy F. Weston, Inc. Work Order: 2284-08-10-0000

Client: USATHAMA-UM

Matrix: SOIL   Lab Sample ID: 8911L453-001

Sample wt/vol: 25.6 (g/mL) G   Lab File ID: M120118

Level: (low/med) LOW   Date Received: 11/10/89

% Moisture: not dec. 0 dec.   Date Extracted: 11/15/89

Extraction: (SepF/Cont/Sonc) SONC   Date Analyzed: 12/02/89

GPC Cleanup: (Y/N) N   pH: 7.0   Dilution Factor: 10.0

Number TICs found: 11

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/wet g

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	DICHLOROPROPANOL	4.32	30000	J
2.	CHLOROPROPANEDIOL	4.97	300000	JB
3.	UNKNOWN	5.32	20000	J
4.	UNKNOWN	10.47	10000	J
5.	TNT	16.27	10000000	J
6.	UNKNOWN	18.95	10000	J
7.	ALKANE	24.73	10000	J
8.	ALKANE	26.85	40000	J
9.	ALKANE	28.23	40000	J
10.	ALKANE	29.93	40000	J
11.	ALKANE	31.98	30000	J

1F

SEMIVOLATILE ORGANICS ANALYSIS SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

CLIENT SAMPLE NO.

SBLK

Lab Name: Roy F. Weston, Inc. Work Order: 2284-08-10-0000

Client: USATHAMA-UM

Matrix: SOIL

Lab Sample ID: 89LE1135-MB1

Sample wt/vol: 30.0 (g/mL) G

Lab File ID: M113011

Level: (low/med) LOW

Date Received: 11/15/89

% Moisture: not dec. 0 dec.

Date Extracted: 11/15/89

Extraction: (SepF/Cont/Sonc) SONC

Date Analyzed: 11/30/89

GPC Cleanup: (Y/N) N pH: 7.0

Dilution Factor: 1.00

Number TICs found: 8

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/wet g

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	UNKNOWN	5.02	600	J
2.	CHLOROPROPANEDIOL	5.35	200	J
3.	ALDOL CONDENSATE	5.48	200	JA
4.	UNKNOWN	18.78	100	J
5.	UNKNOWN	22.37	200	J
6.	UNKNOWN	23.10	100	J
7.	UNKNOWN	25.10	100	J
8.	UNKNOWN	26.73	100	J

Roy F. Weston, Inc. - Lionville Laboratory
VOA ANALYTICAL DATA PACKAGE FOR
USATHAMA-UM

DATE RECEIVED: 11/10/89 RFW LOT # :8911L453

CLIENT ID	RFW #	MTX	PREP #	COLLECTION	EXTR/PREP	ANALYSIS
UMDA LAGOON COMPOSIT	001	S	89LVYA48	10/05/89	N/A	11/22/89
LAB QC:						
VBLK	MB1	S	89LVYA48	N/A	N/A	11/22/89

WESTON

ROY F. WESTON, INC.
Lionville Laboratory

CLIENT: USATHAMA - UM **SAMPLES RECEIVED:** 11-10-89
RFW #: 8911L453, GC/MS VOLATILE
W.O. #: 2284-08-10

NARRATIVE

The set of samples consisted of one soil sample collected on 10-05-89.

The sample was analyzed according to criteria set forth in Method 8240 for TCL Volatile target compounds on 11-22-89.

The following is a summary of the QC results accompanying these sample results and a description of any problems encountered during their analysis:

1. This sample was analyzed out of hold upon client request.

2. Non-target compounds were not detected in this sample.

3. All surrogate recoveries are within EPA QC limits.

4. The blank contains methylene chloride (common laboratory contaminant) at a level less than 4x the CRQL.

for Carter Nulton, Ph.D.
Carter Nulton, Ph.D.
Vice President/Laboratory Manager
Lionville Analytical Laboratory

12-28-89
Date

WESTON

GLOSSARY OF VOA DATA

DATA QUALIFIERS

U = Compound was analyzed for but not detected. The associated numerical value is the estimated sample quantitation limit which is included and corrected for dilution and percent moisture.

J = Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero; for example, if the limit of detection is 10 ug/L and a concentration of 3 ug/L is calculated, it is reported as 3J.

B = This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination. This flag is also used for a TIC as well as for a positively identified TCL compound.

E = Indicates that the compound was detected beyond the calibration range and was subsequently analyzed at a dilution.

I = Interference.

X = Additional qualifiers used as required are explained in the case narrative.

NQ = Result qualitatively confirmed but not able to quantify.

ABBREVIATIONS

BS = Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

BSD = Indicates blank spike duplicate.

MS = Indicates matrix spike.

MSD = Indicates matrix spike duplicate.

DL = Indicates that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

NA = Not applicable.

DF = Dilution factor.

NR = Not required.

Roy F. Weston, Inc. - Lionville Laboratory

Volatiles by GC/MS, HSL List

Report Date: 12/21/89 10:09

RFW Batch Number: 8911L453 Client: USATHAMA-UM Work Order: 2284-08-10-0000 Page: 1a

Sample Information	Cust ID:	UMDA LAGOON COMPOSIT		VBLK	
	RFW#:	001		89LVYA48-MB1	
	Matrix:	SOIL		SOIL	
	D.F.:	1.00		1.00	
	Units:	ug/wet g		ug/wet g	
	Toluene-d8	102	%	98	%
Surrogate	Bromofluorobenzene	85	%	101	%
Recovery	1,2-Dichloroethane-d4	98	%	101	%
Chloromethane		0.01	U	0.01	U
Bromomethane		0.01	U	0.01	U
Vinyl Chloride		0.01	U	0.01	U
Chloroethane		0.01	U	0.01	U
Methylene Chloride		0.03	B	0.02	
Acetone		0.01	U	0.01	U
Carbon Disulfide		0.005	U	0.005	U
1,1-Dichloroethene		0.005	U	0.005	U
1,1-Dichloroethane		0.005	U	0.005	U
1,2-Dichloroethene (total)		0.005	U	0.005	U
Chloroform		0.005	U	0.005	U
1,2-Dichloroethane		0.005	U	0.005	U
2-Butanone		0.01	U	0.01	U
1,1,1-Trichloroethane		0.005	U	0.005	U
Carbon Tetrachloride		0.005	U	0.005	U
Vinyl Acetate		0.01	U	0.01	U
Bromodichloromethane		0.005	U	0.005	U
1,2-Dichloropropane		0.005	U	0.005	U
cis-1,3-Dichloropropene		0.005	U	0.005	U
Trichloroethene		0.005	U	0.005	U
Dibromochloromethane		0.005	U	0.005	U
1,1,2-Trichloroethane		0.005	U	0.005	U
Benzene		0.005	U	0.005	U
Trans-1,3-Dichloropropene		0.005	U	0.005	U
Bromoform		0.005	U	0.005	U
4-Methyl-2-pentanone		0.01	U	0.01	U
2-Hexanone		0.01	U	0.01	U
Tetrachloroethene		0.005	U	0.005	U
1,1,2,2-Tetrachloroethane		0.005	U	0.005	U

*= Outside of EPA CLP QC limits.

Cust ID:	UMDA LAGOON COMPOSIT		VBLK	
RFW#:	001		89LVYA48-MB1	
Toluene	0.002	J	0.005	U
Chlorobenzene	0.005	U	0.005	U
Ethylbenzene	0.005	U	0.005	U
Styrene	0.005	U	0.005	U
Xylene (total)	0.005	U	0.005	U

*= Outside of EPA CLP QC limits.

ROY F. WESTON INC.
LIONVILLE LABORATORY

CLIENT: USATHAMA-UM
RFW #: 8910L200
W.O. #: 2281-08-10-0000

SAMPLES RECEIVED: 10-20-89

METALS NARRATIVE

The following is a summary of the quality control results and a description of any problems encountered during the analysis of this batch of samples:

1. All sample holding times as required by 40CFR136 were met for water samples. Note: Holding times for soil samples have not been promulgated by the USEPA.

2. All calibration verification checks were within the required control limits of 90-110% (85-115% for Hg). Calibration verification is performed using an independent standard purchased from Inorganic Ventures, Inc.

3. All preparation blanks were analyzed below the required detection limit.

4. All laboratory control standards were within the control limits of 80-120%.

   Note: The USEPA-CLP has dropped control limits for silver and antimony due to documented difficulties in obtaining reliable results. WESTON Analytics has adopted the same policy.

5. The analytical methods applied by the laboratory for the determination of metals, are:

As :	EPA 206.2	Hg :	EPA 245.1
Se :	EPA 270.2	ICP Scan :	EPA 200.7
Pb :	EPA 239.2	All others :	EPA 200.7
Tl :	EPA 279.2	EP Leachates (except Hg):	200.7

NOTE: For solid samples, all results are reported on a dry weight basis.

Carter Nulton, Ph.D.
Vice President/Laboratory Manager
Lionville Analytical Laboratory

10-24-89
Date

ROY F. WESTON, INC.

GLOSSARY OF TERMS - INORGANIC REPORTS

DATA QUALIFIERS

U - Indicates that the parameter was not detected at or above the reported limit. The associated numerical value is the sample detection limit.

* - Indicates that the original sample result is greater than 4x the spike amount added. The USEPA-CLP has determined that spike results on samples where this occurs may be unreliable and, therefore, the control limits are not applicable.

ABBREVIATIONS

MB - Method or preparation blank.
MS - Matrix Spike.
MSD - Matrix Spike Duplicate.
REP - Sample Replicate.
LC - Indicates a method LCS or Blank Spike.
NC - Not calculable, result below the detection limit.

LABORATORY CHRONOLOGY AND HOLDTIME REPORT

The test code listed indicates the specific analysis or preparation procedure employed. The codes may be interpreted as follows:

MAAW - Metals prep test for AA digestion, water matrix.
MAAS - Metals prep test for AA digestion, soil matrix.
MICW - Metals prep test for ICP digestion, water matrix.
MICS - Metals prep test for ICP digestion, soil matrix.

M**TO- This type of code indicates a total metal analysis (eg. MAGTO indicates an analysis for total silver).

M**SO- This type of code indicates a soluble metal analysis. (eg. MAGSO indicates an analysis for soluble silver).

M**EP- This type of code indicates an EPTOXICITY metals analysis (eg. MAGEP indicates an analysis for eptox silver).

I**TO- This type of code indicates a non-metallic total analysis. There is also a complimentary soluble analysis for each of these codes (eg. ICNTO indicates an analysis for total cyanide).

A suffix of -R or -S following these codes indicates a replicate or spike analysis respectively.

ROY F. WESTON INC.

INORGANICS DATA SUMMARY REPORT 10/24/89

CLIENT: USATHAMA-UM WESTON BATCH #: 8910L200
WORK ORDER: 2281-08-10-0000

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	REPORTING LIMIT
-001	UMDA LAGOON COMP	SILVER, TOTAL	1.9	u	MG/KG	1.9
		ALUMINUM, TOTAL	4970		MG/KG	38.7
		ARSENIC, TOTAL	2.0	u	MG/KG	2.0
		BARIUM, TOTAL	62.2		MG/KG	38.7
		BERYLLIUM, TOTAL	0.97	u	MG/KG	0.97
		CALCIUM, TOTAL	4250		MG/KG	967
		CADMIUM, TOTAL	0.97	u	MG/KG	0.97
		COBALT, TOTAL	9.7	u	MG/KG	9.7
		CHROMIUM, TOTAL	5.9		MG/KG	1.9
		COPPER, TOTAL	17.0		MG/KG	4.8
		IRON, TOTAL	17300		MG/KG	19.3
		MERCURY, TOTAL	0.10	u	MG/KG	0.10
		POTASSIUM, TOTAL	967	u	MG/KG	967
		MAGNESIUM, TOTAL	3410		MG/KG	967
		MANGANESE, TOTAL	277		MG/KG	2.9
		SODIUM, TOTAL	967	u	MG/KG	967
		NICKEL, TOTAL	7.7	u	MG/KG	7.7
		LEAD, TOTAL	4.5		MG/KG	0.98
		ANTIMONY, TOTAL	15.6		MG/KG	11.6
		SELENIUM, TOTAL	1.2		MG/KG	0.98
		THALLIUM, TOTAL	2.0	u	MG/KG	2.0
		VANADIUM, TOTAL	46.1		MG/KG	9.7
		ZINC, TOTAL	54.3		MG/KG	3.9

ROY F. WESTON INC.

INORGANICS METHOD BLANK DATA SUMMARY PAGE 10/24/89

CLIENT: USATHAMA-UM

WORK ORDER: 2281-08-10-0000

WESTON BATCH #: 8910L200

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	REPORTING LIMIT
BLANK1	89L1101-MB1	SILVER, TOTAL	2.0	u	MG/KG	2.0
		ALUMINUM, TOTAL	40.0	u	MG/KG	40.0
		BARIUM, TOTAL	40.0	u	MG/KG	40.0
		BERYLLIUM, TOTAL	1.0	u	MG/KG	1.0
		CALCIUM, TOTAL	1000	u	MG/KG	1000
		CADMIUM, TOTAL	1.0	u	MG/KG	1.0
		COBALT, TOTAL	10.0	u	MG/KG	10.0
		CHROMIUM, TOTAL	2.0	u	MG/KG	2.0
		COPPER, TOTAL	5.0	u	MG/KG	5.0
		IRON, TOTAL	20.0	u	MG/KG	20.0
		POTASSIUM, TOTAL	1000	u	MG/KG	1000
		MAGNESIUM, TOTAL	1000	u	MG/KG	1000
		MANGANESE, TOTAL	3.0	u	MG/KG	3.0
		SODIUM, TOTAL	1000	u	MG/KG	1000
		NICKEL, TOTAL	8.0	u	MG/KG	8.0
		ANTIMONY, TOTAL	12.0	u	MG/KG	12.0
		VANADIUM, TOTAL	10.0	u	MG/KG	10.0
		ZINC, TOTAL	4.0	u	MG/KG	4.0
BLANK1	89L1100-MB1	ARSENIC, TOTAL	2.0	u	MG/KG	2.0
		LEAD, TOTAL	1.0	u	MG/KG	1.0
		SELENIUM, TOTAL	1.0	u	MG/KG	1.0
		THALLIUM, TOTAL	2.0	u	MG/KG	2.0
BLANK1	89C141B-MB1	MERCURY, TOTAL	0.10	u	MG/KG	0.10
BLANK2	89C141B-MB2	MERCURY, TOTAL	0.10	u	MG/KG	0.10
BLANK3	89C141B-MB3	MERCURY, TOTAL	0.10	u	MG/KG	0.10

ROY F. WESTON INC.

INORGANICS LABORATORY CONTROL STANDARDS REPORT 10/24/89

AMPLE	SITE ID	ANALYTE	SPIKED SAMPLE	SPIKED AMOUNT	UNITS	%RECOV
CS1	89L1101-LC1	SILVER, LCS	113	100	MG/KG	113
		ALUMINUM, LCS	1140	1000	MG/KG	114
		BARIUM, LCS	1040	1000	MG/KG	104
		BERYLLIUM, LCS	51.6	50.0	MG/KG	103
		CALCIUM, LCS	5600	5000	MG/KG	112
		CADMIUM, LCS	59.3	50.0	MG/KG	119
		COBALT, LCS	552	500	MG/KG	110
		CHROMIUM, LCS	110	100	MG/KG	110
		COPPER, LCS	270	250	MG/KG	108
		IRON, LCS	1070	1000	MG/KG	107
		POTASSIUM, LCS	5720	5000	MG/KG	114
		MAGNESIUM, LCS	5480	5000	MG/KG	110
		MANGANESE, LCS	164	150	MG/KG	109
		SODIUM, LCS	5170	5000	MG/KG	103
		NICKEL, LCS	439	400	MG/KG	110
		ANTIMONY, LCS	669	600	MG/KG	112
		VANADIUM, LCS	553	500	MG/KG	111
		ZINC, LCS	236	200	MG/KG	118
LCS2	89L1101-LC2	SILVER, LCS	113	100	MG/KG	113
		ALUMINUM, LCS	1110	1000	MG/KG	111
		BARIUM, LCS	1010	1000	MG/KG	101
		BERYLLIUM, LCS	50.3	50.0	MG/KG	101
		CALCIUM, LCS	5460	5000	MG/KG	109
		CADMIUM, LCS	57.2	50.0	MG/KG	114
		COBALT, LCS	538	500	MG/KG	108
		CHROMIUM, LCS	107	100	MG/KG	107
		COPPER, LCS	264	250	MG/KG	105
		IRON, LCS	1050	1000	MG/KG	105
		POTASSIUM, LCS	5580	5000	MG/KG	112
		MAGNESIUM, LCS	5340	5000	MG/KG	107
		MANGANESE, LCS	160	150	MG/KG	107
		SODIUM, LCS	5030	5000	MG/KG	101
		NICKEL, LCS	430	400	MG/KG	108
		ANTIMONY, LCS	700	600	MG/KG	117
		VANADIUM, LCS	540	500	MG/KG	108
		ZINC, LCS	231	200	MG/KG	115
LCS1	89L1100-LC1	ARSENIC, LCS	5.6	6.0	MG/KG	93.3
		LEAD, LCS	6.3	6.0	MG/KG	105

ROY F. WESTON INC.

INORGANICS LABORATORY CONTROL STANDARDS REPORT 10/24/89

LE	SITE ID	ANALYTE	SPIKED SAMPLE	SPIKED AMOUNT	UNITS	%RECOV
	89L1100-LC1	SELENIUM, LCS	5.2	6.0	MG/KG	87.3
		THALLIUM, LCS	5.4	6.0	MG/KG	89.7
	89L1100-LC2	ARSENIC, LCS	5.6	6.0	MG/KG	93.0
		LEAD, LCS	6.0	6.0	MG/KG	100
		SELENIUM, LCS	5.5	6.0	MG/KG	91.7
		THALLIUM, LCS	5.4	6.0	MG/KG	90.0
	89C141B-LC1	MERCURY, LCS	1.1	1.0	MG/KG	112
	89C141B-LC2	MERCURY, LCS	1.1	1.0	MG/KG	107
	89C141B-LC3	MERCURY, LCS	1.1	1.0	MG/KG	109

ROY F. WESTON INC.

INORGANICS DUPLICATE SPIKE REPORT 10/24/89

IENT: USATHAMA-UM

RK ORDER: 2281-08-10-0000

WESTON BATCH #: 8910L200

MPLE	SITE ID	ANALYTE	SPIKE#1 %RECOV	SPIKE#2 %RECOV	%DIFF
S2	89L1101-LC2	SILVER, LCS	113	113	0.14
		ALUMINUM, LCS	114	111	2.5
		BARIUM, LCS	104	101	2.4
		BERYLLIUM, LCS	103	101	2.6
		CALCIUM, LCS	112	109	2.5
		CADMIUM, LCS	119	114	3.6
		COBALT, LCS	110	108	2.6
		CHROMIUM, LCS	110	107	2.5
		COPPER, LCS	108	105	2.5
		IRON, LCS	107	105	2.2
		POTASSIUM, LCS	114	112	2.4
		MAGNESIUM, LCS	110	107	2.6
		MANGANESE, LCS	109	107	2.4
		SODIUM, LCS	103	101	2.7
		NICKEL, LCS	110	108	2.1
		ANTIMONY, LCS	112	117	4.5
		VANADIUM, LCS	111	108	2.3
		ZINC, LCS	118	115	2.3
CS2	89L1100-LC2	ARSENIC, LCS	93.3	93.0	0.35
		LEAD, LCS	105	100	4.5
		SELENIUM, LCS	87.3	91.7	4.8
		THALLIUM, LCS	89.7	90.0	0.37
CS2	89C141B-LC2	MERCURY, LCS	112	107	4.5

APPENDIX B

ANALYTICAL METHOD FOR TNT, HMX, RDX

EXPLOSIVES IN SOIL

I. SUMMARY

A. Analytes:

HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
RDX	Hexahydro-1,3,5-trinitro-s-triazine
NB	Nitrobenzene
1,3-DNB	1,3-Dinitrobenzene
1,3,5-TNB	1,3,5-Trinitrobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
Tetryl	2,4,6-Trinitrophenylmethylnitramine

B. Matrix: Soil or sediment

C. General Method: An aliquot of soil is extracted with acetonitrile. The acetonitrile is diluted with methanol and water, and the resultant solution is injected onto the HPLC for analysis.

II. APPLICATION

A. Tested Concentration Range:

HMX	1.27-140 ug/g
RDX	0.98- 80.0 ug/g
NB	0.42- 60.0 ug/g
1,3-DNB	0.59- 60.0 ug/g
1,3,5-TNB	2.09- 60.0 ug/g
2,4-DNT	0.42- 60.0 ug/g
2,6-DNT	0.40- 60.0 ug/g
2,4,6-TNT	1.92-100.0 ug/g
Tetryl	0.32- 24.9 ug/g

B. Sensitivity:

Peak Height in mm at an Attenuation of 2^4

HMX	48 mm for 14 ug/g
RDX	48 mm for 8.0 ug/g
NB	26 mm for 6.0 ug/g
1,3-DNB	53 mm for 6.0 ug/g
1,3,5-TNB	44 mm for 6.0 ug/g
2,4-DNT	31 mm for 10.0 ug/g
2,6-DNT	17 mm for 6.0 ug/g
2,4,6-TNT	45 mm for 6.0 ug/g
Tetryl	26 mm for 8.0 ug/g

C. Detection Limits:

HMX	1.27 ug/g
RDX	0.98 ug/g
NB	0.42 ug/g
1,3-DNB	0.59 ug/g
1,3,5-TNB	2.09 ug/g
2,4-DNT	0.42 ug/g
2,6-DNT	0.40 ug/g
2,4,6-TNT	1.92 ug/g
Tetryl	0.32 ug/g

D. Interferences:

1. Any compound that is extracted from soil that gives a retention time similar to the nitro-compounds and absorbs at 250 nm.

E. Analysis Rate:

After instrument calibration, one analyst can analyze two samples in one hour. One analyst can conduct sample preparation at a rate of three samples per hour. One analyst doing both sample preparation and the HPLC analysis can run 16 samples in an 8-hour day.

F. Safety information:

Work in well-ventilated areas. Wear adequate protective clothing to avoid skin contact. Wash skin with soap and water thoroughly immediately after contact.

TNB, HMX, RDX, Tetryl, and TNT's are classified as Explosives A by DOT. Avoid extreme temperatures and pressures.

III. APPARATUS AND CHEMICALS

A. Glassware/Hardware

1. Syringes: 10 uL, 50 uL, 100 uL, 1 mL syringe (Hamilton 1005 TEFLL)

2. Vials with Teflon-lined caps or septa. Nominal volume of 1.8 m L, 4.0 mL and 8.0 mL.

3. B-D Glaspak disposable syringes, 5 mLs, with frosted tip

4. 0.2 micron fluorocarbon filters

5. Micropipettes, 200 uL

6. Hypo needles

7. 2 mL. pipette

B. Instrumentation

1. Perkin-Elmer Series 4 High Performance Liquid Chromatograph (HPLC) equipped with a Perkin-Elmer ISS100 Auto-Injector and Micromeritrics Model 786 UV/VIS variable wavelength detector. Hewlett-Packard 3390 recording integrator in peak height mode was used to record data output. ISS 100 auto injector is equipped with a temperature controlled sample tray jto refrigerate extracts.

2. Analytical Balance

   Capable of weighing 0.01 grams for sample preparation and 0.1 mg for standard preparation. Mettler AE 163 or equivalent.

3. Parameters

   a. Columns:

      1) DuPont Zorbax^R C-8 4.6 mm i.d. x 25 cm HPLC column with a particle size of 5-6 microns.

      2) DuPont Permaphase^R ODS guard column. (optional)

b. Mobile Phase: The water/methanol ratio must be adjusted as described in the calibration Section V.A.5.c to obtain optimum peak separation.

52% methanol

48% water

c. Flow: 1.6 mL/min with a pressure of approximately 2860 psig.

d. Detector: 250 nm

e. Injection Volume: 50 uL

f. Retention Times:

	Minutes
HMX	3.30- 3.60
RDX	4.55- 4.70
NB	7.95- 9.00
1,3-DNB	7.30- 8.00
1,3,5-TNB	6.35- 6.40
2,4-DNT	11.00-13.10
2,6-DNT	10.60-12.40
2,4,6-TNT	10.05-10.90
Tetryl	9.15- 9.70

C. Analytes

1. Chemical Abstracts Registry Numbers

HMX	2691-41-0
RDX	121-82-4
NB	98-95-3
1,3-DNB	99-65-01
1,3,5-TNB	99-35-4
2,4-DNT	121-14-2
2,6-DNT	606-20-2
2,4,6-TNT	118-96-7
Tetryl	35572-78-2

2. Chemical Reactions

a. RDX and HMX can undergo alkaline hydrolysis.

b. RDX and HMX degrade at temperatures greater than 80_C in an organic solvent.

3. Physical Properties

	Formula	Mol. Wt.	M.P. (°C)	B.P. (°C)
HMX	$C_4H_8N_8O_8$	296.6	276	-
RDX	$C_3H_6N_6O_6$	222.12	205	-
NB	$C_6H_5NO_2$	123.11	6	211
1,3-DNB	$C_6H_4N_2O_4$	168.11	90	302
1,3,5-TNB	$C_6H_3N_3O_6$	213.11	122	315
2,4-DNT	$C_7H_6N_2O_4$	182.14	71	300 (decomposes)
2,6-DNT	$C_7H_6N_2O_4$	182.14	66	-
2,4,6-TNT	$C_7H_5N_3O_6$	227.13	82	240 (decomposes)
Tetryl	$C_7H_5N_5O_8$	287.15	131	187

D. Reagents and SARMs:

1. Acetonitrile, distilled in glass for HPLC use
2. Methanol, distilled in glass for HPLC use
3. Water, distilled in glass for HPLC use
4. USATHAMA Standard Soil
5. SARMs

HMX	SARM No.	1217(PA 1303)
RDX	SARM No.	1130(PA 1302)
NB	SARM No.	(PA 1306)
1,3-DNB	SARM No.	2250(PA 1305)
1,3,5-TNB	SARM No.	1154(PA 1300)
2,4-DNT	SARM No.	1147(PA 1298)
2,6-DNT	SARM No.	1148(PA 1299)
2,4,6-TNT	SARM No.	1129(PA 1297)
Tetryl	SARM No.	1149(PA 1301)

IV. CALIBRATION

A. Initial Calibration

1. Preparation of Standards:
   a. Stock calibration solutions containing approximately 10,000 mg/L of a nitro-compound are prepared by accurately weighing ca. 50 mg of a SARM into a 5 mL serum bottle and dissolving the nitro-compound in 5 mL of acetonitrile pipetted into the bottle. All stock solutions prepared in this manner and stored in a freezer ($0^{o}C$ to -4^{o} C) have remained stable for a period of 6 months.

   b. Intermediate Calibration Standards: All compounds appear to be stable for at least 3 months.

      1) Intermediate Calibration Standard A (high level): Combine the appropriate volumes of stock calibration standard as shown below. Dilute to 5 mL with acetonitrile and seal with a Teflon-lined cap. Store in the dark at 0^{o}-4^{o}C. The resulting solution will have the concentrations indicated in the following table.

Nitro-compound	uL of Stock Cal Std	Resulting concentration (ug/mL)
HMX	175	350
RDX	100	200
NB	75	150
1,3-DNB	75	150
1,3,5-TNB	75	150
2,4-DNT	75	150
2,6-DNT	75	150
2,4,6-TNT	125	250
Tetryl	100	200

2) *Intermediate Calibration Standard B (low level):* 1:10 dilution of the Intermediate Calibration Standard A is made in Acetonitrile. Seal with a Teflon-lined cap and store in the dark at 0°-4°C. The resulting solution will have the following concentrations:

Nitro-Compound	Resulting conc. (ug/mL)
HMX	35.0
RDX	20.0
NB	15.0
1,3-DNB	15.0
1,3,5-TNB	15.0
2,4-DNT	15.0
2,6-DNT	15.0
2,4,6-TNT	25.0
Tetryl	20.0

c. Working Calibration Standards: Using the following table, prepare a series of ten calibration standards. Place the mobile phase into a 1-mL serum vial. Inject the indicated volumes of intermediate calibration standard A or B into the acetonitrile with a microliter syringe. Seal the vial with a teflon-lined septum and cap. Mix well. These solutions are prepared fresh daily and kept in the dark.

WORKING CALIBRATION STANDARDS

	Amt. (uL) Intermed. Cal. Std. to Add			Resulting Concentration (ug/L)			
Conc.	A	B	Amt. (uL) Mobile Phase to Add	HMX	2,4,6-TNT	Tetryl RDX	1,3-DNB 1,3,5-TNB 2,6-DNT 2,4-DNT
0	0	0	2.0	-	-	-	-
0.2 X	-	1.0	999.0	35	25	20	15
0.5 X	-	2.5	997.5	87.5	62.5	50	37.5
1 X	-	5	995.0	175	125	100	75
2 X	-	10	990.0	350	250	200	150
5 X	-	25	975.0	875	625	500	375
10 X	5	-	995.0	1750	1250	1000	750
20 X	10	-	990.0	3500	2500	2500	1500
50 X	25	-	975.0	8750	6250	5000	3750
100 X	50	-	950.0	17500	12500	10000	7500

2. Instrument Calibration

   a. Set up the instrument according to the manufacturer's recommendations.

   b. Mobile Phase is analyzed as a blank to verify a stable baseline.

   c. Analyze the medium calibration standard (10X) to verify peak separation and retention times.

   d. Analyze the calibration standards prepared in Section IV.A.1.

3. Analysis of Calibration Data

   a. Tabulate the calibration standard concentration versus the peak height response for each calibration standard.

   b. Perform a linear regression analysis on the calibration data plotting peak height vs. concentration in ug/l.

4. Calibration Checks

   a. After completion of analyses of samples, a calibration standard at the highest concentration is analyzed. The response must agree within 25% for that concentration from the first seven calibration curves. Thereafter, the response must agree within two standard deviations of the mean response for that concentration. If it does not, the calibration standard will be reanalyzed. If the calibration standard fails this test, initial calibration must be performed, and all samples analyzed since the last acceptable calibration must be reanalyzed.

   b. No certified calibration check standards are available for these compounds.

B. Daily Calibration

1. Prior to analyses each day, a high calibration standard will be analyzed. For the first seven determinations at this concentration, the response must agree within 25% of the mean of all previous responses. After seven determinations, the response must agree within +/- two standard deviations of the mean response for previous determinations at this concentration.

2. If the calibration standard fails this test, it will be reanalyzed. If the calibration standard fails the second test, the system will have failed daily calibration, and initial calibration will be performed.

3. After completion of sample analyses each day, the high calibration standard will be analyzed again. The response for this calibration standard will be subjected to the criteria discussed in Section IV.B.1, above. If the response fails the criteria, the standard will be reanalyzed. If the second response fails the test, the system will have failed calibration, and initial calibration will be performed. All samples analyzed since the last acceptable calibration must be reanalyzed.

V. Certification Testing

A. Control Spikes:

To a series of ten 5-mL serum vials, approximately one gram of soil is accurately weighed into each vial. Using a syringe, the volumes of intermediate calibration standard indicated in the following table are injected onto the soil. The serum vial is covered with a septum and shaken until the soil no longer looks wet (approximately 60 seconds). The sample must equilibrate at least one hour. The septum is removed and the indicated amount (see Table below) of acetonitrile is pipetted onto the soil. The septum is replaced and the vial is capped. The sealed sample is shaken by hand for approximately 2-3 minutes. The sample is prepared via the procedure given in this method, to give the target concentrations in the following table.

CONTROL SPIKES

Conc.	Amt. (uL) Intermed. Cal. Std. to Add A	B	Amt. (uL) Aceto Nitrile to Add	Resulting Concentration (ug/g) HMX	2,4,6 TNT	Tetryl RDX	1,3-DNB 1,3,5-TNB 2,6-DNT 2,6-DNT NB
0	0	0	2000	0	0	0	0
0.2 X	-	8.0	1992	0.28	0.2	0.16	0.12
0.5 X	-	20	1980	0.70	0.5	0.4	0.3
1 X	4	-	1996	1.40	1.0	0.8	0.6
2 X	8	-	1992	2.80	2.0	1.6	1.2
5 X	20	-	1980	7.0	5.0	4.0	3.0
10 X	40	-	1960	14.0	10.0	8.0	6.0
20 X	80	-	1920	28.0	20.0	16.0	12.0
50 X	200	-	1800	70.0	50.0	40.0	30.0
100 X	400	-	1600	140.0	100.0	80.0	60.0

VI. SAMPLE HANDLING STORAGE

A. Sampling Procedure: The stability of explosives in soil is not truly known. Precautions should be taken to avoid prolonged exposure to light and heat.

B. Containers: Wide-mouth amber glass bottles with teflon-lined lids.

C. Storage Conditions: Samples should be maintained at 4_ C from the time of collection to the time of analysis. No chemical preservatives are necessary.

D. Holding Time Limits: 7 days to extraction; 40 days to analysis from the time of extraction.

E. Solution Verification: No certified check standards are available.

VII. PROCEDURE

A. Separations

1. Accurately weigh 1 gram of soil into a 5-mL serum vial and pipette 4 mL of acetonitrile onto the soil.

2. Place a septum and cap on the vial and shake the vial thoroughly by hand for 2-3 minutes.

3. The extract is then filtered using the following technique.

   A 5-mL syringe is fitted with a needle. After the extract is drawn into the syringe barrel, a Fluorocarbon 0.2 micron disposable filter is attached in place of the needle. The sample is then slowly forced through the filter into a 4.0 mL teflon capped vial and stored until the extract is diluted and analyzed by HPLC. (Step 4-C.)

4. Preparation of sample extracts and spikes for injection is performed the day of analysis.

   a. Using a disposable micropipette, accurately measure 200 uL of filtered extract into a 1-mL vial. Accurately measure 600 uL of a 33% methanol/67% water solutiojn onto the filtered sample. This will produce 800 uL of extracted sample in mobile phase.

   b. Place a septum cap on the vial. Shake the vial well to thoroughly mix. Store in the dark at 0^{o}-4^{o} C until ready to analyze.

B. Chemical Reactions - None. Compounds are read directly.

C. Instrumental Analysis:

   1. Set the chromatographic conditions as follows:

	Time (minutes)	Flow (mLs/min.)	MeCN %	MeOH %	HOH %
Equilibrium	2	1.6	16	34	50
Analysis Run	20	1.6	16	34	50

   2. All standards and extracts should be in chilled tray (4^{o} C)

   3. Using the auto-injector manufacturer's recommended procedure, introduce 50 uL of the medium level calibration standard into the

chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes and separation of the nitrobenzene and tetryl. If necessary, adjust the water/ methanol ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally, the column ages rapidly the first 24 hours, after which it is fairly stable.

4) Once good peak separation is obtained, introduce 50 uL of each working calibration standard and sample into the chromatographic system using the auto-injector manufacturer's recommended procedure.

VIII. CALCULATIONS

A. The diluted extract concentration is read or calculated from the instrument calibration curve.

B. $$\text{Sample Concentration (ug/g)} = \text{extract conc} \times \frac{B \times D}{A \times C}$$

where:

A = sample weight (dry weight)

B = mL acetonitrile used to extract sample

C = mL acetonitrile extract diluted into mobile phase

D = final volume in mL of mobile phase prepared for injection

NOTE: When samples are prepared according to this method (1 gram extracted into 8 mL of mobile phase), the above calculation becomes:

Sample Concentration (ug/g) = extract conc (ug/l) X 0.008

IX. DAILY QUALITY CONTROL

A. Control Samples

1. Intermediate Spiking Standard A and B are made according to Section IV just as calibration standards.

2. Daily control samples are prepared in a manner identical to that described in Section V. A total of three control spikes are required on a daily basis: two at 10X and one at 2X. They will have the following concentrations.

Conc.	Amt (uL) Intermed. Spiking A to add to 2.0 mls Acetonitrile	HMX	2,4,6-TNT	Tetryl RDX	2,4-DNT 2,6-DNT 1,3-DNB 1,3,5-TNB NB
2X	8	2.8	2.0	1.6	1.2
10X	40	14.0	10.0	8.0	6.0

3. At least one method blank using the USATHAMA Standard Soil is also analyzed with each analytical lot.

4. At least one matrix spike (actual sample) at 10X is analyzed for each analytical lot or at a frequency of 10%, whichever is more frequent.

B. Control Charts:

1. Average Percent Recovery (X)

   a. Percent recoveries for the 10X certification spikes from days 1 and 2 are averaged to obtain the first value to be plotted.

   b. Percent recoveries for the 10X certification spikes from days 3 and 4 are averaged to obtained the second value to be plotted.

   c. Percent recoveries for the method spikes closest to ;the certification 10X concentration from the first day of analyses are averaged to obtain the third value to be plotted.

   d. Values from a, b, and c are averaged to determine the central line of the control chart.

   e. Differences in percent recoveries for each pair of values in a, b, and c are averaged to obtain R.

   f. The upper and lower warning limits are +/- 1.25 R from the central line.

g. The upper and lower control limits are +/- 1.88 R from the central line.

2. Difference in percent recoveries (R)

   a. The value for R obtained in Section IX.B.1.e, above, is the base line of the control chart.

   b. The warning limit is 2.511 R.

   c. The control limit is 3.267 R.

3. Three Point Moving Average X

   a. The average percent recovery from the 5 ug/g concentration from the first three days of certification testing is the first point to be plotted.

   b. Subsequent points to be plotted are the average percent recoveries from the 5 ug/g concentration from the next group of three determinations (e.g., certification days 2, 3, and 4; certification days 3 and 4 and the first day of analysis; certification day 4, day 1 of analysis, and day 2 of analysis; etc.)

   c. The central point on the control chart is the average of the plotted points and changes with each added point.

   d. The range for each point is the difference between the highest and lowest values in each group of three determinations. The average range (MAR) is used to define the warning and control limits.

   e. The upper and lower warning limits are +/- 0.682 MAR, respectively.

   f. The upper and lower control limits are +/- 1.023 MAR, respectively.

4. Three point Moving Average R:

   a. The base line is the MAR.

   b. The warning limit is 2.050 MAR.

   c. The control limit is 2.575 MAR.

5. Certified Calibration Check Standard:

   a. If available, two certified calibration check standards are analyzed with samples.

   b. For the first 20 determinations, results must fall within the acceptable range specified by the source of the standard.

   c. After 20 determinations, the mean value of the 20 determinations is used as the central line of a control chart.

   d. Warning limits are +/- two standard deviations.

   e. Control limits are +/- three standard deviations.

X. REFERENCES

A. USATHAMA Method 2C Cyclotrimethylenetrinitramine (RDX) in Soil and Sediment Samples, 12-3-80.

B. USATHAMA Method 8H Explosives in Water by HPLC, 12-27-82.

XI. DATA

A. Off-the-Shelf Analytical Reference Materials Characterization: Not Applicable

B. Initial Calibration

1. Response versus concentration data: See attached.

2. Response versus concentration graphs: See attached.

3. LOF Tests: Not applicable.

4. ZI Tests: Not applicable.

C. Daily Calibration

1. Response: Not applicable.

2. Required percentage or two standard deviation limits: Not applicable.

D. Standard Certification Samples

1. Tabulation and graph of found versus target concentrations: See attached.

2. LOF and ZI tests for the pooled data: See attached.

3. Calculated least squares linear regression line, confidence bounds, reporting limit, accuracy, standard deviation, percent imprecision, and percent inaccuracy: See attached.

4. Chromatograms: Attached

APPENDIX C

STATISTICAL ANALYSES OF UMDA STATIC PILES FOR DIFFERENCES IN EXPLOSIVES CONCENTRATIONS BETWEEN SAMPLING DAYS

STATIC PILE 1

----------GLM ANOVA------------------------------------

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report

ANOVA Table for Response Variable: HMX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	13329.32	3332.33	9.88	0.0001
ERROR	20	6743.628	337.1814		
TOTAL(Adj)	24	20072.95			

Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	25	94.388	
A: DAY			
0	5	119.6	8.21196
10	5	124.4	8.21196
20	5	70.08	8.21196
44	5	84.44	8.21196
90	5	73.42	8.21196

GLM ANOVA (Multiple Comparisons)

Scheffe's Procedure

Response Variable: HMX Factor(A,DAY) Error Term: ERROR

Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(20)	70.08	...SS
B(90)	73.42	...SS
C(44)	84.44	....S
D(0)	119.6	SS...
E(10)	124.4	SSS..

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report

ANOVA Table for Response Variable: RDX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	1068477	267119.3	19.52	0.0000
ERROR	20	273666.8	13683.34		
TOTAL(Adj)	24	1342144			

Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	477.44	
A: DAY			
0	5	775.6	52.31317
10	5	647	52.31317
20	5	427.6	52.31317
44	5	324.4	52.31317
90	5	212.6	52.31317

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	212.6	...SS
B(44)	324.4	...SS
C(20)	427.6	....S
D(10)	647	SS...
E(0)	775.6	SSS..

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	3621969	905492.3	82.08	0.0000
ERROR	20	220634	11031.7		
TOTAL(Adj)	24	3842603			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	25	392.964	
A: DAY			
0	5	1144	46.9717
10	5	270.2	46.9717
20	5	271.2	46.9717
44	5	172.62	46.9717
90	5	106.8	46.9717

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	106.8	....S
B(44)	172.62	....S
C(10)	270.2	....S
D(20)	271.2	....S
E(0)	1144	SSSS.

```
-----GLM ANOVA--------------------
Expected Mean Squares ... Balanced Case

Source      DF  Expectation of Mean Square (S stands for ERROR).
A           4   S+nA
S           20  S
Analysis of Variance Report
ANOVA Table for Response Variable: HMX
Source          DF    Sum-Squares  Mean Square   F-Ratio   Prob>F
Error Term
A (DAY      ) 4      15180.16     3795.04       11.76    0.0000
ERROR
ERROR           20     6454.8       322.74
TOTAL(Adj)      24     21634.96

Means & Standard Errors for Y = HMX
Term Count    Mean            Std.Error
ALL   25      151.04
A: DAY
0      5      180.2           8.034177
10     5      125.2           8.034177
20     5      127.4           8.034177
44     5      180.6           8.034177
90     5      141.8           8.034177

GLM ANOVA                       (Newman / Keul's Range Test)
Response Variable: HMX    Factor(A,DAY)     Error Term: ERROR
Summary Results Á= .05          Level Codes
Code(Level)       Mean          ABCDE
A(10)             125.2         ...SS
B(20)             127.4         ...SS
C(90)             141.8         ...SS
D(0)              180.2         SSS..
E(44)             180.6         SSS..

GLM ANOVA                       (Multiple Comparisons)
Scheffe's Procedure
Response Variable: HMX    Factor(A,DAY)     Error Term: ERROR
Summary Results Á= .05          Level Codes
Code(Level)       Mean          ABCDE
A(10)             125.2         ...SS
B(20)             127.4         ...SS
C(90)             141.8         ....S
D(0)              180.2         SS...
E(44)             180.6         SSS..

Expected Mean Squares ... Balanced Case
Source      DF  Expectation of Mean Square (S stands for ERROR).
A           4   S+nA
S           20  S
```

Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	790287.1	197571.8	8.59	0.0003
ERROR					
ERROR	20	459770.8	22988.54		
TOTAL(Adj)	24	1250058			

Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	837.08	
A: DAY			
0	5	1008	67.8064
10	5	972.8001	67.8064
20	5	723.4	67.8064
44	5	939.2	67.8064
90	5	542.0001	67.8064

GLM ANOVA (Newman / Keul's Range Test)
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	542.0001	..SSS
B(20)	723.4	..SSS
C(44)	939.2	SS...
D(10)	972.8001	SS...
E(0)	1008	SS...

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	542.0001	..SSS
B(20)	723.4	.....
C(44)	939.2	S....
D(10)	972.8001	S....
E(0)	1008	S....

STATIC PILE 2

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Data Base Name C:\ncss\static2
Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	8.0802E07	2.0200E07	185.73	0.0000
ERROR					
ERROR	20	2175296	108764.8		
TOTAL(Adj)	24	8.2978E07			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	25	1451.6	
A: DAY			
0	5	4984	147.4888
10	5	1114	147.4888
20	5	719	147.4888
44	5	240.7999	147.4888
90	5	200.2002	147.4888

GLM ANOVA (Newman / Keul's Range Test)
C:\ncss\static2
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	200.2002	...SS
B(44)	240.7999	..SSS
C(20)	719	.S..S
D(10)	1114	SS..S
E(0)	4984	SSSS.

GLM ANOVA (Multiple Comparisons)
C:\ncss\static2 Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	200.2002	...SS
B(44)	240.7999	...SS
C(20)	719	....S
D(10)	1114	SS..S
E(0)	4984	SSSS.

GLM ANOVA------------------------------------
Data Base Name C:\ncss\static2
Analysis of Variance Report
ANOVA Table for Response Variable: HMX

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	15180.16	3795.04	11.76	0.0000
ERROR					
ERROR	20	6454.8	322.74		
TOTAL(Adj)	24	21634.96			

Data Base Name C:\ncss\static2
Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	25	151.04	
A: DAY			
0	5	180.2	8.034177
10	5	125.2	8.034177
20	5	127.4	8.034177
44	5	180.6	8.034177
90	5	141.8	8.034177

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: HMX Factor(A,DAY) Error Term: ERROR
Summary Results à= .05

Code(Level)	Mean	Level Codes ABCDE
A(10)	125.2	...SS
B(20)	127.4	...SS
C(90)	141.8	....S
D(0)	180.2	SS...
E(44)	180.6	SSS..

Data Base Name C:\ncss\static2
Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Data Base Name C:\ncss\static2
Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	790287.1	197571.8	8.59	0.0003
ERROR	20	459770.8	22988.54		
TOTAL(Adj)	24	1250058			

Data Base Name C:\ncss\static2
Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	837.08	
A: DAY			
0	5	1008	67.8064
10	5	972.8001	67.8064
20	5	723.4	67.8064
44	5	939.2	67.8064
90	5	542.0001	67.8064

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results à= .05

Code(Level)	Mean	Level Codes ABCDE
A(90)	542.0001	..SSS
B(20)	723.4	.....
C(44)	939.2	S....
D(10)	972.8001	S....
E(0)	1008	S....

STATIC PILE 3

GLM ANOVA--------------------

Data Base Name C:\ncss\static3

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report

ANOVA Table for Response Variable: HMX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	2046.16	511.54	0.73	0.5833
ERROR	20	14053.6	702.68		
TOTAL(Adj)	24	16099.76			

Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	25	184.36	
A: DAY			
0	5	193.8	11.85479
10	5	174.6	11.85479
20	5	174	11.85479
44	5	195.2	11.85479
90	5	184.2	11.85479

GLM ANOVA (Newman / Keul's Range Test)

C:\ncss\static3

Response Variable: HMX Factor(A,DAY) Error Term: ERROR

Summary Results Á= .05

Code(Level)	Mean	Level Codes ABCDE
A(20)	174	.....
B(10)	174.6	.....
C(90)	184.2	.....
D(0)	193.8	.....
E(44)	195.2	.....

GLM ANOVA (Multiple Comparisons)

C:\ncss\static3

Scheffe's Procedure

Response Variable: HMX Factor(A,DAY) Error Term: ERROR

Summary Results Á= .05

Code(Level)	Mean	Level Codes ABCDE
A(20)	174	.....
B(10)	174.6	.....
C(90)	184.2	.....
D(0)	193.8	.....
E(44)	195.2	.....

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	243163.8	60790.96	2.10	0.1192
ERROR					
ERROR	20	579842	28992.1		
TOTAL(Adj)	24	823005.8			

Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	1016.08	
A: DAY			
0	5	1076	76.14735
10	5	1178	76.14735
20	5	960.8001	76.14735
44	5	963.6	76.14735
90	5	902	76.14735

GLM ANOVA (Newman / Keul's Range Test)
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	902	.....
B(20)	960.8001	.....
C(44)	963.6	.....
D(0)	1076	.....
E(10)	1178	.....

GLM ANOVA (Multiple Comparisons)
C:\ncss\static3
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .1 Level Codes

Code(Level)	Mean	ABCDE
A(90)	902	.....
B(20)	960.8001	.....
C(44)	963.6	.....
D(0)	1076	.....
E(10)	1178	.....

Data Base Name C:\ncss\static3
Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Data Base Name C:\ncss\static3
Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	9.8297E07	2.4574E07	169.76	0.0000
ERROR					
ERROR	20	2895209	144760.4		
TOTAL(Adj)	24	1.0119E08			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	25	2451.24	
A: DAY			
0	5	5716	170.1531
10	5	3322	170.1531
20	5	2370	170.1531
44	5	517.2	170.1531
90	5	331	170.1531

GLM ANOVA (Newman / Keul's Range Test)
C:\ncss\static3
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	331	..SSS
B(44)	517.2	..SSS
C(20)	2370	SS.SS
D(10)	3322	SSS.S
E(0)	5716	SSSS.

GLM ANOVA (Multiple Comparisons)
C:\ncss\static3
Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	331	..SSS
B(44)	517.2	..SSS
C(20)	2370	SS.SS
D(10)	3322	SSS.S
E(0)	5716	SSSS.

STATIC PILE 4

Data Base Name C:\ncss\static4

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	6	S+nA
S	28	S

Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	6	6541097	1090183	1.38	0.2589
ERROR					
ERROR	28	2.2197E07	792782.9		
TOTAL(Adj)	34	2.8739E07			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	35	11393.71	
A: DAY			
0	5	11320	398.1916
2	5	11580	398.1916
4	5	11700	398.1916
8	5	11880	398.1916
16	5	11740	398.1916
20	5	10900	398.1916
44	5	10636	398.1916

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .1 Level Codes

Code(Level)	Mean	ABCDEFG
A(44)	10636	....
B(20)	10900	....
C(0)	11320	....
D(2)	11580	....
E(4)	11700	....
F(16)	11740	....
G(8)	11880	....

Data Base Name C:\ncss\static4
Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	6	S+nA
S	28	S

Data Base Name C:\ncss\static4

Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	6	27074.29	4512.381	0.30	0.9334
ERROR	28	426040	15215.71		
TOTAL(Adj)	34	453114.3			

Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	35	1222.857	
A: DAY			
0	5	1234	55.16469
2	5	1258	55.16469
4	5	1222	55.16469
8	5	1236	55.16469
16	5	1246	55.16469
20	5	1184	55.16469
44	5	1180	55.16469

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .1 Level Codes

Code(Level)	Mean	ABCDEFG
A(44)	1180	....
B(20)	1184	....
C(4)	1222	....
D(0)	1234	....
E(8)	1236	....
F(16)	1246	....
G(2)	1258	....

Data Base Name C:\ncss\static4
Description Data base created at 17:23:20 on 01-22-1991

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	6	S+nA
S	28	S

Analysis of Variance Report

ANOVA Table for Response Variable: HMX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	6	27074.29	4512.381	0.30	0.9334
ERROR	28	426040	15215.71		
TOTAL(Adj)	34	453114.3			

Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	35	1222.857	
A: DAY			
0	5	1234	55.16469
2	5	1258	55.16469
4	5	1222	55.16469
8	5	1236	55.16469
16	5	1246	55.16469
20	5	1184	55.16469
44	5	1180	55.16469

Scheffe's Procedure
Response Variable: **HMX** Factor(A,DAY) Error Term: ERROR
Summary Results Á= .1 Level Codes

Code(Level)	Mean	ABCDEFG
A(44)	1180	....
B(20)	1184	....
C(4)	1222	....
D(0)	1234	....
E(8)	1236	....
F(16)	1246	....
G(2)	1258	....

STATIC PILE 5

GLM ANOVA----Date/Time 02-07-1991 11:03:13
Data Base Name C:\ncss\static5

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	2.0878E08	5.2196E07	170.12	0.0000
ERROR	20	6136490	306824.5		
TOTAL(Adj)	24	2.1492E08			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	25	3381.72	
A: DAY			
0	5	7908	247.7194
10	5	5058	247.7194
20	5	3242	247.7194
44	5	526.2	247.7194
90	5	174.4001	247.7194

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Fesults Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	174.4001	..SSS
B(44)	526.2	..SSS
C(20)	3242	SS.SS
D(10)	5058	SSS.S
E(0)	7908	SSSS.

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report
ANOVA Table for Response Variable: HMX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	2.0878E08	5.2196E07	170.12	0.0000
ERROR	20	6136490	306824.5		
TOTAL(Adj)	24	2.1492E08			

	A	B	C	D	E	F
41	3/16/91					

Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	25	3381.72	
A: DAY			
0	5	7908	247.7194
10	5	5058	247.7194
20	5	3242	247.7194
44	5	526.2	247.7194
90	5	174.4001	247.7194

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: HMX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(90)	174.4001	..SSS
B(44)	526.2	..SSS
C(20)	3242	SS.SS
D(10)	5058	SSS.S
E(0)	7908	SSSS.

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	894204.2	223551	8.50	0.0004
ERROR	20	525730.4	26286.52		
TOTAL(Adj)	24	1419935			

Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	1024.76	
A: DAY			
0	5	1178	72.50726
10	5	1278	72.50726
20	5	1002.8	72.50726
44	5	740.8001	72.50726
90	5	924.2001	72.50726

Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCDE
A(44)	740.8001	...SS
B(90)	924.2001	....S
C(20)	1002.8	.....
D(0)	1178	S....
E(10)	1278	SS...

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	3	S+nA
S	16	S

Analysis of Variance Report
ANOVA Table for Response Variable: TNT

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	3	4.2011E07	1.4003E07	567.60	0.0000
ERROR					
ERROR	16	394750.9	24671.93		
TOTAL(Adj)	19	4.2406E07			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	20	942.5125	
A: DAY			
0	5	3452	70.24519
10	5	165.08	70.24519
20	5	63.17603	70.24519
44	5	89.79388	70.24519

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: TNT Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCD
A(20)	63.17603	...S
B(44)	89.79388	...S
C(10)	165.08	...S
D(0)	3452	SSS.

STATIC 6

Data Base Name C:\ncss\static6

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Date/Time 02-11-1991 14:48:34
Data Base Name C:\ncss\static6
Description Data base created at 17:32:03 on 01-22-1991

Analysis of Variance Report
ANOVA Table for Response Variable: HMX

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	4	20155.36	5038.84	3.93	0.0164
ERROR					
ERROR	20	25666	1283.3		
TOTAL(Adj)	24	45821.36			

Data Base Name C:\ncss\static6
Means & Standard Errors for Y = HMX

Term	Count	Mean	Std.Error
ALL	25	317.16	
A: DAY			
0	5	309.6	16.02061
10	5	372	16.02061
20	5	289.6	16.02061
44	5	309.8	16.02061
90	5	304.8	16.02061

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: HMX Factor(A,DAY) Error Term: ERROR
Summary Results à= .05

Code(Level)	Mean	Level Codes ABCDE
A(20)	289.6	....S
B(90)	304.8	.....
C(0)	309.6	.....
D(44)	309.8	.....
E(10)	372	S....

Data Base Name C:\ncss\static6
Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	4	S+nA
S	20	S

Data Base Name C:\ncss\static6
Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	4	1573263	393315.8	5.08	0.0055
ERROR	20	1549667	77483.36		
TOTAL(Adj)	24	3122930			

Data Base Name C:\ncss\static6
Means & Standard Errors for Y = RDX

Term	Count	Mean	Std.Error
ALL	25	1593.52	
A: DAY			
0	5	1572	124.4856
10	5	1974	124.4856
20	5	1556	124.4856
44	5	1191.6	124.4856
90	5	1674	124.4856

```
GLM ANOVA                          (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX    Factor(A,DAY)    Error Term: ERROR
Summary Results à= .05         Level Codes
Code(Level)      Mean          ABCDE
A(44)            1191.6        ....S
B(20)            1556          .....
C(0)             1572          .....
D(90)            1674          .....
E(10)            1974          S....

Data Base Name C:\ncss\static6
Expected Mean Squares ... Balanced Case
Source     DF  Expectation of Mean Square (S stands for ERROR).
A          4   S+nA
S          20  S

Analysis of Variance Report
ANOVA Table for Response Variable: TNT
Source         DF    Sum-Squares   Mean Square   F-Ratio   Prob>F
Error Term
A (DAY      ) 4      3.0025E08     7.5062E07     108.06    0.0000
ERROR
ERROR          20    1.3892E07     694642
TOTAL(Adj)     24    3.1414E08

Means & Standard Errors for Y = TNT
Term Count    Mean            Std.Error
ALL    25     5815.2
A: DAY
0       5     9858            372.731
10      5     9440            372.731
20      5     5956            372.731
44      5     1736            372.731
90      5     2086            372.731

GLM ANOVA                          (Multiple Comparisons)
Scheffe's Procedure
Response Variable: TNT    Factor(A,DAY)    Error Term: ERROR
Summary Results à= .05         Level Codes
Code(Level)      Mean          ABCDE
A(44)            1736          ..SSS
B(90)            2086          ..SSS
C(20)            5956          SS.SS
D(10)            9440          SSS..
E(0)             9858          SSS..
```

MC-1

```
C:\ncss\mc-1
Expected Mean Squares ... Balanced Case
Source     DF  Expectation of Mean Square (S stands for ERROR).
A          3   S+nA
S          16  S
```

Analysis of Variance Report
ANOVA Table for Response Variable: **HMX**

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	3	25575.89	8525.298	63.62	0.0000
ERROR					
ERROR	16	2144.132	134.0083		
TOTAL(Adj)	19	27720.03			

Data Base Name C:\ncss\mc-1
Description Data base created at 17:37:56 on 01-22-1991

Means & Standard Errors for Y = **HMX**

Term	Count	Mean	Std.Error
ALL	20	125.215	
A: DAY			
0	5	169.2	5.177031
10	5	140.2	5.177031
20	5	71.06	5.177031
44	5	120.4	5.177031

GLM ANOVA (Multiple Comparisons)
C:\ncss\mc-1
Scheffe's Procedure
Response Variable: **HMX** Factor(A,DAY) Error Term: ERROR
Summary Results Á= .05 Level Codes

Code(Level)	Mean	ABCD
A(20)	71.06	.SSS
B(44)	120.4	S..S
C(10)	140.2	S..S
D(0)	169.2	SSS.

Data Base Name C:\ncss\mc-1

Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	3	S+nA
S	16	S

Data Base Name C:\ncss\mc-1

Analysis of Variance Report
ANOVA Table for Response Variable: RDX

Source	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
Error Term					
A (DAY)	3	25575.89	8525.298	63.62	0.0000
ERROR					
ERROR	16	2144.132	134.0083		
TOTAL(Adj)	19	27720.03			

Means & Standard Errors for Y = **RDX**

Term	Count	Mean	Std.Error
ALL	20	125.215	
A: DAY			
0	5	169.2	5.177031
10	5	140.2	5.177031
20	5	71.06	5.177031
44	5	120.4	5.177031

GLM ANOVA (Multiple Comparisons)
Scheffe's Procedure
Response Variable: RDX Factor(A,DAY) Error Term: ERROR

Summary Results Á= .05

Code(Level)	Mean	Level Codes ABCD
A(20)	71.06	.SSS
B(44)	120.4	S..S
C(10)	140.2	S..S
D(0)	169.2	SSS.

Data Base Name C:\ncss\mc-1
Expected Mean Squares ... Balanced Case

Source	DF	Expectation of Mean Square (S stands for ERROR).
A	3	S+nA
S	16	S

Analysis of Variance Report

ANOVA Table for Response Variable: TNT

Source Error Term	DF	Sum-Squares	Mean Square	F-Ratio	Prob>F
A (DAY) ERROR	3	4.2011E07	1.4003E07	567.60	0.0000
ERROR	16	394750.9	24671.93		
TOTAL(Adj)	19	4.2406E07			

Means & Standard Errors for Y = TNT

Term	Count	Mean	Std.Error
ALL	20	942.5125	
A: DAY			
0	5	3452	70.24519
10	5	165.08	70.24519
20	5	63.17603	70.24519
44	5	89.79388	70.24519

GLM ANOVA (Multiple Comparisons)

Scheffe's Procedure

Response Variable: TNT Factor(A,DAY) Error Term: ERROR

Summary Results Á= .01

Code(Level)	Mean	Level Codes ABCD
A(20)	63.17603	...S
B(44)	89.79388	...S
C(10)	165.08	...S
D(0)	3452	SSS.