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Task Order - 8 Field Demonstration - Composting of Explosives- Contaminated Sediments at the Louisiana Army Ammunition Plant (LAAP)

Contract Number DAAK-11-85-D-007
Report Number AMXTH-JR-TE-88242

September 1988

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FIELD DEMONSTRATION - COMPOSTING OF EXPLOSIVES-
CONTAMINATED SEDIMENTS AT THE LOUISIANA
ARMY AMMUNITION PLANT (LAAP)

Final Report

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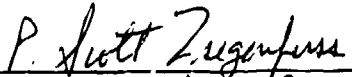
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Prepared for:

United States Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground
Edgewood Area, Maryland

Contract Number DAAK-11-85-D-0007
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SECTION 1

EXECUTIVE SUMMARY

A field-scale demonstration of static pile composting to decontaminate explosives-contaminated sediment was conducted at the Louisiana Army Ammunition Plant (LAAP). Composting as used at LAAP is a biological treatment process in which contaminated soil or sediment is mixed with bulking agents/carbon sources (organic materials such as manure) to enhance microbial metabolism in degrading and stabilizing soil contaminants.

Temperature was the primary test variable investigated. For both sets of two compost piles each, one pile was maintained at approximately the mesophilic temperature optimum (35°C) and one at approximately the thermophilic temperature optimum (55°C). Compost piles self-heat when energy released from microbial metabolism of organic matter is trapped within the compost matrix. Therefore, no external heat sources were required. Vacuum (drawn) aeration was used to remove excess heat and to maintain aerobic conditions within the compost piles.

Lagoon sediments that were previously contaminated with TNT, RDX, HMX, and tetryl were mixed with horse manure, straw, alfalfa, horse feed, and fertilizer and were composted for 153 days. The initial concentration of explosives in the mixture to be composted was approximately 17,000 mg/kg. After 153 days of composting, the concentrations of solvent-extractable total explosives were reduced to 376 and 74 mg/kg in the mesophilic and thermophilic compost piles, respectively. Mean percent reductions of extractable TNT, RDX, and HMX in the mesophilic pile were 99.6, 94.8, and 86.9 weight percent, respectively. Corresponding values for the thermophilic pile were 99.9, 99.1, and 96.5 weight percent.

Contaminant degradation for TNT, RDX, and HMX approximated first-order decay kinetics, with correlation coefficients ranging from 0.94 to 0.99. Diamino-nitrotoluenes and aminodinitrotoluenes (known TNT biotransformation products) were detectable in the initial mixture to be composted, increased in concentration during the first 4 weeks of composting, and decreased to low mg/kg levels thereafter.

These data indicate that composting is a feasible full-scale remediation technology for decontaminating explosives-contaminated soils and sediments. Further investigation into optimizing the materials balance and soil loading rate for mixtures to be composted, minimizing bulking agent usage, and developing a design and operation management plan for a full-scale composting facility is warranted. In addition, the compost residue should be subjected to a toxicity evaluation and more extensively analyzed to determine the final fates of HMX, RDX, TNT, and tetryl.

SECTION 2**INTRODUCTION****2.1 STATEMENT OF THE PROBLEM**

The manufacture and handling of explosives and propellants has resulted in soil and sediment contamination at U.S. Army munitions facilities, often as a result of previously acceptable waste disposal practices. The United States Army Toxic and Hazardous Materials Agency (USATHAMA) is currently investigating several technologies for decontaminating explosives-contaminated soils. Among these candidate technologies is composting. USATHAMA has previously conducted laboratory- and pilot-scale tests of this technology (Isbister et al., 1982; Doyle et al., 1986). The results of these studies were positive and warranted a field-scale demonstration. This report describes the results of that field demonstration.

2.2 BACKGROUND INFORMATION

Composting is a process by which organic materials are biodegraded by microorganisms, resulting in the production of organic and/or inorganic by-products 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 is initiated by mixing biodegradable organic matter (explosives in the present study) with organic carbon sources and bulking agents, which are added to enhance the porosity of the mixture to be composted. Bulking agents may also provide additional organic carbon to the microorganisms. In this report, the term bulking agents is used to refer to materials that provide both porosity and degradable organic matter.

The environment in a compost matrix 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.

Composting is applicable to the remediation of soils contaminated with any biodegradable compound(s). Materials and facilities required include a biodegradable organic substrate, bulking agents to increase the porosity and organic carbon concentration of the mixture to be composted, water, a containment structure to prevent contaminant migration, mixing equipment,

and a means to provide oxygen to the composting materials. Parameters that affect the efficiency of the composting process include temperature, moisture content, 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.

Microorganisms that catalyze the composting process and degrade organic constituents are present in the materials used to prepare a compost mixture. No supplemental organisms are typically required. Special circumstances may exist where supplemental microorganisms may be useful, but this was not the case in the present study.

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. Consequently, costs 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. This level of technology is often referred to as a "windrow" system, so named 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 effect aeration and heat removal. This level of technology is often referred to as a "static pile."

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 continuous treatment process. This type of system is often referred to as "in-vessel" 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.

Since sludge and refuse are generated continuously, these objectives are best met by a composting system designed for relatively rapid turnover of incoming wastes. The rate of waste disposal must approximate the rate of waste loading for wastewater and refuse facilities to operate efficiently. In contrast, the primary objective of hazardous materials composting is to convert hazardous substances into innocuous products for ultimate disposal. Rapid processing is desirable, but remains secondary to successful treatment of the waste. Thus, while hazardous materials composting systems share many of the characteristics of sludge and refuse composting systems, operational parameters will differ according to the primary objective of the process.

Composting is a combination of biological and engineering processes. Biological aspects of the process that require management include optimizing the environmental conditions required to enhance microbial growth and to maximize contaminant destruction within the compost pile. Engineering aspects that require attention include materials handling, composting facility design and operation, and process control systems. Both biological and engineering requirements must be addressed in order to provide a cost-effective and successful treatment process.

Previous studies have demonstrated aerobic biotransformation of explosives and propellants. Successful composting of explosives- and propellant-contaminated soils has been achieved at both laboratory- and pilot-scales. These studies indicated that composting is a feasible technology for the treatment of soils contaminated with explosives and propellants. To assess composting at full scale, WESTON conducted a field-scale demonstration project on-site at the Louisiana Army Ammunition Plant (LAAP). This report details the findings of that field demonstration project and includes recommendations for future work and full-scale implementation.

2.3 LITERATURE SUMMARY

A brief summary of previous research on the biotransformation and composting of 2,4,6-trinitrotoluene (TNT), hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazocine (HMX), and N-methyl-N,2,4,6-tetranitroaniline (tetryl) is presented here. An extensive literature review, recommended for a clearer understanding of the technical issues discussed in this report, is included in this report as Appendix C. The structures of the contaminants of concern are presented in Figure 2-1.

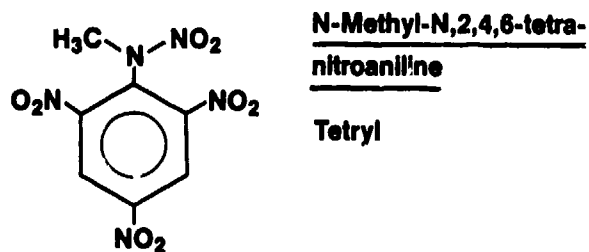
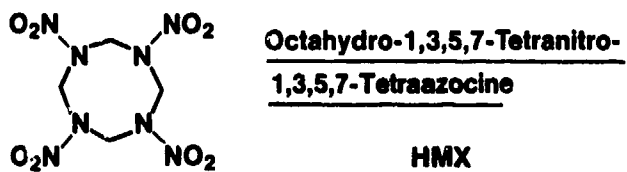
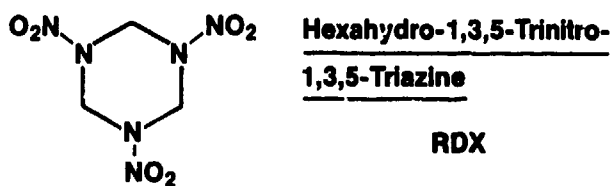
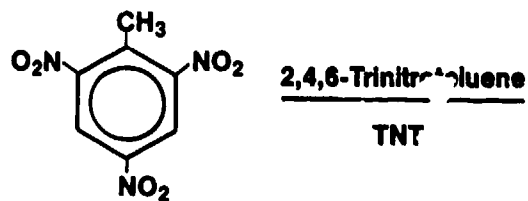


Figure 2-1. Structures of explosives of concern.

Trinitrotoluene (TNT) is the primary military explosive. The symmetrical alpha isomer (2,4,6-TNT) is the compound sought for munitions use. Previous research has indicated that TNT is microbially transformed but is not completely mineralized to inorganic products. In studies using radiolabeled (^{14}C) TNT, very low levels of $^{14}\text{CO}_2$ production have been reported.

Conditions that have been found to enhance the biotransformation of TNT include high organic carbon concentration, aerobic conditions, and the presence of readily biodegradable co-substrates. Laboratory- and pilot-scale composting have demonstrated extensive reduction in the concentration of solvent-extractable TNT after 6 weeks of composting.

Microbes generally catalyze nitro group reduction of the TNT molecule. A number of TNT biotransformation products are known. Evidence exists that these metabolic products are adsorbed strongly to organic materials. A greater percentage of these transformation products has been found to bind with increasing compost age and stability.

Anaerobic conditions have been found to enhance the biotransformation of RDX. Co-metabolism facilitated by yeast extract or high TOC levels, as well as low redox potential, has been shown to enhance RDX degradation. Laboratory-scale composting studies with ^{14}C -RDX have demonstrated high levels of $^{14}\text{CO}_2$ production (37 to 46 percent of initial ^{14}C activity added), suggesting ring cleavage and complete mineralization. Biotransformation products of RDX include mono- and dinitroso RDX derivatives, methanol, formaldehyde, hydrazine, and dimethyl hydrazines.

Complete biodegradation of HMX has not been demonstrated in either soil or aqueous systems, but up to 53 percent removal of HMX has been achieved in a pilot-scale activated sludge treatment system. HMX degradation is facilitated by anaerobic conditions, but at a slower rate than that observed for RDX. Like RDX, high TOC concentration and low redox potential enhance HMX degradation. Biotransformation products from HMX include mono- and dinitroso HMX derivatives, methanol, and formaldehyde.

Little information was located on tetryl biotransformation. In laboratory-scale composting studies, 80 to 90 percent of the ^{14}C tetryl initially spiked into compost was detected as unextractable residue after 56 days of composting. Production of $^{14}\text{CO}_2$ accounted for only 3 to 5 percent of the initial ^{14}C activity over the same period.

The success of bench- and pilot-scale composting studies by the Atlantic Research Corporation (ARC) (Isbister et al., 1982; Doyle et al., 1986) was primarily responsible for the initiation of the present study. Laboratory-scale studies were conducted using artificially heated composts in closed vessels.

Carbon-14 radiolabeled TNT and RDX were utilized to determine the degree of mineralization and biotransformation of these compounds in horse feed/alfalfa compost. After 6 weeks of composting, solvent-extractable TNT had been reduced 83 percent from an initial concentration of 10,000 mg/kg. No significant quantities of $^{14}\text{CO}_2$ were produced from ^{14}C -TNT, suggesting that ring cleavage and mineralization did not occur. An initial TNT concentration of 20,000 mg/kg was reduced 99.9 percent after 3 weeks in the pilot-scale study. In general, accumulation of amino-dinitrotoluenes and diamino-nitrotoluenes in compost did not occur; however, trace quantities were detected in one sample.

After 6 weeks of laboratory-scale composting, an initial RDX concentration of 10,000 mg/kg in compost was reduced 78 percent. Significant quantities of $^{14}\text{CO}_2$ were produced from ^{14}C -RDX, suggesting that ring cleavage and mineralization had occurred. In the pilot-scale study, 82 percent reduction in solvent-extractable RDX occurred after 6 weeks.

These data suggested that composting may be a cost-effective alternative to incineration for decontaminating explosives-contaminated sediments and soils. The present study was initiated to evaluate composting at field scale.

2.4 SITE BACKGROUND

Construction of the Louisiana Army Ammunitions Plant was started in 1941 and completed in 1942. The facility was built to load and to pack ordnance for the U.S. Army. Explosives have not been manufactured at the facility, but are brought in and utilized in loading, assembling, and packing lines.

Initially, the area where the composting field demonstration was conducted was used as a burning grounds to dispose of out-of-specification ordnance. During the mid-1940's, new burning pits were constructed and the previous burning pits were converted to wastewater lagoons.

The lagoons were used to dispose of wastewater generated during wash down of the munitions loading lines. Equipment used to load munitions was washed with water, and the resulting wastewater contained high concentrations of suspended explosives. These suspended organics give the water a pinkish color; consequently, these wastewaters are commonly referred to as "pink water."

Pink water was transported to the unlined lagoons by tank trucks and was dumped into individual lagoons via concrete spillways. Suspended explosives settled to the bottom of the lagoons, and the water evaporated. Generally, the lagoons remained filled with water from rainfall. Over the period of

approximately 30 years during which pink water was disposed of in the lagoons, high concentrations of explosives accumulated in the upper sediment layers at the bottom of the lagoons. The highest concentrations (approximately 300,000 to 600,000 mg explosive/kg sediment) accumulated near the lagoon spillways (Doyle et al., 1986). Large particles of pure-phase explosives are also present in the sediments.

In the mid-1970's, a carbon adsorption plant was constructed at LAAP to treat pink water, and disposal in the lagoons was halted completely by 1981. In October 1984, the pink water lagoon site at LAAP was proposed for inclusion on the U.S. EPA National Priority List (NPL). Currently, that status is unchanged. Soils and sediments at the site are currently being remediated by incineration.

2.5 OBJECTIVES

The primary objective of this study was to evaluate the utility of aerated static pile composting as a technology for remediating soils and sediments contaminated with the explosives TNT, HMX, RDX, and tetryl. Secondary objectives included a comparison of the efficacy of mesophilic (35°C) versus thermophilic (55°C) composting, evaluating different materials handling and process control strategies, and determining transformation products when Standard Analytical Reference Materials (SARMs) were available. Aspects that were not a part of this investigation included:

- Toxicological evaluation of the final compost residue.
- Determination of a maximum soil loading rate.
- Minimization of carbon supplement and bulking agent utilization.
- Analytical characterization of the compost for analytes for which SARMs could not be found.
- Determination of an engineering design and management plan for full-scale implementation.

SECTION 3**MATERIALS AND METHODS**

This section contains information on materials and methods that pertain to all four compost piles studied at LAAP. Information specific to compost piles 1 and 2 or compost piles 3 and 4 is presented in Sections 4 and 5, respectively.

3.1 COMPOST TEST PADS, ROOFS, AND ELECTRICAL SUPPLY

Two 8-inch-thick concrete test pads (18 x 30 feet) were constructed. The pads were located 20 feet apart in a flat area adjacent to the pink water lagoons (see Figure 3-1). The pads contained a 2-inch-deep drainage channel located on the perimeter of three sides of each pad. The drainage channels were piped into a 6- x 8- x 6-foot sump located below grade level to catch liquid draining from the piles. Water was pumped from the sump and was reapplied to the compost piles or, on one occasion, treated in the carbon adsorption wastewater treatment system. The sump was covered with a corrugated tin roof.

The composting test pads were covered with an open-sided, corrugated tin roof (approximate 16-foot eave height). This roof provided protection from precipitation and minimized the amount of water collected in the sump. LAAP provided 110- and 220-volt power and a weather-proof electrical control panel adjacent to the pads (see Figure 3-2).

3.2 BULKING AGENTS/CARBON SOURCES

A mixture of horse manure and soiled bedding material (straw) was obtained from Louisiana Downs racetrack and nearby training tracks at a cost of approximately \$3.00 per cubic yard. The mixture had been collected from horse stalls within 1 week prior to delivery to LAAP. Visually, the material appeared to consist mainly of straw.

Alfalfa hay and horse feed were obtained from a local supplier for \$6.00 per bale and \$260.00 per ton, respectively. Alfalfa was finely chopped in an incinerator feed system (see Subsection 3.3) prior to use in the mixture to be composted. The brand of horse feed used was Purina Balanced Blend 14. The horse feed contained greater than 14 percent crude protein, 2 percent crude fat, and 12 percent crude fiber. The horse feed also contained molasses; calcium and sodium carbonate; vitamins A, B12, D3, and E; sodium selenite; riboflavin; niacin; manganese oxide; zinc oxide; and copper sulfate in unspecified amounts. Sawdust, wood chips, and baled straw, obtained from local suppliers, were used to construct bases and insulating covers for the compost piles (see Figure 3-3).

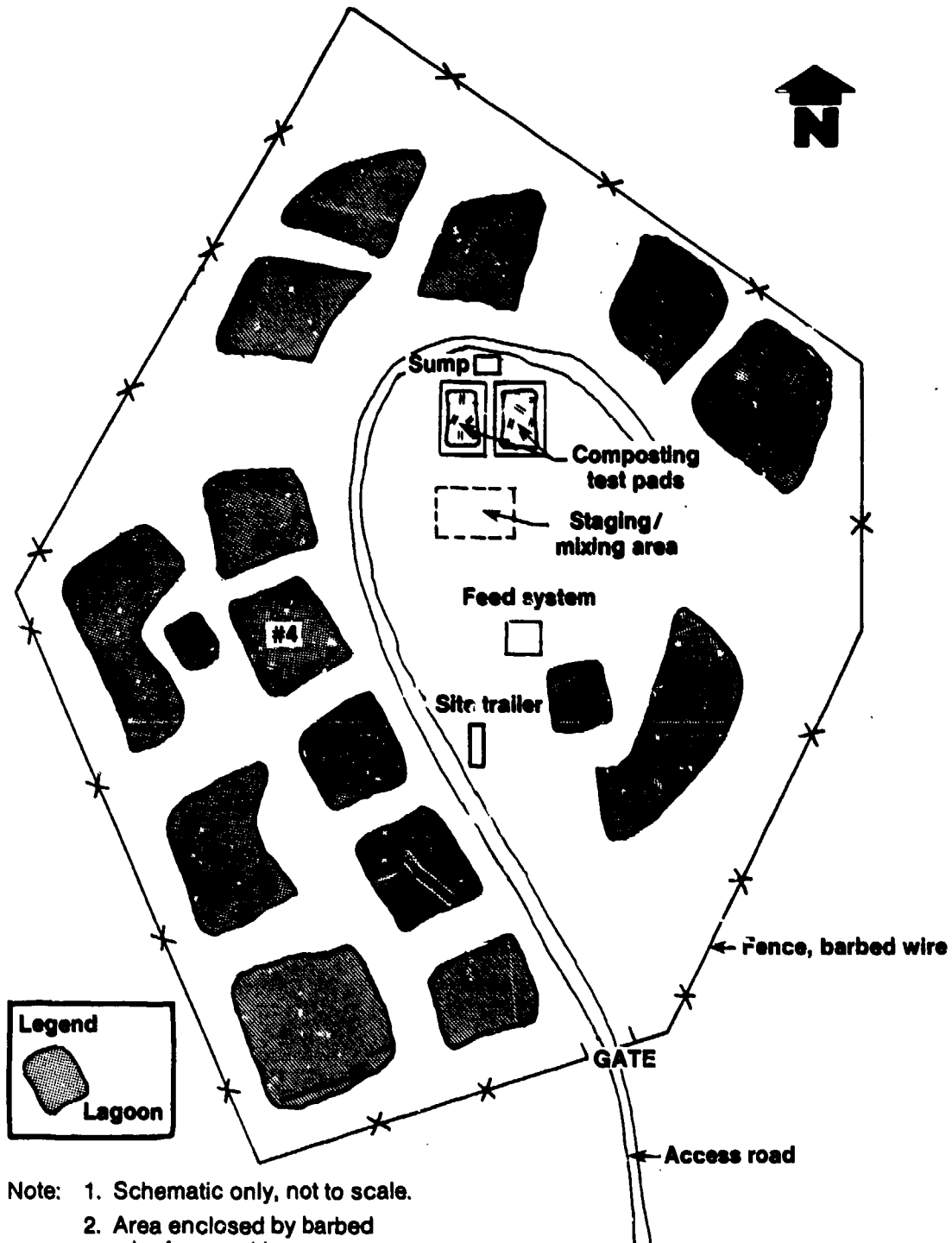
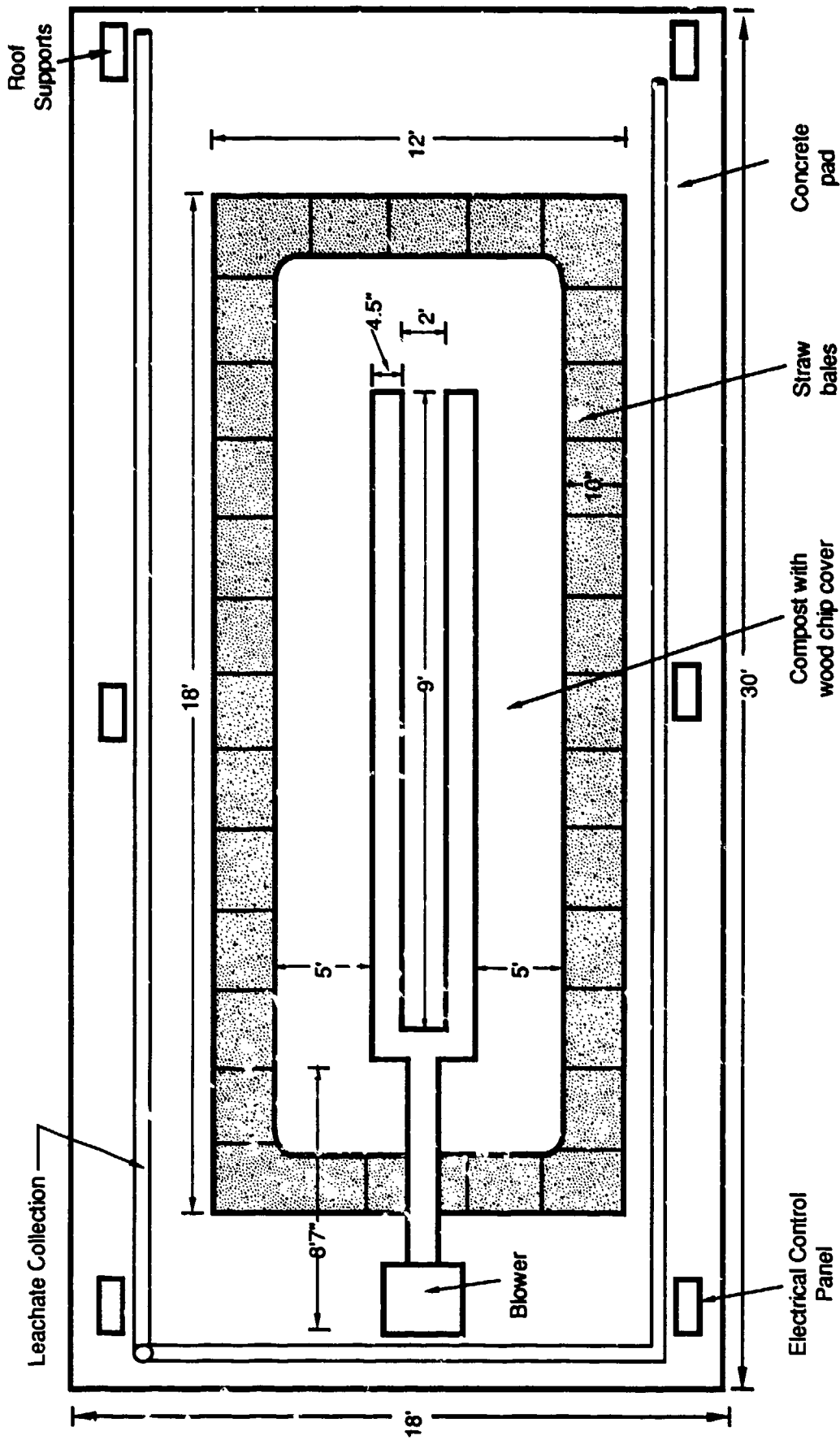


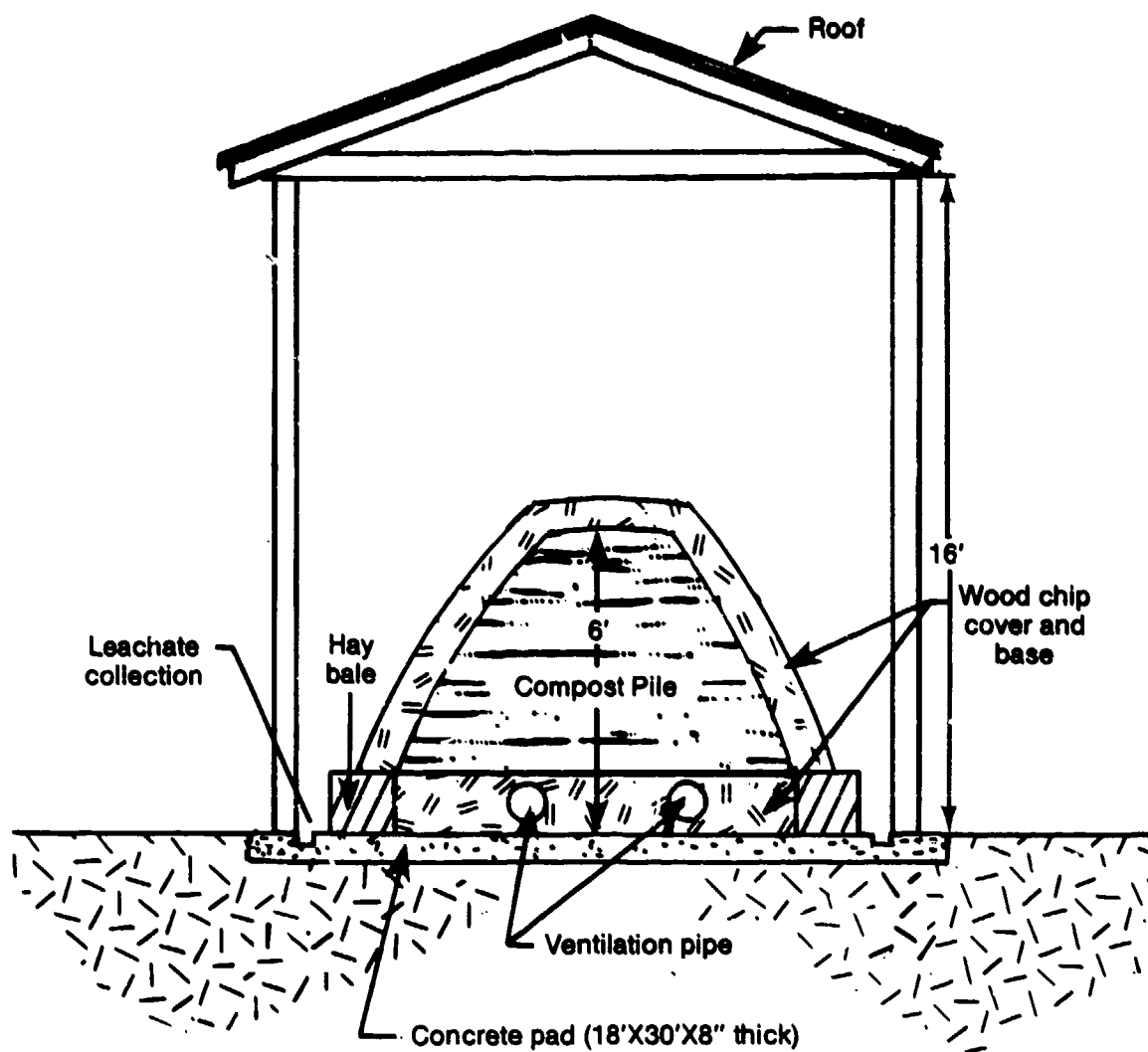
Figure 3-1. Location of composting test pads in Area P, pink water lagoons, Louisiana Army Ammunition Plant.



Note: Schematic only, not to scale.

13-249

Figure 3-2. Overview schematic diagram of compost pile, Louisiana Army Ammunition Plant.



Note: Schematic only, not to scale.

Figure 3-3. Cross-sectional schematic of compost pile with roof, Louisiana Army Ammunition Plant.

3.3 MIXING SYSTEM

A mechanical feed system, developed initially to meter explosives-contaminated soil into an incinerator, was used to homogenize sediment and to mix the material to be composted during compost pile construction. This system was chosen for use because it was already on-site and had been previously approved for use with explosives-contaminated soil by the Department of Defense Explosives Safety Board. The feed system consisted of three major components (see Figure 3-4):

- **Live-Bottom Hopper** - The live-bottom hopper consisted of a loading bin, into which materials to be mixed were placed, with four metering screws in the bottom to control the material transfer rate into the cross conveyor.
- **Cross Conveyor** - Materials from the live-bottom hopper discharged into a twin-screw cross conveyor that transferred the material horizontally to the feed conveyor.
- **Feed Conveyor** - This twin-screw conveyor was designed to deposit soil directly into an incinerator. In this field demonstration, soil and compost were discharged into the bucket of a front-end loader for transport to either the mixing or compost pads.

A bermed, 16-foot-long by 8-foot-wide concrete mixing pad was located adjacent to the mixing system. Bulking agents and sediment were mixed on this pad. Water draining from this pad was directed into a pink water lagoon through an existing concrete channel.

3.4 TEST SEDIMENT

Excavations of test sediment were performed near the spillway in pink water lagoon number 4 (see Figure 3-1) using a tracked excavator. Excavated sediment was placed in the bed of a dump truck parked over the sluiceway leading into the lagoon. The bed of the truck was elevated slightly to allow water to drain from the soil back into the lagoon.

The first excavation took place on 7 December 1987. Approximately 15 cubic yards of sediment were excavated. The sediment was drained, homogenized in the mixing system, sampled for explosives analysis, covered with a plastic tarp, and stored on the concrete mixing pad adjacent to the mixing system. This sediment was used to prepare the mixture to be composted for piles 1 and 2 (see Table 3-1).



Photograph by WESTON, 1988

Figure 3-4. Feed system at Louisiana Army Ammunition Plant.



Table 3-1

**Concentrations of Explosives in Sediment Collected
from LAAP Pink Water Lagoon No. 4
(concentrations in mg/kg)**

Contaminant	<u>Date Sediment Collected</u>	
	12/7/87	2/2/88
2,4,6-TNT	15,500	56,800
RDX	8,930	17,900
HMX	1,130	2,390
Tetryl	88	650

On 2 February 1988, three additional batches (2 cu yd each) of sediment were excavated, processed in the feed system, sampled, and stored on the mixing pad. The batch with the highest explosives concentration was incorporated into the mixture to be composted for piles 3 and 4 (see Table 3-1).

3.5 MATERIALS HANDLING EQUIPMENT

Two front-end loaders, equipped with 1-cubic-yard buckets, and a tracked excavator were used to transport sediment and compost at the field site. Smaller-scale materials handling operations were performed with hand tools.

3.6 COMPOST TEMPERATURE MONITORING

Two dual-channel strip-chart recorders (Model RD-2080, Omega Engineering, Stamford, Connecticut) were used to continuously record temperatures within the compost piles. Two 6-foot-long landfill thermocouple probes (type K, Atkins Technical, Gainesville, Florida) were placed in each pile and were connected to the temperature recorder. One probe was placed 2 feet inside each pile, 3 feet above pad level, and 2 feet from the blower end of the pile. The other probes were inserted into the center of each pile.

In addition to the continuous temperature recorder, a landfill probe equipped with a hand held digital thermometer was used to monitor compost temperatures on a daily basis. A temperature profile of the compost piles was made by manually inserting the probe and measuring the temperature at six points within each pile. Two measurements, one in the upper and one in the lower part of the pile, were made at three longitudinal locations in each pile: the toe (end near the blower), the middle, and the heel (end away from the blower). Thus, six discrete temperature measurements were made daily in each pile.

3.7 COMPOST TEMPERATURE/AERATION CONTROL

Each compost pile contained a system of perforated and non-perforated polyethylene drainage pipe (4-inch diameter) placed on top of the wood chip bases and connected to an explosion-proof, Peerless PW-12 radial-blade blower (single-phase, 2 hp, 860 SCFM, 6-inch static pressure). The blowers were used to pull air through the compost piles to promote aeration and to remove excess heat (see Figure 3-2).

Blower cycling was controlled by both timer and temperature feedback systems. The temperature feedback system consisted of soil thermistors that measured compost temperature and panel-mounted Fenwal series 551 thermistor sensing temperature controllers. Timer control was obtained by programmable timer relays mounted in the thermistor control panels.

The thermistor controllers had low- and high-temperature set point values that were operator-set. The low and high set points on the thermistor controller in the mesophilic compost piles were 31°C and 35°C, respectively. The corresponding values for the thermophilic piles were 51°C and 55°C. After pile construction, the blowers were placed on timer control (2 minutes on/60 minutes off) to provide oxygen to the piles while minimizing heat removal during the startup phase. After the piles reached operating temperature (2 to 3 days), blower control was automatically transferred to the temperature feedback system.

The temperature feedback system provided three functions. When the pile temperature as registered by the thermistor was: below the low set point, the blower operated on timer control; between the low and high set points, the blower did not operate; above the high set point, the blower operated continuously until the compost temperature (as monitored by the thermistors) fell below the high set point.

When the compost temperature was below the low set point, the blower operated intermittently (timed on/off cycling) to aerate the pile with minimal cooling. When the compost temperature was between the low and high set points, the blowers did not operate in order to allow the compost pile to reach the high set point. When the compost temperature exceeded the high set point, the blowers ran continuously to lower the compost temperature to just below the high set point. When the blowers were on, compost temperatures were lowered by both evaporation and the passage of cool ambient air through the pile. In this way, compost temperatures could theoretically be controlled near the optimum levels. The temperature ranges sought during this study were $35 \pm 4^\circ\text{C}$ and $55 \pm 4^\circ\text{C}$.

3.8 MICROBIAL ENUMERATION

The population density of heterotrophic microorganisms was determined for compost samples from piles 3 and 4. Ten grams of compost was aseptically transferred into 90 ml of sterile 0.1 M K_2HPO_4 buffer and was agitated by hand for 2 minutes. Large particles were allowed to settle for 2 minutes after agitation. The compost extract was serially diluted into a sterile phosphate buffer (1 ml extract into 9 ml buffer) to a dilution of 10^{-9} . Duplicate 100-microliter (μ L) aliquots of each extract dilution were spread plated onto nutrient agar plates (Difco Laboratories) using a spread plating turntable (Fisher Scientific) and a bent glass rod. Single plates (one from each duplicate set) were incubated at 35°C and 55°C for 5 days. Total colony counts were made after 2 and 5 days of incubation. The total number of microbial (bacterial and fungal) colonies on each plate was used to calculate the number of colony forming units (cfu) per gram of dry compost.

3.9 ANALYTICAL METHODS

3.9.1 TOC and Metals

Analyses for lead, selenium, and arsenic were conducted by procedures in Standard Methods for Chemical Analysis of Water and Wastes (U.S. EPA 600/4-79-020, 1979) (see Table 3-2).

Table 3-2

Analytical Methods for TOC, TKN, and
Metals Analyses of Compost

Analysis	Methodology Used
TOC	Loss-On-Ignition (Stromm, 1976)
Lead	Method 239.2 (U.S. EPA, 1979)
Mercury	ICP Analysis Method 200.7 (U.S. EPA)
Selenium	Method 270.2 (U.S. EPA, 1979)
Arsenic	Method 206.2 (U.S. EPA, 1979)

3.9.2 Explosives

Compost samples were analyzed for TNT, RDX, HMX, tetryl, and transformation compounds by USATHAMA Method LW02 (see Appendix A), modified for the extraction and analysis of compost.

3.9.2.1 Sample Extraction and Preparation

- Approximately 10 grams of wet compost was accurately weighed into a wide-mouth glass jar with a Teflon-lined screw cap.
- 40.0 mL of acetonitrile 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 (um) Teflon filter and was retained in a 4-mL autosampler vial.
- At the time of analysis, 200 uL of the extract was diluted with 600 uL of acetonitrile.

3.9.2.2 Percent Solids Determination

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

3.9.2.3 High-Performance Liquid Chromatograph (HPLC) Analysis

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

- Column: Zorbax ODS, 250 mm x 4.6 mm, 5 micron.
- Detector: UV absorbance at 250 nm.
- Mobile phase: 50 percent water, 34 percent methanol, 16 percent acetonitrile.
- Flow rate: 1.6 mL/min.
- Injection volume: 50 uL.

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 was used to quantify sample response.

Standard	HMX	RDX	Tetryl	2,4,6-TNT
0.5x	1.27	0.98	5.00	1.92
1x	2.54	1.96	10.0	3.84
2x	5.08	3.92	20.0	7.68
5x	12.7	9.80	50.0	19.2
10x	25.4	19.6	100	38.4
20x	50.8	39.2	200	76.8
50x	127	98.0	500	192
100x	254	196	1,000	284

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

- Full calibration curve and linear regression for all analytes.
- QA/QC samples (see Subsection 3.9.2.4).
- 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:

$$\text{sample response (ppm)} \times D \times 1/W = \text{mg/kg analyte}$$

Where:

D = dilution of extract
W = dry weight of sample

3.9.2.4 QA/QC Samples

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

- Method blank.
- 2x standard spike.
- 10x standard spike.
- 10x standard spike duplicate.

Samples were prepared as in Subsection 3.9.2.1, 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.

3.9.3 Transformation Compounds

Samples were extracted as per Subsection 3.9.2.1. Instrument conditions for the HPLC analysis of transformation products are as follows:

- Column: Zorbax C8, 250 mm x 4.6 mm, 5 micron.
- Detector: UV absorbance at 250 nm.
- Mobile phase: 35 to 42 percent acetonitrile, 65 to 58 percent water.
- Flow rate: 1.6 mL/min.
- Injection volume: 10 uL.

Instrument calibration was performed by analyzing the following series of standards that cover the entire calibration range. Standard concentrations are in units of mg/kg based on a 10-gram compost sample with no dilution. Linear regression was performed for each calibration curve and was used to quantify sample response.

2,4-Diamino-6-nitrotoluene and 2,6-diamino-4-nitrotoluene co-eluted on the chromatographs, and data are reported as total diamino-nitrotoluenes.

Standard	DANT*	2A-4,6-DNT**	4A-2,6-DNT***
0.5x	1.23	1.30	0.50
1x	2.46	2.60	0.99
2x	4.92	5.20	1.98
5x	12.3	13.0	4.95
10x	24.6	36.0	9.90
20x	49.2	52.0	19.8
50x	123.0	130.0	49.5
100x	246.0	260.0	99.0

*diamino-nitrotoluene

**2-amino-4,6-dinitrotoluene

***4-amino-2,6-dinitrotoluene

The protocol for daily sample analysis was as follows:

- Full calibration curve and linear regression for each analyte were performed.

- Sample extracts were analyzed.
- Final 2x and 10x calibration standards were analyzed.

Calculations were carried out as per Subsection 3.9.2.3.

3.10 REACTIVITY TESTING

The U.S. EPA Office of Solid Waste Management has accepted data from a procedure consisting of the following five separate tests to define the reactivity of solid materials:

- Stability test.
- Detonation test.
- Spark test.
- Impact test.
- Card gap test.

Procedures used for conducting these tests are presented in Appendix E. All reactivity tests were performed by Allegany Ballistics Laboratory (Rocket Center, West Virginia).

3.11 COMPOST PILES AND SAMPLING

Four compost piles were constructed at LAAP during the period from December 1987 to July 1988. The first set of compost piles (numbers 1 and 2) was set up on 11 December 1987 and was terminated on 13 January 1988 (33-day test period). The second set of compost piles (numbers 3 and 4) was set up on 25 February 1988 and was terminated on 27 July 1988 (153-day test period).

Samples were taken from the compost piles by a soil auger (Forestry Suppliers, Inc.). The auger had a 3-inch diameter stainless steel auger bucket and a 5-foot-long extension handle. Cover materials on the compost piles were scraped away using a shovel to expose the compost before sampling. The auger was inserted approximately 24 to 30 inches into the compost pile, and a core sample was removed. Samples were taken along the longitudinal axes of the piles, 2 to 3 feet below the apex. At least three, and as many as nine, core samples were removed from each compost pile and were analyzed at each sampling time point. Appendix B lists all samples taken from compost piles 1 through 4 at each time point in the study. Samples were placed in a refrigerator immediately after removal from the piles and were shipped to WESTON's laboratory by overnight freight using chain-of-custody procedures.

SECTION 4

COMPOST PILES 1 AND 2

4.1 COMPOST PILE DESIGN, CONSTRUCTION, AND OPERATION

4.1.1 Test Variables

The test variable in compost piles 1 and 2 was temperature. Pile 1 was operated to maintain compost temperatures in the mesophilic range ($35\pm 4^{\circ}\text{C}$). Pile 2 was operated to maintain compost temperatures in the thermophilic range ($55\pm 4^{\circ}\text{C}$).

4.1.2 Test Sediment and Bulking Agents

The mixture to be composted in piles 1 and 2 consisted of sediment from lagoon no. 4 (see Table 3-1), sawdust, wood chips, and straw/manure. Bulk density measurements were obtained for the sediment, sawdust, wood chips, straw/manure, and mixture to be composted. A spring scale (50-lb capacity) was tared to the weight of a hanging steel bucket, and the mass of three separate bucketfuls of tap water (21°C) was recorded. The volume of the bucket was calculated from these data using the known density of water. Triplicate samples of the compost materials were subsequently weighed, and the bulk densities were determined (see Table 4-1).

Table 4-1

Bulk Densities of Materials Used in Compost Piles 1 and 2

Material	Mean Bulk Density (lb/cu yd)
Straw/manure mixture	190
Sediment	2,300
Sawdust (dry)	600
Sawdust (wet)	760
Mixture to be composted	1,200

Prior to mixing the materials to be composted in piles 1 and 2, four small (0.5-cu ft) trial compost mixtures were hand-mixed and visually inspected for moisture content, porosity, and homogeneity. Based on this visual inspection, sediment analytical data, and previous pilot-scale studies (Doyle et al., 1986), a materials balance for the compost mixture components was developed.

Explosives-mediated microbial toxicity was a concern in developing the materials balance for the mixture to be composted. Doyle et al. (1986) demonstrated successful composting of TNT, RDX, HMX, and tetryl in compost mixtures containing up to approximately 57,000 ppm total explosives, with no apparent microbial inhibition. Sediment used in piles 1 and 2 of the present study contained approximately 25,600 mg/kg total explosives. This concentration was reduced by dilution when bulking agents were mixed with the sediment during preparation of the mixture to be composted. Thus, based on Doyle et al.'s data, toxicity concerns were not believed to be a limiting factor with regard to the proportion of soil intended for use in the mixture to be composted.

On the recommendation of a composting consultant retained for technical advise during the test period for piles 1 and 2, the proportion of sediment incorporated into the mixture to be composted was increased to elevate the moisture content (the sediment contained more moisture than the bulking agents) and explosives concentration. A high explosives concentration was desired to provide a large analytical "window" in which contaminant degradation could be tracked. The materials balance utilized for piles 1 and 2 is presented in Table 4-2.

Table 4-2
Materials Balance of Mixture
to be Composted in Piles 1 and 2

Material	Volume (cu yd)	Mass (lb)	Volume (percent)	Mass (percent)
Sediment	14	32,500	36	79
Sawdust	7	5,320	18	13
Straw/manure	<u>18</u>	<u>3,360</u>	<u>46</u>	<u>8</u>
Total	39	41,180	100	100

The calculated and measured bulk densities of the mixture to be composted were 1,060 and 1,200 lb/cu yd, respectively. The calculated bulk density was determined by dividing the total mass of the mixture to be composted (41,180 lb) by the volume of the mixture to be composted (39 cu yd). The measured bulk density value was slightly higher as a result of volume reduction after the materials were mixed together.

A major concern was that incomplete mixing could result in a heterogeneous distribution of explosives in the mixture to be composted. Heterogeneity could introduce significant variability in explosives concentrations between replicate compost samples, making it difficult to interpret changes in contaminant concentrations over time.

To account for this potential problem, approximately 0.25 cu yd of the final compost mixture was removed from the bulk pile after mixing and was homogenized with hand tools for 1 hour to obtain a small amount of homogeneous material. Fist-sized aliquots of this fine mixture were placed in small nylon bags, and the bags were then placed in the compost piles. The bags were placed in a row along the center longitudinal axis of the compost piles, approximately 2 feet below the apex of the mixture to be composted. If analyses of the bagged material revealed consistent explosives concentrations while bulk material from the piles showed greater variability in explosives concentrations, it could be assumed that a better mixing system or procedure was required to obtain a homogeneous mixture to be composted.

In addition to the bags of homogenized mixture to be composted, other bags were prepared that contained the fine mixture and hardwood chips that had been inoculated previously with the fungus, Phanerochaete chrysosporium. Previous research has shown that this fungus is capable of biodegrading explosives and other organic compounds normally thought to be recalcitrant to biological degradation. Inoculated bags were placed in the mesophilic compost pile (no. 1) only. Triplicate bags of the compost/wood chip mixture were sampled and analyzed for explosives on days 0 and 33 of the study.

4.1.3 Compost Mixing/Pile Construction

Piles 1 and 2 were constructed by the following steps:

- Sediment, excavated from lagoon no. 4 and homogenized 2 weeks previously, was processed through the feed system twice and was returned to the mixing pad.
- Sawdust, straw/manure, and 50 pounds of potassium/nitrogen/phosphorus (13/13/13) fertilizer were added to the sediment, and the entire mixture was mixed with the excavator and front-end loaders for 30 minutes.
- The mixture to be composted was processed through the feed system twice.
- Wood chip bases were constructed on the concrete compost pads. The bases were approximately 8 inches thick, 18 feet long, 10 feet wide, and oval in shape.

- Aeration piping was placed on the wood chip bases, connected to the blowers, and covered with approximately 3 inches of additional wood chips to prevent compost from entering the pipes. Pipe connections were secured with snap connectors and duct tape. Non-perforated pipe was used from the blowers through the "T" junction in the toe of each pile, and perforated pipe was used from the junction to the capped ends (see Figure 3-2). (Note: "toe" refers to the end of the compost pile adjacent to the blower; "heel" refers to the end away from the blower.)
- The mixture to be composted was transported to the composting pads in 1-cubic-yard front-end loader buckets. Loads were alternated between the two piles until both had received 14 cubic yards of material. Nylon bags containing the fungus-inoculated wood chip/compost mixture were placed along the centerline axis of pile 1. Nylon bags that contained finely mixed compost only were placed along the center axes of both piles. Nylon strings with identification labels were attached to all nylon bags and were run out the sides of the piles to facilitate sampling. Bags were placed in each pile after the seventh load of compost had been added. Seven more loads were added afterward.
- The compost piles were covered with a 1-foot-deep layer of sawdust to increase heat retention. The sawdust was covered with several inches of wood chips to prevent the sawdust from being blown away by high winds.
- Thermocouple and thermistor probes were inserted into the piles, and the temperature chart recorders were started at a chart paper feed rate of 1 cm/hr.
- Temperature controllers were set to timer operation (2 minutes on/60 minutes off).

4.1.4 Operations Schedule

Compost piles 1 and 2 were sampled immediately after pile construction and after 33 days of composting (see Table 4-3).

Table 4-3

Operations Schedule for Compost Piles 1 and 2

Day	Date	Event(s)
0	11 Dec 87	Piles constructed, temperature recorders and controllers started, time 0 samples taken. Analyses: explosives, intermediates, metals.
33	13 Jan 88	5-week samples taken. Analyses: explosives, intermediates. Piles terminated.

Samples of bulk material from the compost piles were removed with a 3-inch diameter soil auger. Bags were removed by digging, using the attached nylon strings as a guide. Samples were refrigerated immediately after removal from the piles and were shipped overnight to WESTON's Fate and Effects Laboratory.

4.2 RESULTS

4.2.1 Compost Temperatures

Temperatures ranged from 7°C to 48°C in pile 1 and from 21°C to 51°C in pile 2 over the test period (see Figure 4-1).

4.2.2 Compost Moisture Content

The moisture content of the compost in piles 1 and 2 ranged from 26 to 20 percent over the test period (see Table 4-4). The initial moisture content had been established on-site based on the recommendations of a composting consultant. His recommendation was based on visual inspection of the mixture to be composted as well as the ability of the mixture's constituents to stick together.

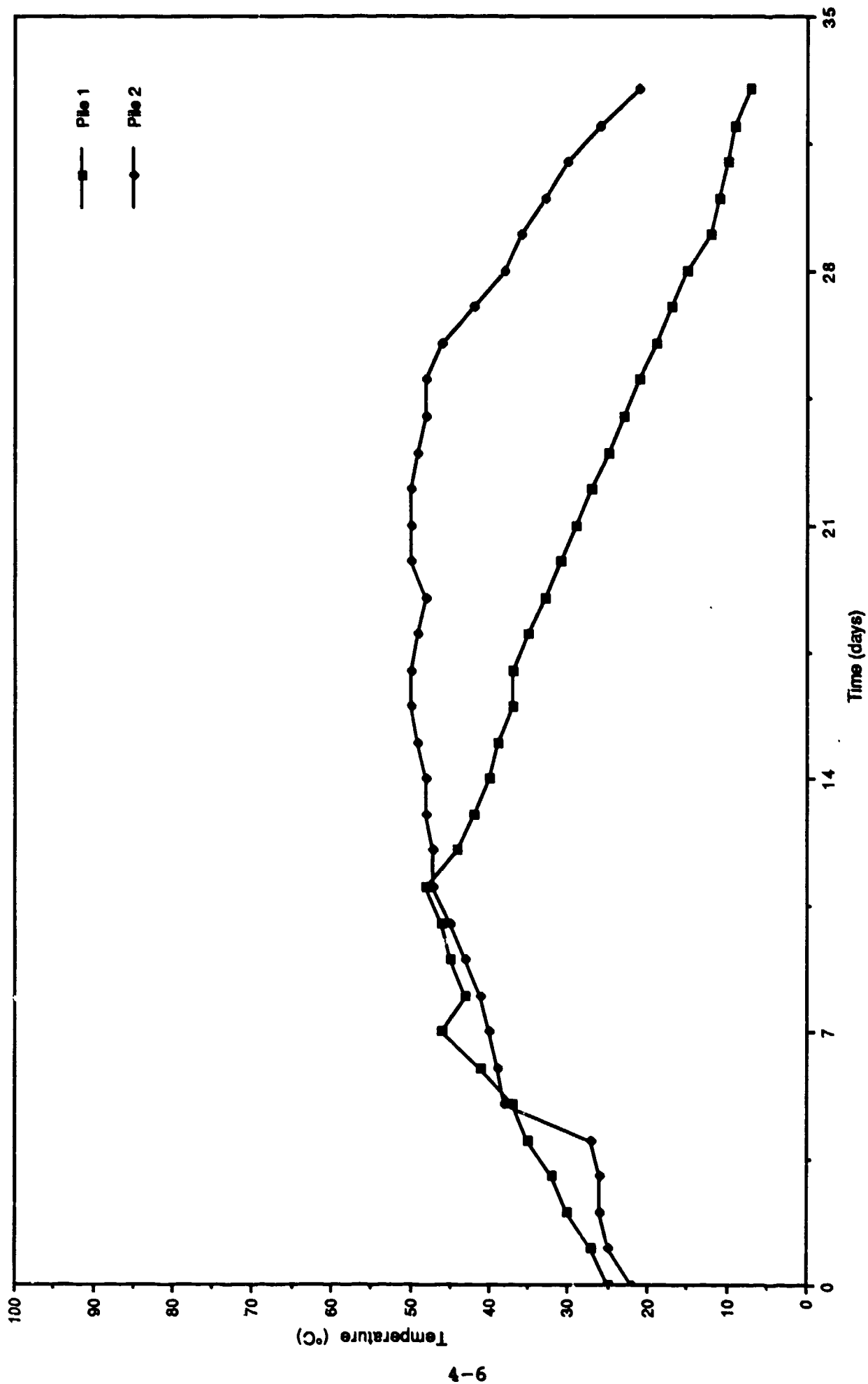


Figure 4-1. Temperatures of compost in piles 1 and 2.

Table 4-4

Moisture Content of Piles 1 and 2 Compost

Day	Mean Percent Moisture in Compost (weight percent)	
	Pile 1	Pile 2
0	26	26
33	20	21

4.2.3 Microbial Plate Counts

Plate count enumerations were not conducted on compost from piles 1 and 2.

4.2.4 Fate of Sediment Contaminants

Mean percent reductions of TNT in compost piles 1 and 2 were 64 and 84 percent, respectively (see Figure 4-2). Smaller percent reductions were noted for bagged compost (see Figure 4-2). No significant change in RDX or HMX concentrations was observed in either pile (see Figures 4-3 and 4-4). Error bars in these figures indicate one standard deviation from the mean explosives concentration.

No change in the concentrations of selected inorganic contaminants was noted in compost piles 1 and 2 (see Table 4-5).

Table 4-5

Inorganic Contaminants in Piles 1 and 2

Pile	Day	Concentration in Compost (mg/kg)			
		Arsenic	Mercury	Lead	Selenium
1	0	ND	0.8	20.0	ND
2	0	ND	0.8	19.0	ND

ND = Not detected

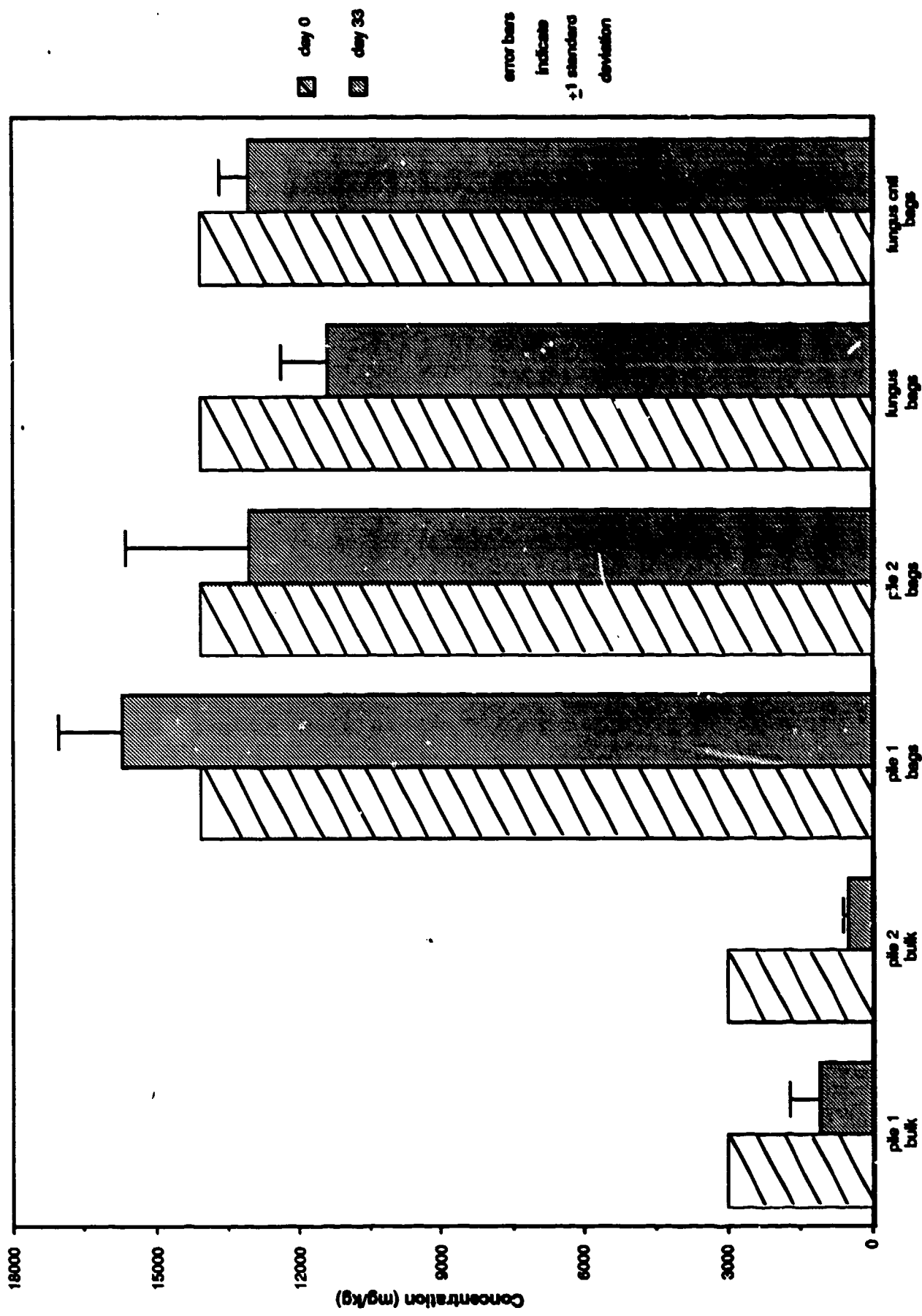


Figure 4-2. Concentration of TNT in compost piles 1 and 2.

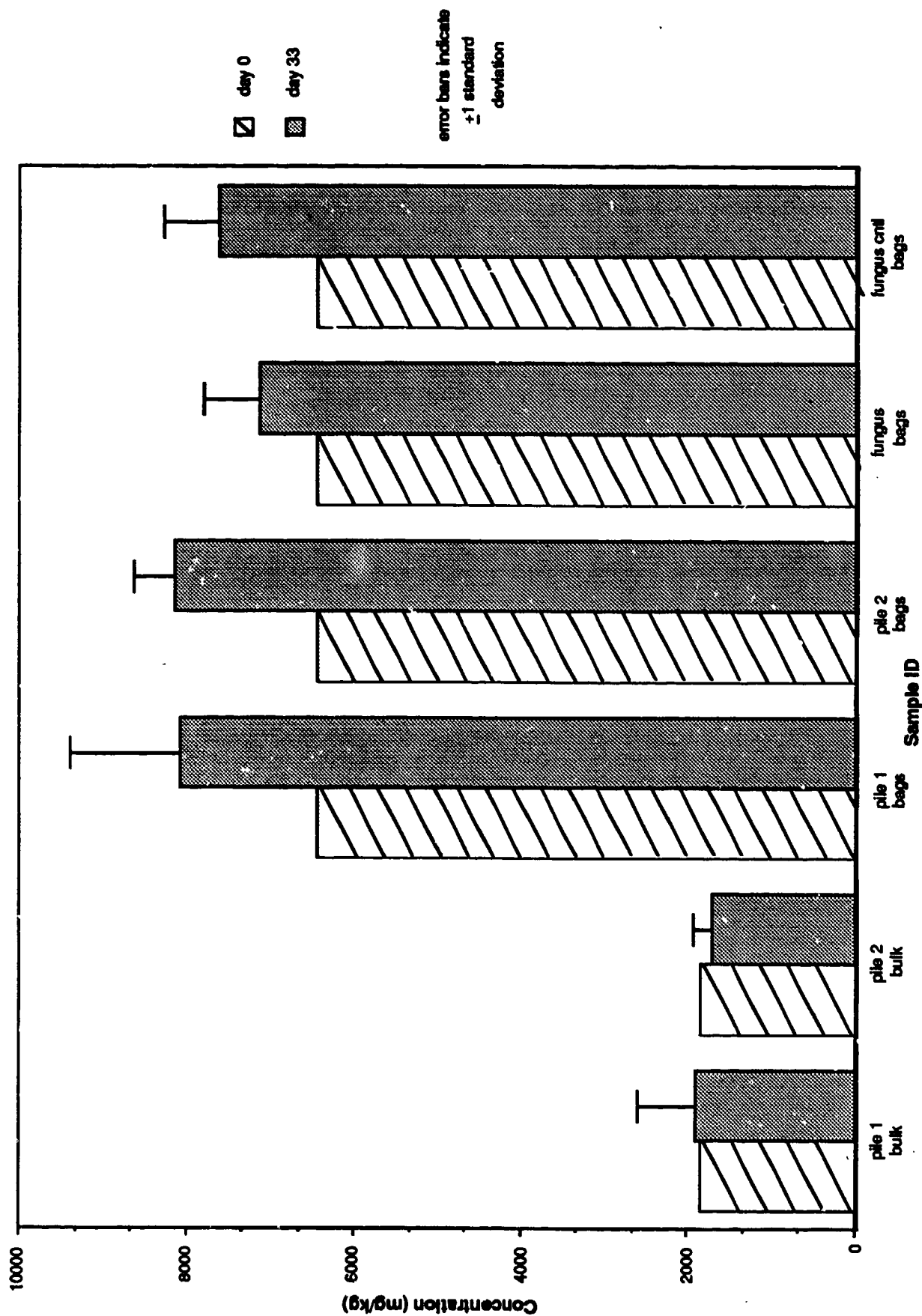


Figure 4-3. Concentration of RDX in compost piles 1 and 2.

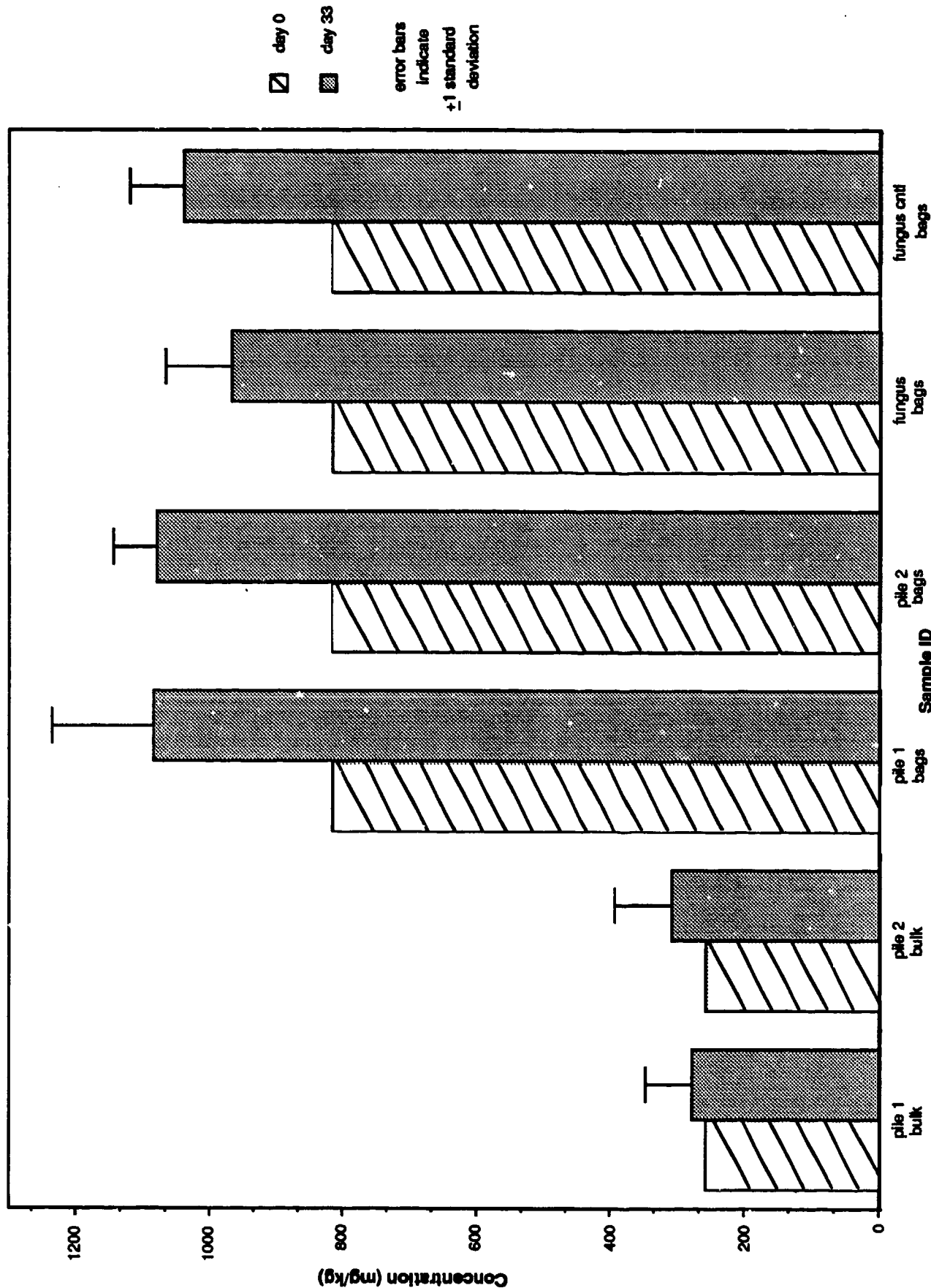


Figure 4-4. Concentration of HMX in compost piles 1 and 2.

4.2.5 Reactivity Testing

Reactivity tests were not conducted on compost residue from piles 1 and 2.

4.2.6 Nonquantitative Observations

Moderate to heavy fungal growth was evident in both compost piles at the time of termination. This fungal growth penetrated into clumps of compost. Compost from both piles appeared quite dry and had changed little in appearance over the test period.

4.3 DISCUSSION

The overall performance of compost piles 1 and 2 was poor. Figure 4-1 indicates that heat production within the compost was less than optimal. Temperatures in pile 1 (mesophilic) exceeded 30°C for only 18 days, after which compost temperatures dropped to near ambient levels. Pile 2 (thermophilic) maintained reasonable temperatures (45 to 50°C) for approximately 17 days. This duration is not without precedent since sludge composting times in the noncuring phase can be in this general timeframe. However, the intensity and duration of composting in piles 1 and 2 were far less than expected.

The two main factors that probably contributed to the lack of heat production were compost moisture content and the proportion of soil added to the initial compost mixture. Moisture in compost piles 1 and 2 ranged from 20 to 26 weight percent over the 33-day test period (see Table 4-4). The initial moisture content had been set as described in Subsection 4.2.2.

Compared to manure, horse feed, hay, and other organic bulking agents, sediment and soil are poor sources of organic carbon. The mixture to be composted in piles 1 and 2 contained approximately 79 percent (by weight) sediment. Thus, the concentration of biodegradable organic carbon in the compost mixture was probably too low to sustain intense microbial activity. The lack of sustainable heat production observed in piles 1 and 2 supports this conclusion.

Of the three major contaminants present in the test sediment (TNT, RDX, and HMX), only TNT was significantly degraded in compost piles 1 and 2 (see Figures 4-2 through 4-4). The percent reduction of TNT was greater in the thermophilic pile (no. 2) than in the mesophilic pile (no. 1), suggesting a positive relationship between compost temperature and the extent of TNT reduction.

No enhancement of contaminant degradation was noted in the bags of finely mixed compost that were inoculated with white rot fungus (see Figures 4-2 through 4-4). Fungal growth was widespread within pile 1 when the piles were terminated, suggesting

the presence of fungal spores in the bulking agents. However, the fungi were not identified. These observations indicate that inoculation of the compost with fungal spores is not necessary. However, these bags do not represent a fair experimental test of the capability of P. chrysosporium because of the problems discussed previously.

Concentrations of TNT, RDX, and HMX were approximately four times higher in the homogenized bagged compost than in the bulk compost from piles 1 and 2 (see Figures 4-2 through 4-4). This difference suggests that the mixing of the bulk mixture to be composted was inadequate. Sediment particles up to 4 inches in diameter remained in the bulk mixture to be composted after mixing. Large sediment particles were incorporated into the volume of compost mixture that was removed from the bulk pile, homogenized by hand, and placed in nylon bags. Incorporation of these sediment particles increased the total explosives concentration in bagged compost significantly. Exclusion of large sediment particles (that contained high concentrations of explosives) from bulk compost samples probably caused the lower explosives concentrations in samples of bulk compost as compared to the finely mixed, bagged compost.

Based on the concentrations of explosives in test sediment (see Table 3-1) and the materials balance for the mixture to be composted (see Table 4-2), a theoretical total explosives concentration in the mixture to be composted was determined by multiplying the total explosives concentration in the test sediment by the mass percent of sediment in the mixture to be composted. The theoretical total explosives concentration in the mixture to be composted was: $25,650 \text{ mg/kg} \times 0.79 = 20,260 \text{ mg/kg}$. Measured total explosives concentrations at time zero in bulk and bagged compost material were approximately 5,150 and 21,100 mg/kg, respectively (Figures 4-2 through 4-4). The observed concentration of total explosives in bagged compost more closely approximates the theoretical explosives concentration based on sediment dilution.

This calculation demonstrates the importance of the mixing equipment and procedures in preparing a homogeneous mixture to be composted. Comparison of contaminant concentrations in bulk-mixed and hand-mixed mixtures to be composted may be used to evaluate the efficiency of the bulk mixing system in future sediment composting studies.

As a result of the data obtained from compost piles 1 and 2, the materials balance for the mixture to be composted in piles 3 and 4 was prepared so that moisture content and organic carbon concentration would not limit composting activity. A decision was made to add water to the point where the mixture to be composted was saturated.



SECTION 5

COMPOST PILES 3 AND 4

5.1 COMPOST PILE DESIGN, CONSTRUCTION, AND OPERATION

5.1.1 Test Variables

Two new piles (nos. 3 and 4) were constructed after piles 1 and 2 were terminated. Temperature remained the test variable between the two new compost piles (mesophilic versus thermophilic).

5.1.2 Test Sediment and Bulking Agents

Alfalfa and horse feed were incorporated into the new compost mixture to increase the concentration of biodegradable organic carbon in the mixture to be composted. A new manure/straw mixture was obtained from a local training track in an attempt to obtain a higher proportion of manure in the mixture. Bulk densities of the bulking agents used in piles 3 and 4 are presented in Table 5-1.

Table 5-1

Bulk Densities of Materials Used in Compost Piles 3 and 4

Material	Mean Bulk Density (lb/cu yd)
Straw/manure	160
Alfalfa	70
Horse feed	950
Mixture to be composted	268

The sediment content of the mixture to be composted was substantially reduced from that used in piles 1 and 2 to increase the proportion of biodegradable organic carbon to biologically inert materials in the mixture to be composted (see Table 5-2).

The reduced proportion of sediment incorporated into the material to be composted raised concerns that the explosives concentration of the mix would be low. Consequently, three separate batches of sediment were excavated and analyzed for explosives prior to preparation of the mixture to be composted. The batch with the highest concentration (see Table 3-1) was

used to prepare the mixture to be composted; the other two batches of sediment were returned to lagoon no. 4.

Table 5-2

**Materials Balance of Mixture to be Composted in
Piles 3 and 4**

Material	Volume (cu yd)	Mass (lb)	Volume (percent)	Mass (percent)
Sediment	1	2,300	3	24
Alfalfa	13	940	38	10
Straw/manure	16	2,480	47	25
Horse feed	<u>4</u>	<u>4,000</u>	<u>12</u>	<u>41</u>
Total	34	9,720	100	100

5.1.3 Compost Mixing/Pile Construction

The mixture to be composted in piles 3 and 4 was prepared as follows:

- Sediment (excavated and homogenized 2 weeks previously from lagoon no. 4 and analyzed for explosives content) was reprocessed through the feed system once.
- The straw/manure mixture and alfalfa were processed individually through the feed system once.
- The horse feed was mixed into the sediment on the mixing pad using the excavator bucket. This process served to reduce the moisture content of the sediment and reduced the average sediment particle size to less than 0.5-inch diameter.
- The straw/manure; alfalfa; and 35 pounds of fertilizer that contained nitrogen, potassium, and phosphorus (13/13/13) were added to the mixture.
- The mixture was mixed using the excavator and front-end loaders for 30 minutes.
- The mixture was processed through the feed system once.

The compost piles were constructed as described in Subsection 4.1.3, with the following exceptions:

- Each load of compost was moistened with water from two garden hoses as it was delivered to the pads. Approximately 400 gallons of water were applied to each compost pile.
- The final volume of the mixture to be composted was 12 cubic yards per pile.
- The wood chip bases were 16 feet long, 9 feet wide, and 8 inches thick.
- No test bags of fungus-inoculated wood chips or finely homogenized compost were placed in piles 3 and 4.
- Bales of straw were stacked on their sides around the compost piles to form a retaining wall for the piles. This was done to minimize short-circuiting of air and to prevent the insulating blanket from sliding down the sides of the compost piles.

5.1.4 Operations Schedule

Pile construction was completed and the temperature control systems and recorders started on 25 February 1988. Samples from compost piles 3 and 4 were analyzed for contaminant concentration nine times over a 153-day test period (see Table 5-3).

5.2 RESULTS

5.2.1 Compost Temperatures

The following temperature records were maintained for piles 3 and 4:

- Temperature recorders - Continuous strip-chart. LED readouts read/recorded daily.
- Thermistors - Read/recorded daily (these probes were part of the temperature control system).
- Hand-held landfill thermocouple probes - Temperature profile of each pile made daily.
- Ambient high and low air temperatures - Read/recorded daily.

Table 5-3

Operation Schedule of Compost
Piles 3 and 4

Day	Date	Event
0	25 Feb 1988	Piles set up, temperature recorders and control systems started. Time zero compost and leachate samples taken. Analyses: explosives, intermediates, TOC.
14	10 Mar 1988	Two-week samples taken. Analyses: explosives, intermediates.
33	29 Mar 1988	Five-week samples taken. Analyses: explosives, intermediates, TOC. Compost was transported from test pads, remixed, and fortified with organic carbon (500 lb horse feed, 1 bale alfalfa, and 15 lb fertilizer per pile). Piles were rewatered during reconstruction on the test pads.
55	20 Apr 1988	Eight-week samples taken. Analyses: explosives, intermediates.
60	25 Apr 1988	Nine-week samples taken. Analyses: explosives, intermediates. Compost remixed and rewatered only, no organic carbon supplements added.
82	17 May 1988	Twelve-week samples taken. Analyses: explosives, intermediates.
111	15 June 1988	Sixteen-week samples taken. Piles remixed and rewatered. Analyses: explosives, intermediates, TOC.
126	30 June 1988	Eighteen-week samples taken. Analyses: explosives, intermediates, TOC.
139	13 July 1988	Twenty-week samples taken. Analyses: explosives, intermediates, TOC.
153	27 July 1988	Twenty-two-week samples taken. Analyses: explosives, intermediates, TOC.

Data obtained with the hand-held landfill probe were considered the most representative of the piles as six individual data points were taken within each pile on a daily basis. Temperature data are presented in Figures 5-1 through 5-11 as follows:

Pile	Figure	Data
Ambient	5-1	High and low ambient air temperatures during the field demonstration
3	5-2	Mean daily compost temperatures as registered with landfill probe
3	5-3	Daily thermistor readings
3	5-4	Condensed strip-chart recorder record
3	5-5	Comparison of temperatures in top and bottom of pile (landfill probe)
3	5-6	Comparison of temperatures in toe ¹ and heel ² of pile (landfill probe)
4	5-7	Mean daily temperatures as registered with hand-held landfill probe
4	5-8	Daily thermistor readings
4	5-9	Condensed strip-chart recorder record
4	5-10	Comparison of temperatures in top and bottom of pile (landfill probe)
4	5-11	Comparison of temperatures in toe and heel of pile (landfill probe)

¹Toe - End of pile near blower.

²Heel - End of pile away from blower.

5.2.2 Compost Moisture Content

The moisture content of compost samples removed from piles 3 and 4 ranged from 25 to 56 weight percent (see Figures 5-12 and 5-13).

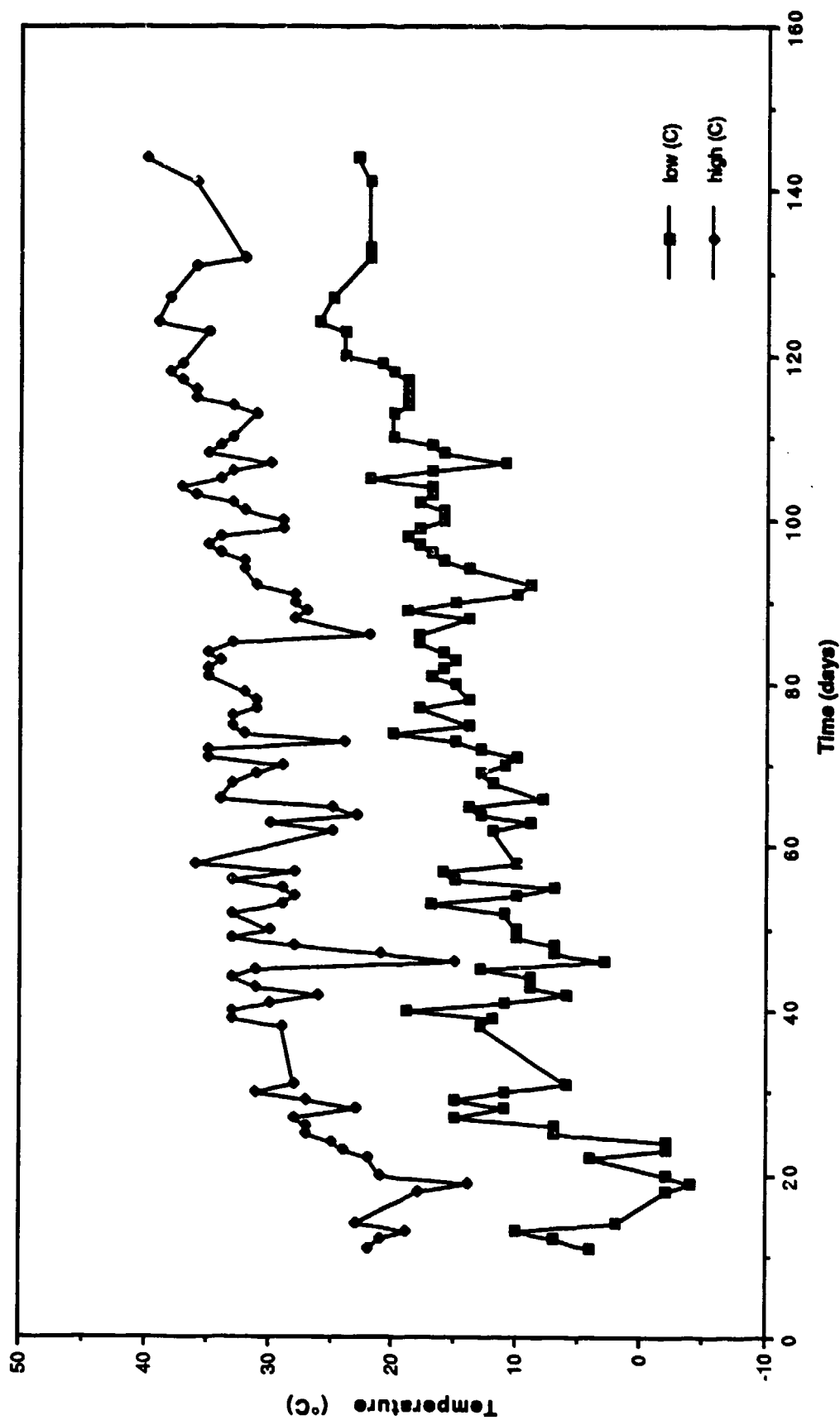


Figure 5-1. High and low ambient temperature during test period.

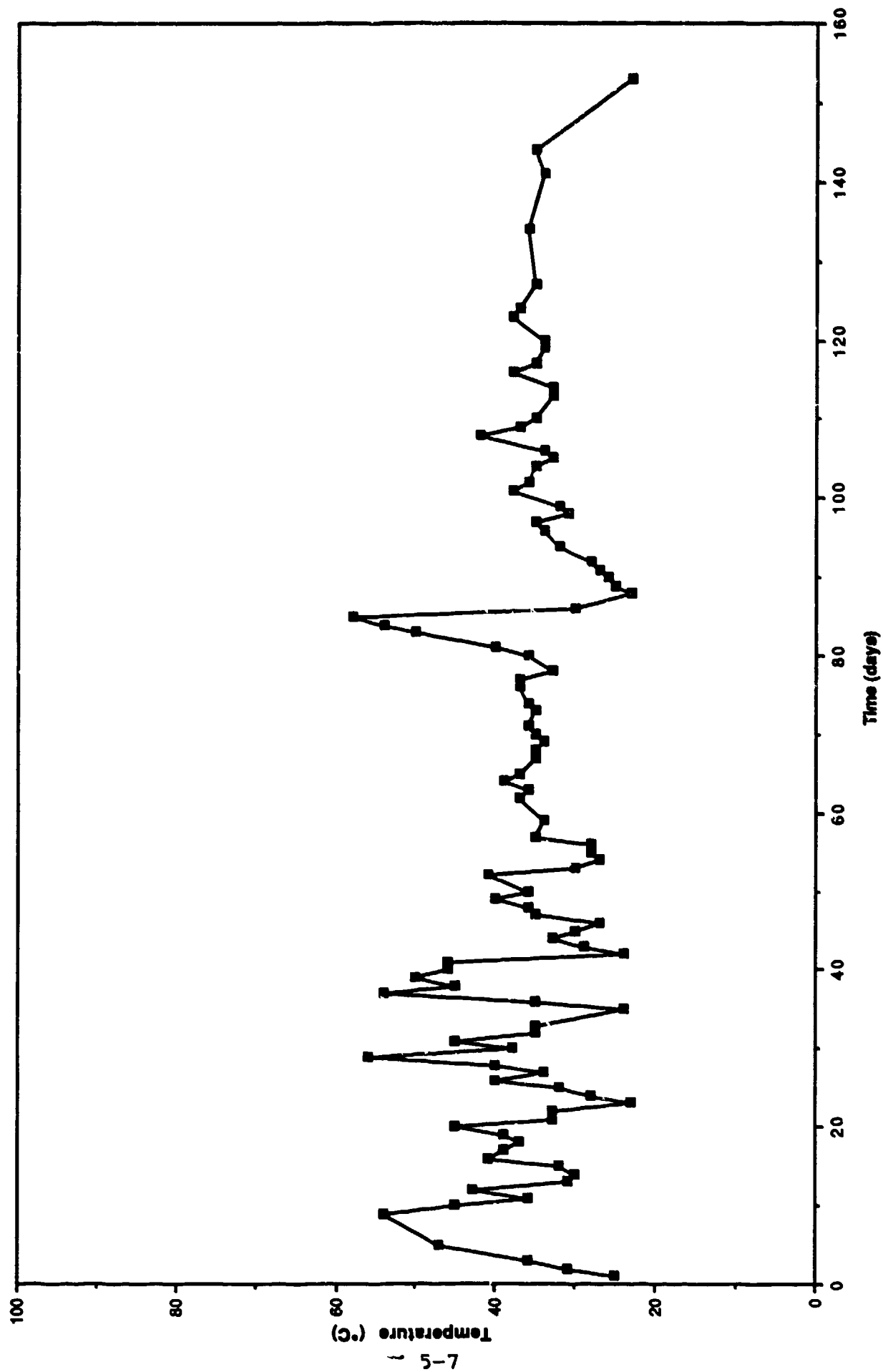


Figure 5-2. Mean compost temperature in pile 3.

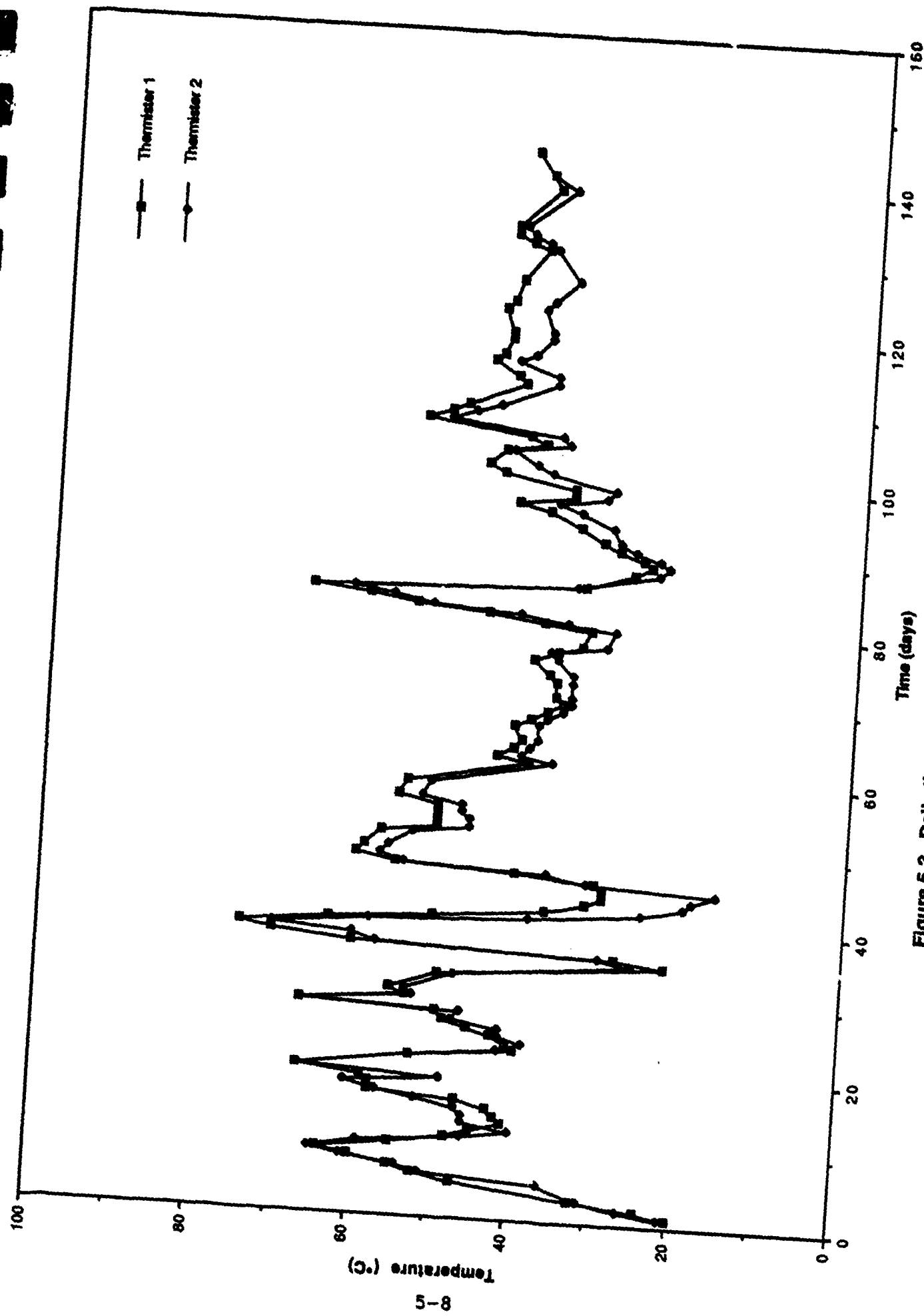


Figure 5-3. Daily thermistor readings for compost pile 3.

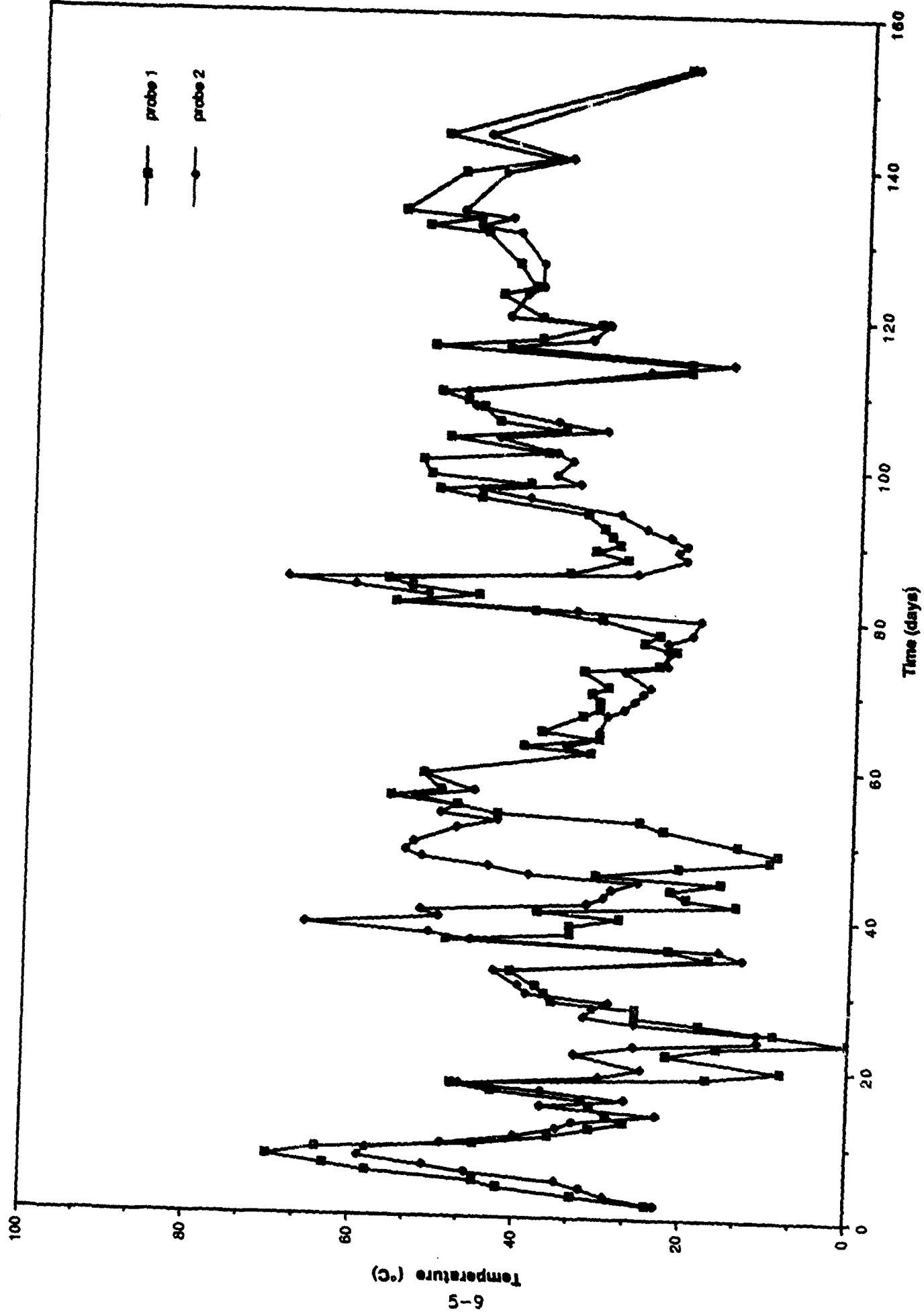


Figure 5-4. Condensed temperature strip chart for compost pile 3.

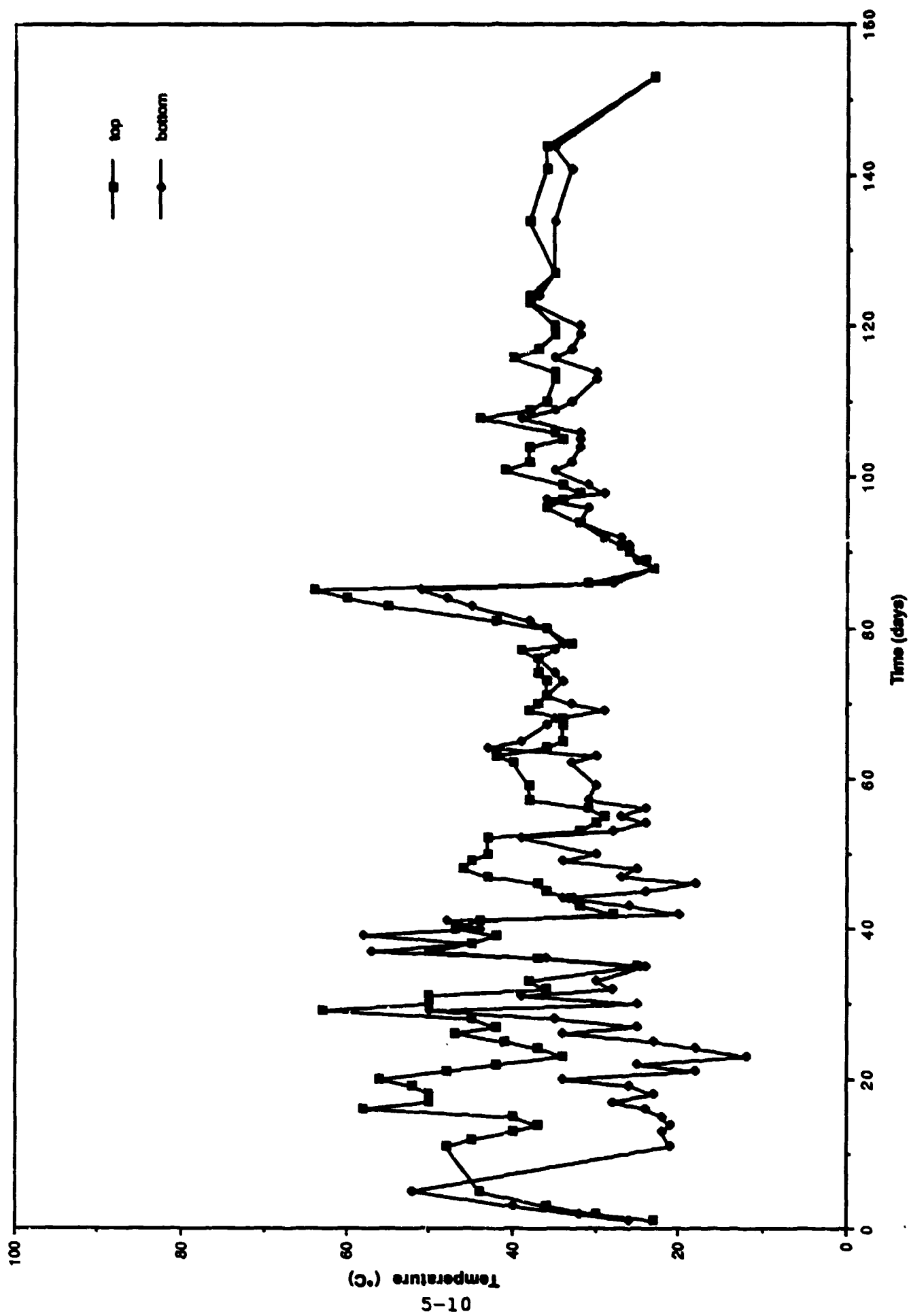


Figure 5-5. Temperature in top and bottom of compost pile 3.

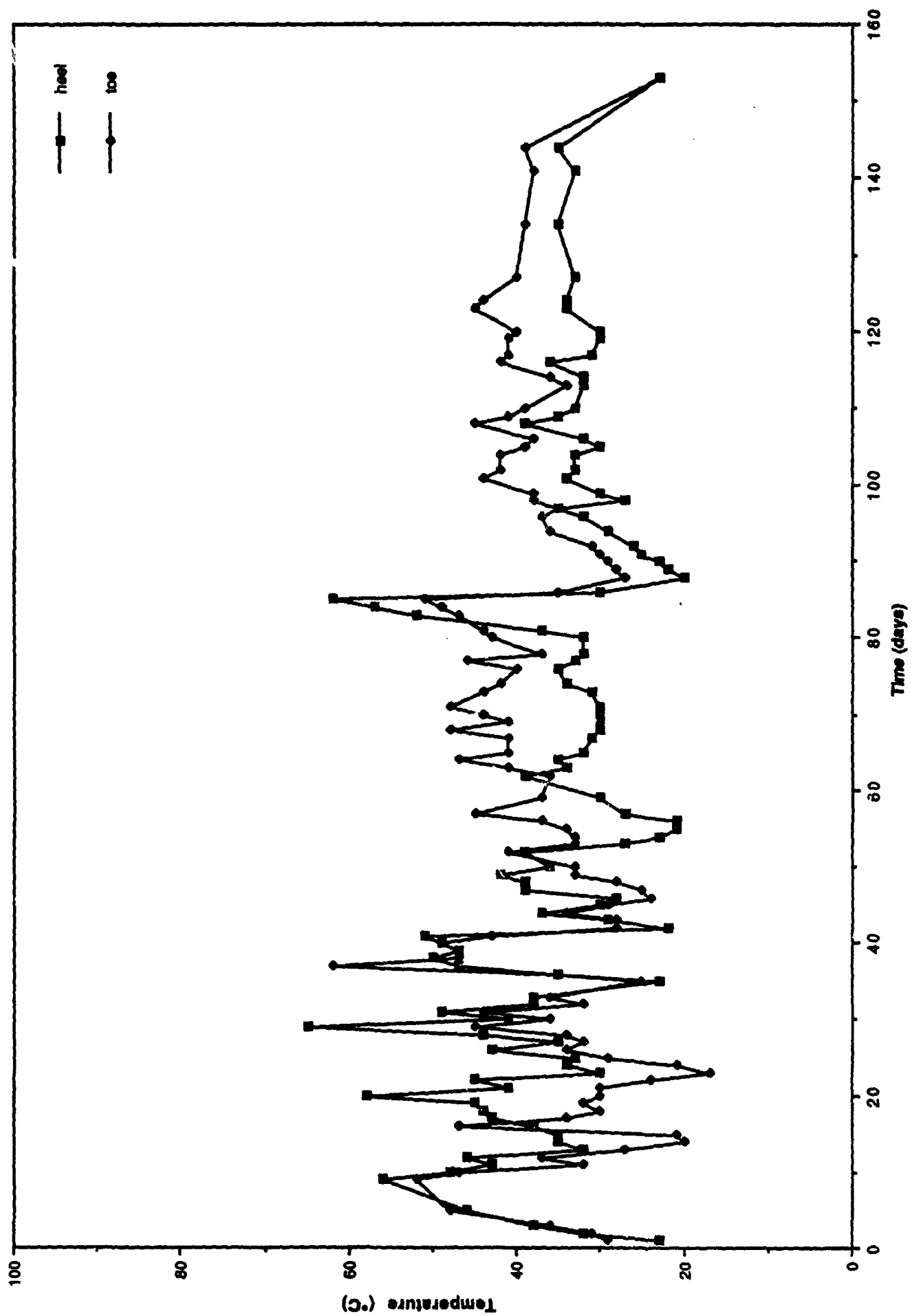


Figure 5-6. Mean temperature in toe and heel of compost pile 3.

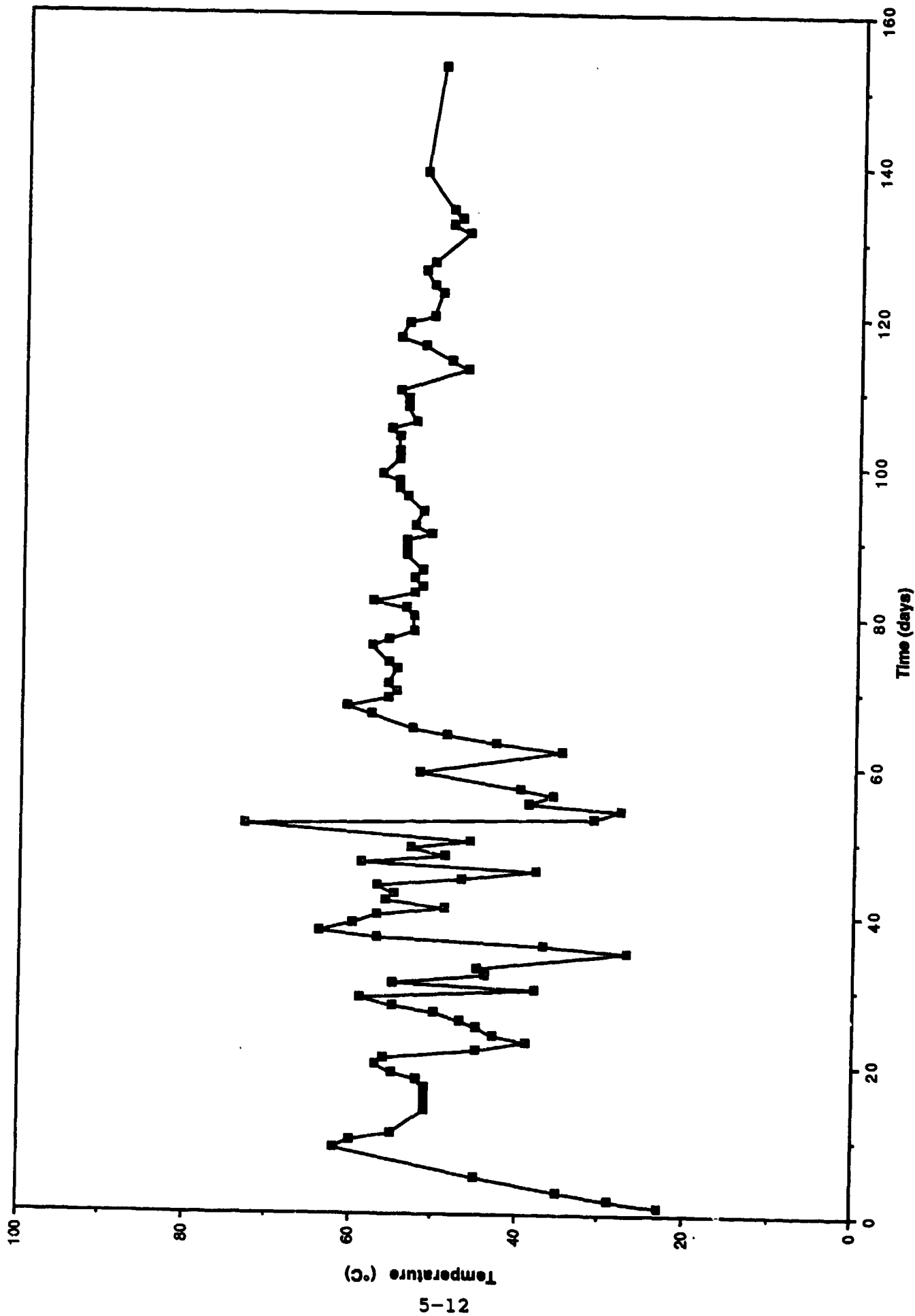


Figure 5-7. Mean compost temperature in pile 4.

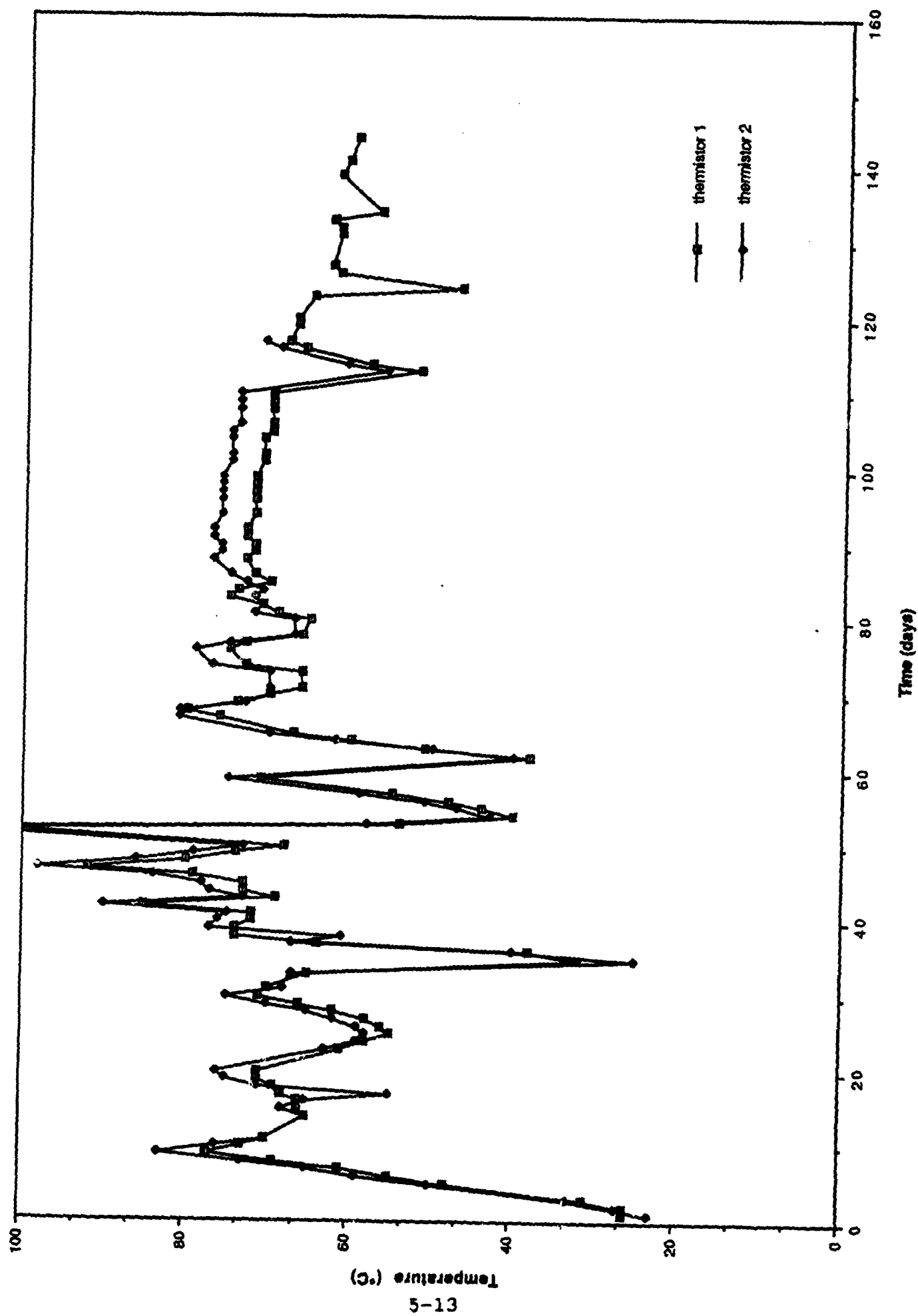


Figure 5-8. Daily thermistor readings for compost pile 4.

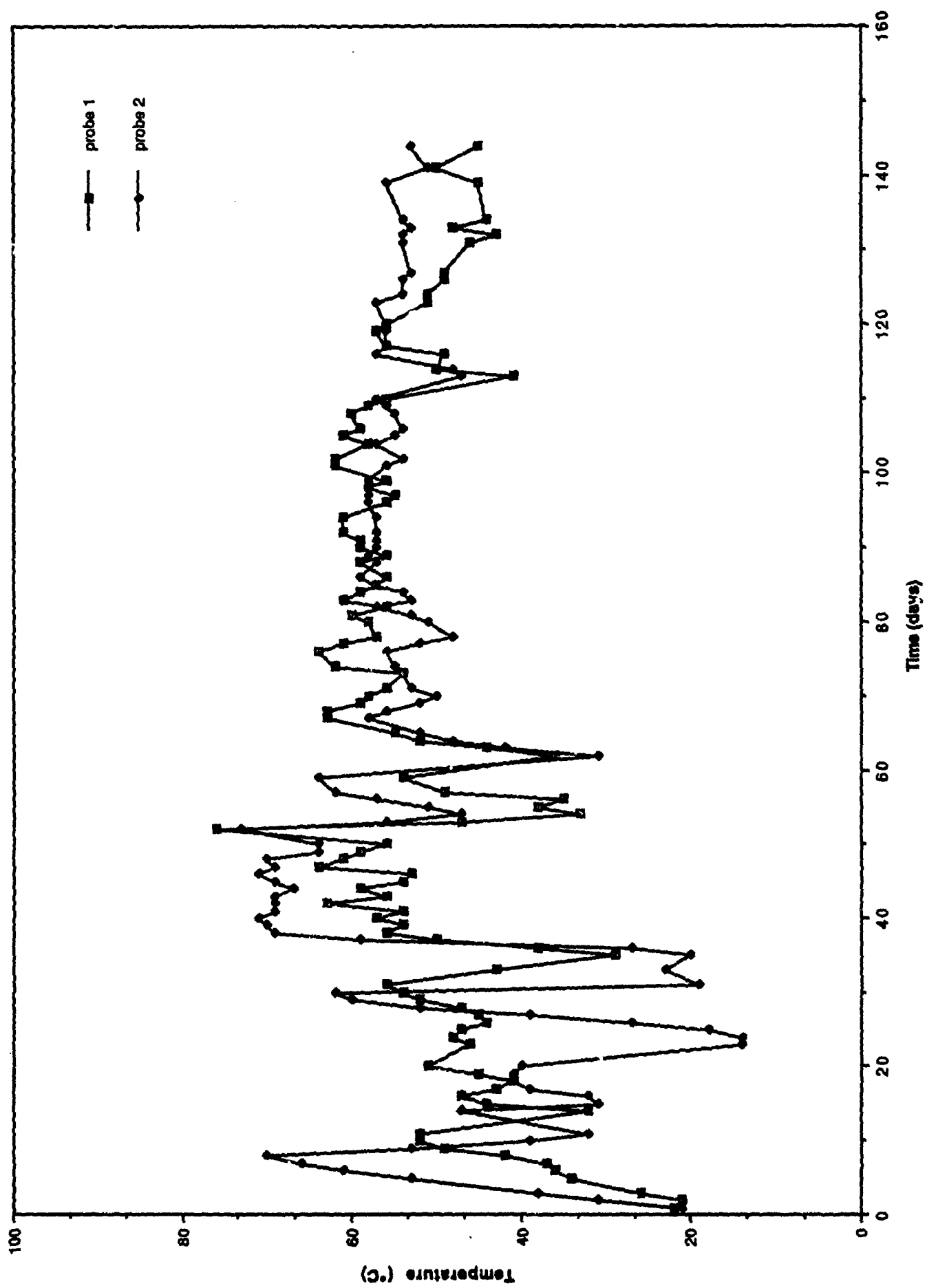


Figure 5-9. Condensed temperature strip chart for compost pile 4.

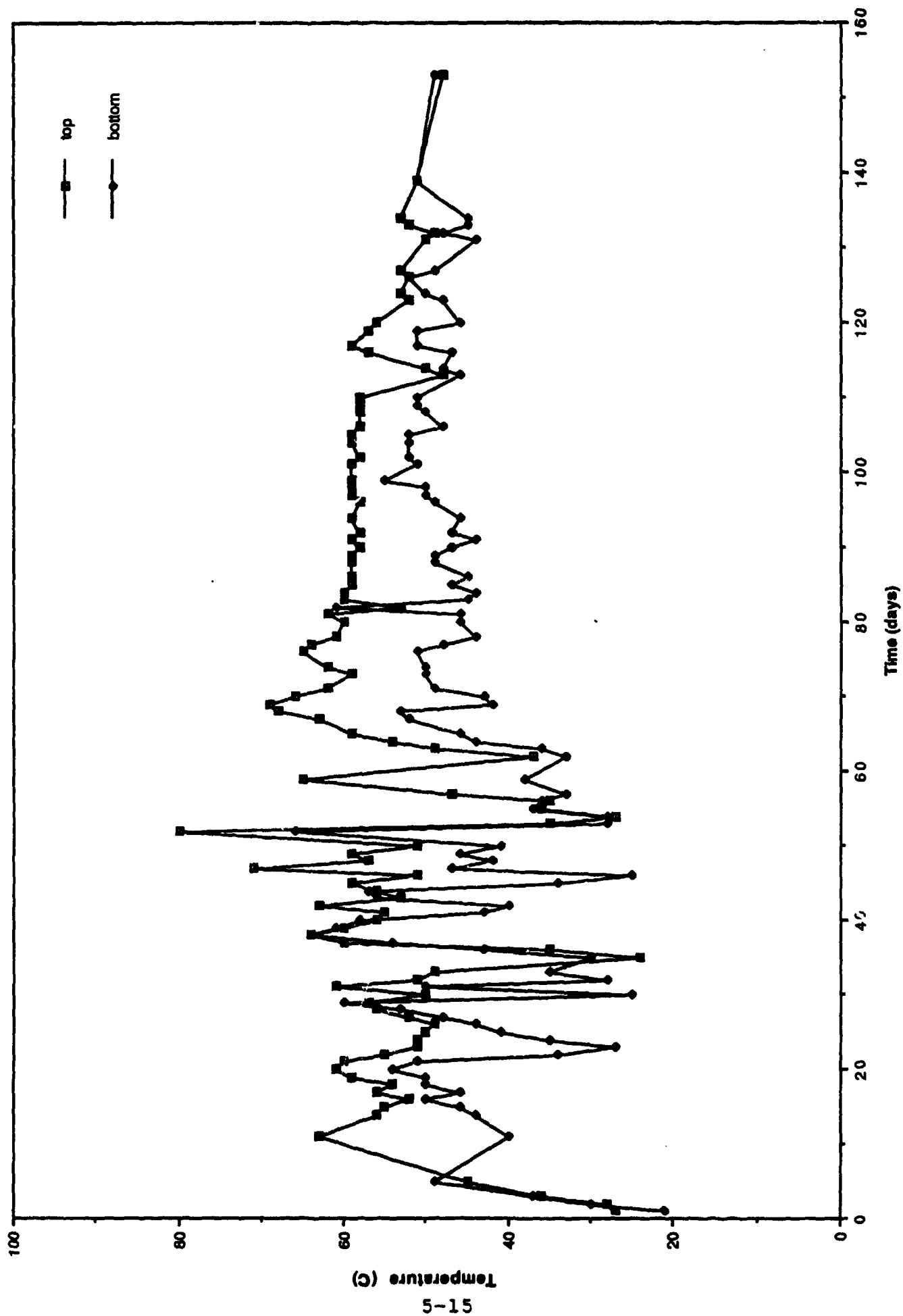


Figure 5-10. Temperature in top and bottom of compost pile 4.

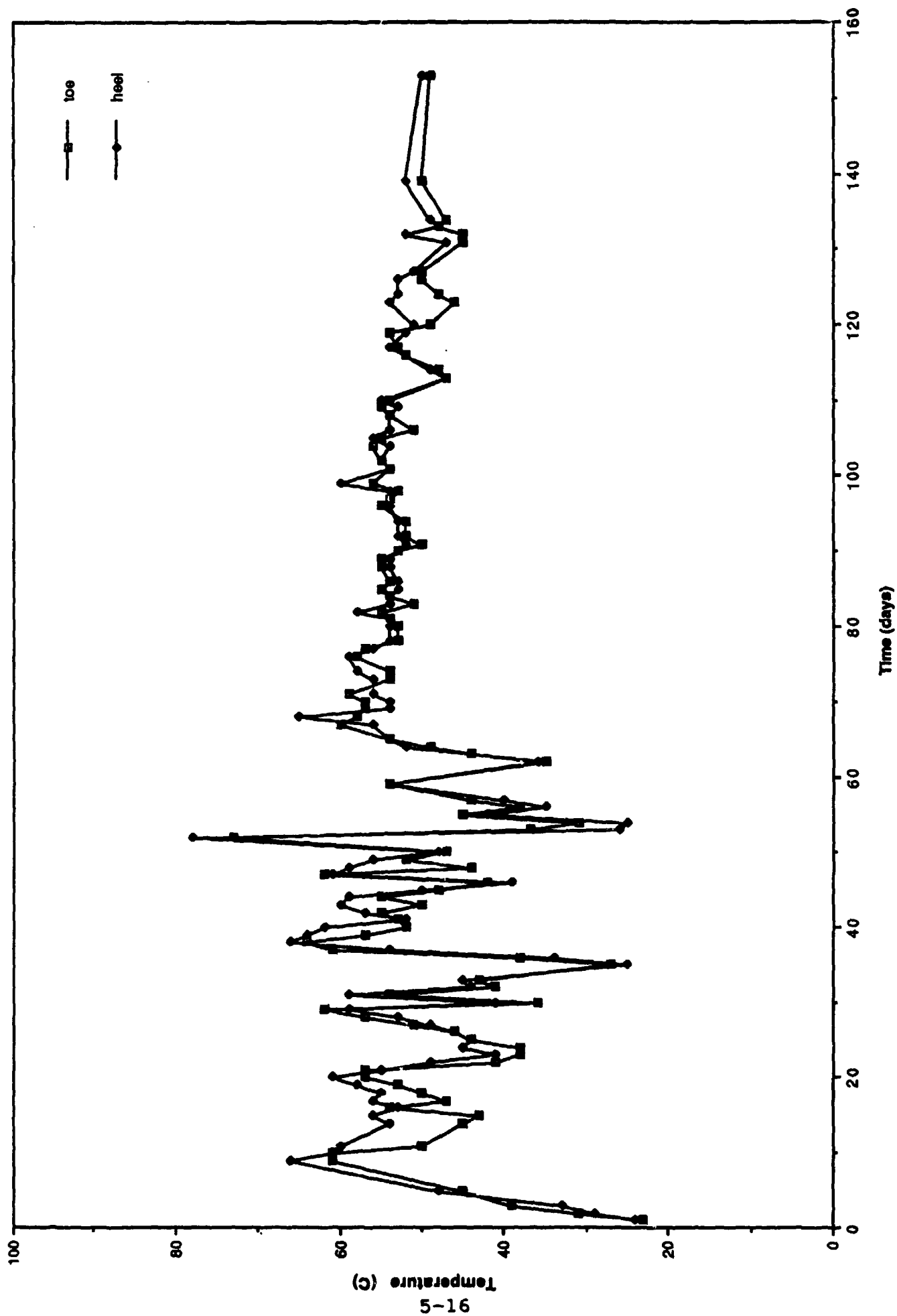


Figure 5-11. Temperature in toe and heel of compost pile 4.

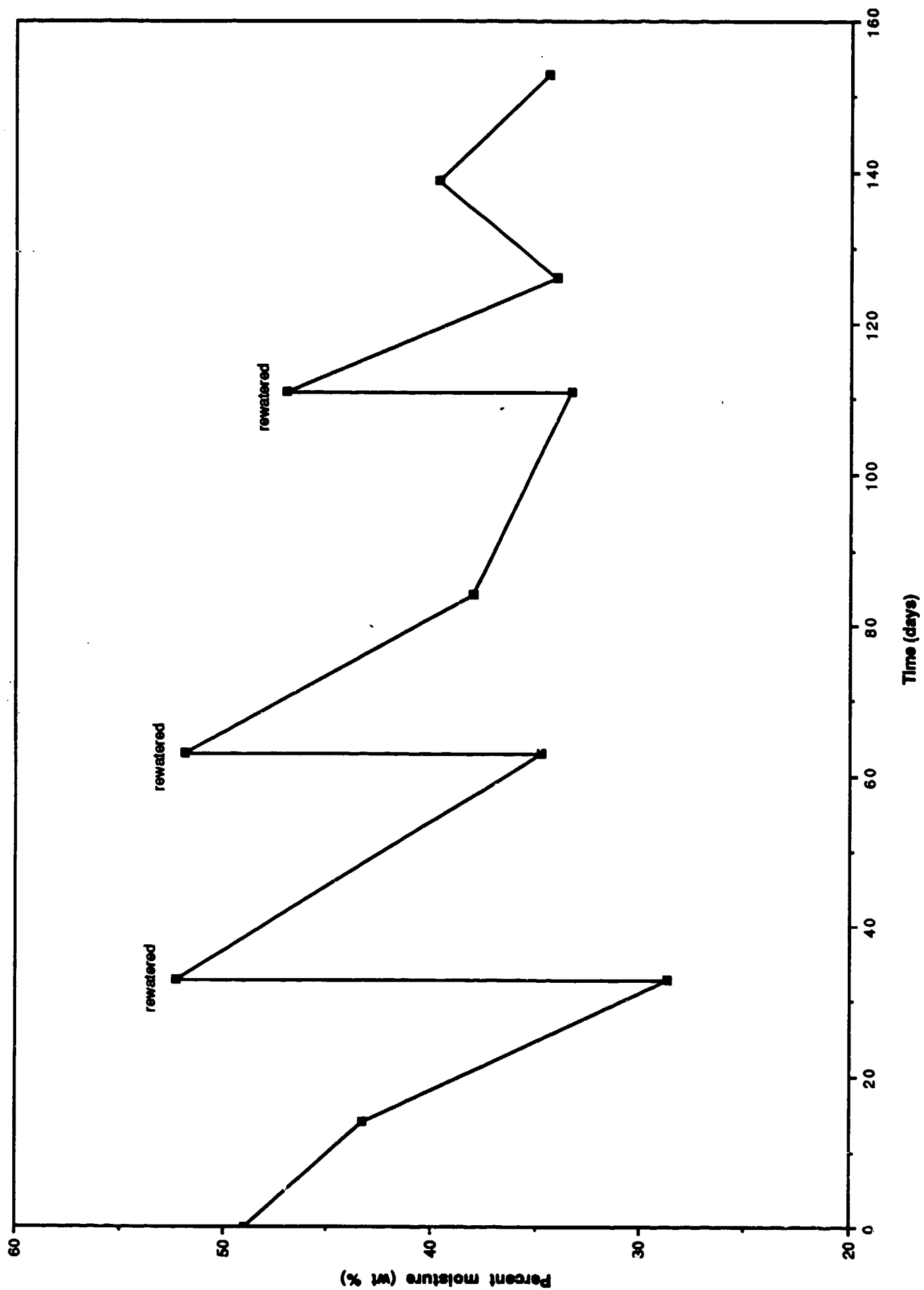


Figure 5-12. Moisture content of compost pile 3.

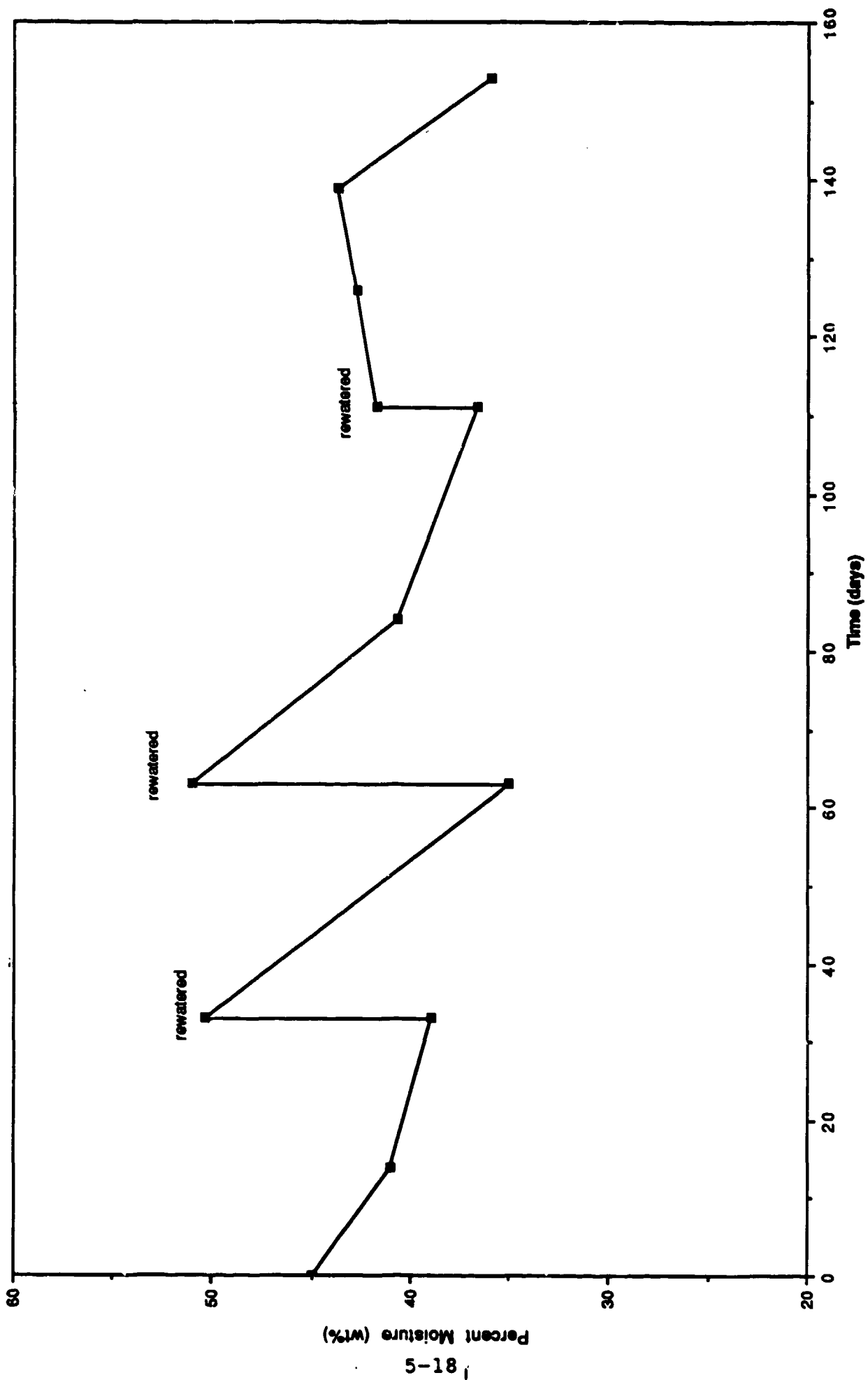


Figure 5-13. Moisture content of compost pile 4.

5.2.3 Microbial Enumeration

Spread-plate microbial enumerations conducted on serially diluted compost extracts confirmed the existence of heterotrophic microorganisms capable of growth at 35°C and 55°C in compost piles 3 and 4 (see Figures 5-14 and 5-15).

5.2.4 Fate of Sediment Contaminants

Total explosives concentrations in piles 3 and 4 at the beginning of the study were 16,460 and 17,870 mg/kg, respectively. After 153 days, when the study was terminated, total explosives concentrations in piles 3 and 4 were 326 mg/kg and 74 mg/kg, respectively. Percent reduction in total explosives was 98.0 percent for pile 3 and 99.6 percent for pile 4.

When the field demonstration was terminated after 153 days, the bulk density and total volume of the compost remaining in pile 3 were 270 pounds per cubic yard and 10 cubic yards, respectively. The corresponding values for pile 4 were 268 pounds per cubic yard and 9 cubic yards. Based on the initial compost volume of 12 cubic yards per pile and the bulk density of 268 pounds per cubic yard, the volume and mass of compost pile 3 were reduced 17 percent, and the volume and mass of compost pile 4 were reduced 25 percent. Based on the initial and final bulk densities, compost volumes, and explosives concentrations, total masses of 23.6 and 26.0 kilograms of explosives were degraded in compost piles 3 and 4 over the 153-day test period, respectively.

Samples of water from the sump were analyzed for explosives and transformation products on days 0, 16, 22, and 153. The analytes were nondetectable in all sump water samples.

Linear plots of contaminant concentration versus time were prepared for TNT, RDX, HMX, and intermediates for each compost pile. These data are presented in Figures 5-16 through 5-29 as follows:

Pile	Figure	Contaminant
3	5-16	TNT
3	5-17	RDX
3	5-18	HMX
3	5-19	Total explosives
4	5-20	TNT
4	5-21	RDX
4	5-22	HMX
4	5-23	Total explosives
3	5-24	Total diamino-nitrotoluenes
3	5-25	2-Amino-4,6-dinitrotoluene

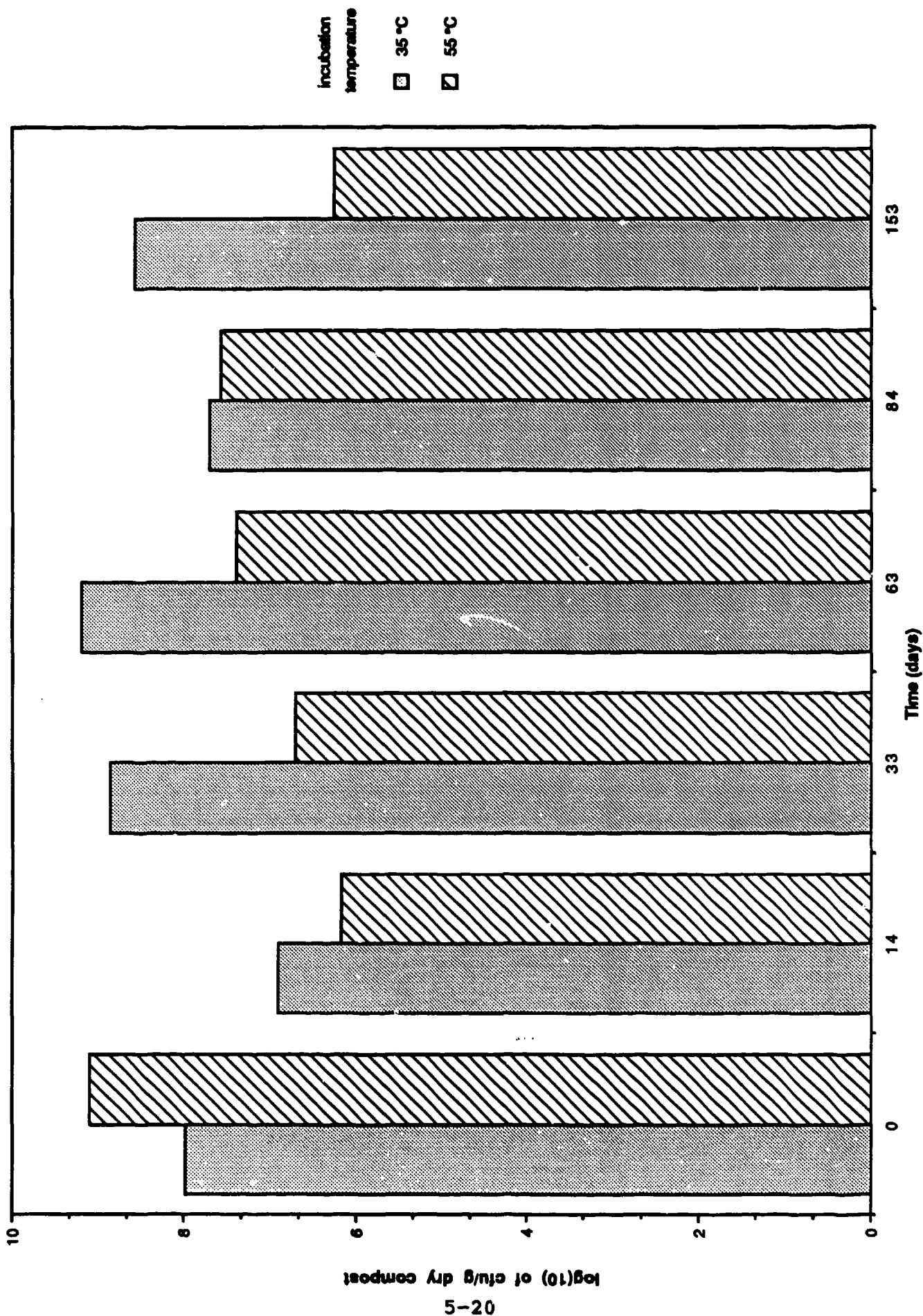


Figure 5-14. Population density of heterotrophic microorganisms in compost pile 3.

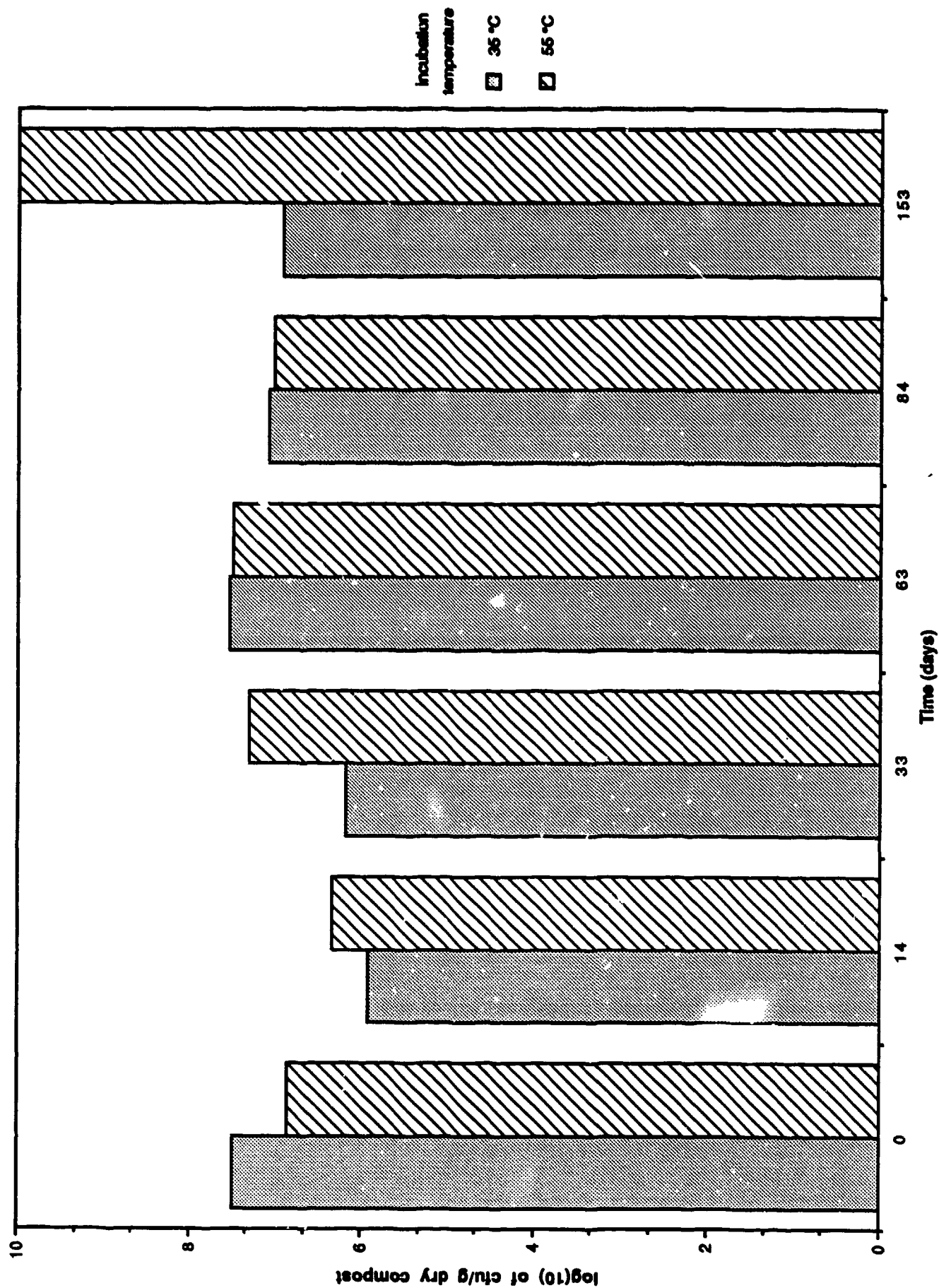


Figure 5-15. Population density of heterotrophic microorganisms in compost pile 4.

Pile	Figure	Contaminant
3	5-26	4-Amino-2,6-dinitrotoluene
4	5-27	Total diamino-nitrotoluenes
4	5-28	2-Amino-4,6-dinitrotoluene
4	5-29	4-Amino-2,6-dinitrotoluene

No data are presented for tetryl as this compound was below detection limits in all compost samples analyzed.

Mean concentrations and standard deviations of TNT, RDX, and HMX at each time point were calculated as the mean and standard deviation of all samples analyzed for the particular contaminant at the specified time point. The number of samples per time point was at least three and as many as nine (see Appendix B). Mean concentrations and standard deviations of total explosives were determined by summing the concentrations of TNT, RDX, and HMX in each sample and calculating the mean and standard deviation of these sums for each sampling (time) point.

Degradation of TNT, RDX, and HMX approximated first-order decay kinetics in both compost piles. An exponential curve fitting routine was run on the data as shown in Figures 5-16 to 5-23. The correlation coefficient (R) values for nonlinear regression ranged from 0.94 to 0.99. Copies of Figures 5-16 through 5-23 with nonlinear regression data and fitted curves are presented in Appendix B. Calculated first-order decay constants and half-lives for TNT, RDX, and HMX in compost piles 3 and 4 are presented in Table 5-4.

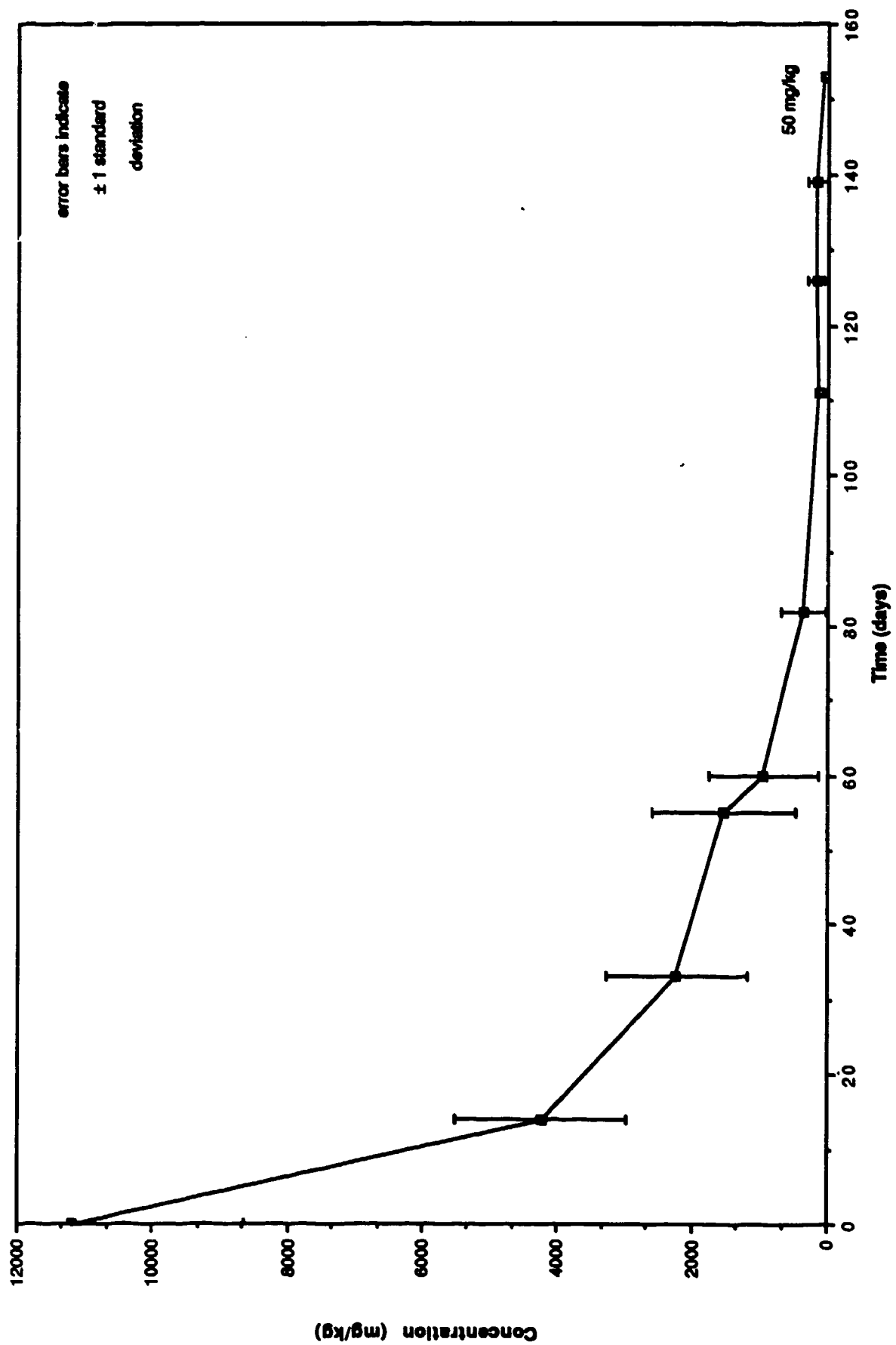


Figure 5-16. Concentration of TNT in compost pile 3.

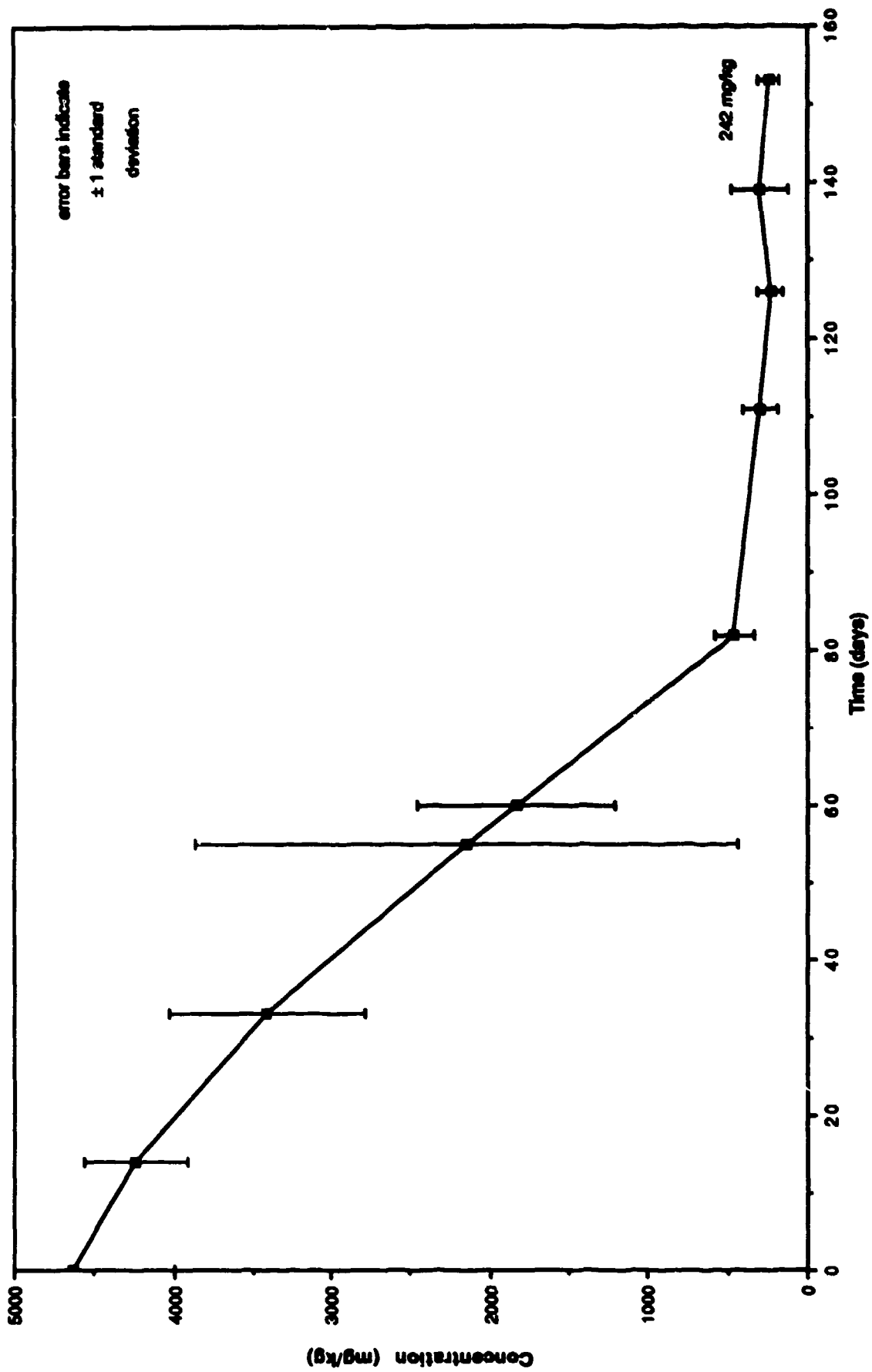


Figure 5-17. Concentration of RDX in compost pile 3.

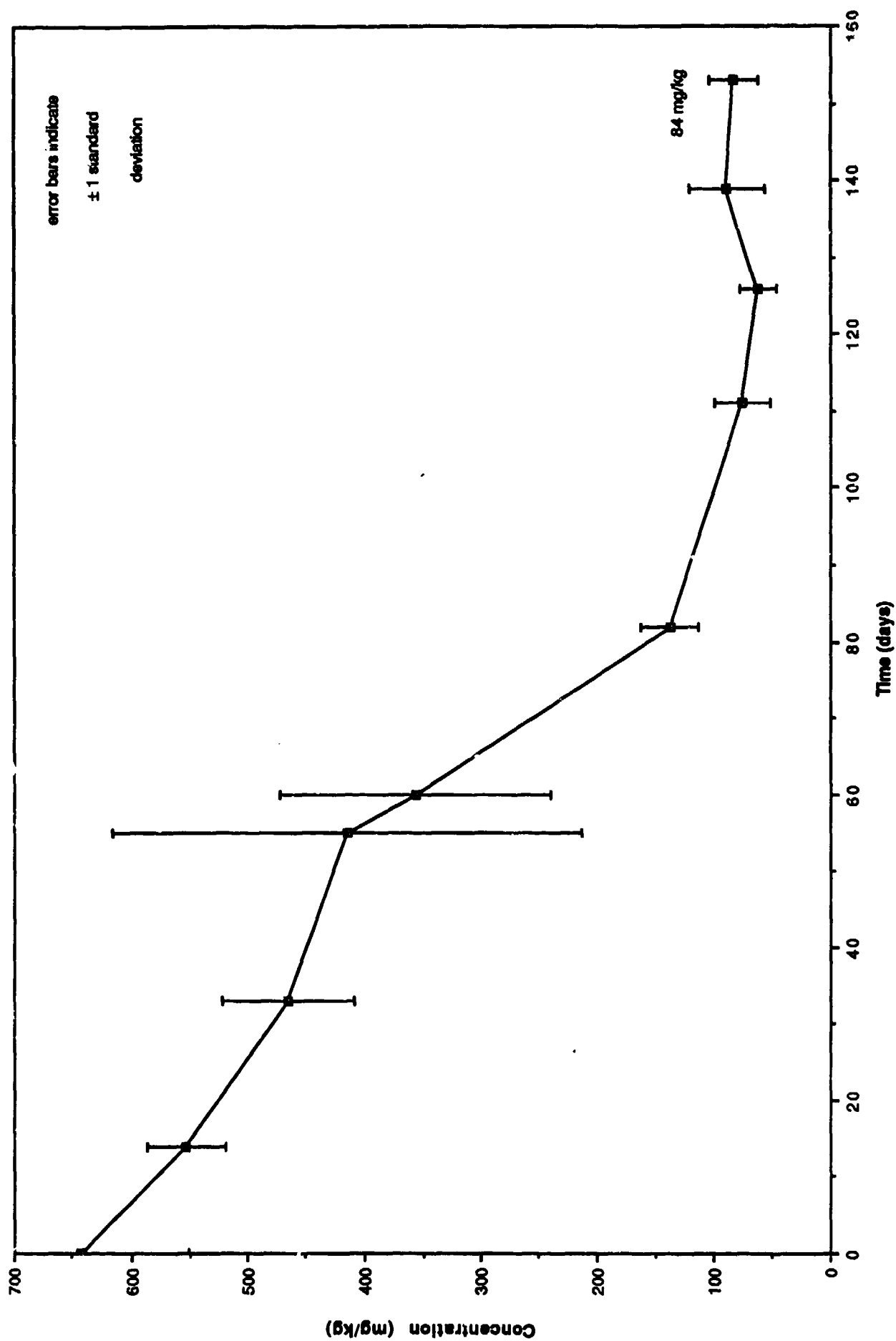


Figure 5-18. Concentration of HMX in compost pile 3.

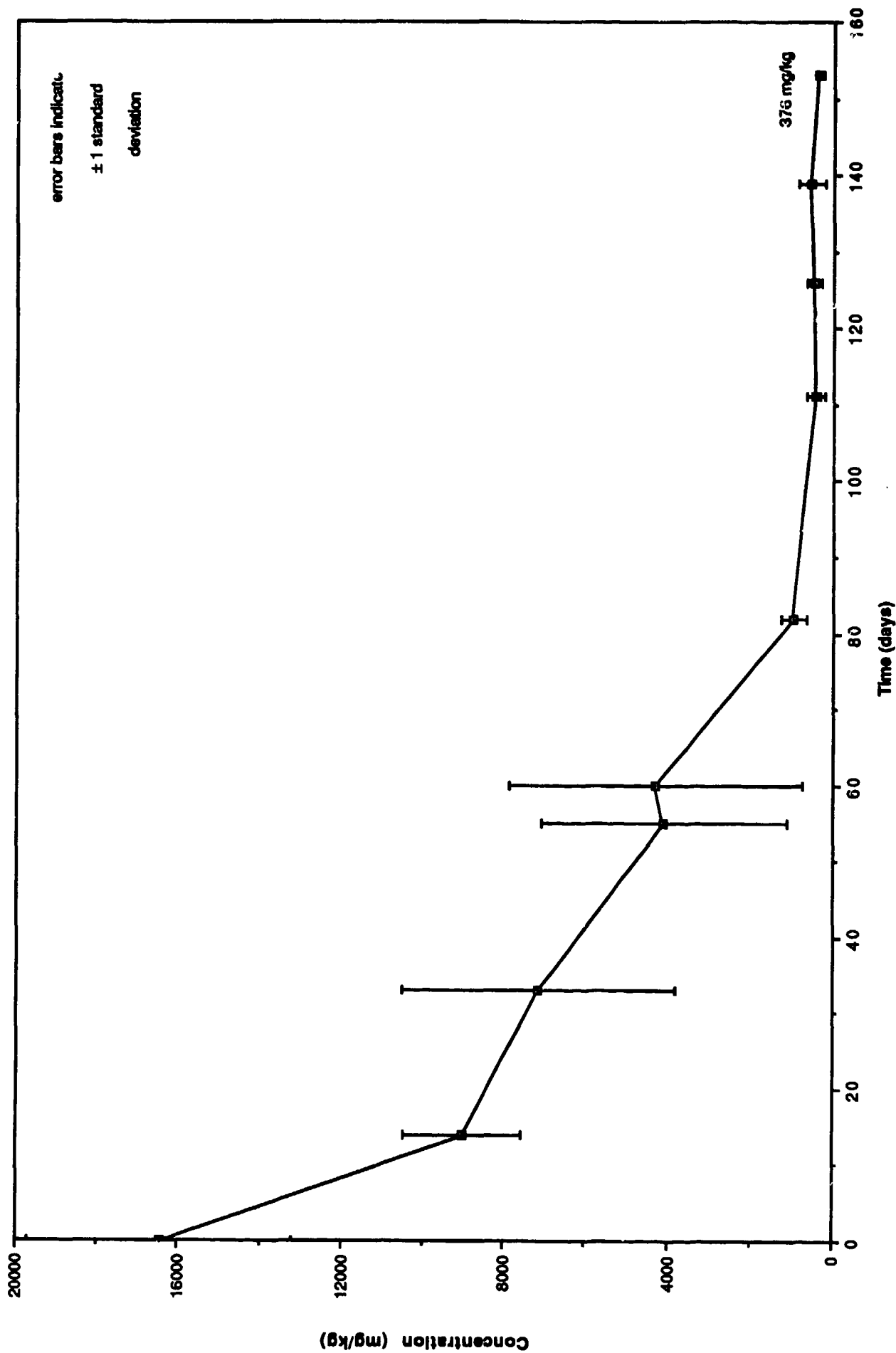


Figure 5-19. Mean concentration of total explosives in compost pile 3.

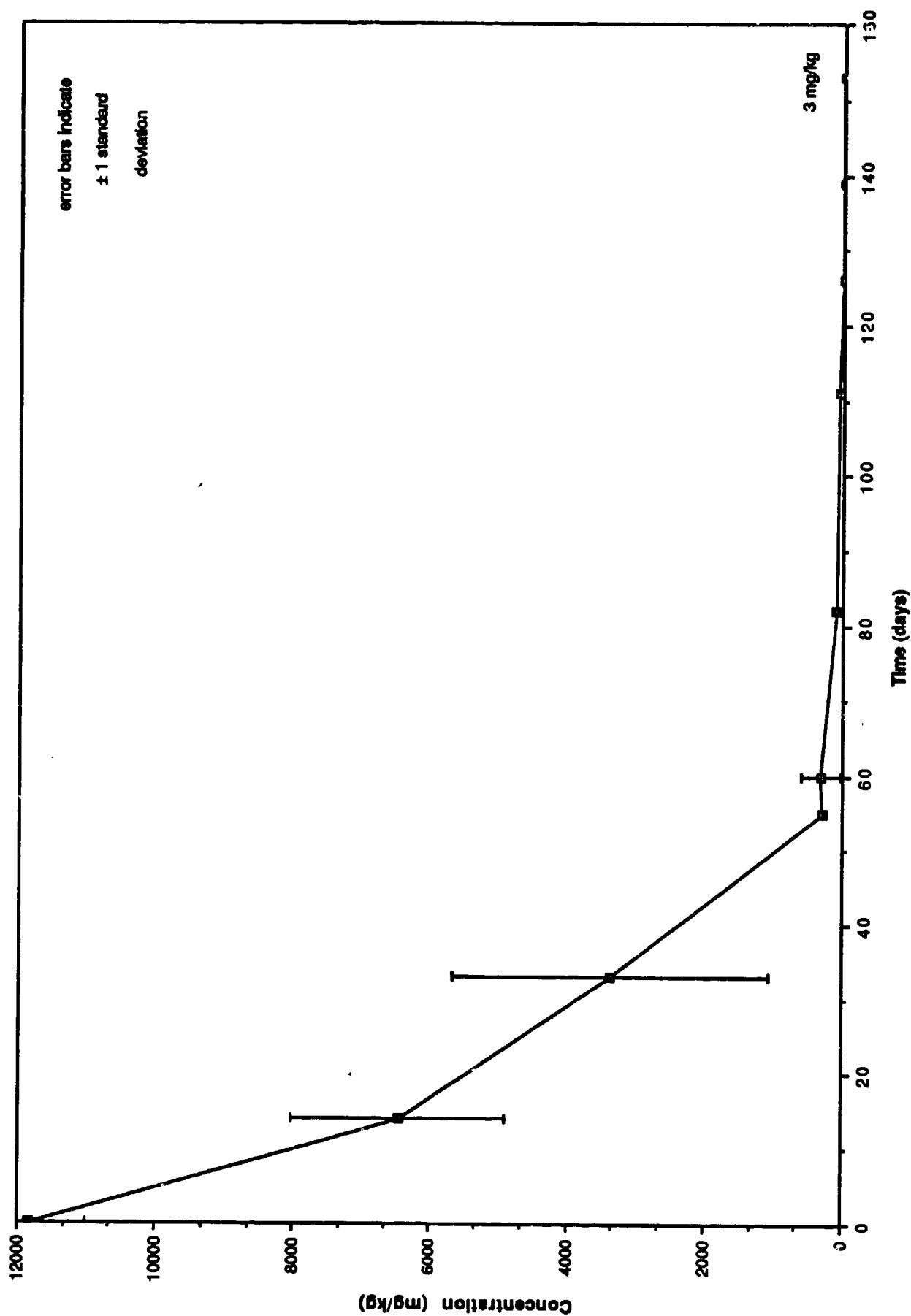


Figure 5-20. Concentration of TNT in compost pile 4.

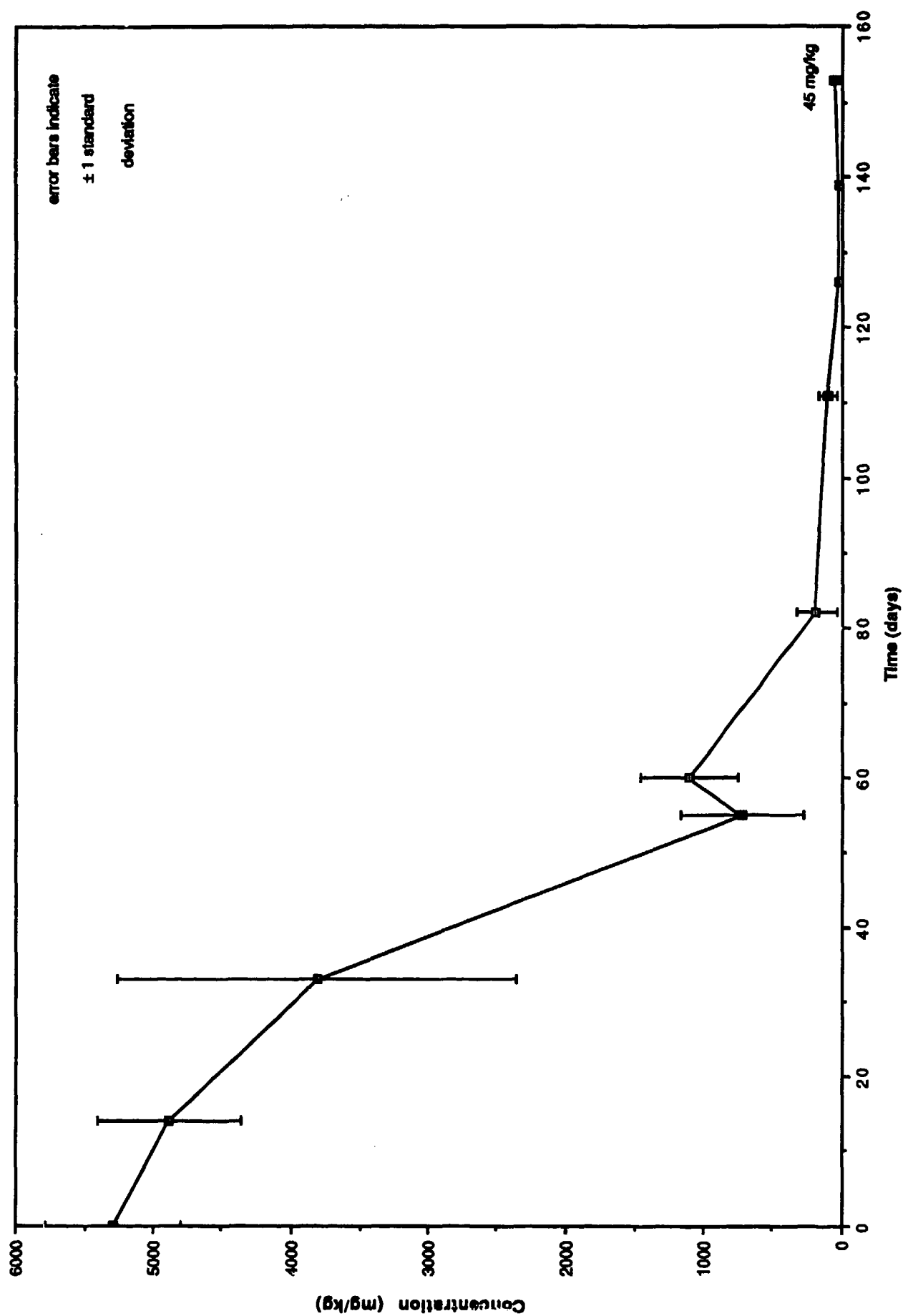


Figure 5-21. Concentration of RDX in compost pile 4.

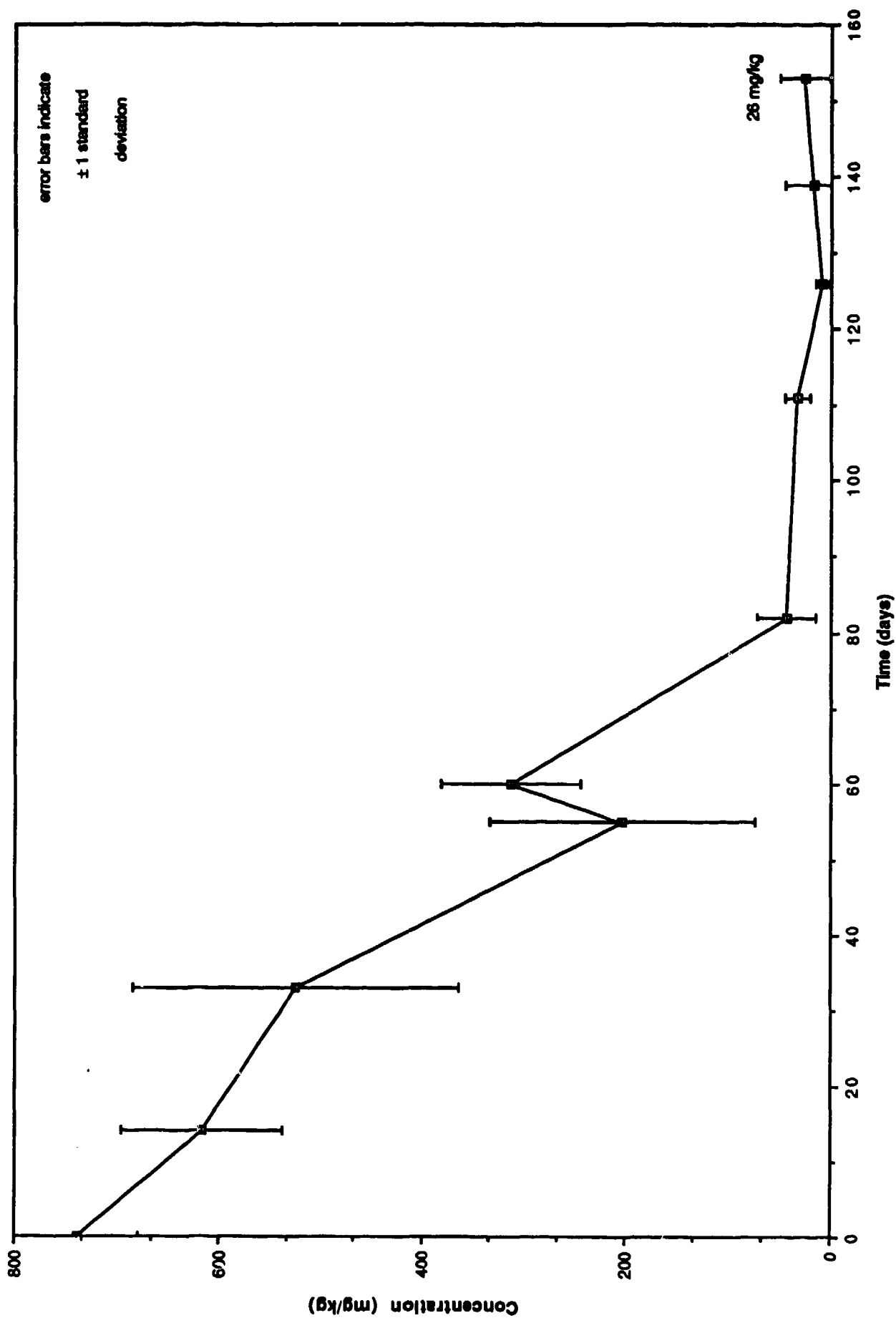


Figure 5-22. Concentration of HMX in compost pile 4.

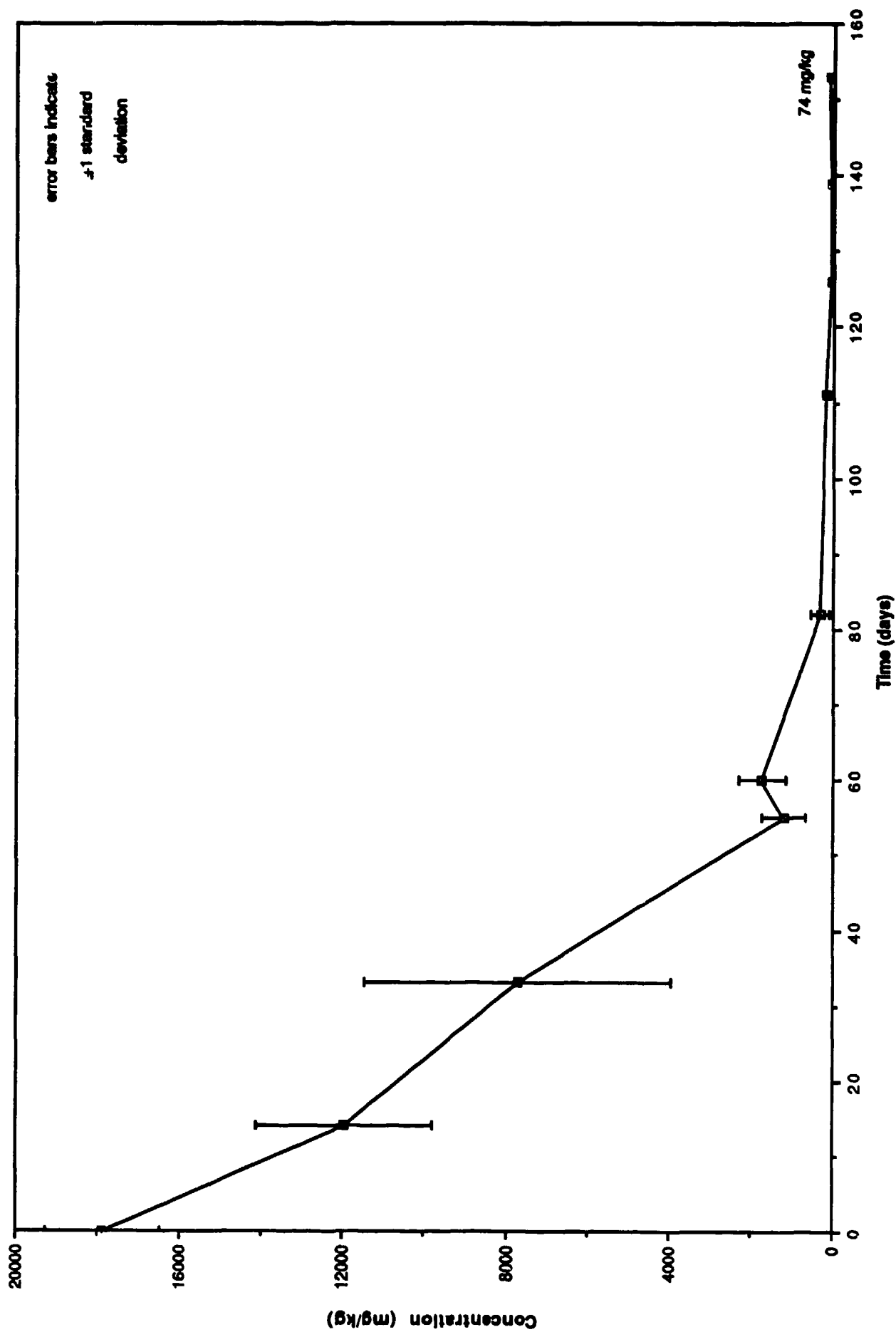


Figure 5-23. Mean concentration of total explosives in compost pile 4.

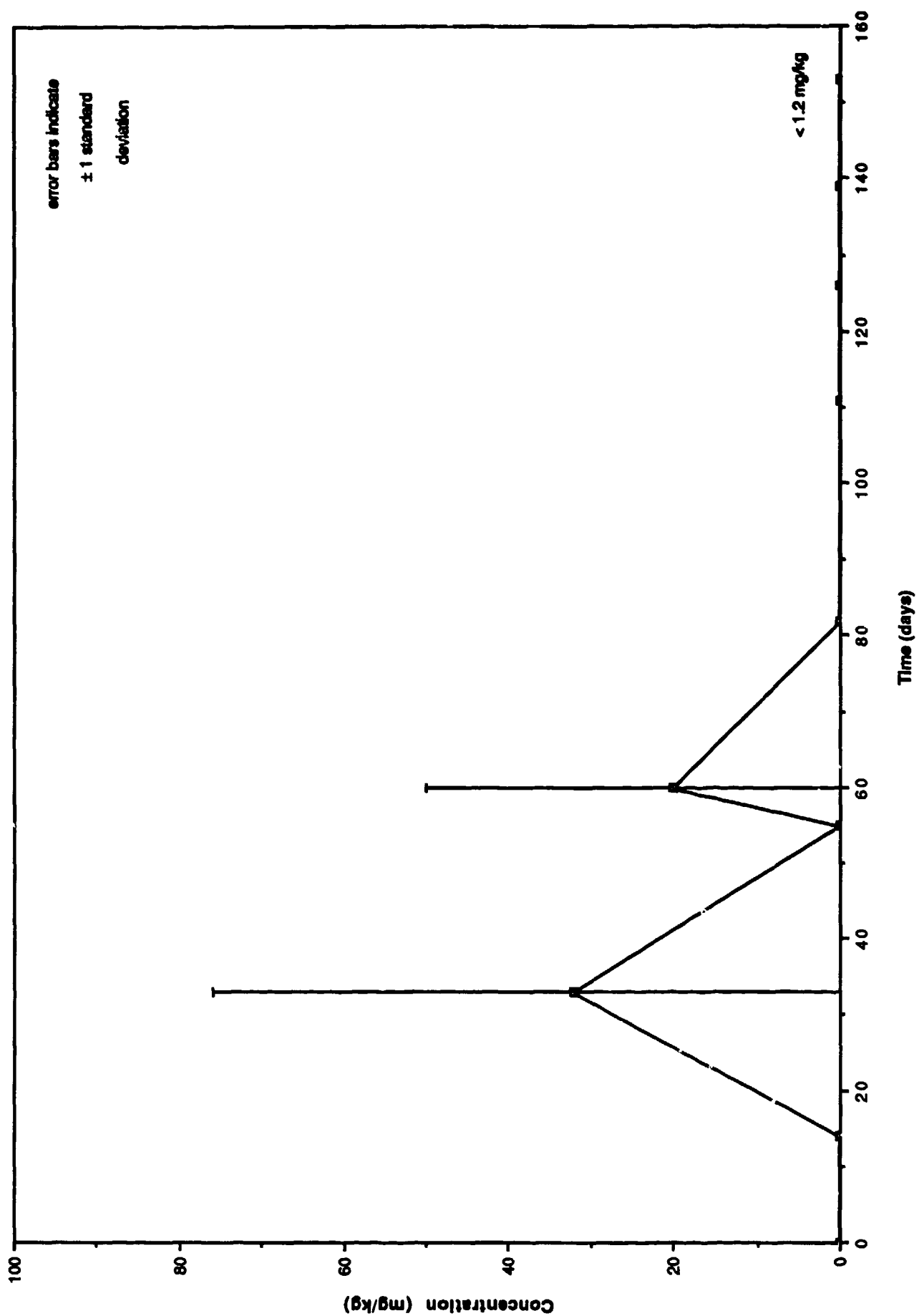


Figure 5-24. Concentration of total diaminonitrotoluenes in compost pile 3.

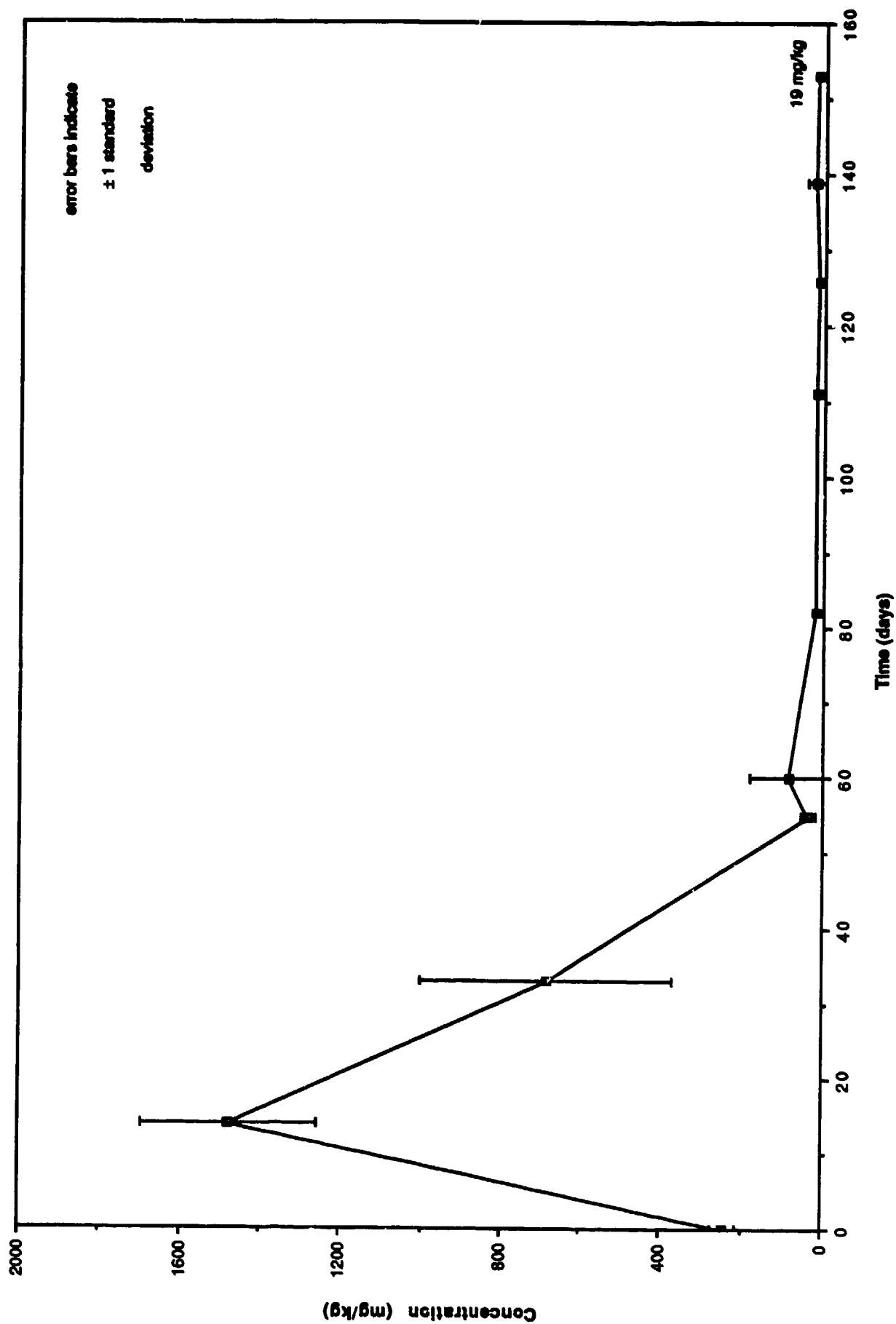


Figure 5-25. Concentration of 2-amino-4,6-dinitrotoluene in compost pile 3.

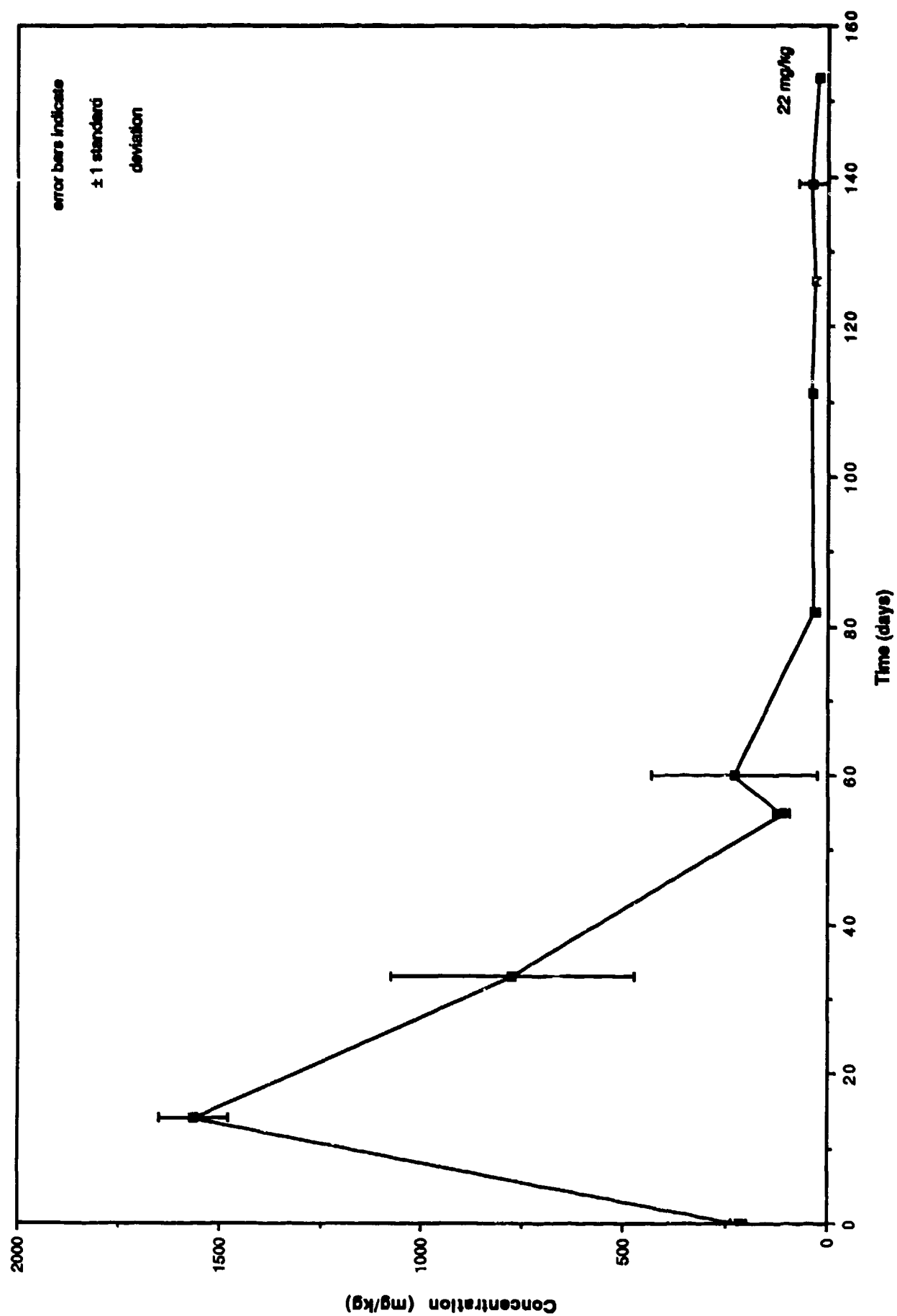


Figure 5-26. Concentration of 4-amino-2,6-dinitrotoluene in compost pile 3.

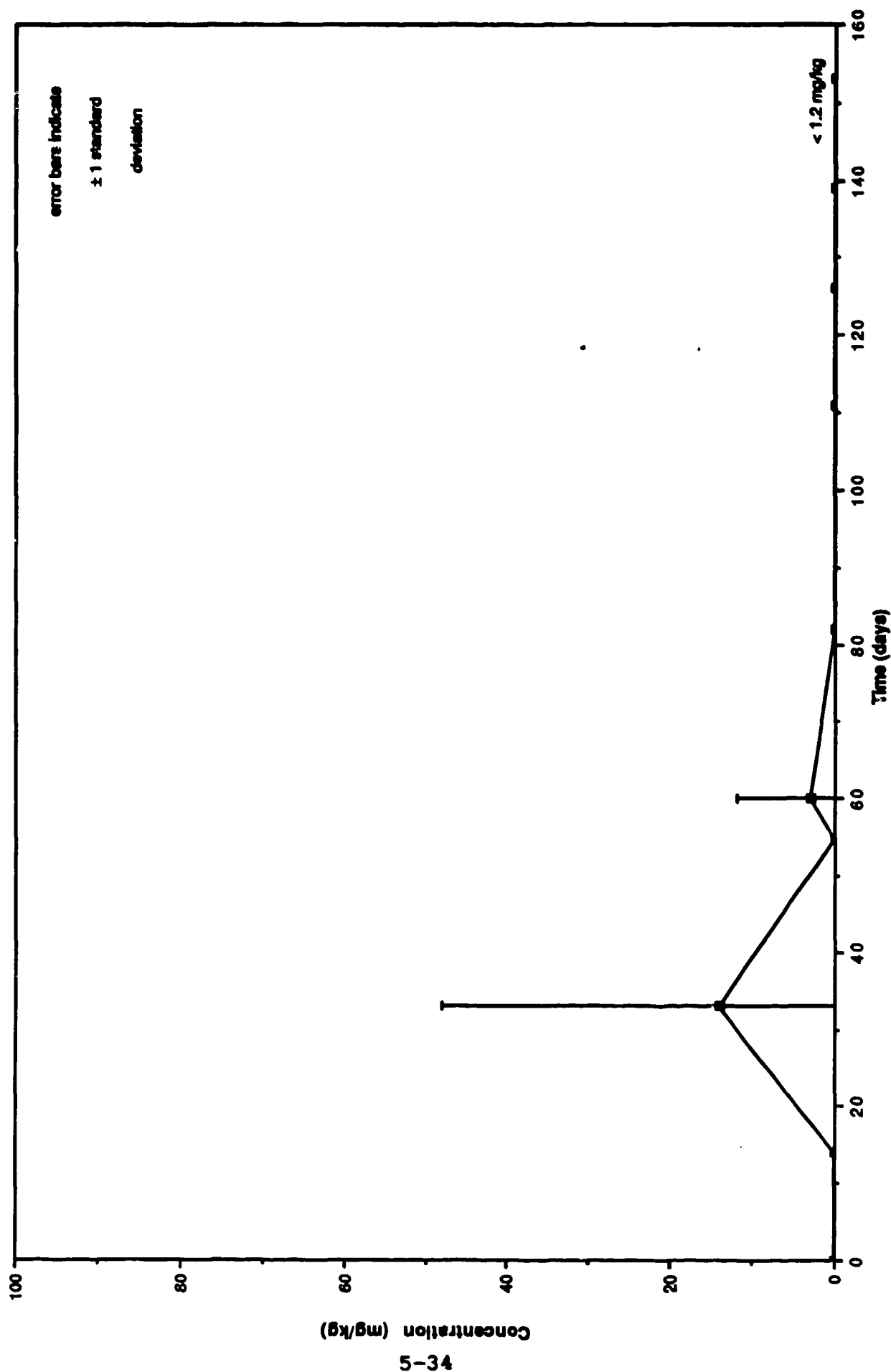


Figure 5-27. Concentration of total diammonitrotoluenes in compost pile 4.

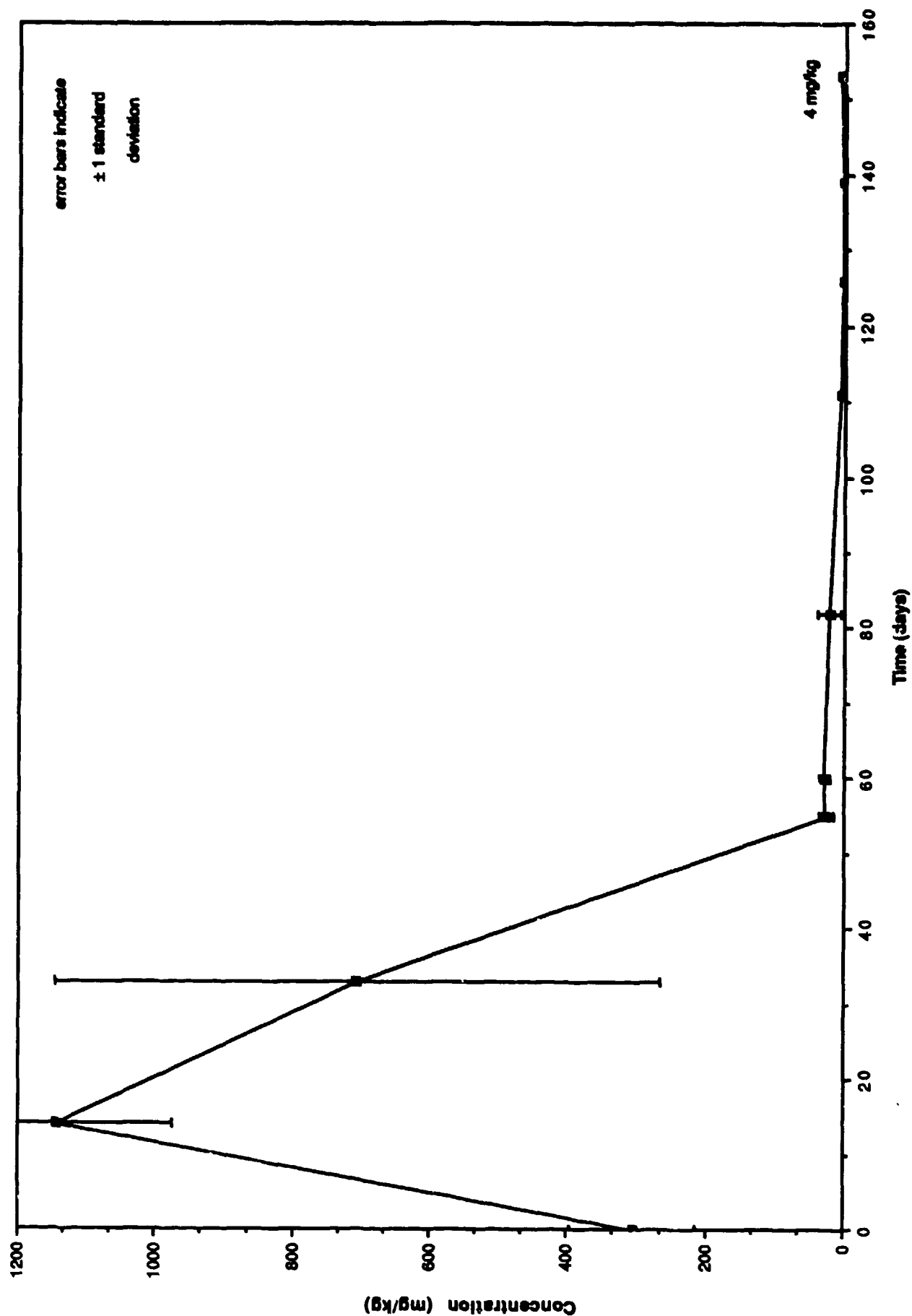


Figure 5-28. Concentration of 2-amino-4,6-dinitrotoluene in compost pile 4.

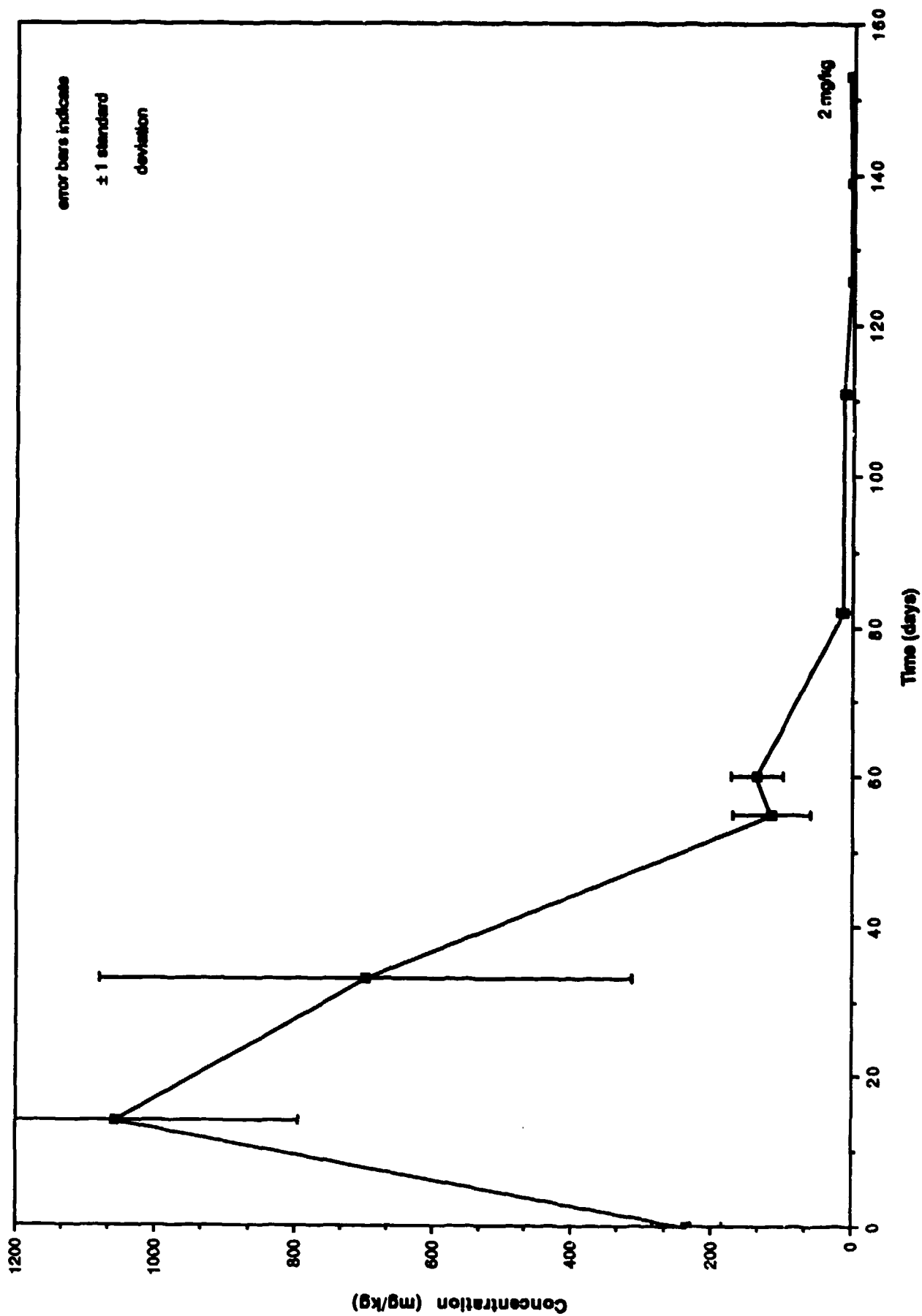


Figure 5-29. Concentration of 4-amino-2,6-nitrotoluene in compost pile 4.

Table 5-4

**Calculated Decay Constants (k) and Half-Lives
(t 1/2) for TNT, RDX, HMX, and Total Explosives
in LAAP Compost Piles 3 and 4**

Compound	Pile 3		Pile 4	
	k (per day)	t 1/2 (days)	k (per day)	t 1/2 (days)
TNT	0.032	21.9	0.058	11.9
RDX	0.023	30.1	0.040	17.3
HMX	0.016	42.0	0.030	22.8
Total explosives	0.026	26.6	0.043	16.2

5.2.5 Reactivity Testing

Compost residue samples from piles 3 and 4 were subjected to the following tests: thermal stability, detonation (no. 8 cap), spark, ABL impact, and U.S. Gap (see Appendix D). The following results were obtained:

- Thermal Stability -- No reaction was observed when the compost residue samples from piles 3 and 4 were placed in an oven and heated to 167°F for a period of 49.5 hours.
- Detonation (No. 8 Cap) Test -- No reaction was observed when the compost residue samples from piles 3 and 4 were subjected to the detonation (no. 8 cap) test.
- Spark Test -- No reaction was observed for the compost residue samples from piles 3 and 4 during the spark test.
- ABL Impact Test -- Three criteria (auditory response, visual response, and production of combustion products) were used. The threshold initiation level (TIL) for compost residue samples from pile 3 was determined to be 41 centimeters based upon production of combustion products (CO₂, CO, NO, NO₂, and N₂O). No visible or auditory reaction was observed, but the LIRA infrared analyzer detected the presence of combustion products at all test heights greater than 41 centimeters. The TIL is the maximum level of input stimulus that gives no response in 20 trials.

Compost residue from pile 4 showed no response of any type at the 120-centimeter drop height in 20 trials. The TIL value is therefore greater than 120 centimeters. The 120-centimeter drop height is the maximum input stimulus of which the ABL Impact Machine is capable.

- U.S. Gap Test -- Compost residue samples from piles 3 and 4 showed no indication of a reaction when subjected to the U.S. Gap test.

5.2.6 Nonquantitative Observations

The appearance of the compost changed considerably over the 153-day test period. When the compost was initially mixed, it had a highly fibrous appearance, a rough texture, and it smelled conspicuously of the manure, urine, and feed used to prepare it. After approximately 100 days the compost had become more soil-like and less fibrous in appearance. At the end of the test period, the compost had both the appearance and smell of loamy soil, suggesting that the bulking agents were highly degraded.

5.2.7 Concentrations of Metals in Sump Water and Compost

Samples of water from the sump were analyzed for metals at the terminal time point in the study. Potassium, magnesium, sodium, aluminum, iron, and calcium were the only metals detected at greater than 1 mg/L in the sump water (see Table 5-5).

No metals were detected in compost samples from piles 3 and 4 using the Toxicity Characteristic Leaching Procedure Test (EPA, 1979) (see Table 5-5).

5.3 DISCUSSION

5.3.1 Fate of Sediment Contaminants

The concentrations of solvent-extractable TNT, RDX, and HMX were significantly reduced during the 153-day test period of compost piles 3 and 4 (see Figures 5-16 to 5-23). Fate mechanisms that may have been responsible for contaminant reduction include: sorption of explosives and transformation products to the compost matrix; incorporation of explosives and transformation products into environmentally stable molecules; and mineralization of explosives to carbon dioxide, water, and other inorganics.

Previously published literature indicates that biotransformation of TNT, RDX, and HMX does occur. However, with the exception of RDX (Doyle et al., 1986), significant mineralization of these compounds has not been demonstrated (see Appendix C). Work by Kaplan and Kaplan (1983) indicated that incorporation

Table 5-5

**Mean Concentrations of Metals in Sump Water and
Compost from Piles 3 and 4 at the End of the Study**

Sump Water	Concentration (ug/L)	Detection Limit (ug/L)
Silver	ND	10.0
Aluminum	10,180	200.0
Arsenic	ND	10.0
Barium	331	200.0
Beryllium	ND	5.0
Calcium	41,200	5,000.0
Cadmium	ND	5.0
Cobalt	ND	50.0
Chromium	24.5	10.0
Copper	69.6	25.0
Iron	7,140	100.0
Mercury	0.86	0.20
Potassium	234,500	5,000.0
Magnesium	14,600	5,000.0
Manganese	634	15.0
Sodium	87,300	5,000.0
Nickel	82.0	40.0
Lead	ND	5.0
Antimony	47.6	40.0
Selenium	ND	5.0
Thallium	ND	10.0
Vanadium	ND	50.0
Zinc	589	20.0

Compost from Piles 3 and 4 - Toxicity Leaching Procedure

Silver	ND	500.0
Arsenic	ND	500.0
Barium	ND	1000.0
Cadmium	ND	100.0
Chromium	ND	500.0
Mercury	ND	0.20
Lead	ND	500.0
Selenium	ND	100.0

ND - Not detected

of chemically reactive transformation products into the compost matrix increased with increasing compost age and was the primary fate process involved in explosives composting. Thus, it appears that mineralization of explosives may have played a small role in the present study.

The production of organic metabolites from TNT, RDX, and HMX is well documented (Kaplan and Kaplan, 1982c; Won et al., 1974; Won and Heckley, 1974). In the present study, four known TNT metabolites (2-amino-4,6-dinitrotoluene; 4-amino-2,6-dinitrotoluene; 2,4-diamino-6-nitrotoluene; and 2,6-diamino-4-nitrotoluene) were detectable in initial compost samples, increased in concentration over the first several weeks of the test period, and decreased to low mg/kg levels thereafter. These data do not support one environmental fate process over another.

Previous research on explosives composting with ^{14}C -labeled test materials has demonstrated extensive (and apparently non-reversible) binding of ^{14}C activity to a compost matrix over time (Isbister et al., 1982; Doyle et al., 1986). In one study, less than 1 percent of the initial ^{14}C activity (spiked as ^{14}C -TNT into compost) was recovered as $^{14}\text{CO}_2$ after 6 weeks of composting, while 66 percent of the ^{14}C activity became bound to the compost matrix and was unextractable.

The total organic carbon (TOC) concentration of compost from piles 3 and 4 was approximately 20,000 to 30,000 mg/kg. The capacity for sorption of organic chemicals onto an environmental matrix is highly dependent on the TOC concentration of the matrix. Consequently, the compost used in the present study had a high capacity to adsorb organics. The bulking agents used to prepare the compost mixture (manure, hay, and horse feed) all consist primarily of plant products. As such, these materials contain proteins, polysaccharides, and other high molecular weight organic compounds. Given the high sorptive capacity of compost and the presence of high molecular weight organics in the matrix, sorption and incorporation of explosives into environmentally stable macromolecules may be a major fate process in explosives composting. This conclusion is supported by previous research (Isbister et al., 1982; Kaplan and Kaplan, 1983).

Calculated first-order decay rate constants in the present study were lower than those determined by Doyle et al. (1986). However, calculated half-lives for TNT, RDX, and HMX compare favorably with the results of Doyle et al. Data from these studies indicate that TNT degrades at the highest rate of the three compounds, followed by RDX and HMX, respectively.

Composting is a biologically mediated process. Microbial metabolism produces heat and is responsible for the self-heating compost environment. This warming is a characteristic of the composting process, and intensifies the rate of chemical and

biological reactions. Microbial metabolism drives the composting process, but chemical binding and stabilization of the explosives (probably catalyzed by or resulting from the biological reactions) may be the primary fate processes involved in reducing explosives concentrations. Radioisotopes and sophisticated analytical chemistry techniques are required to resolve this issue.

5.3.2 Temperature/Aeration Control and Monitoring

Both the rate and extent of contaminant degradation in compost piles 3 and 4 indicate that the thermophilic temperature range is superior to the mesophilic temperature range for explosives composting (see Figures C-16 to C-29 and Table 5-4). Whether increased rates of contaminant loss in the thermophilic pile are a result of the density and species of microorganisms associated with thermophilic temperatures and/or enhancement of the chemical binding/sorption process at elevated temperatures cannot be deduced from the present study.

If enhanced sorption results from higher compost temperatures, maintaining compost piles at temperatures exceeding the 55°C thermophilic optimum may be beneficial. However, above 60°C the microbes responsible for producing heat can be adversely affected. In addition, volatilization of certain contaminants or their breakdown products from compost may become significant at higher temperatures.

Periodic calibration checks of the temperature feedback control thermistors and the strip-chart recorder thermocouple probes indicated that the thermocouple probes gave more accurate and consistent temperature readings than the thermistors. As a result of these findings, control over the on/off cycling of the blowers was transferred from feedback control to timer control 2 weeks into the test period. The timed cycling of the blowers was manually set in response to compost temperatures as registered by the strip-chart recorder and the hand-held landfill probe thermometer.

Accurate maintenance of temperature in the compost piles was highly dependent on the skill and experience of the operator who adjusted the timers. The plots of mean compost temperatures during the test period support this conclusion (see Figures 5-2 to 5-11). Wide variations in compost temperatures were evident during the first part of the study as deviations in compost temperature were overcompensated for when the timers were adjusted. Compost temperatures stabilized considerably during the latter part of the study as the operator became more experienced.

Profiles of compost temperatures taken with the landfill probe and hand-held digital thermometer indicated that compost temperatures varied widely between different locations within the

piles. The top portions of both piles were generally warmer than the bottom portions (see Figures 5-5, 5-6, 5-10, and 5-11). Two nonmutually exclusive explanations may account for this observation. First, air would naturally pass through the area of least resistance when flowing from the outside, through the compost, and into the pipes. The path of least resistance in the present study would be the shortest possible distance through the compost matrix (i.e., at the base of the piles, see Figure 3-3). The cooling effect caused by increased air flow would result in lower compost temperatures in the base of the piles. Second, heat generated in the base of the piles would be expected to rise to the upper portion of the piles when the blowers were not operating.

5.3.3 Compost Moisture Content and Remixing

Periodic moistening of the compost was required to maintain an appropriate moisture content. The evaporative effect of pulling relatively dry ambient air through the compost piles was substantial. During periods following rewatering of the piles, large volumes of dense steam were expelled from the blowers.

Periodic remixing of the compost also facilitates the efficiency of the composting process. Remixing served to homogenize the compost and maximized contact of sediment contaminants with active microbial biomass. As compost temperatures were quite variable depending on location in the piles, remixing also ensured a more even exposure of a given volume of compost to warm areas in the compost pile.

5.3.4 Compost Reactivity

The compost residue from pile 4 was nonreactive in all five reactivity tests employed. The residue from pile 3 was completely nonreactive in four of the five tests. In the ABL impact test, the residue from pile 3 had a TIL of 41 centimeters for one of the three criteria used. For the other two criteria (visual and auditory response), the residue from pile 3 was nonreactive.

These data indicate that composting at thermophilic temperatures is superior to composting at mesophilic temperatures. In addition, these data demonstrate that composting can be used to transform reactive sediments into a nonreactive residue.

5.3.5 Microbial Populations

Several tentative trends are evident from the data on microbial populations in both the mesophilic and thermophilic compost piles. However, caution must be used in interpreting these data. Plate counts may have a great deal of variation among replicates. In addition, the degree of selectivity for growth on an agar medium may differ at 35°C and 55°C, as well as for mesophiles versus thermophiles.

The overall population densities in compost were higher than commonly observed in soils and appeared to be higher in the mesophilic pile. The data suggest enhancement of growth of mesophilic and thermophilic organisms in the mesophilic and thermophilic piles, respectively. The fact that the absolute population density of microorganisms was higher in the mesophilic pile while the rate and extent of contaminant degradation were higher in the thermophilic pile suggests that the density of microorganisms may not be the primary factor affecting the destruction of sediment contaminants.

5.3.6 Nonquantitative Observations

The change in the odor and appearance of the compost with time confirmed the role of a metabolically active microbial population in degrading and stabilizing the biodegradable substrates present in the initial mixture to be composted.

The volume of the compost piles was visibly reduced over the test period. Factors contributing to the volume loss included: compaction and settling of the compost matrix, water loss, and conversion of biodegradable organics to gaseous by-products.



SECTION 6

CONCLUSIONS AND RECOMMENDATIONS

The results of this field demonstration indicate that composting is a feasible technology for reducing the extractable explosives/intermediates concentrations in contaminated soils and sediments. This indicates that composting may be a suitable technology for remediating explosives-contaminated soil and sediment. The compost residue, however, must be acceptable for disposal in a manner which makes composting cost effective.

Extensive reductions in the concentrations of solvent-extractable TNT, RDX, and HMX were observed in both mesophilic and thermophilic compost piles. Calculated first-order decay rate constants and half-lives suggest that explosives degraded at a higher rate under thermophilic conditions. Percent reductions in the concentrations of individual and total explosives indicate that the extent of explosive degradation is greater under thermophilic conditions as well. Previous research suggests that a large portion of the observed reduction in contaminant concentration may be attributed to chemical sorption and/or binding of reactive biotransformation products as opposed to complete microbial mineralization of parent compounds. The acceptability of the compost residue for land disposal is a key concern. Additional chemical characterization of the residue as well as residue toxicity studies are recommended.

Temperature and contaminant reduction data from the present study indicate that the optimal soil loading rate for explosives composting lies between the levels used in piles 1 and 2 (79 weight percent) and piles 3 and 4 (25 weight percent). Successful composting will likely occur at sediment loading rates up to, or possibly exceeding, 50 weight percent (provided that parameters such as moisture and contaminant concentration are within favorable ranges). Maximizing the proportion of sediment or soil in mixtures to be composted will enhance the economic feasibility of the treatment process by minimizing bulking agent/carbon source usage, as well as treatment time for a given site. The quality of the organic carbon required to prepare the mixture to be composted and to facilitate degradation of the contaminants should also be investigated since this will directly affect costs.

The use of wood chips, sawdust, or other materials to form a base and insulating cover for compost piles should be discontinued. A portion of this material inevitably becomes incorporated into the compost during remixing. Thus, previously uncontaminated and generally nondegradable materials become contaminated and increase the volume of waste to be treated.

Concrete composting pads with aeration pipes located below grade would eliminate the need for wood chip bases and would also reduce short-circulating of air through the base material. A form-fitting cover of fiberglass or other suitable insulation would provide an inert, air-permeable, insulative blanket for compost piles. This type of cover could be used repeatedly.

As noted in Subsection 5.3, short-circuiting of air in the compost matrix almost certainly occurred during the present study.

Designing the shape of and supporting structure for a compost pile to force air to flow through the compost in one direction only would minimize short-circuiting. An enclosed vessel or bin with an aeration pipe below the bottom of the mixture to be composted and an open top would likely resolve this problem.

Triangular-shaped (cross-section) static piles are probably not ideal for a full-scale treatment process, although they served well to demonstrate proof of concept in the present study. Actual site remediation calls for a composting system capable of handling a relatively continuous influx of material to be decontaminated. A series of four or more vessels or bins as described previously would allow composts of increasing age to be in process and moved, mixed, and moistened periodically, as well as allow for semicontinuous loading of incoming wastes.

The compost piles constructed at LAAP tended to dry out rapidly, creating less than optimal conditions for microbial metabolism and contaminant destruction. Saturating all air entering the compost piles with water vapor may be a useful addition to the treatment process. Alternatively, liquid-phase water could be continuously or semicontinuously applied to compost piles, but it could cause leachate production and may not be evenly distributed. The compost mixture should be periodically mixed to achieve good particle size reduction and surface contact. Water can be added relatively easily at these remixing time points.

Developing a mixing system suitable for processing explosives-contaminated soils is a requirement. The system must achieve good homogeneity, handle materials with high bulk densities such as soils, and meet all safety criteria.

An improved, automated temperature control and monitoring system is required. An improved system would consist of at least six thermocouple probes placed in each compost pile and a microcomputer-based monitoring/control system to regulate the operation of the blower(s). At the basic level of operation, temperatures measured by the thermocouples would be averaged by the computer, and this average temperature would be used to control the cycling of the blowers. Enhancements could include

differential aeration to discrete areas within the pile in response to temperature and moisture conditions, as well as automatic recording and plotting of compost temperatures using a spreadsheet software program.

The final compost residue from the present study should be subjected to toxicological and additional analytical characterization to determine whether it may be disposed as a nonhazardous material. Important parameters in need of investigation include: mutagenicity of compost and leachate, toxicity of compost and leachate to aquatic and terrestrial biota, and biochemical oxygen demand and chemical composition of leachate. A complete analytical survey of the compost residue to determine the exact fate of explosives is desirable, but conclusive results may be difficult or impossible to obtain. Regulatory data requirements for desired residue disposal methods should be identified in specific regions of the country where implementation of composting is proposed.

In conclusion, the present study demonstrated extensive reduction of solvent-extractable explosives in compost after approximately 5 months of field-scale treatment. The exact fate of the explosive contaminants could not be determined, but previous research suggests that both microbial degradation and chemical sorption/binding were the major fate processes.

Chemical and toxicological analyses are needed to fully define the nature of the compost residue in order to identify residue disposal options. Refinements in the materials balance for the material to be composted, process control strategy, and materials handling will serve to further optimize process and cost efficiencies.



SECTION 7

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APPENDIX A

USATHAMA ANALYTICAL METHOD LW02

3833B

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 μ L, 50 μ L, 100 μ L, 1 mL syringe (Hamilton 1005 TEFL)
2. Vials with Teflon-lined caps or septa. Nominal volume of 1.8 mL, 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 μ L
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 Micromeritics 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 ODS 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.

44-55% water
28-34% methanol
16-22% acetonitrile

- 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:

	<u>Minutes</u>
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

	<u>Formula</u>	<u>Mol. Wt.</u>	<u>M.P. (°C)</u>	<u>B.P. (°C)</u>
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	5	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°C to -4°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°C to -4°C. The resulting solution will have the concentrations indicated in the following table.

<u>Nitro-compound</u>	<u>uL of Stock Cal Std</u>	<u>Resulting concentration (ug/mL)</u>
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:

<u>Nitro-Compound</u>	<u>Resulting conc. (ug/mL)</u>
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-INB 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.

E. 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

Resulting Concentration (ug/g)

Conc.	Amt. (uL) Intermed. Cal. Std. to Add		Amt. (uL) Aceto Nitrile to Add	Resulting Concentration (ug/g)				1,3-DNB 1,3,5-TNB 2,6-DNT 2,6-DNT NB
	A	B		HMX	2,4,6 TNT	Tetryl RDX		
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
X	4	-	1996	1.40	1.0	0.8		0.6
2 X	8	-	1992	2.80	2.0	1.6		1.2
X	20	-	1980	7.0	5.0	4.0		3.0
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
0 X	400	-	1600	140.0	100.0	80.0		60.0

I. 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.

II. PROCEDURE

A. Separations

1. Accurately weigh 1 gram of soil into a 5-mL serum vial and pipette 2 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 solution 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°-4° 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 %	H ₂ O %
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° 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 tetraol. 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.

I. CALCULATIONS

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

- B. Sample Concentration (ug/g) = 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:

$$\text{Sample Concentration (ug/g)} = \text{extract conc (ug/l)} \times 0.008$$

K. 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					2,4-DNT 2,6-DNT 1,3-DNB 1,3,5-TNB NB	
	Conc.	HMX	2,4,6-TNT	Tetryl RDX			
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)

- Percent recoveries for the 10X certification spikes from days 1 and 2 are averaged to obtain the first value to be plotted.
- Percent recoveries for the 10X certification spikes from days 3 and 4 are averaged to obtain the second value to be plotted.
- 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.
- Values from a, b, and c are averaged to determine the central line of the control chart.
- Differences in percent recoveries for each pair of values in a, b, and c are averaged to obtain R.
- The upper and lower warning limits are $\pm 1.25 R$ from the central line.

- g. The upper and lower control limits are $\pm 1.88 R$ 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 $\pm 0.682 MAR$, respectively.
- f. The upper and lower control limits are $\pm 1.023 MAR$, 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 B

RAW DATA

3833B

LAAP Data File - Piles 1 and 2

Temperature and Analytical Data

USATHAMA 2281-04-08

Temperature data in deg. C		
Test	Pile 1	Pile 2
Day	1	2
0	25	22
1	27	25
2	30	26
3	32	26
4	35	27
5	37	38
6	41	39
7	46	40
8	43	41
9	45	43
10	46	45
11	48	47
12	44	47
13	42	48
14	40	48
15	39	49
16	37	50
17	37	50
18	35	49
19	33	48
20	31	50
21	29	50
22	27	50
23	25	49
24	23	48
25	21	48
26	19	46
27	17	42
28	15	38
29	12	36
30	11	33
31	10	30
32	9	26
33	7	21

Analytical Data				Client		Concs.(mg/kg)			
Day	Date	batch	smpl	ID		HMXX	RDX	TNT	tetryl
0	12/10/87	8712-043	1	S-1		1130	8930	15500	88
0	12/10/87	8712-043	2	FM-1		936	7410	14800	44
0	12/10/87	8712-043	3	FM-2		692	5490	13300	34
0	12/10/87	8712-043	4	P2-1		222	1530	2630	4
0	12/10/87	8712-043	5	P2-2		289	2120	3310	6
33	1/13/88	8801-221	1	P1-1		255	1640	936	<16
33	1/13/88	8801-221	2	P1-2		202	1190	370	<16
33	1/13/88	8801-221	3	P1-3		370	2860	1920	<16
33	1/13/88	8801-221	4	P2-4		276	1680	645	<16
33	1/13/88	8801-221	5	P2-5		423	1970	440	<16
33	1/13/88	8801-221	6	P2-6		221	1470	322	<16
33	1/13/88	8801-221	7	B-1		1150	8360	16200	118
33	1/13/88	8801-221	8	B-6		921	6750	16100	18
33	1/13/88	8801-221	9	B-9		1300	10100	17000	21
33	1/13/88	8801-221	10	B-12		966	7060	13500	24
33	1/13/88	8801-221	11	B-17		1150	8700	17400	40
33	1/13/88	8801-221	12	B-18		1100	8270	11000	17
33	1/13/88	8801-221	13	B-20		1090	8240	11500	50
33	1/13/88	8801-221	14	B-21		972	7360	12300	36
33	1/13/88	8801-221	15	F-1		931	7030	10700	33
33	1/13/88	8801-221	16	F-2		1100	8000	12800	59
33	1/13/88	8801-221	17	F-3		866	6390	10700	72
33	1/13/88	8801-221	18	FC-1		1090	7940	13500	23
33	1/13/88	8801-221	19	FC-2		1100	8200	13400	168
33	1/13/88	8801-221	20	FC-3		930	6710	12200	<16

DATA SUMMARY-			HMX	
	day 0		day 33	
sample	mean	stdev	mean	stdev
pile1	256		276	70
pile2	256		307	85
bags1	814		1084	151
bags2	814		1078	65
fungus	814		966	99
f cntl	814		1040	78

DATA SUMMARY-			RDX	
	day 0		day 33	
sample	mean	stdev	mean	stdev
pile1	1825	295	1897	706
pile2	1825	295	1707	205
bags1	6450	960	8068	1320
bags2	6450	960	8143	487
fungus	6450	960	7140	662
f cntl	6450	960	7617	650

DATA SUMMARY-			TNT	
	day 0		day 33	
sample	mean	stdev	mean	stdev
pile1	2970		1075	640
pile2	2970		469	133
bags1	14050		15700	1317
bags2	14050		13050	2554
fungus	14050		11400	990
f cntl	14050		13033	591

LAAP pile 3 data

LAAP ANALYTICAL DATA LOG
Compost Pile #3 (mesophilic)

A= 2,4-diamino-6-nitrotoluene
 2,6-Diamino-4-nitrotoluene
 B= 2-amino-4,6-dinitrotoluene
 C= 4-amino-2,6-dinitrotoluene

RFW Batch #	Wk.	Sample ID	Concentration (ug/g)				Intermediates		
			HMX	RDX	TNT	tetryl	A	B	C
8803-616	0	P3-1	752	5310	14480	< 50	<10	225	201
		P3-2	652	4800	10800	< 50	<10	288	243
		P3-3	526	3780	8280	< 50	<10	217	210
		Mean	643	4630	11187	#####	#####	243	218
		Stdev	92	636	2546	#####	#####	32	18
8803-755	2	3-2wk-1	548	4240	5900	< 50	<10	1200	1470
		3-2wk-2	556	4640	3960	< 50	<10	1500	1680
		3-2wk-3	514	3840	2840	< 50	<10	1730	1540
		Mean	553	4240	4233	#####	#####	1477	1563
		Stdev	34	327	1264	#####	#####	217	87
8804-981	5	3-5wk-1	437	3153	1826	< 50	<10	583	457
		3-5wk-2	553	4430	3880	< 50	18	1310	973
		3-5wk-3	483	3460	2290	< 50	<10	605	716
		3-5wk-4	527	4060	2380	< 50	<10	950	918
		3-5wk-5	489	3890	3560	< 50	<10	595	519
		3-5wk-6	446	2800	624	< 50	122	55	237
		3-5wk-7	360	2520	950	< 50	<10	778	1030
		3-5wk-8	435	3000	2270	< 50	73	629	909
		3-5wk-9	887	6040	8590	< 50	79	671	1230
		Mean	466	3414	2223	#####	73	686	777
		Stdev	57	622	1055	#####	37	315	300
8804-177	8	3-8wk-1	228	735	548	< 50	<12	26	89
		3-8wk-2	696	4570	3010	< 50	<12	19	112
		3-8wk-3	321	1160	1030	< 50	<12	59	141
		Mean	415	2155	1529	#####	#####	35	114
		Stdev	202	1716	1065	#####	#####	17	21
8804-216	9	3-9wk-1	478	2790	2500	< 50	<12	157	299
		3-9wk-2	116	635	213	< 50	<12	23	66
		3-9wk-3	291	1460	262	< 50	25	27	155
		3-9wk-4	492	2460	1600	< 50	<12	53	163
		3-9wk-6	370	1630	409	< 50	96	333	767
		3-9wk-7	281	1540	518	< 50	<12	22	82
		3-9wk-8	404	2230	407	< 50	31	54	105
		3-9wk-9	422	1910	1700	< 50	27	48	244
		3-9wk-5	1130	6940	5570	< 50	<12	53	182
		Mean	357	1832	951	#####	45	86	229
		Stdev	116	630	805	#####	30	96	203
8805-427	12	3-12wk-1	137	444	345	< 50	<12	19	35
		3-12wk-2	97	230	405	< 50	<12	13	13
		3-12wk-3	158	609	120	< 50	<12	20	44
		3-12wk-1p	160	427	177	< 50	<12	16	34
		3-12wk-2p	160	433	1044	< 50	<12	13	30
		3-12wk-3p	115	594	89	< 50	<12	13	39
		mean	138	456	363	#####	#####	16	33
		stdev	24	126	325	#####	#####	3	10

LAAP pile 3 data

8806-724	16	3-16wk-6	75	380	151	< 50	< 1.2	16	39
		3-16wk-8					< 1.2	18	54
		3-16wk-10	109	418	110	< 50	< 1.2	17	45
		3-16wk-11	46	191	284	< 50	< 1.2	9	20
		3-16wk-12	62	202	52	< 50	< 1.2	18	34
		3-16wk-13	37	131	31	< 50	< 1.2	12	26
		3-16wk-22	92	300	155	< 50	< 1.2	19	48
		3-16wk-24	103	437	92	< 50	< 1.2	18	39
		3-16wk-25	81	324	106	< 50	< 1.2	20	47
		mean	76	298	123	#####	#####	16	39
		stdev	24	106	73	#####	#####	3	10
8806-890	18	3-18wk-1p	71	180	14	< 5	< 1.2	10	25
		3-18wk-2p	72	343	318	< 5	< 1.2	21	37
		3-18wk-1c	63	244	202	< 5	< 1.2	21	38
		3-18wk-2c	69	278	79	< 5	< 1.2	9	31
		3-18wk-3c	33	106	273	< 5	< 1.2	8	21
		mean	62	230	177	#####	#####	14	30
		stdev	15	81	115	#####	#####	6	7
8807-018	20	3-20wk-1c	135	599	430	< 10	< 1.2	66	111
		3-20wk-2p	95	199	12	< 10	< 1.2	10	19
		3-20wk-2c	42	109	200	< 10	< 1.2	13	21
		3-20wk-3p	104	399	13	< 10	< 1.2	7	16
		3-20wk-3c	69	181	103	< 10	< 1.2	12	18
		mean	89	297	153	#####	#####	22	37
		stdev	32	179	155	#####	#####	22	37
8807-140	22	TD-3-1	95	313	10	< 5	< 1.2	15	20
		TD-3-2	76	267	24	< 5	< 1.2	14	19
		TD-3-3	69	182	36	< 5	< 1.2	21	21
		TD-3-4	118	304	134	< 5	< 1.2	34	31
		TD-3-5	60	144	45	< 5	< 1.2	11	19
		mean	84	212	50	#####	#####	19	22
		stdev	21	67	44	#####	#####	8	5

LAAP pile 3 data

GRAPH DATA - TNT			
day	mean	mean+stdev	mean-stdev
0	11187	13733	8641
14	4273	5497	2969
33	2223	3277	1168
55	1529	2595	464
60	951	1756	147
82	363	689	38
111	123	196	50
126	177	292	62
139	153	308	-3
153	50	94	6

GRAPH DATA - diamino-NT's			
day	mean	mean+stdev	mean-stdev
0	#DIV/0!	#DIV/0!	#DIV/0!
14	#DIV/0!	#DIV/0!	#DIV/0!
33	73	110	36
55	#DIV/0!	#DIV/0!	#DIV/0!
60	45	74	15
82	#DIV/0!	#DIV/0!	#DIV/0!
111	#DIV/0!	#DIV/0!	#DIV/0!
126	#DIV/0!	#DIV/0!	#DIV/0!
139	#DIV/0!	#DIV/0!	#DIV/0!
153	#DIV/0!	#DIV/0!	#DIV/0!

GRAPH DATA - RDX			
day	mean	mean+stdev	mean-stdev
0	4630	5266	3994
14	4240	4567	3913
33	3414	4036	2792
55	2155	3871	439
60	1832	2462	1202
82	456	582	330
111	298	404	192
126	230	312	149
139	297	476	119
153	242	309	175

GRAPH DATA - 2-amino-4,6-DNT			
day	mean	mean+stdev	mean-stdev
0	243	275	212
14	1477	1694	1260
33	686	1002	371
55	35	52	17
60	86	181	-10
82	16	19	13
111	16	20	13
126	14	20	8
139	22	44	-1
153	19	27	11

GRAPH DATA - HMX			
day	mean	mean+stdev	mean-stdev
0	643	736	551
14	553	586	519
33	466	523	410
55	415	617	213
60	357	473	241
82	138	162	114
111	76	100	51
126	62	75	47
139	89	121	57
153	84	104	63

GRAPH DATA - 4-amino-2,6-DNT			
day	mean	mean+stdev	mean-stdev
0	218	236	200
14	1563	1651	1476
33	777	1076	477
55	114	135	93
60	229	432	26
82	33	42	23
111	39	49	29
126	30	37	24
139	37	74	0
153	22	27	17

LAAP pile 4 data

LAAP Analytical Data Log
Compost Pile #4 (thermophilic)

A= 2,4-diamino-6-nitrotoluene
2,6-Diamino-4-nitrotoluene
B= 2-amino-4,6-dinitrotoluene
C= 4-amino-2,6-dinitrotoluene

RFW Batch#	Wk.	Sample ID	Concentration (ug/g)				Intermediates		
			MPX	RDX	TNT	etryl	A	B	C
8803-616	0	P4-1	796	5780	12720	< 50	<10	225	177
		P3-2	766	5480	12080	< 50	<10	258	229
		P4-3	654	4620	10720	< 50	<10	431	297
		Mean	739	5293	11840	####	####	305	234
		Stdev	61	492	834	####	####	90	49
8803-755	2	4-2WK-1	522	4180	4440	< 50	<10	1060	1080
		4-2WK-2	716	5420	8280	< 50	<10	1377	1370
		4-2WK-3	612	5040	6660	< 50	<10	993	724
		Mean	617	4880	6460	####	####	1143	1058
		Stdev	79	519	1574	####	####	167	264
8804-981	5	4-5WK-1	510	3320	1770	< 50	<10	310	360
		4-5WK-2	575	4270	4530	< 50	<10	1050	665
		4-5WK-3	358	2550	3660	< 50	<10	580	427
		4-5WK-4	665	5240	4230	< 50	<10	1580	1230
		4-5WK-5	517	2860	1020	< 50	15	254	406
		4-5WK-6	566	4720	3200	< 50	<10	826	546
		4-5WK-7	373	2610	3020	< 50	<10	533	688
		4-5WK-8	849	6730	8650	< 50	<10	1070	1520
		4-5WK-9	299	1990	306	< 50	110	150	441
		Mean	524	3810	3376	####	63	706	698
8804-177	8	4-8WK-1	368	1330	239	< 50	<12	24	174
		4-8WK-2	51	305	316	< 50	<12	13	40
		4-8WK-3	194	518	290	< 50	<12	37	129
		Mean	204	718	282	####	####	25	114
		Stdev	130	442	32	####	####	10	56
8804-216	9	4-9WK-1	217	777	190	< 50	<12	31	142
		4-9WK-2	324	757	65	< 50	<12	36	117
		4-9WK-3	302	1020	183	< 50	<12	20	180
		4-9WK-4	401	1230	927	< 50	<12	35	91
		4-9WK-5	395	1720	226	< 50	<12	20	88
		4-9WK-6	400	1600	645	< 50	<12	26	141
		4-9WK-7	265	773	59	< 50	<12	20	121
		4-9WK-8	220	781	361	< 50	30	20	134
		4-9WK-9	295	1290	92	< 50	<12	41	210
		Mean	313	1105	305	####	30	28	136
8805-427	12	4-12wk-1	102	460	265	< 50	<12	59	39
		4-12wk-2	30	205	38	< 50	<12	13	5
		4-12wk-3	25	46	38	< 50	<12	13	5
		4-12wk-1p	25	22	72	< 50	<12	13	8
		4-12wk-2p	52	202	38	< 50	<12	13	9
		4-12wk-3p	25	178	38	< 50	<12	13	8
		mean	43	186	82	####	####	21	12
		stdev	28	143	83	####	####	17	12

LAAP pile 4 data

8806-724	16	4-16wk-1	34	88	3.1	< 50	< 1.2	5.4	12
		4-16wk-3	41	29	162	< 50	< 1.2	8.9	29
		4-16wk-5	6.4	36	153	< 50	< 1.2	2	4.6
		4-16wk-16	28	103	5.8	< 50	< 1.2	5	10
		4-16wk-17	35	90	16	< 50	< 1.2	6	11
		4-16wk-18	37	204	8.4	< 50	< 1.2	4.8	7.9
		4-16wk-26	28	78	16	< 50	< 1.2	6.7	9.4
		4-16wk-28							
		4-16wk-30	49	183	7	< 50	< 1.2	7	15
		mean	32	101	46	#####	#####	6	12
		stdev	12	59	64	#####	#####	2	7

8806-890	18	4-18wk-1p	<2.5	7	12		< 1.2	4.6	2.3
		4-18wk-2p	5	12	<3.8		< 1.2	0	3.1
		4-18wk-1c	5	13	27		< 1.2	9	4
		4-18wk-2c	7	14	<3.8		< 1.2	0	3.5
		4-18wk-3c	21	63	<3.8		< 1.2	2.2	4.2
		mean	10	22	20	#####	#####	3	3
		stdev	7	21	8	#####	#####	3	1

8807-018	20	4-20wk-1p	72	65	3	<10	<1.2	5	2
		4-20wk-2p	1	2	1	<10	<1.2	<1.3	3
		4-20wk-1c	2	13	2	<10	<1.2	9	4
		4-20wk-2c	<2.5	1	<3.8	<10	<1.2	<1.3	3
		4-20wk-3c	9	12	18	<10	<1.2	2	4
		mean	21	19	6	#####	#####	5	3
		stdev	30	24	7	#####	#####	3	1

8807-140	22	TD-4-1	10	19	6	<5	< 1.2	5	6
		TD-4-2	23	35	1	<5	< 1.2	3	4
		TD-4-3	20	37	3	<5	< 1.2	3	3
		TD-4-4	72	123	4	<5	< 1.2	7	8
		TD-4-5	7	9	2	<5	< 1.2	2	3
		mean	26	45	3	#####	#####	4	5
		stdev	24	41	2	#####	#####	2	2

LAAP pile 4 data

GRAPH DATA - TNY			
day	mean	mean+stdev	mean-stdev
0	11840	12674	11006
14	6460	8034	4886
33	3376	5678	1075
55	282	314	250
60	305	585	26
82	82	164	-1
111	46	111	-18
126	20	27	12
139	6	13	-1
153	3	5	1

GRAPH DATA - diamino-NT's			
day	mean	mean+stdev	mean-stdev
0	#DIV/0!	#DIV/0!	#DIV/0!
14	#DIV/0!	#DIV/0!	#DIV/0!
33	63	110	15
55	#DIV/0!	#DIV/0!	#DIV/0!
60	30	30	30
82	#DIV/0!	#DIV/0!	#DIV/0!
111	#DIV/0!	#DIV/0!	#DIV/0!
126	#DIV/0!	#DIV/0!	#DIV/0!
139	#DIV/0!	#DIV/0!	#DIV/0!
153	#DIV/0!	#DIV/0!	#DIV/0!

GRAPH DATA - RDX			
day	mean	mean+stdev	mean-stdev
0	5293	5785	4802
14	4880	5399	4361
33	3810	5267	2353
55	718	1159	276
60	1105	1459	752
82	186	328	43
111	101	160	43
126	22	43	1
139	19	42	-5
153	45	85	4

GRAPH DATA - 2-amino-4,6-DNT			
day	mean	mean+stdev	mean-stdev
0	305	395	214
14	1143	1311	976
33	706	1146	266
55	25	34	15
60	28	35	20
82	21	38	4
111	6	8	4
126	3	7	0
139	5	8	2
153	4	6	2

GRAPH DATA - HMX			
day	mean	mean+stdev	mean-stdev
0	739	800	678
14	617	696	537
33	524	684	363
55	204	334	75
60	313	382	244
82	43	71	15
111	32	44	21
126	10	16	3
139	21	51	-9
153	26	50	3

GRAPH DATA - 4-amino-2,6-DNT			
day	mean	mean+stdev	mean-stdev
0	234	283	185
14	1058	1322	794
33	698	1081	315
55	114	170	59
60	136	173	99
82	12	24	0
111	12	19	5
126	3	4	3
139	3	4	2
153	5	7	3

exponential decay regression analysis

LAAP Compost Pile 3

TNT

non-linear regression curve values

day	conc	$(\ln(C_t) - \ln(C_0))/t$	k	t 1/2
0	7394			
20	4000	-0.030718725	0.030718725	22.56432131
40	2089	-0.031599584	0.031599584	21.93532582
60	1107	-0.031650253	0.031650253	21.90020448
80	545	-0.032595479	0.032595479	21.26513232
100				
mean			0.031641011 /day	21.91624723 days

LAAP Compost Pile 4

TNT

non-linear regression curve values

day	conc	$(\ln(C_t) - \ln(C_0))/t$	k	t 1/2
0	11500			
20	3874	-0.054402974	0.054402974	12.74097969
40	945	-0.062472935	0.062472935	11.0951596
60				
80				
100				
mean			0.058437954 /day	11.91806964 days

exponential decay regression analysis

LAAP Compost Pile 3

RDX

non-linear regression curve values

day	conc	$\ln(C_t) - \ln(C_0) / t$	k	t 1/2
0	5561			
20	3509	-0.023150749	0.023150749	29.9475942
40	2231	-0.022833201	0.022833201	30.356987
60	1404	-0.022940877	0.022940877	30.2145018
80	877	-0.023087828	0.023087828	30.022191
100				
mean			0.02303164 /day	30.1335685 days

LAAP Compost Pile 4

RDX

non-linear regression curve values

day	conc	$\ln(C_t) - \ln(C_0) / t$	k	t 1/2
0	7725			
20	3439	-0.040464055	0.040464055	17.1299487
40	1579	-0.039691752	0.039691752	17.4632548
60	689	-0.040282931	0.040282931	17.2069701
80				
100				
mean			0.040146246 /day	17.2667245 days

exponential decay regression analysis

LAAP Compost File 3

HMX

non-linear regression curve values

day	conc	$\ln(Ct) - \ln(Co))/t$	k	t 1/2
0	728			
20	524	-0.016702917	0.016302917	42.5167595
40	376	-0.016449022	0.016449022	42.1391126
60	269	-0.016547311	0.016547311	41.8888119
80	191	-0.016690957	0.016690957	41.5283058
100	140	-0.016459076	0.016459076	42.1133716
mean			0.016489856 /day	42.0372723 days

LAAP Compost File 4

HMX

non-linear regression curve values

day	conc	$\ln(Ct) - \ln(Co))/t$	k	t 1/2
0	933			
20	512	-0.030004029	0.030004029	23.1018036
40	271	-0.030907159	0.030907159	22.4267513
60				
80				
100				
mean			0.030455594 /day	22.7642775 days

exponential decay regression analysis

LAAP Compost Pile 3

Total Explosives

non-linear regression curve values

day	conc	$(\ln(C_t) - \ln(C_0))/t$	k	t 1/2
0	14700			
20	8735	-0.026025478	0.026025478	26.6334088
40	5185	-0.026051941	0.026051941	26.6063543
60	3029	-0.026326916	0.026326916	26.3284609
80	1836	-0.026003228	0.026003228	26.656198
100	1194	-0.025105385	0.025105385	27.6095024
mean			0.025902589 /day	26.5561055 days

LAAP Compost Pile 4

Total Explosives

non-linear regression curve values

day	conc	$(\ln(C_t) - \ln(C_0))/t$	k	t 1/2
0	16500			
20	8000	-0.04191646	0.04191646	16.5363962
40	3388	-0.042438274	0.042438274	16.3330672
60	1368	-0.043407015	0.043407015	15.968552
80	586	-0.043152578	0.043152578	16.0627062
100				
mean			0.042728582 /day	16.2251804 days

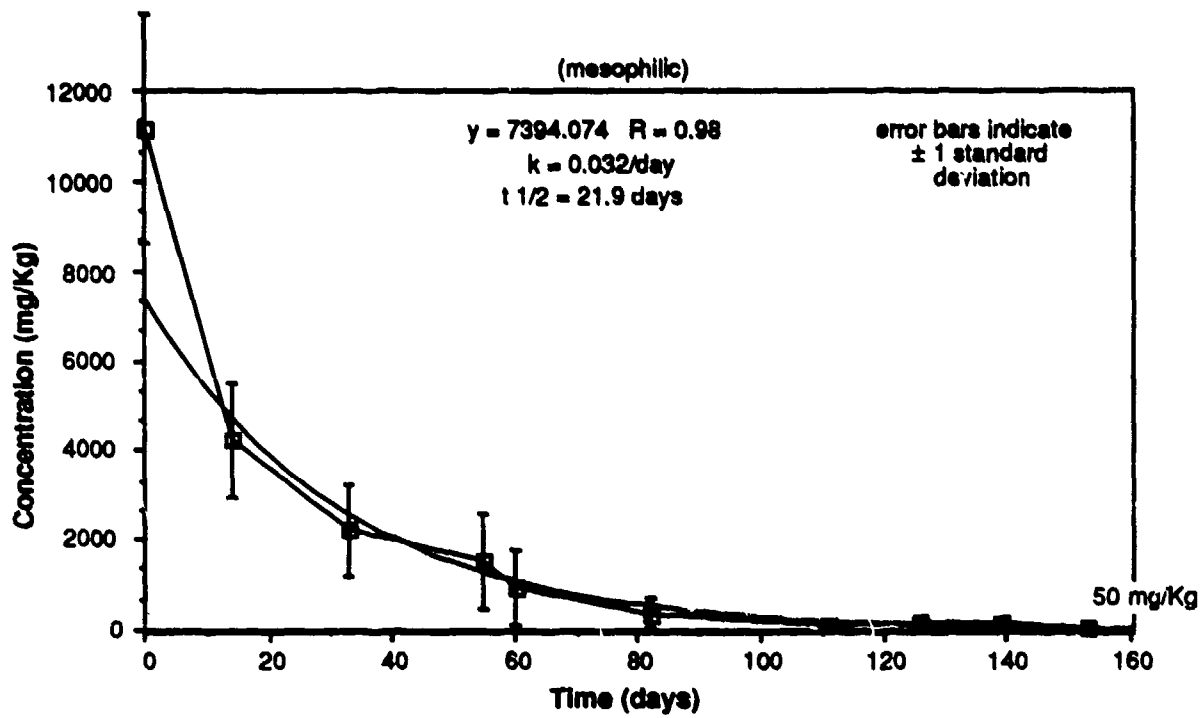


Figure C.16. Concentrations of TNT in LAAP Compost Pile 3.

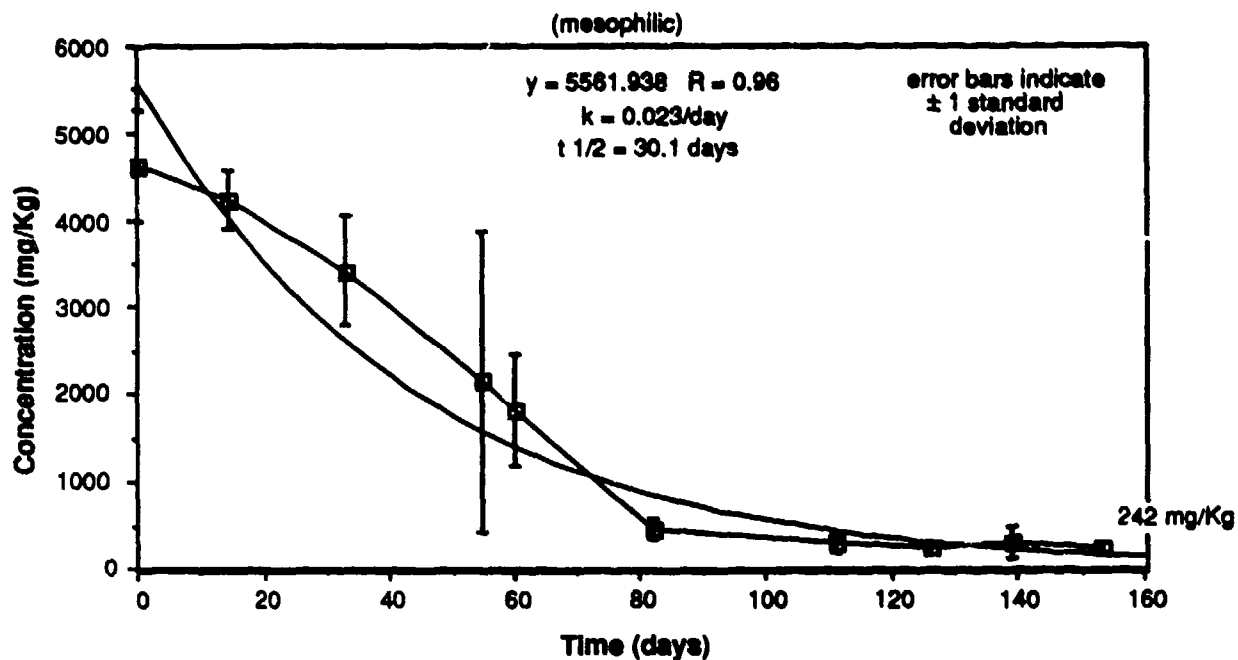


Figure C.17. Concentrations of RDX in LAAP Compost Pile 3.

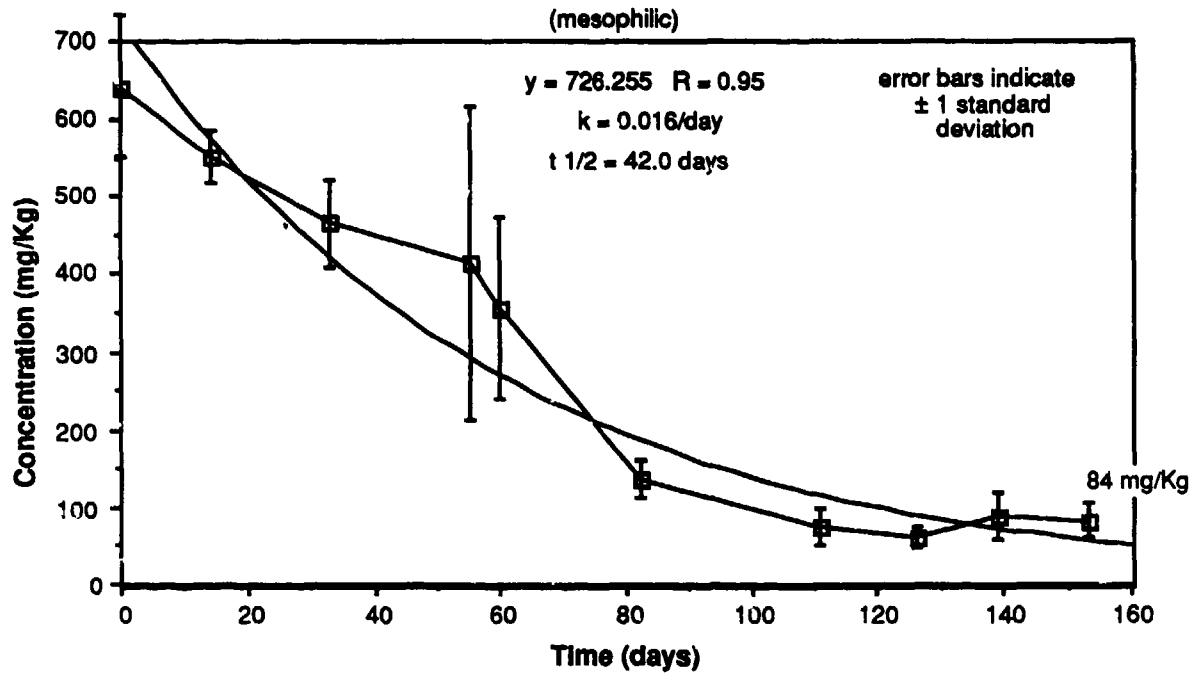


Figure C.18. Concentrations of HMX in LAAP Compost Pile 3.

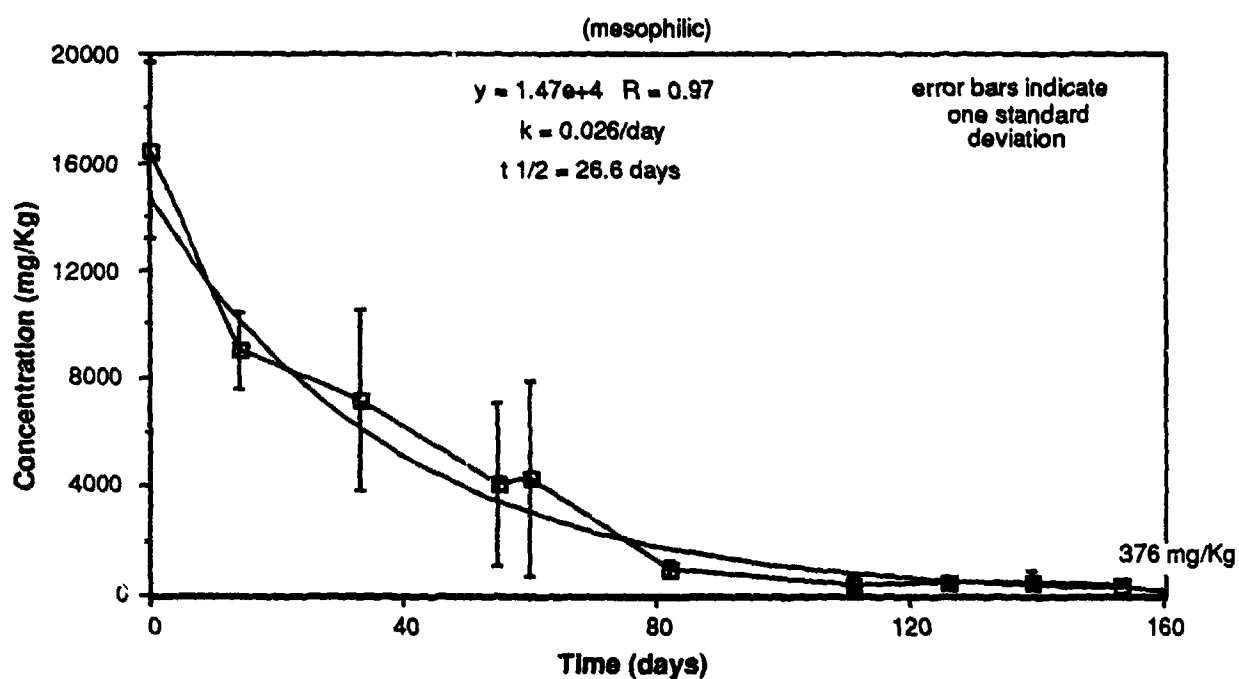


Figure C.19. Mean Concentrations of Total Explosives in LAAP Compost Pile 3.

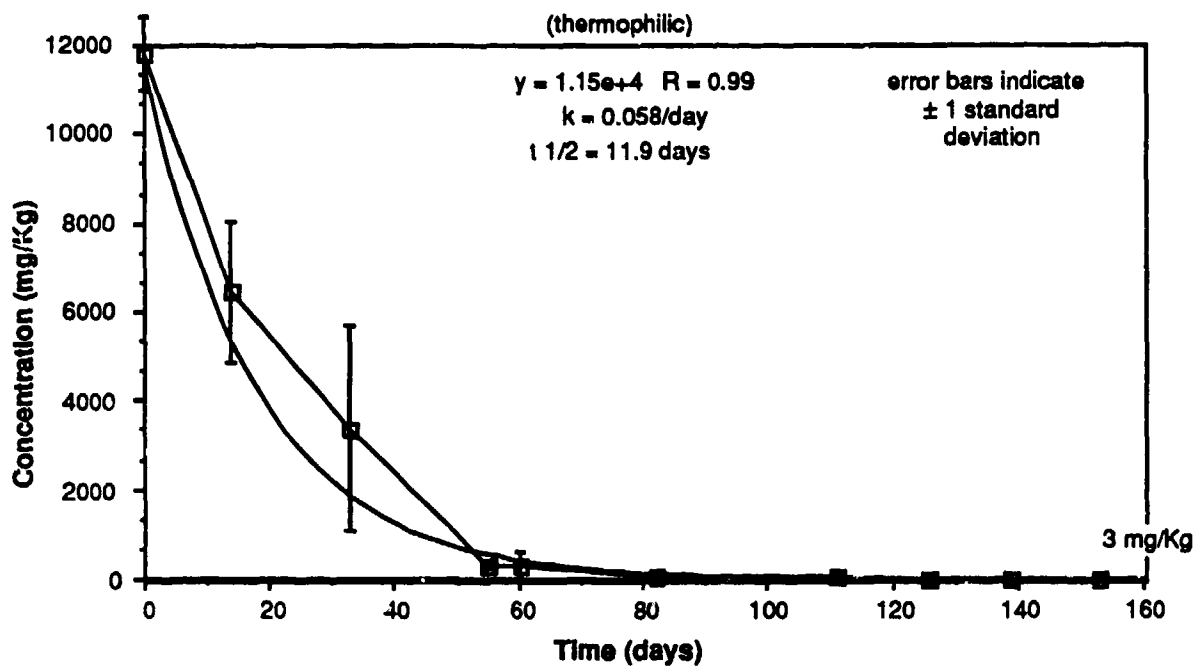


Figure C.20. Concentrations of TNT in LAAP Compost Pile 4.

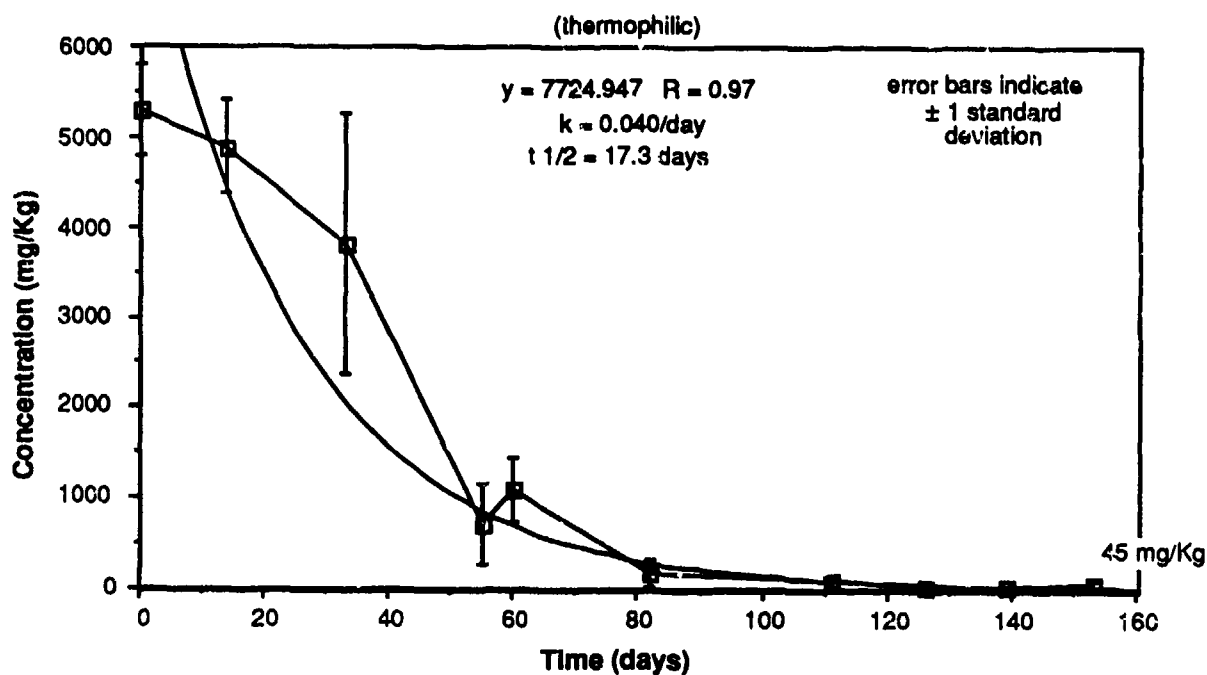


Figure C.21. Concentrations of RDX In LAAP Compost Pile 4.

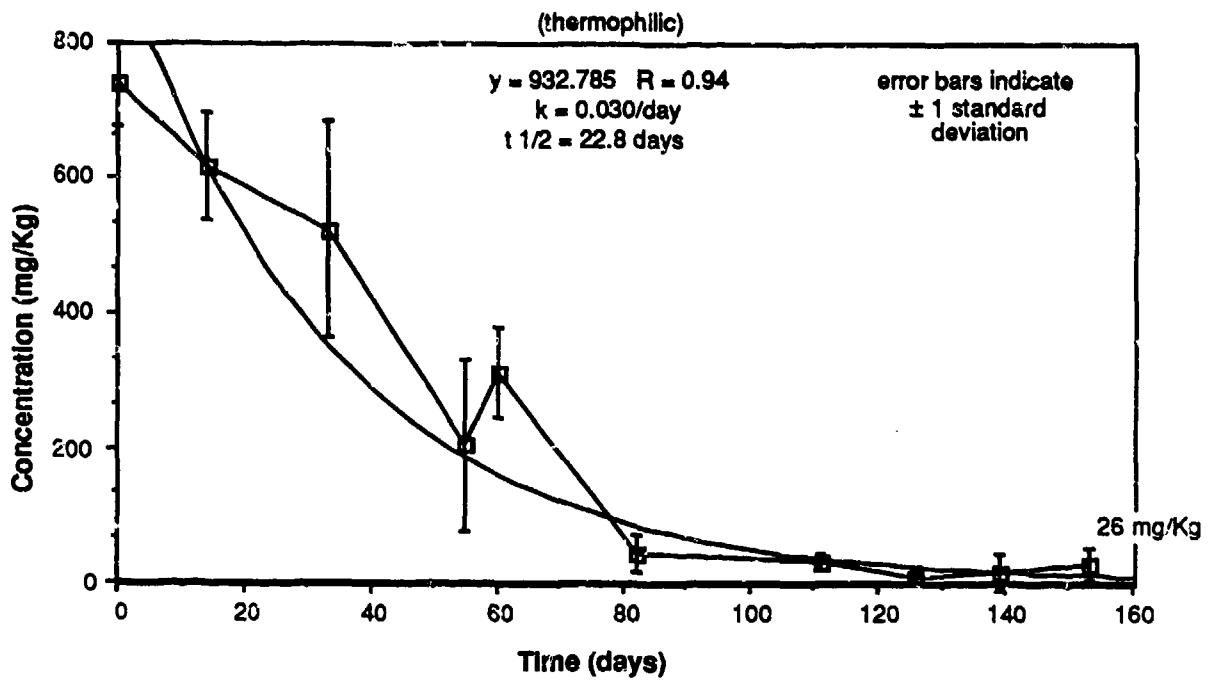


Figure C.22. Concentrations of HMX in LAAP Compost Pile 4.

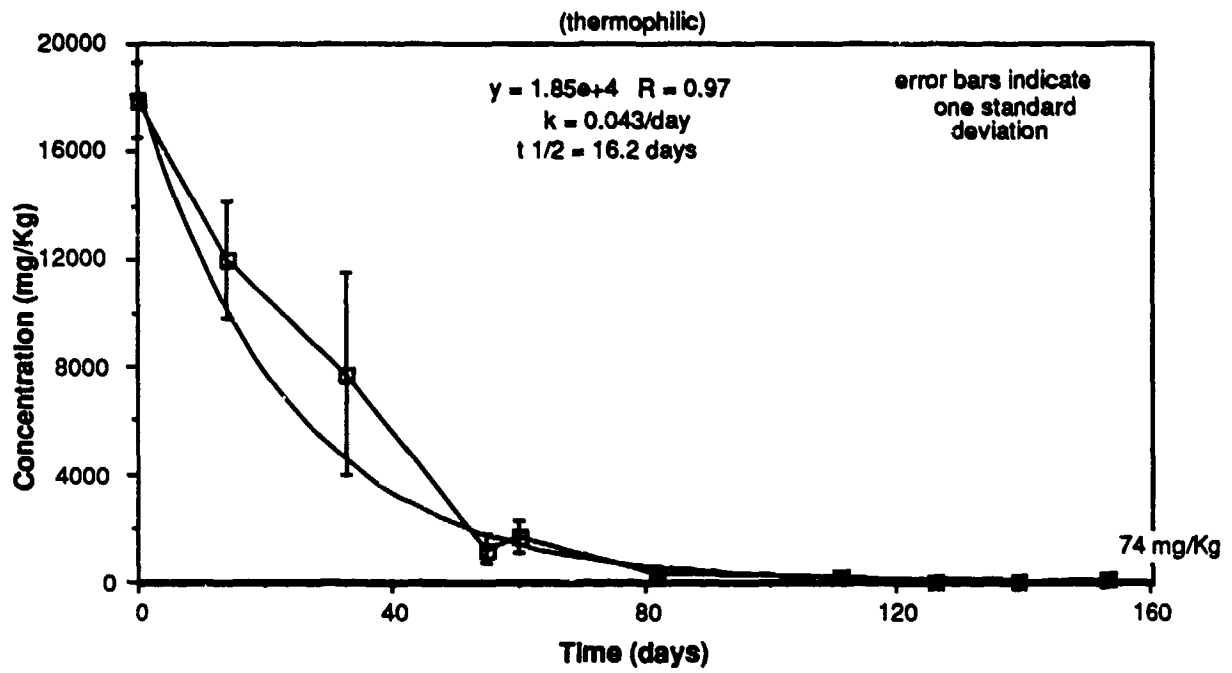


Figure C.23. Mean Concentrations of Total Explosives in LAAP Compost Pile 4.

LAAP pile 3 data

LAAP - Composting Temperature Data - File 3 (mesophilic)

all data in degrees C

		thermistor		probes		landfill probe data												lateral average			horizontal average			ambient	
day	date	high	low	#1	#2	toe top	toe mid	toe low	mid top	mid mid	mid low	heel top	heel mid	heel low	pile mean	toe	mid	heel	top	mid	low	low	mid	high	
0	2/25"																								
1	26	20	21			26	30	31	23	22	23	20	23	25	25	29	23	23				23	25	26	
2	27	24	26	24	23	31	29	32	28	30	33	31	32	32	31	31	30	32				30	30	32	
3	28	32	31	33	29	32	35	41	38	25	40	38	36	40	36	36	34	38				36	32	40	
4	29			42	32																				
5	"3/1"	47	36	45	35	48	48	47	42	46	57	43	43	53	47	48	48	46				44	46	52	
6	2	52	51	58	46																			9	
7	3	55	54	63	51																			15	
8	4	60	61	70	59																			11	
9	5	64	65	64	58		52			54			56		54	52	54	56				54		4	
10	6	55	59	45	49		47			40			48		45	47	40	48				45		4	
11	7	48	46	36	40	42	37	18	47	40	15	54	46	29	36	32	34	43				48	41	21	
12	8	45	40	31	35		37		45				46		43	37	45	46				45	42	7	
13	9	41	46	27	33	32		22	46		20	41.8		22.8	31	27	33	32				40		22	
14	10	42	46	29	23	21	23	15	45	39	23	45	35	26	30	20	36	35				37	32	21	
15	11	43	47	31	37	22	25	16	51	40	24	46	35	25	32	21	38	35				40	33	22	
16	12	47	52	32	27	72		22	54		23	49		26	41	47	39	38				58		24	
17	13	58	57	43	37	45		22	52		29	54		32	39	34	41	43				50		28	
18	14	58	61	48	47	42		17	51		21	58		30	37	30	36	44				50		23	
19	15	59	49	17	30	43		20	56		26	57		33	39	32	41	45				52		26	
20	16	67	67	8	25	48		11	59		38	62		54	45	30	49	58				56		34	
21	17					45		14	42		16	57		24	33	30	29	41				48		18	
22	18	53	53	22	33	33		14	42		21	50		39	33	24	32	45				42		25	
23	19	40	42	16	26	28		6	33		12	42		17	23	17	23	30				34		12	
24	20	41	39	0	11	31		11	40		17	41		27	28	21	29	34				37		18	
25	21	43	42	9	11	41		16	41		28	42		24	32	29	35	33				41		23	
26	22	46	42	18	26	47		20	48		41	46		40	40	34	45	43				47		34	
27	23	49	48	26	32	44		19	40		29	41		28	34	32	35	35				42		25	
28	24	50	47	26	31	42		26	46		39	48		40	40	34	43	44				45		35	
29	25	67	67	36	29	60		30	64		54	64		65	56	45	59	65				63		50	
30	26	54	53	37	39	51		20	49		23	51		31	38	36	36	41				50		25	
31	27	56	54	38	40	52		36	48		36	51		46	45	44	42	49				50		39	
32	28			33	37	27	36	39	28	40	46	29		35	35	32	34	38				36	41	28	
33	29	50	48	41	43	38	43	28	37	31	25	39	38	36	35	36	31	38				38	37	30	
34	30																								
35	31	22	26	17	13	27	23	25	26	23	21	21	21	27	24	25	23	23				25	22	24	
36	"4/1"	28	30	22	16	35	30	39	38	30	34	39	31	35	35	35	34	35				37	30	36	
37	2	61	58	49	46	59		65	48		56	43		51	54	62	52	47				50		57	
38	3	71	61	34	71	48		45	47		30	40		60	45	47	39	50				45		45	
39	4	75	71	34	66	38		56	48		63	39		54	50	47	56	47				42		58	
40	5	64	59	28	50	44		53	49		29	48		50	46	49	39	49				47		44	
41	6	51	39	38	52	41		44	44		46	46		55	46	43	45	51				44		48	
42	7	37	25	14	32	27		29	28		14	28		16	24	28	21	22				28		20	
43	8	32	20	20	30	29		26	35		24	31		27	29	28	30	29				32		26	
44	9	30	19	22	29	36		32	29		28	33		41	33	34	29	37				33		34	
45	10	30	16	16	26	28		30	41		23	39		20	30	29	32	30				36		24	
46	11	31	32	31	39	35		13	41		20	35		20	27	24	31	28				37		18	
47	12	41	37	21	44	28		21	47		37	53		24	35	25	42	39				43		27	
48	13	56	55	10	52	31		24	56		25	52		26	36	28	41	39				46		25	
49	14	61	58	9	54	31		34	52		36	52		32	40	33	44	42				45		34	
50	15	60	57	14	53	37		29	45		35	46		25	36	33	40	36				43		30	
51	16																								
52	17	58	54	23	48	51		31	40		48	39		38	41	41	44	39				43		39	
53	18	51	47	26	43	36		30	32		29	29		24	30	33	31	27				32		28	
54	19	51	47	43	50	36		29	30		21	25		21	27	33	26	23				30		24	
55	20	51	48	48	48	32	37	34	34	28	25	20	21	21	28	34	29	21				29		27	
56	21	51	48	56	53	44		29	28		22	22		20	28	37	25	21				31		24	
57	22	56	53	50	46	52		38	35		28	26		28	35	45	32	27				38		31	

LAAP pile 3 data

58	23																		
59	24	55	52	52	52	44	30	41	31	29	30	34	37	36	30	38	30	10	36
60	25																		
61	26																		
62	27	40	37	32	32	38	34	41	30	42	35	37	36	36	39	40	33	12	25
63	28	44	41	40	35	48	33	40	26	38	30	36	41	33	34	42	30	9	30
64	29	42	40	31	31	42	51	33	41	33	36	39	47	37	35	36	43	13	23
65	30	41	39	38	31	41	40	32	45	30	33	37	41	39	32	34	39	14	25
66	"5/1"																	8	34
67	2	42	39	33	30	42	39	31	36	28	34	35	41	34	31	34	36		
68	3	40	38	31	28	47	48	28	26	28	31	35	48	27	30	34	35	12	33
69	4	38	36	31	27	50	31	33	27	30	30	34	41	30	30	38	29	13	31
70	5	36	35	32	26	53	35	30	33	29	31	35	44	32	30	37	33	11	29
71	6	37	35	30	25	53	42	28	33	28	32	36	48	31	30	36	36	10	35
72	7																	13	35
73	8	37	35	33	28	48	40	30	30	30	32	35	44	30	31	36	34	15	24
74	9	38	35	24	23	46	37	32	34	32	35	36	42	33	34	37	35	20	32
75	10																	14	33
76	11	40	37	22	23	43	37	35	38	33	36	37	40	37	35	37	37		
77	12	37	38	26	23	54	38	32	32	31	34	37	46	32	33	39	35	18	31
78	13	34	31	24	20	38	35	31	33	30	33	33	37	32	32	33	34	14	31
79	14																		32
80	15	33	30	31	19	45	41	32	33	31	33	36	43	33	32	36	36	15	
81	16	39	36	39	34	47	41	39	37	39	35	40	44	38	37	42	38	17	35
82	17	46	42	56	52													16	35
83	18	55	53	46	52	52	42	56	48	57	46	50	47	52	52	55	45	15	34
84	19	61	58	54	61	56	41	62	51	62	51	54	49	57	57	60	48	16	35
85	20	68	63	57	69	59	43	68	53	66	58	58	51	61	62	64	51	18	33
86	21	34	35	35	27	38	31	27	21	28	32	30	35	24	30	31	28	18	22
87	22																		
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89	24	26	24	32	22	30	26	22	25	21	23	25	28	24	22	24	25	19	27
90	25	27	25	29	21	31	27	24	26	22	24	26	29	25	23	26	26	15	28
91	26	30	28	30	23	32	27	25	27	24	25	27	30	26	25	27	26	10	28
92	27	32	30	31	26	33	28	28	28	26	26	28	31	28	26	29	27	9	31
93	28																		
94	29	35	31	33	29		36	30	30	29	29	31	36	30	29	30	32	14	32
95	30																	16	32
96		39	35	46	40	41	32	34	31	33	31	34	37	33	32	36	31	17	34
97		43	38	51	46	46	32	38	33	37	33	37	39	36	35	40	33	18	35
98		36	32	40	34	42	34	28	27	27	27	31	38	28	27	32	29	19	34
99		36	31	52	37	42	33	30	30	29	30	32	38	30	30	34	31	18	29
100																		16	29
101	5	45	39	43	35	50	37	37	35	35	33	38	44	36	34	41	35	16	32
102	6	47	41	38	37	47	37	34	30	33	32	36	42	32	33	38	33	18	33
103	7																	17	36
104	8		44	50	44	49	35	33	30	33	32	35	42	32	33	38	32	17	37
105	9	40	37	36	31	42	36	30	29	29	31	33	39	30	30	34	32	22	34
106	10		38	44	37	42	33	32	32	32	31	34	38	32	32	35	32	17	33
107	11																	11	30
108	12	55	52	46	47	49	41	44	39	40	37	42	45	42	39	44	39	16	35
109	13	52	49	48	48	43	39	36	32	35	34	37	41	34	35	38	35	17	34
110	14	50	46	51	48	43	35	34	32	32	33	35	39	33	33	36	33	20	33
111	15																		
112	16																		
113	17	43	39	21	26	37	31	35	29	34	30	33	34	32	32	35	30	20	31
114	18	44	39	21	16	39	32	34	28	32	31	33	36	31	32	35	30	19	33
115	19																	19	36
116	20	47	44	52	43	46	37	37	34	37	34	38	42	36	36	40	35	19	36
117	21	46	42	39	33	46	36	33	32	31	31	35	41	33	31	37	33	19	37
118	22																	20	38
119	23	45	40	32	31	44	37	32	31	30	29	34	41	32	30	35	32	21	37
120	24	45	40	39	43	45	35	31	31	30	30	34	40	31	30	35	32	24	
121	25																		
122	26																		

LAAP pile 3 data

123	27	46	41	44	41	46	44	34	36	33	34	38	45	35	34	38	38	24	35
124	28	45	40	40	39	47	40	33	35	33	35	37	44	34	34	38	37	26	39
125	29																		
126	30																		
127	7/1"	44	37	42	39	42	37	31	34	31	34	35	40	33	33	35	35	25	38
128	2																		
129	3																		
130	4																		
131	5	41	40	46	42														36
132	6	43	41	53	47													22	32
133	7	45	43	47	43													22	
134	8	45	44	56	49	43	35	35	34	35	35	36	39	35	35	38	35		
135	9																		
136	10																		
137	11																		
138	12																		
139	13	40	38	49	44														
140	14																		
141	15	41	41	38	36	40	35	34	31	34	32	34	38	33	33	36	33	22	36
142	16																		
143	17																		
144	18	43		51	46	40	37	33	33	35	34	35	39	33	35	36	35	23	40
145	19																		
146	20																		
147	21																		
148	22																		
149	23																		
150	24																		
151	25																		
152	26																		
153	27			22	21	22	23	23	22	22	23	24	23	23	23	23	23		

LAAP pile 4 data

LAAP - Composting Temperature Data - Pile 4 (thermophilic)

All Data in Degrees C

		Thermistor		Probes		Landfill Probe Data												Lateral			Horizontal			Ambient	
day	date	high	low	1	2	toe top	toe mid	toe low	mid top	mid mid	mid low	heel top	heel mid	heel low	Pile mean	average toe	average mid	average heel	average top	average mid	average low	low	high		
0	"2/25"																								
1	26	26	23	22	21	26	22	20	27	21	20	28	21	23	23	23	23	24	27	21	21				
2	27	26	27	21	31	33	29	30	28	26	26	24	28	35	29	31	27	29	28	28	30				
3	28	31	33	26	38	39	37	41	37	32	33	32	30	37	35	39	34	33	36	33	37				
4	29																								
5	"3/1"	48	50	34	53	47	42	46	42	40	47	45	44	55	45	45	43	48	45	42	49				
6	2	55	59	3	61																				
7	3	61	65	37	66																				
8	4	69	73	42	70																				
9	5	77	83	49	53		61			58			66		62	61	58	66		62					
10	6	73	76	52	39		61			59			61		60	61	59	61		60					
11	7	70	70	52	32	55	62	34	68	63	38	67	64	48	55	50	56	60	63	63	40	3.5	22		
12	8						59			62			62									7	21		
13	9						54			58			57									10	19		
14	10	65	65	32	47	43	48	44	63	60	42	61	54	47	51	45	55	54	56	54	44	1.5	23		
15	11	66	68	44	31	43	45	41	61	58	44	61	56	52	51	43	54	56	55	53	46				
16	12	66	65	47	32	50		58	50	43	37		49	51	51	54	47	53	52		50				
17	13	68	55	43	39	50		43	57	43	60		52	51	51	47	50	56	56		46				
18	14	69	71	41	41	51		48	56	48	55		54	52	50	52	55	54		50	-2	18			
19	15	71	73	45	41	55		50	58	48	63		53	55	53	53	58	59		50	-4	14			
20	16	71	76	51	40	59		55	59	49	64		58	57	57	54	61	61		54	-1.5	21			
21	17					55		58	62	48	62		48	56	57	55	55	60		51					
22	18					50		32	56	32	58		39	45	41	44	49	55		34	4	22			
23	19	61	63	46	14	49		26	53	25	51		31	39	38	39	41	51		27	-1.5	24			
24	20	58	59	48	14	49		27	53	37	50		40	43	38	45	45	51		35	-2	25			
25	21	55	58	47	18	51		37	51	44	47		41	45	44	48	44	50		41	7	27			
26	22	56	59	44	27	50		42	51	45	47		45	47	46	48	46	49		44	7	27			
27	23	58	62	45	39	54		47	51	51	51		47	50	51	51	49	52		48	15	28			
28	24	62	65	47	52	58		56	56	52	55		51	55	57	54	53	56		53	11	23			
29	25	66	70	52	60	58		65	55	57	59		59	59	62	56	59	57		60	15	27			
30	26	71	75	54	62	51		20	49	23	51		31	38	36	36	41	50		25	11	31			
31	27	70	68	56	19	59		48	64	44	60		57	55	54	54	59	61		50	6	28			
32	28					44	50	28	56	55	27	52	51	29	44	41	46	44	51	52	28				
33	29	65	67	43	23	44	50	34	55	51	38	49	52	34	45	43	48	45	49	51	35				
34	30																								
35	31	32	25	29	20	24	26	31	25	31	30	24	23	28	27	27	29	25	24	27	30				
36	"4/1"	38	40	38	27	34	34	45	34	36	50	37	31	33	37	38	40	34	35	34	43				
37	2	64	67	50	59	62		59	59		54	58		49	57	61	57	54	60		54				
38	3	74	61	56	69	63		64	65	63	65		66	64	64	64	66	64		64	12.5	29			
39	4	74	77	54	70	55		59	62	58	62		66	60	57	60	64	60		61	12	33			
40	5	72	76	57	71	52		52	59	56	57		66	57	52	58	62	56		58	19	33			
41	6	72	75	54	69	59		47	56	28	49		55	49	53	42	52	55		43	11	30			
42	7	85	90	63	69	65		45	67	70	31	58	69	43	56	55	56	57	63		40	6	26		
43	8	69	73	56	69	56		43	53	56	51		68	55	50	55	60	53		56	9	31			
44	9	73	77	59	67	63		46	55	58	51		66	57	55	57	59	56		57	9	33			
45	10	73	78	54	69	60		36	58	27	60		39	47	48	43	50	59		34	13	31			
46	11	79	84	53	71	58		25	47	21	48		30	38	42	34	39	51		25	3	14.5			
47	12	92	98	64	69	74		49	71	39	68		54	59	62	55	61	71		47	6.5	21			
48	13	80	86	61	70	52		36	61	29	57		60	49	44	45	59	57		42	6.5	28			
49	14	74	79	59	64	54		50	64	35	58		54	53	52	50	56	59		46	10	33			
50	15	68	73	56	64	53		41	56	33	45		50	46	47	45	48	51		41	10	30			
51	16																								
52	17	104	112	76	73	79		66	80	58	81		74	73	73	69	78	80		66	11	32.5			
53	18	54	58	47	56	40		34	35	26	29		23	31	37	31	26	35		28	16.5	29			
54	19	40	43	33	47	29		32	26	28	26		24	28	31	27	25	27		28	10	27.5			
55	20	44	47	38	51	40	52	44	31	29	26	36	48	42	39	45	29	42	36		37	7	29		

LAAP pile 4 data

56	21	48	51	35	57	34	42	33	35	39	30	36	38	34	35	35	36	15	33
57	22	55	59	49	62	49	39	43	30	48	31	40	44	37	40	47	33	16	28
58	23																	10	36
59	24	71	75	54	64	66	42	63	33	67	40	52	54	48	54	65	38		
60	25																		
61	26																		
62	27	38	40	36	31	36	33	36	33	38	34	35	35	35	36	37	33	12	25
63	28	51	50	44	42	47	41	51	30	50	37	43	44	41	44	49	36	9	30
64	29	60	62	52	48	53	45	54	40	56	48	49	49	47	52	54	44	13	23
65	30	67	70	55	52	59	48	60	43	59	48	53	54	52	54	59	46	14	25
66	"5/1"																	8	34
67	2	76	81	63	58	64	56	62	52	63	48	58	60	57	56	63	52		
68	3	80	81	63	56	69	46	66	53	70	60	61	58	60	65	68	53	12	33
69	4	74	73	59	52	69	45	68	44	71	37	55	57	56	54	69	42	13	31
70	5	70	70	58	50	66	47	65	43	68	39	55	57	54	54	66	43	11	29
71	6	66	70	56	53	62	55	63	43	62	49	56	59	53	56	62	49	10	35
72	7																	13	35
73	8	66	70	54	54	59	48	60	49	59	53	55	54	55	56	59	50	15	24
74	9	73	77	62	55	60	48	63	49	62	54	56	54	56	58	62	50	20	32
75	10																	14	33
76	11	75	79	64	56	65	50	65	52	65	52	58	58	59	59	65	51		33
77	12	73	75	61	52	63	50	64	46	64	48	56	57	55	56	64	48	18	31
78	13	66	67	57	48	59	46	62	41	63	45	53	53	52	54	61	44	14	31
79	14																		32
80	15	65	67	58	51	58	48	61	43	61	46	53	53	52	54	60	46	15	
81	16	69	72	60	53	60	48	63	43	62	46	54	54	53	54	62	46	17	35
82	17	71	71	56	57	48	38	60	58	61	62	58	55	60	58	53	61	16	35
83	18	75	72	61	53	58	43	58	48	63	45	53	51	53	54	60	45	15	35
84	19	74	71	59	54	57	50	62	37	62	46	52	54	50	54	60	44	16	35
85	20	70	73	57	57	57	52	60	42	59	47	53	55	51	53	59	47	18	33
86	21	72	75	56	59	57	50	60	39	59	46	52	54	50	53	59	45	19	22
87	22																		
88	23	73	77	59	57	58	51	60	48	60	47	54	55	54	54	59	49	15	28
89	24	72	76	56	58	58	52	60	47	60	47	54	55	54	54	59	49	19	28
90	25	72	76	59	57	57	48	59	46	59	46	53	53	53	53	58	47	15	29
91	26	73	77	59	57	58	42	59	44	59	45	51	50	52	52	59	44	11	28
92	27	73	77	61	57	56	48	60	46	59	47	53	52	53	53	58	47	10	32
93	28																		
94	29	72	76	61	57	57	46	60	46	59	46	52	52	53	53	59	46	14	32
95	30																		
96	31	72	76	56	58	50	51	58	47	59	48	54	55	53	54	58	49	18	35
97	"6/1"	72	76	55	58	57	50	60	53	59	48	55	54	57	54	59	50	18	36
98	2	72	76	58	58	58	48	60	53	60	48	55	53	57	54	59	50	20	30
99	3	72	76	56	58	58	53	60	53	59	60	57	56	57	60	59	55	18	30
100	4																		
101	5	71	75	62	56	57	51	60	53	59	49	55	54	57	54	59	51	16	32
102	6	71	75	62	54	57	52	59	53	59	50	55	55	56	55	58	52	18	33
103	7																		
104	8	71	75	58	57	58	53	60	53	58	50	55	56	57	54	59	52	17	38
105	9	70	75	61	55	57	53	60	53	60	51	56	55	57	56	59	52	22	34
106	10	70	74	59	54	57	44	59	51	59	48	53	51	55	54	58	48	18	34
107	11																		
108	12	70	74	60	55	57	50	59	52	58	49	54	54	56	54	55	50	16	36
109	13	70	74	58	56	57	52	59	52	58	48	54	55	56	53	58	51	17	34
110	14	70	74	56	57	56	52	59	52	60	49	55	54	56	55	58	51	21	33
111	15																		
112	16																		
113	17	52	56	41	47	49	45	51	44	50	46	48	47	48	48	50	45	20	31
114	18	58	61	50	48	51	45	54	46	52	50	50	48	50	51	52	47	19	33
115	19																		
116	20	66	69	49	57	55	48	59	48	57	46	52	52	54	52	57	47	19	36
117	21	68	71	56	56	56	50	61	54	59	48	55	53	58	54	59	51	20	37
118	22																		
119	23	67		57	56	55	52	60	55	56	47	54	54	58	52	57	51	21	37

LAAP pile 4 data

120	24	67	56	56	51	47	60	47	57	45	51	49	54	51	56	46	24
121	25																
122	26																
123	27	65	51	57	49	42	53	50	55	53	50	46	52	54	52	48	24
124	28	47	51	54	48	48	56	50	54	51	51	48	53	53	53	50	26
125	29																
126	30	62	49	54	49	51	50	52	55	54	54	54	52	52	52	50	25
127	7/1"	63	49	53	52	47	55	50	52	50	51	50	53	51	53	49	38
128	2																
129	3																
130	4																
131	5	62	46	54	48	41	50	48	51	43	47	45	49	47	50	44	36
132	6	62	43	54	48	42	49	48	51	53	49	45	49	52	49	48	33
133	7	63	48	53	53	42	50	48	52	44	48	48	49	48	52	45	22
134	8	57	44	54	52	41	55	48	52	45	49	47	52	49	53	45	23
135	9																
136	10																
137	11																
138	12																
139	13	62	45	56	49	50	50	50	55	55	54	54	48	52	50	53	27
140	14																
141	15	61	50	51													36
142	16																
143	17																
144	18	60	45	53													40
145	19																
146	20																
147	21																
148	22																
149	23																
150	24																
151	25																
152	26																
153	27				48	51	49	45	54	50	51	52	48	50	49	50	

LAAP Pile 3 data

LAAP COMPOSTING PROJECT - Compost Moisture - Pile #3 (mesop

NOTE: "Week.001 indicates a post-mix sampling

DATE	TIME ELAPSED	% MOISTURE	MEAN % MOISTURE
Feb29	wk 0	46	49.0
		48	
		53	
Mar10	wk 2	44	43.3
		40	
		46	
Mar28	wk 5	25	28.7
		28	
		33	
Mar28	wk 5.001	56	52.3
		48	
		53	
Apr25	wk 9	32	34.7
		31	
		41	
May19	wk 12	42	38.0
		28	
		44	
Jun15	wk 16	29	33.3
		35	
		36	
Jun15	wk 16.001	43	47.0
		48	
		50	
Jun30	wk 18	30	34.0
		32	
		40	
Jul13	wk 20	41	39.7
		41	
		37	
Jul-27	wk 22	36	34.7
		34	
		34	

DATA FOR GRAPHS	
Time (days)	% Moisture
0	49.0
14	43.3
35	28.7
35.001	52.3
63	34.7
84	38.0
112	33.3
112.001	47.0
126	34.0
140	39.7
153	34.4

LAAP COMPOSTING PROJECT-compost moisture - File #4

NOTE: "Week.001 indicates a post-mix sampling

DATE	TIME ELAPSED	% MOISTURE	MEAN % MOISTURE
Feb29	wk 0	45	45.0
		42	
		48	
Mar10	wk 2	39	41.0
		41	
		43	
Mar28	wk 5	39	39.0
		44	
		34	
Mar28	wk 5.001	51	50.3
		48	
		52	
Apr25	wk 9	38	35.0
		41	
		26	
May19	wk 12	44	40.7
		42	
		36	
Jun15	wk 16	36	36.7
		30	
		44	
Jun15	wk 16.001	40	41.7
		42	
		43	
Jun30	wk 18	42	42.7
		44	
		42	
Jul13	wk 20	45	43.7
		43	
		43	
Jul-27	wk 22	35.9	36.0
		36.1	
		35.9	

DATA FOR GRAPHS	
Time (days)	% Moisture
0	45.0
14	41.0
35	39.0
35.001	50.3
63	35.0
84	40.7
112	36.7
112.001	41.7
126	42.7
140	43.7
153	36

APPENDIX C

LITERATURE REVIEW

3833B

LITERATURE REVIEW

This review summarizes previous work on the biodegradation of 2,4,6-trinitrotoluene (TNT), cyclotrimethylene trinitramine (RDX), cyclotetramethylene tetranitramine (HMX), and n-methyl-N,2,4,6-tetranitroaniline (tetryl). Work on the composting of explosives is included. The literature review is not exhaustive. Opinions and conclusions discussed in the text are the interpretations of the original investigators. WESTON has, however, included statements that either clarify specific points or present an overall sense of perspective regarding the individual studies.

Trinitrotoluene (TNT)

Trinitrotoluene is the primary military explosive. It is synthesized in a continuous process where three nitro groups are attached to the aromatic ring of toluene. The symmetrical alpha form (2,4,6-TNT) is the form sought for munitions use. Of the four explosives being investigated in the present study, TNT is the most studied.

Initial reports of microbial degradation of TNT were made by Rogovskaya (Spanggard et al., 1980a), who observed the biodegradation of TNT present at 5 mg/L. Chambers et al. (1963) adapted microbial communities from soil, compost, and mud to utilize phenol. Using respirometry and these adapted organisms, they observed a slow but significantly higher oxygen uptake rate with 2,4,6-TNT present than in control systems, thereby demonstrating TNT oxidation. Enzinger (1970) acclimated sewage treatment plant microbes to 2,4,6-TNT. When cultured in a nutrient broth (trypticase soy), these microbes decreased the TNT concentration from 100 to 1.25 ppm in 5 days. The cell biomass contained only trace amounts of 2,4,6-TNT, indicating sorption of TNT was not a major factor in this disappearance.

Using a two-stage model wastewater treatment reactor, Bringmann and Kuehn (Spanggard et al., 1980a) observed that 99.6 percent of a 128-mg/L 2,4,6-TNT concentration was transformed after passage through the second stage. The first stage was aerated and contained an Azotobacter species, while the second stage contained a conventional activated sludge microflora.

Strong evidence that TNT transformation is a co-metabolic process was provided by Osmon and Klausmeier (1972). They observed that flasks containing 100 ppm TNT, mineral salts, and yeast extract lost greater than 99 percent of the TNT initially added after 6 days when inoculated with either sewage effluent, soil, or pond water. On the other hand, no TNT disappearance was observed in flasks when the yeast extract was not included. Nay et al. (1974) observed that TNT waste could be biologically treated in combination with domestic waste.

Won et al. (1974) and Won and Heckley (1974) provided the first evidence that microbes could use TNT as a sole carbon source by demonstrating that three pseudomonad-like organisms could oxidize TNT. Sediment and aquatic TNT enrichment cultures were able to degrade TNT in a basal salts medium, but required the addition of glucose or nitrogenous substances (yeast extract) for accelerated transformation. In a medium supplemented with a 0.5-percent yeast extract, 80 mg/L of TNT was completely degraded.

Similar results were reported by Traxler et al. (1974). They isolated a number of gram-negative bacteria from various sources capable of using TNT as a sole source of carbon and nitrogen. They also determined that yeast extract (100 ug/mL) stimulated TNT utilization. They found that 62 percent of the initial TNT was removed from a yeast extract-supplemented medium in 20 hours. Nitrate was detected in the medium, indicating that nitro groups were removed from the ring. When ^{14}C -TNT studies were performed, they detected the incorporation of TNT into the cellular material of two isolates. They also detected $^{14}\text{CO}_2$ and concluded that ring cleavage had occurred. The amount of ^{14}C detected as $^{14}\text{CO}_2$, however, represented a very low percentage of the ^{14}C initially added as ^{14}C -TNT (0.3 to 1.2 percent). The authors conducted studies that led them to believe that fixation of $^{14}\text{CO}_2$ by cells metabolizing TNT accounted for this low amount of free $^{14}\text{CO}_2$. In addition, Traxler (1975) found that washed cells effectively removed TNT from media with or without organic nutrients. After 18 hours, 94 percent of the TNT was removed by a cell mass of 14 mg/mL. At lower cell concentrations, removal was poor.

Weitzel et al. (1975) reported that sediment bacteria transformed TNT in a medium not supplemented with organic materials. A TNT concentration of 10 mg/L was reduced from 3.3 to 0.6 mg/L after 5 days. When glucose, peptone, and fatty acids were added, a 10 mg/L initial TNT concentration was reduced to 0.1 mg/L in 5 days.

Naumora et al. (1979) isolated a Pseudomonas denitrificans strain from soil contaminated with industrial waste that transformed TNT concentrations as high as 200 mg/L to reduced metabolites within 4 days.

McCormick et al. (1976) showed that the nitro groups on TNT were reduced by both aerobic and anaerobic microbial enzyme systems. Depending on the reducing potential of the system, one, two, or three of the nitro groups were reduced to amino groups. They did not, however, find any evidence for biological cleavage of the ring and subsequent degradation of the aromatic TNT nucleus. The biological reduction of nitroaromatic compounds was believed to represent only a superficial modification of the molecule.

Jerger et al. (1976) conducted laboratory biodegradation studies with benthic microorganisms. They found that transformation of TNT was a fairly rapid process, with acclimated and unacclimated populations transforming 70 to 90 percent of 10 ppm and 100 ppm TNT concentrations in 3 to 5 days. Anaerobic degradation also occurred, with these microbes tolerating greater than 200 ppm TNT after acclimation. An added carbon source appeared to be a prerequisite for microbial TNT transformation.

Parrish (1977) screened 190 fungi (from 98 genera) and reported that 183 were able to transform TNT in a basal salts medium containing glucose and 100 mg/L TNT. No evidence was found for ring cleavage using ^{14}C -TNT, and only reduction of the nitro group at the 4 position was observed.

Carpenter et al. (1978) investigated the fate of ^{14}C -labeled 2,4,6-TNT in an activated sludge system. No significant $^{14}\text{CO}_2$ was formed despite the absence of ^{14}C -TNT in the aerated reactor after 3 to 5 days. The radioactivity was found as ^{14}C -labeled transformation products that were evenly divided between the floc and supernatant. Evidence indicated that TNT transformation products formed polymeric conjugates with lipids, fatty acids, and proteins. These conjugates were resistant to further biodegradation. No evidence was found to indicate that the TNT aromatic nucleus was cleaved or ultimately degraded.

Hoffsommer et al. (1978) studied the biogradability of aqueous TNT solutions in a 3-year investigation of a 3,000-gallon pilot-scale oxidation ditch treatment system. The most efficient transformation occurred with activated sludge microbes and supplemental nutrients. TNT, at a concentration between 10 and 50 ppm, was 97 percent biotransformed when continuously fed into the aerated ditch lagoon supplemented with cornsteep water. No evidence for aromatic ring cleavage was found in a ^{14}C -TNT support study. Less than 0.4 percent of the added ^{14}C -TNT was converted to $^{14}\text{CO}_2$, with the remainder being distributed between the solid (bacterial floc) and aqueous phases.

Osmon and Andrews (1978) investigated the feasibility of disposing solid TNT using land application and composting systems. These studies were done on a laboratory scale. In soil, microorganisms biotransformed TNT to partial decomposition products. Manipulation of soil moisture, nutrient concentration and type, and the degree of aeration did not facilitate complete degradation. Soil application was concluded not to be suitable for disposal of large quantities of solid TNT. In compost, biotransformation also occurred, but the conversion products formed in the soil system could not be located in solvent extracts or in trapped effluent gases. Only "traces" (not quantified) of TNT were detected in the compost residue. This was

considered presumptive evidence for either ring cleavage and complete degradation or incorporation of the TNT products into biomass. No definitive ^{14}C -TNT experiments were performed.

Hale et al. (1979) monitored the downward movement of TNT and its transformation products through soil (coarse-, medium-, and fine-texture) columns. They found very little movement of these compounds over 6 months, but suggested the need for more precise and longer term experiments. They suggested that their findings were due to either soil fixation or limited water solubility. They also performed ^{14}C experiments, but the quantity of ^{14}C in each compartment at the end of the experiment was too low to quantify, and, therefore, no meaningful results were obtained.

Spanggord et al. (1980b) studied the major transformation and transport processes affecting the environmental fate of TNT in aquatic systems. A variety of microbes possessed a nitroreductase enzyme system enabling them to reduce the nitro group of TNT to an amino group in rich media, but not when TNT was the sole carbon and energy source. Results indicated only very slow TNT transformation under either aerobic or anaerobic conditions without additional nutrients such as yeast extract or nutrient broth. Rapid transformation (half-lives of several hours) occurred only with additional nutrients and greater than 10^9 microbial cells/mL. ^{14}C -TNT studies verified that the aromatic nucleus was not cleaved. Biotransformation of TNT in natural waters was found to be 1,000-fold slower than phototransformation. Burlinson (1980) also found that the biotransformation of TNT was significantly less than phototransformation in natural water. A 20-mg/L TNT concentration required greater than 30 days for 90-percent biological removal.

Klausmeier et al. (1982) examined the composting of solid TNT on a laboratory scale. During a 74-day composting cycle, the TNT concentration decreased from 1 percent to 0.002 percent. The atmosphere surrounding the TNT compost contained less than 0.66 ng of nitro-bodies per liter.

Kaplan and Kaplan (1982a) emphasized that the transformation pathways of TNT in ambient environments are the same as in thermophilic biotransformation of ^{14}C -TNT under composting conditions (55°C). TNT was found to be transformed under thermophilic conditions, but no $^{14}\text{CO}_2$ was produced. The transformation products appeared to bind to the organic matter fraction, with a progressively greater percentage of the ^{14}C being bound or conjugated to the organic matter fractions with increasing compost age and stability.

Kaplan and Kaplan (1982c) indicated that the composting work of Ocman and Andrews (1978), which suggested TNT composting may be feasible, was terminated before a definitive determination of the fate of the TNT substrate and transformation products could

be made. In addition, they noted the inability of Osmon and Andrews to trace TNT transformations by chemical methods.

To clarify the binding potential between TNT/TNT reduction products and soil components (such as humic acids), Kaplan and Kaplan (1983) conducted additional research. They found that unreactive arylnitro groups became more reactive once microbially reduced to the corresponding arylamine. In addition, they found that some microbial transformation products (2,4DA and 2,6DA; see Table C-1) were active binding species. A significant proportion of the ^{14}C -TNT in compost in their test systems was bound and not extractable with polar or nonpolar solvents. They stressed that these results, which indicate the potential for soil binding, must be taken into consideration prior to interpreting the results of TNT biodegradation studies.

Isbister et al. (1982) studied the degradation of ^{14}C -TNT in laboratory-scale (50-gram dry weight) compost systems incubated at 55°C . The initial concentration of TNT was 1.0 percent, and the compost was continuously aerated. Solvent extracts were analyzed after 3 and 6 weeks of composting. The TNT concentration was decreased by 50 percent after 3 weeks and by 82.6 percent after 6 weeks. The decrease in extractable TNT was paralleled by a decrease in solvent-extractable ^{14}C and by an increase in the residual (nonextractable) ^{14}C activity from 6.1 percent at 0 weeks to 66.5 percent at 6 weeks. No TNT reduction products were found after 3 weeks, but one of the three replicate composts did contain approximately 1 percent of the ^{14}C activity as 4-amino-2,6-dinitrotoluene and 2-amino-4,6-dinitrotoluene after 6 weeks. In all test systems, complete destruction of TNT by cleavage of the aromatic ring did not appear to occur. Recovery of ^{14}C as $^{14}\text{CO}_2$ was less than 1.0 percent.

Isbister et al. (1982) also conducted greenhouse-scale (10-kg dry weight) composting studies. TNT (2 percent of initial concentration in the mixture to be composted) was mixed with hay, horse feed, and seed compost. Additional hay, horse feed, and moisture were added during the 6-week test period. The temperature within the compost varied, but was generally above 55°C and reached greater than 70°C frequently. TNT concentrations were below the detection limit at both 3 and 4 weeks.

Spanggard et al. (1983) examined the persistence of TNT in waste disposal lagoons at the Louisiana Army Ammunition Plant. The half-life of TNT resulting from biotransformation was calculated to be 69 days. In experiments using lagoon water, the rate constant for TNT biotransformation at 50 ppm TNT was only one-third of that observed at 4.2 ppm. Kaplan et al. (1984) also studied the influence of TNT concentration, as well as other environmental factors, on biotransformation of TNT. TNT

transformation in soil proceeded at the highest rate at 0.1 percent TNT and was progressively lower at 1.0 percent and 10.0 percent. Their results indicated that high concentrations of TNT, low microbial populations, lower temperatures, and dry soil lengthened the period of time when TNT would remain unchanged.

Bell et al. (1984) studied munitions wastewater treatment in semicontinuous activated sludge treatment systems for 30 months. No significant removal of TNT occurred under anoxic conditions at concentrations below 5 mg/L. The rate of TNT removal was a function of biodegradable COD, with the rate of removal being reduced to an extremely low level or zero when the COD was exhausted. When COD remained available, TNT removal was complete, and TNT metabolites were not identified.

Greene et al. (1985) added TNT (70 mg/L) to soil columns in a pink water mixture also containing RDX (30 mg/L), HMX (4 mg/L), and 2,4-DNT (0.01 mg/L). The soil columns were run continuously for 140 days at a loading rate of either 40 or 110 mL/day. Almost all of the TNT fed into the columns either remained in the soil or was biotransformed. Microbially produced intermediates were found in the leachate, and supplemental carbon did not appear to be a requirement for transformation of TNT. Based on these results, land application of TNT was believed to pose a threat to groundwaters and soils because of intermediate and parent compound toxicity.

Doyle et al. (1986) conducted laboratory-scale composting studies using ^{14}C -labeled TNT, HMX, RDX, tetryl, and nitrocellulose. They also conducted pilot-scale (approximately 500 pounds) composting using the same explosives. Both studies used contaminated soil from a U.S. Army ammunition plant as a test matrix.

In the laboratory experiments, hay/horse feed and sewage sludge/wood shavings composts were tested with soil additions of 10, 18, and 25 percent on a dry weight basis. The 50- or 70-gram composts were incubated at 60°C for 6 to 10 weeks. TNT was transformed rapidly in sewage sludge composts but not in hay/horse feed composts with higher rates of sediment addition. No TNT transformation products (amine derivatives of DNT) accumulated. Most of the ^{14}C activity was recovered in the unextracted residue. Cleavage of the aromatic ring appeared insignificant as less than 2.5 percent of the ^{14}C added was recovered as $^{14}\text{CO}_2$.

In the pilot-scale systems, TNT was removed effectively in the hay/horse feed compost with 11 percent sediment, which averaged 45 to 55°C. In the most active of the two hay/horse feed pilot tanks, TNT was reduced from 53,019 ppm to 897 ppm during the 7-week composting period. The final concentration of 2-amino-4, 6-dinitrotoluene in the two tanks was 107 and 199 ppm. The final

concentrations of 4-amino-2,6-dinitrotoluene were 10 and 25 ppm. A manure compost proved more effective, decreasing TNT from 31,021 ppm to 138 ppm in 56 days. The two amino derivatives totaled 124 ppm at day 56. Moisture and supplementary carbon were added to these pilot systems during the test period.

The aforementioned reports do not demonstrate conclusively either the mineralization of TNT to innocuous end products or cleavage of the TNT aromatic ring. A number of biotransformation products have repeatedly been recovered in a variety of degradation study systems.

Kaplan and Kaplan (1982c) have proposed a number of TNT reduction products and suggested the biotransformation pathways presented in Figure C-1. Table C-1 lists acronyms for TNT transformation products as used throughout this text.

Table C-1

Acronyms for TNT Transformation Products

Acronym	Compound
2A	2-amino-4,6-dinitrotoluene
4A	4-amino-2,6-dinitrotoluene
2,4DA	2,4-diamino-6-nitrotoluene
2,6DA	2,6-diamino-4-nitrotoluene
4OHA	4-hydroxylamino-2,6-dinitrotoluene
4,4'AZ	2,2',6,6'-tetranitro-4,4'-azoxytoluene
2,2'AZ	4,4',6,6'-tetranitro-2,2'-azoxytoluene
2,4'AZ	2',4,6,6'-tetranitro-2,4'-azoxytoluene

The initial reactions in TNT transformation are stepwise reductions of the TNT nitro groups to yield amino and hydroxylamino compounds, which may then undergo additional reactions to yield azoxy compounds (Carpenter et al., 1978). These authors speculate that the resistance of the TNT aromatic ring to cleavage may be a function of either the resistance of a highly substituted molecule to enzymatic hydroxylation, or the formation of resistant polyamide conjugates.

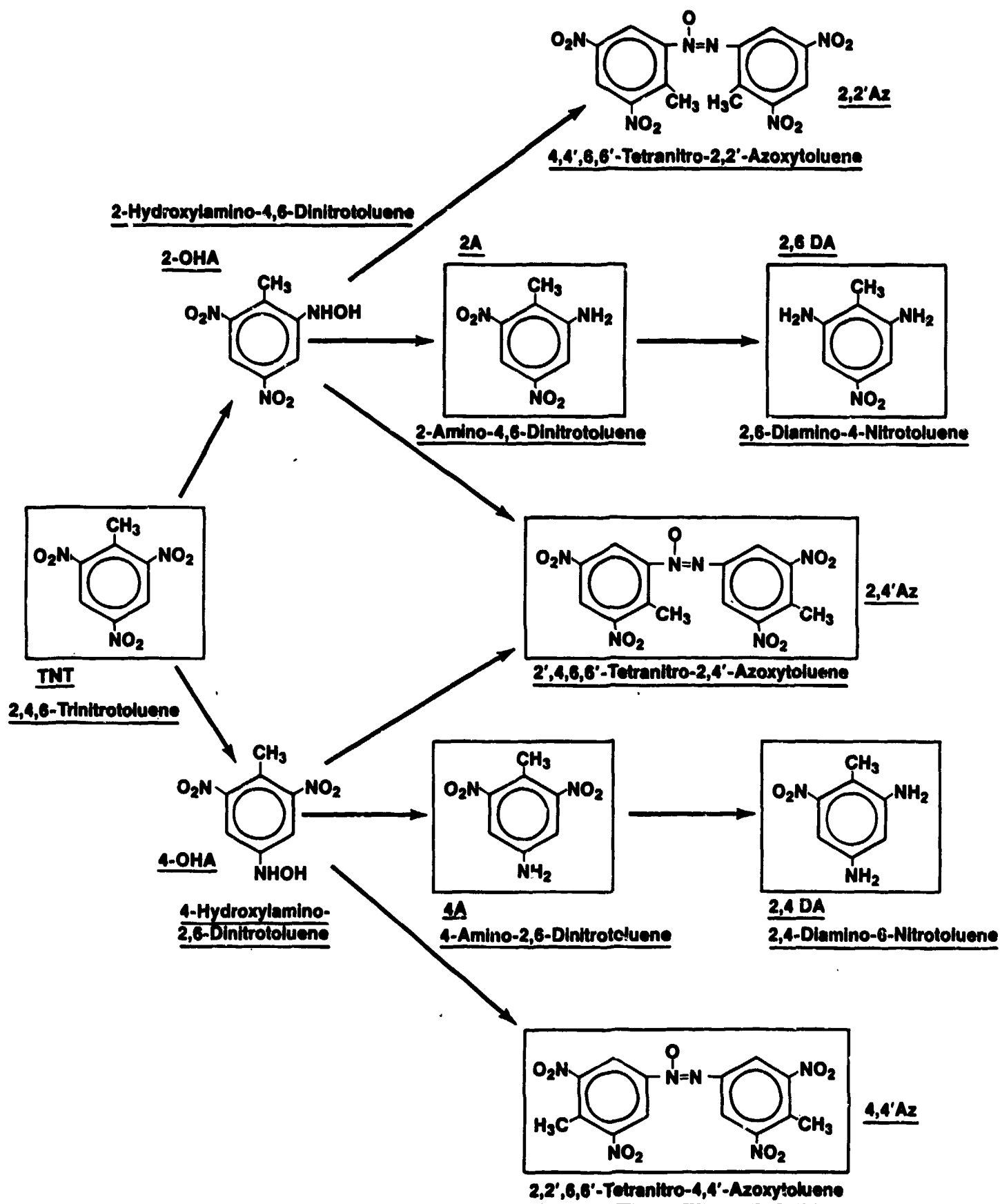


Figure C-1. Biotransformation scheme for TNT in compost.

Won et al. (1974) and Won and Heckley (1974) found that the following intermediates were produced in microbial cultures that contained TNT:

Acronym	Compound
4AZ	2,2',6,6'-tetranitro-4-azoxytoluene
6AZ	2,2',4,4'-tetranitro-6-azoxytoluene
2A	4,6-dinitro-2-aminotoluene
4A	2,6-dinitro-4-aminotoluene
6A	2,4-dinitro-6-aminotoluene
4OHA	4-hydroxylamino-2,6-dinitrotoluene
6OHA	6-hydroxylamino-2,4-dinitrotoluene
6A	6-amino-2,4-dinitrotoluene

Burlinson (1980) found that the major intermediates in aerated river water were 2A and 4A. Spanggord et al. (1983) found this to be true in TNT waste disposal lagoons. Spanggord et al. (1980b) found that these intermediates were produced in aquatic systems in combination with 2,4DA, 2,6DA, and 4,4'AZ. Hale et al. (1979) detected many of the aforementioned compounds, as well as dinitrobenzaldehyde and 2,4,6-trinitrobenzaldehyde, in soil columns leached with TNT.

Hoffsommer et al. (1978) found that the two monoamino and two diamino isomers comprised 12 percent of the biotransformation products formed in a simulated activated sludge system. Par-rish (1977) found that fungi transformed TNT to 4A, 4OHA, and 4,4'AZ. Naumora et al. (1979) found that a Pseudomonad trans-formed TNT to 4A, 2A, 4AZ, and 6AZ. Jerger et al. (1976) found that sediment populations transformed TNT to 4OHA, 4A, 2A, 4,4'AZ, 2,4AZ, and 2-hydroxylamino-4,6-dinitrotoluene.

In summary, TNT has been found to be biotransformed, but not mineralized to innocuous end products such as carbon dioxide and water, by a diverse group of microorganisms in a number of environments. These microbes generally catalyze nitro group reduction, but no conclusive evidence exists for aromatic ring cleavage. As a result, a number of metabolites are produced, and evidence exists that these metabolites are tightly bound to organic materials.

RDX

Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) is an explosive widely used for military purposes. During manufacture, load, assembly, and pack operations, RDX may be discharged to the environment in process wastewater.

The absence of oxygen has frequently been cited as a prerequisite for RDX biodegradation. Hoffsommer et al. (1978) found no disappearance of RDX in an activated sludge system, both when RDX was the only compound present and when it was in the presence of TNT. Other researchers have observed that RDX was not readily biotransformed in water under aerobic conditions (Spanggard et al., 1980). Conversely, Soli (1973) detected the disappearance of RDX when it was incubated anaerobically with purple photosynthetic bacteria. Approximately 97 percent of the RDX (20 mg/L) was transformed after 5 days of incubation. It was hypothesized that the strong reducing conditions of the photosynthetic culture were responsible for destruction of the RDX molecule.

RDX biodegradation appears to rely on co-metabolism, requiring the presence of alternative organic substrates. In an environmental fate study (Sikka et al., 1978), a concentration of 10 ppm RDX was decreased only when sediments were added to river water and after a 20-day lag period. During a 1-month period, no loss of RDX occurred in media unsupplemented with yeast extract. The sediment may have provided essential nutrients, co-metabolic substrates, or metabolically capable microorganisms. Spanggard et al. (1980b) detected no significant RDX transformation in anaerobic flasks without added yeast extract. In flasks containing RDX and yeast extract, RDX was transformed in 2 to 3 days.

Further evidence that the absence of oxygen is required for RDX biodegradation was presented by other research groups. McCormick et al. (1981) reported the complete disappearance of RDX (50 to 100 ug/mL) after 4 days of incubation in anaerobic sewage sludge. Bell et al. (1984) reported the removal of RDX under anaerobic conditions in a bench-scale semicontinuous activated sludge treatment system. The initial concentration of RDX was approximately 5 mg/L. RDX decreased to 3.05 mg/L within approximately 11 hours and no further biodegradation occurred, apparently because organic nutrients were exhausted in this batch experiment. No significant removal of RDX was observed under aerobic conditions in a comparable system.

McCormick et al. (1984) compared the efficiency of RDX biodegradation in batch and continuous culture systems. In anaerobic batch cultures using sewage sludge as an inoculum and incubated at 37°C, biodegradation of RDX occurred through reduction of the nitro groups. No activity was observed under aerobic conditions. RDX was also degraded in an anaerobic, denitrifying, continuous culture system using simulated wastewater. RDX completely disappeared after 3 weeks under conditions of high total organic carbon (TOC, 3,135 mg carbon per liter) and low redox potential (Eh, -200 to -400 mv). Under similar conditions, but with a lower TOC, only 75 to 85 percent of the RDX disappeared in 5 weeks. No buildup of transformation products was observed in the continuous culture systems.

Some work has been done on the biological degradation of RDX in soil. In a soil enrichment study, RDX disappeared, but conclusive evidence for RDX degradation by microorganisms was not obtained (Osmon and Klausmeier, 1973). Greene et al. (1985) reported limited biodegradation of RDX in soil columns using low flow rates and dextrose as a carbon supplement. The influent contained 99 mg/L of RDX and the effluent 59 mg/L, a 40 percent reduction.

Isbister et al. (1982) followed the degradation of ^{14}C -RDX and RDX in laboratory- and greenhouse-scale composting experiments, respectively. More details of this study are provided in the TNT review section. In the laboratory ^{14}C -RDX experiment, RDX was reduced by 31.2 percent after 3 weeks and by 70.3 percent after 6 weeks. At 6 weeks, 55.8 percent of the ^{14}C was recovered as $^{14}\text{CO}_2$, 21.6 percent was present in the solvent extract, and 16.1 percent remained in the compost. The high percentage of $^{14}\text{CO}_2$ recovered indicated that cleavage of the RDX molecule occurred. In the greenhouse-scale experiments, RDX was reduced by 31 percent after 3 weeks and by 82 percent following 6 weeks of composting.

Doyle et al. (1986) conducted laboratory- and pilot-scale composting experiments with ^{14}C -RDX and RDX, respectively. More details of this study are provided in the TNT review section. In the laboratory studies, RDX was significantly transformed. $^{14}\text{CO}_2$ production accounted for 46.5 and 37.2 percent of the ^{14}C added as ^{14}C -RDX in hay/horse feed and sewage sludge composts, respectively. In those same composts, 5.6 and 11.0 percent of the ^{14}C added as ^{14}C -RDX were recovered in the extract, and 29.7 and 54.6 percent remained as unextracted residue. In the pilot-scale study, RDX was reduced from approximately 7,400 ppm to approximately 400 ppm after 56 days in the manure compost.

RDX transformation products have also been detected. In batch cultures using anaerobic sewage sludge as inoculum, biodegradation of RDX produced mono and dinitroso derivatives. Under reducing conditions, further reductions led to the formation of formaldehyde and methanol. Traces of hydrazine, 1,1-dimethylhydrazine, and 1,2-dimethylhydrazine also were detected, but these were shown to be biodegradable under either aerobic or anaerobic conditions (McCormick et al., 1984). Greene et al. (1985) reported mono and dinitroso intermediates in the leachates of soil columns treated with RDX.

In summary, RDX has been reported to resist aerobic biological degradation. RDX has been reported to biodegrade under certain anaerobic conditions in the presence of sufficient organic nutrients. However, RDX was transformed in composts considered to be aerobic. It is possible that anaerobic microsites existed within these composts. The most efficient RDX biological treatment tested was an anaerobic, denitrifying continuous culture system operated with high TOC and low redox potential. When RDX degradation was incomplete, nitroso intermediates were produced.

HMX

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazocine (HMX) is produced as an explosive and is also formed as a by-product of RDX production. It has a higher melting point than RDX and was named HMX for "high-melting explosive" (Bell et al., 1984). During the manufacture of RDX, up to 12 mg/L of HMX may be discharged to the environment in process wastewater (Jackson et al., 1978). This is well above the level of 0.3 mg/L for RDX/HMX that has been proposed by the Army Medical Bioengineering Research and Development Laboratory to protect aquatic life (Burrows, 1982).

Early work suggested that RDX and HMX could be at least partially degraded by an aquatic microflora (Small and Rosenblatt, 1974; Green, 1972). Fifty-three percent of HMX and 42 percent of RDX were removed from Holston Army Ammunition Plant (HAAP) Area B wastes in pilot-plant studies using an activated sludge reactor dominated by Pseudomonas and Alcaligenes species (Green, 1972). Stillwell (1977) presented data indicating that RDX and HMX could be biologically removed from pilot-plant units at HAAP. Two treatment schemes were investigated: 1) denitrification plus activated sludge; and 2) denitrification plus aerobic trickling filter plus activated sludge. Both systems were effective in reducing RDX concentrations, but the latter system was more effective in HMX removal.

More recent work has been done on the biodegradation of HMX in aqueous systems. Using anaerobic batch cultures, gradual reduction of HMX was observed, but no activity was detected under aerobic conditions (McCormick et al., 1984). These authors also inoculated a nutrient broth containing 50 mg/L of HMX with anaerobic sewage sludge. Subsequent HPLC analysis showed a reduction of HMX, from an initial concentration of 50 ppm to 25 ppm after 5 days and 15 ppm after 25 days. The initial rate of disappearance of HMX was less than that found for RDX and leveled off such that complete elimination of HMX did not occur even after several weeks of incubation.

As an extension of the aforementioned batch culture study, the fate of HMX in anaerobic, denitrifying, continuous culture systems using simulated wastewater was studied by McCormick et al. (1984). Anaerobic sewage sludge obtained from the Nut Island Sewage Treatment Plant, Boston, Massachusetts, was used as an inoculum. The retention time in the continuous culture system was 10 to 14 days, and various influent nutrient solutions were tested. These included nutrient broth, alkaline hydrolyzed sludge, molasses, and basal salts medium. These data indicated that HMX was not completely degraded. Under optimal conditions of high total organic carbon (TOC, 3,135 mg carbon per liter) and low redox potential (Eh, -200 to -400 mv), HMX had slightly greater than 90 percent disappearance after 40 days. Similar results were found in experiments using other media with different levels of TOC and redox potential.

McCormick et al. (1984) found that HMX and acetylated HMX remained unaltered after passage through a full-scale anaerobic treatment system.

HMX was utilized in a bench-scale study to evaluate the potential for treatment of munitions wastewater in a semicontinuous activated sludge treatment system. HMX removal remained at 34 to 35 percent with a cycle ratio of 3 hours aeration/2 hours anoxic (Bell et al., 1984). Batch studies were performed in order to investigate the removal of HMX in greater detail. The results of these studies confirmed the previous work of McCormick et al. (1984). No removal or degradation of HMX occurred in a 14-hour period under aerobic conditions. Under anaerobic conditions, the HMX content decreased during the first 11 hours, with no additional removal. The authors believe that the abrupt halt in removal was due to the exhaustion of organic nutrients.

Smith et al. (1985) studied HMX in experiments on the biological treatability of waste streams from propellant and explosives manufacture. Preliminary cost studies indicated that a rotating biological contractor (RBC) would be the preferred technology for treatment of these wastewaters, provided technical feasibility could be demonstrated. However, both RDX and HMX flowed through the pilot unit essentially unchanged, while TNT photolyzed in the influent tank.

Greene et al. (1985) studied the feasibility of land farming HMX. Simulated pink water containing TNT, RDX, HMX, and 2,4-DNT was continuously applied to a number of soil columns at different flow rates, with and without glucose as a carbon supplement. Within the timeframe of the study (110 days) there appeared to be little evidence of degradation of HMX under any of the column conditions studied.

Doyle et al. (1986) conducted laboratory- and pilot-scale composting experiments on ^{14}C -HMX and HMX, respectively. More details of this study are provided in the TNT review section. In the laboratory studies, the results were complicated by the fact that 40.3 percent of the ^{14}C in the spiking solution was ^{14}C -RDX and 53.4 percent was ^{14}C -HMX. In the sewage sludge compost with 10 percent sediment, 31.1 percent of the ^{14}C added was recovered as $^{14}\text{CO}_2$, 27.1 percent was recovered in the extract, and 42.8 percent was found in the unextracted residue. In the pilot-scale system using a manure compost, HMX was reduced from approximately 1,550 ppm to approximately 300 ppm after 56 days of composting.

Biotransformation products have been identified from HMX. McCormick et al. (1984) extracted and identified mono and dinitroso derivatives of HMX from batch cultures under anaerobic conditions. Methanol was also detected in reaction mixtures

and is presumed to have arisen through reduction of formaldehyde, a primary HMX biotransformation product. Formaldehyde, however, was not detected in the reaction mixture, nor was methane found in headspace gas samples taken from above the HMX mixture.

In summary, complete biodegradation of HMX has not been observed experimentally in either aqueous or soil systems, but has been presumed in compost systems. Partial degradation of HMX, at a slower rate than RDX, has been observed under optimal treatment conditions in an anaerobic continuous culture system with high TOC and low redox potential. Degradation products include mono and dinitroso derivatives of HMX, as well as methanol and formaldehyde.

Tetryl

Very little information could be located on tetryl. Doyle et al. (1986) conducted laboratory- and pilot-scale studies on ^{14}C -tetryl and nonlabeled tetryl, respectively. More details of this study are provided in the TNT review section. In the laboratory experiments, loss of ^{14}C -tetryl was considered "substantial" and did not vary significantly with composting treatments. However, very little of the added ^{14}C was recovered as $^{14}\text{CO}_2$. In the hay/horse feed and sewage sludge composts (with 10 percent sediment addition), $^{14}\text{CO}_2$ accounted for 5.0 and 3.2 percent of the recovered ^{14}C and the unextractable residue for 84.2 and 92.6 percent, respectively. In the pilot-scale manure compost, tetryl was reduced from approximately 950 ppm to approximately 100 ppm after 56 days of composting.



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APPENDIX D
PROCEDURES FOR REACTIVITY TESTING

3833B

HERCULES INCORPORATED
HERCULES MISSILES, ORDNANCE & SPACE GROUP
ALLEGANY BALLISTICS LABORATORY
P. O. BOX 210
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SENSITIVITY TESTING
OF
COMPOSTED SAMPLES

OCTOBER 20, 1988

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PREPARED FOR

WESTON
WESTON WAY
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HERC NO. 88-61

SUMMARY

The composted samples were subjected to the following tests: Thermal Stability, Detonation (#8 Cap), Spark, ABL Impact, and U.S. Gap.

Thermal Stability - No reaction was observed when the Weston cooler 3 and cooler 4 samples were placed in an oven and heated to 167°F for a period of 49.5 hours.

Detonation (#8 Cap) Test - No reaction was obtained when the cooler 3 and the cooler 4 composted samples were subjected to the detonation (#8 Cap) test.

Spark Test - No reaction was observed for either sample - cooler 3 or cooler 4 - during the spark test.

ABL Impact Test - The cooler 3 sample threshold initiation level (TIL) was determined to be 41 centimeters. No visible reaction was observed at drop heights greater than 41 centimeters but the LIRA infrared analyzer detected the presence of combustion products at all test levels greater than 41 centimeters. The TIL is the maximum level of input stimulus that gives no response in 20 trials.

The cooler 4 sample showed no response at the 120 centimeter drop height in 20 trials. The TIL value is therefore greater than or equal to 120 centimeters. The 120 centimeter drop height is the maximum input stimulus the ABL Impact Machine is capable of.

U.S. Gap Test - The cooler 3 and cooler 4 samples showed no indication of a reaction when subjected to the U.S. Gap test.

INTRODUCTION

Thermal Stability, Detonation (#8 Cap), Spark Test, ABL Impact, and U.S. Gap Test were performed on cooler 3 and cooler 4 composted samples supplied by Weston. This report documents those results.

DISCUSSION

1. Thermal Stability

A. Test Results

No reaction was observed for either the cooler 3 or the cooler 4 samples.

B. Test Apparatus and Procedure

The test procedure consists of heating an explosion-proof oven to 167°F, filling a 4-ounce cup with sample (approximately 30 grams), placing the cup with sample into the heated oven for a period of not less than 48 hours. The sample is weighed prior to heating and after heating to determine any mass loss.

2. Detonation (#8 Cap)

A. Test Results

The results were negative (no reaction) for both the cooler 3 and the cooler 4 samples.

B. Test Apparatus and Procedure

The Detonation test consists of placing approximately 50 grams of the sample to be tested in a paper cup. The paper cup is placed on a 3/8" by 2" by 2" mild steel plate which is located on top of a nominal 4" lead cylinder. The lead cylinder is the witness to explosion/detonation reactions. A number 8 blasting cap is placed in the metal cap holder above the top surface of the test sample and ignited.

3. Spark Test

A. Test Results

No reaction was observed for the cooler 3 and cooler 4 samples.

B. Test Apparatus and Procedure

The Spark test consists of placing approximately 50 grams of the sample to be tested in a paper cup. The paper cup is placed on a 3/8" by 2" by 2" mild steel plate which is located on top of a nominal 4" lead cylinder. The lead cylinder is the witness to explosion/detonation reactions. A MIAI squib igniter is placed in direct contact with the top surface of the test sample and ignited.

4. ABL Impact

A. Test Results

A TIL of 41 centimeters was obtained for the cooler 3 sample. No visible reaction was observed at higher levels of input stimuli but the LIRA infrared analyzer detected combustion products. Visual inspection of the sample revealed the presence of white particles, possible RDX, which could explain the impact sensitivity.

No reaction was observed in the cooler 4 sample at the highest level of input stimulus - drop height of 120 centimeters.

B. Test Apparatus and Procedure

The test apparatus was designed and built at ABL. The test consists of spreading a monolayer of sample on the top surface of the impact anvil. The impact hammer is placed in direct contact with the top surface of the test sample. A 4.0 kilogram weight is raised to a predetermined height and released. The weight falls the prescribed distance and strikes the hammer. A reaction is determined by a LIRA infrared analyzer and observations made by the technician performing the test.

5. U.S. Cap

A. Test Results

No reaction was observed for either the cooler 3 or the cooler 4 composted sample.

B. Test Apparatus and Procedure

The test sample is placed in a cold-drawn seamless carbon steel cylinder 16 inches long x 1.875 inches outside diameter. The cylinder has a wall thickness of 0.219 inches. The sample is loaded to the density attained by tapping the cylinder until further settling becomes imperceptible. The bottom of the cylinder is closed with two layers of polyethylene sheet 0.003 inches in thickness. The polyethylene is secured with rubber bands and polyvinyl chloride electrical tape. The sample is subjected to the shock wave generated by the detonation of a pentolite pellet 2 inches in diameter by 2 inches in length. The pentolite pellet is initiated by a number 8 cap butted against the pellet and held in place by a wooden cap holder. A cellulose acetate cylinder 2 inches by 2 inches is placed between the pentolite pellet and the sample tube. A continuous velocity probe is located on the inner surface of the sample cylinder. In addition, a 6 inch square mild steel witness plate is mounted at the opposite end from the pentolite pellet and separated from it by a 0.063 inch thick spacer.

A reaction is determined by a stable propagation velocity of greater than 1.5 km/sec and or a clean hole punched in the witness plate. Also, the sample tube should be fragmented along its entire length. If any two of the above three criteria (velocity probe, witness plate, and tube fragmentation) are met the reaction is considered positive.

CONCLUSIONS

The Weston composted samples showed no indication of reactivity during testing with the exception of the cooler 3 sample's sensitivity to impact stimulus.