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COMPOSTING EXPLOSIVES/ORGANICS CONTAMINATED SOILS

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Commander
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Aberdeen Proving Ground, Maryland 21010

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TNT; 2,4,6-trinitrotoluene
RDX; hexahydro-1,3,5-trinitro-1,3,5-triazine
HMX; octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
tetryl; N-methyl-N,2,4,6-tetranitroaniline
Nitrocellulose
TCE; trichloroethylene
biodegradation
sediment
soil
composting
explosives

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

The primary objectives of this study were to determine if sediment contaminated with TNT, RDX, HMX, and tetryl from Louisiana AAP, soil contaminated with nitrocellulose from Badger AAP, and soil contaminated with TCE from Letterkenny AD can be decontaminated using composting. Laboratory studies were conducted using ^{14}C-tracers to evaluate contaminated breakdown in two types of compost (hay-horse feed and sewage sludge-wood chips) with contaminated sediment from these Army installations added as 10, 18, and 25% of the compost dry mass.

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Results indicated that the Louisiana AAP sediment at high rates of addition had a detrimental effect on the breakdown of explosives. However, RDX and TNT degraded rapidly from compost where the sediment addition was at the 10% level. Tetryl breakdown was not strongly affected by sediment loading; greater than 90% loss of tetryl occurred in all treatment after 44 days of composting. HMX breakdown was relatively slow. Nitrocellulose degradation was unaffected by sediment loading rates and decontamination of the Badger AAP soil appeared to be complete within 4 weeks. No accumulation of toxic or otherwise harmful degradation products was detected in composted Louisiana AAP or Badger AAP soil.

Pilot-scale composting tests were conducted using 500 gallon self-sustaining composts. ~~In the~~ hay-horse feed composts containing 11% Louisiana AAP soil, explosive losses followed first-order kinetics with half-lives of 1.6, 3.0, and 4.7 weeks for TNT, RDX, and HMX, respectively. Breakdown of explosives in sewage sludge composts amended with 16% Louisiana AAP sediment was insignificant. A third mixture of compost materials (manure-hay-saw dust) amended with 12% Louisiana AAP sediment was tested. First order half-lives for TNT, RDX, HMX, and tetryl were 1.0, 2.5, 3.3, and 1.2 weeks, respectively. Nitrocellulose in the Badger AAP sediment was rapidly broken down: 100% decontamination within 3 weeks in hay-horse feed composts, and 92-97% decontamination in the sewage sludge composts within 4 weeks. Loss of explosives or heavy metals in compost leachate was low in all tests.

Laboratory studies with the Letterkenny soil demonstrated that TCE was rapidly volatilized from thermophilic composts.

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EXECUTIVE SUMMARY

Laboratory scale and pilot scale studies were conducted to evaluate composting as a decontamination method to treat sediments and soils containing hazardous waste materials. All studies were conducted using contaminated sediment/soil from U.S. Army installations. Sediment from lagoons at Louisiana Army Ammunition Plant (LAAP) contained high concentrations of TNT and RDX, and moderate levels of HMX and tetryl. Soil from Badger Army Ammunition Plant (BAAP) contained moderate to high levels of nitrocellulose. The degradation of the explosives in these sediments/soils during composting was studied in a series of laboratory experiments using ^{14}C-labeled tracers to follow the fate of each explosive. Two types of composts (hay-horse feed and sewage sludge-wood shavings) and three rates of sediment/soil addition to the compost (10, 18 and 25% dry weight basis) were utilized in these studies. The composts were incubated at 60°C with continuous aeration for 6-10 weeks. Offgasses from the composts were monitored for ^{14}C and at the completion of the incubation, composts were analyzed for the explosives, extractable ^{14}C-labeled degradates and unextracted residual ^{14}C.

TNT degraded rapidly in all the sewage sludge composts, but breakdown in the hay-horse feed compost was adversely affected by the higher rates of sediment addition. Accumulation of TNT transformation products (amino derivatives of DNT) did not occur. Most of the ^{14}C from degraded TNT was recovered as unextracted residue. Cleavage of the benzene ring during TNT breakdown did not appear to be significant.

RDX was almost completely degraded in composts amended with sediment at the 10% rate during 10 weeks of incubation. Increased rates of sediment addition significantly decreased the rate of RDX breakdown in both hay-horse feed and sewage sludge composts; although the inhibitory effect was much more pronounced in the hay-horse feed composts. Substantial losses of ^{14}C from the composts as $^{14}CO_2$ demonstrated that RDX is completely metabolized to natural products.

HMX did not degrade in the hay-horse feed composts, but levels were reduced 30-50% during 10 weeks of incubation in the sewage sludge composts. HMX losses were lowest in the composts with the higher rates of sediment addition.

Tetryl was highly susceptible to degradation by composting; 90-100% tetryl loss was demonstrated after composting for 44 days. Apparent rates of tetryl breakdown were not strongly influenced by the sediment loading rates. Recovery of ^{14}C as $^{14}CO_2$ or extractable degradates was insignificant. Most of the ^{14}C from degraded ring-labeled tetryl was found as unextracted residue.

Nitrocellulose degraded rapidly with substantial evolution of $^{14}CO_2$. The patterns of $^{14}CO_2$ evolution indicated that essentially all the nitrocellulose was degraded within 4 weeks.

Pilot scale composting of the LAAP and BAAP sediments was carried out using 500 gallon self-sustaining composts that were aerated intermittantly. Breakdown of explosives in the hay-horse feed compost (11% LAAP sediment) followed first order kinetics. The half-lives for TNT, RDX, and HMX were 1.6, 3.0, and 4.7 weeks, respectively. No loss of explosives in the sewage sludge compost (16% LAAP sediment) was observed during 7 weeks of composting. A third type of compost (manure mixed with hay and saw dust) was tested with the LAAP sediment (12% sediment). Loss of LAAP explosives was greatest in this compost. Half-lives of TNT, RDX, HMX, and tetryl were 1.0, 2.5, 3.3, and 1.2 weeks, respectively. Nitrocellulose degradation in hay-horse feed composts (15% BAAP sediment) was complete within 3 weeks. In the sewage sludge composts 92-97% degradation occurred with 4 weeks. Leaching of explosives and heavy metals from the composts was minimal and should not constitute an environmental hazard.

Soil from Letterkenny Army Depot contaminated with TCE was tested to determine the fate of TCE during composting. TCE was found to volatilize from compost materials within a few hours when the temperature was held at 60°C.

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I. INTRODUCTION

A. Background

Operations at a number of military installations have resulted in the release of toxic materials or hazardous substances onto soil or sediment within these installations. These released substances include a variety of explosives, solvents, pesticides and other organics, as well as a number of inorganic materials, such as heavy metals and mineral acids. Concentrations of pollutants vary from low ppb levels up to the point where more than half the weight of the sediments is composed of contaminants. Low level contamination is generally found in soils around manufacturing and handling facilities where release of the pollutants was inadvertent. These lightly contaminated areas can account for the majority of the contaminated land mass. Areas of heavy contamination are largely confined to spillways and lagoons utilized over extended periods to channel and contain wastewaters from the installations' operations, an accepted practice at the time the facilities were constructed and operated. Contaminants in some of the heavily polluted areas have been leached to varying degrees. In some situations, contaminants have reached ground water and are migrating horizontally toward potable water supplies. Near term action is necessary at these sites to eliminate the source of ground water pollution, i.e., remove or clean the contaminated sediments. Ultimately all soil/sediments, including the lightly contaminated soils, will have to be removed or decontaminated to be able to release the land for unrestricted use.

Previous work has demonstrated that composting is an effective means of degrading TNT and RDX. Composting can be setup as an on site treatment. Compost materials (hay, straw, wood chips, manure, sewage sludge, etc.) can be obtained locally at low or no cost, and composting facilities can be rapidly constructed at minimal costs. Major equipment items needed to handle the compost are off-the-shelf items that can be readily transported from site to site as needed. Relatively few personnel are needed to maintain a composting facility, and training for most of the personnel is minimal. In short, composting has significant economic and portability advantages over other currently available technologies. However, additional experimentation was deemed necessary to demonstrate the ability of the composting process to handle the wide variety of contaminants found in the lagoon sediments at various Army Ammunition Plants and depots.

The previous work on composting of RDX and TNT was purposely designed to avoid many of the complicating factors that will be encountered in an actual composting operation. In these early studies, the contaminated sediment was made by adding solutions of a single explosive to an uncontaminated sandy soil that had a low adsorptive capacity. In the present studies described in this report, the scope of the work was expanded to examine the breakdown of TNT, RDX, tetryl, HMX, nitrocellulose, and TCE in actual sediments from military installations. In some cases, these sediments contained multiple contaminants, including the contaminants being studied as well as other organics, pesticides, heavy metals, etc. The explosives are not uniformly dispersed in the sediment, but are found in large nodules as well as being adsorbed to the soil surface. The sediments and soils used in the studies contained substantially more clay and/or organic matter than the sand used in the initial study, thus increasing possibility of strong adsorption of the contaminants onto the soil. Both the presence of nodules of explosives and strong adsorption of the explosives to the soil could protect the explosives from microbial and chemical attack in the compost.

B. Objectives

The present experimental work, conducted under contract No. DAAK11-84-C-0057 with USATHAMA, was aimed at studying the effects of multiple contamination on the composting process. This work was divided into three phases:

- field survey to locate and quantitate the contaminants in lagoon or soil samples collected at three Army Installations described in Section II
- laboratory tests presented in Section III to determine the fate of each contaminant during composting
- pilot scale composting trials discussed in Section IV.

The primary objectives in the laboratory tests were to determine:

- if TNT, tetryl, RDX, and HMX in a Louisiana AAP sediment could be simultaneously degraded by composting
- if nitrocellulose in a Badger AAP soil could be metabolized by composting
- and to determine the fate of TCE in compost.

Within these objectives, information was obtained on the relative rates of explosive breakdown and the resultant degradates were characterized. Secondary objectives were to determine the efficiency of the composting process as a function of contaminated sediment loading and the composting materials.

The primary objectives of the pilot scale studies were to obtain kinetic data on the loss of explosives during composting and to verify the findings from the laboratory studies.

II. FIELD SURVEYS

Field surveys were conducted at three different installations to determine the amount of hazardous materials or explosives in the soils or lagoons and to identify which soils or sediments would be suitable for the pilot-scale composting studies. The three installations which were visited were Badger Army Ammunition Plant in Baraboo, Wisconsin; Louisiana Army Ammunition Plant near Shreveport, Louisiana; and Letterkenny Army Depot in Chambersburg, Pennsylvania. At each site, surface and ground water samples were taken. Core samples were also taken at depths to 12 inches at various locations. These samples were packed in dry ice to maintain the samples at 4°C and shipped via freight to ARC's main laboratories for analyses.

B. Badger Army Ammunition Plant

Badger AAP was visited on September 11, 1984 to locate potentially contaminated areas and obtain samples for analysis. Badger has a series of four large lagoons which receive nitrocellulose production waste waters via a creek directly from the production lines. As shown in Figure 1, the first lagoon encompasses 25 acres. This lagoon resembles a field with a small stream trickling through it. The 25 acre lagoon ends in a sluice gate which guides the water into another series of three lagoons which eventually empty into Gruber's Grove Bay. The last two lagoons have been dredged and the dredgings placed in mounds along the side of the lagoons. All lagoons and dreding mounds support grass growth and are used for grazing of cattle.

Samples were taken in the creek below the sewage treatment plant, at the entrance to the 25 acre lagoon, at the gate from the 25 acre lagoon, in the dredgings ponds and at the entrance to Gruber's Grove Bay. The analyses of the samples are presented in Table 1. The high level analysis are understated because of problems with dilutions with the old nitrocellulose analytical method using the acidic sulfanilamide reagent. These problems have been overcome with a modified analysis method (see Appendix F).

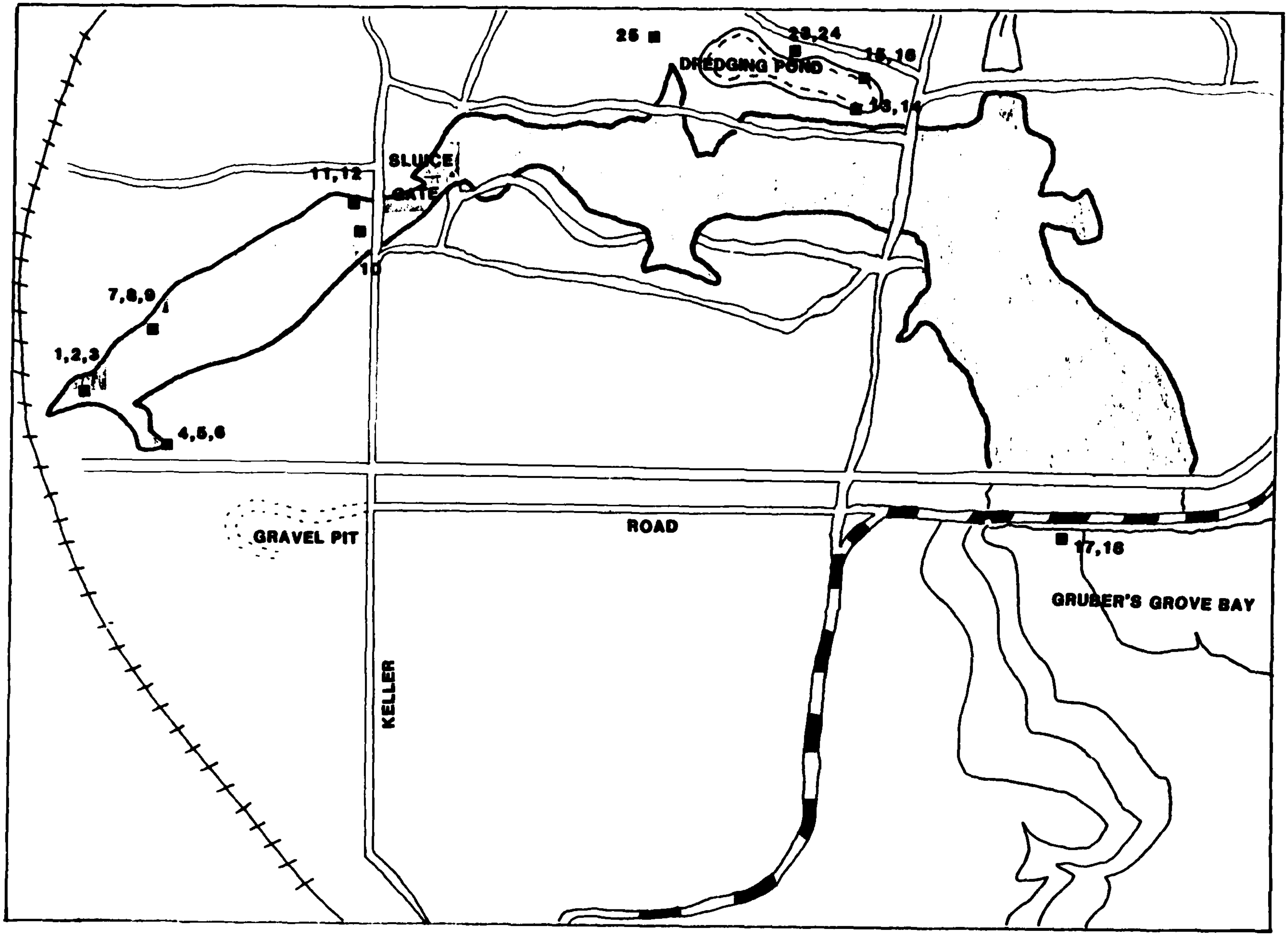

Figure 1. Layout of the Nitrocellulose Wastewater Lagoons at Badger AAP.

Table I. Analysis of Samples Collected from Badger AAP **

Sample #	Site Description	Nitrocellulose Concentration ppm
1	Top 6" of core, creek leading into 25 A pond	1,326.0
2	Bottom 6" of core, creek leading into 25 A pond	6,277.0
3	Skim top of sediment, creek leading into 25 A pond	< 17.2*
4	Surface sample at end of right fork in 25 A pond inlet	90.0
5	Bottom 6" of core, at end of right fork in 25 A pond inlet	518.0
6	Top 6" of core at end of right fork in 25 A pond inlet	148.1
7	Surface samples at end of left fork in 25 A pond inlet	88.8
8	Bottom 6" of core at end of left fork in 25 A pond inlet	3,498.0
9	Top 6" of core at end of left fork in 25 A pond inlet	592.0
10	Surface sample from right side of sluice gate in creek bed at far end of 25 A pond	1,690.0
11	Bottom 6" of core, creek bed at end of 25 A pond	Sample jar broken in shipment
12	Top 6" of core, creek bed at end of 25 A pond	5,867.0
13	Bottom 6" of core, east side of dredgings pit (50 yds toward road from labeled site)	3,148.4
14	Top 6" of core, east side of dredgings pit (50 yds toward road from labeled site)	1,684.0
15	Bottom 6" of core, 1' from marked sampling site in dredgings pit	Sample jar broken in shipment

** See Appendix F for Analysis Procedures

Table I. Analysis of Samples Collected from Badger AAP (continued)

Sample #	Site Description	Nitrocellulose Concentration ppm
16	Top 6" of core, 1' from marked sampling site in dredgings pit	523.6
17	Bottom 6" of core, final effluent before Gubers grove Bay	< 17.2
18	Top 6" of core, final effluent before Gubers Grove Bay	< 17.2
19	Bottom 6" of core, outlet of 84" line from treatment plant	< 17.2
20	Top 6" of core, outlet of 84" line from treatment plant	< 17.2
21	Bottom 6" of core, approximately 1/2 way between treatment plant and pond #1, in creek bed	< 17.2
22	Top 6" of core, approximately 1/2 way between treatment plant and pond #1, in creek bed	< 17.2
23	Bottom 6" of core, dredgings pond north of #4 about 100' down from stake	< 17.2
24	Top 6" of core, dredgings pond north of #4 about 100' down from stake	< 17.2

* Detection limit for nitrocellulose in soil.

B. Louisiana Army Ammunition Plant

Louisiana AAP was visited on September 13, 1984. Louisiana has 16 lagoons of approximately one-two acres each which contain varying amounts of TNT, RDX, HMX and tetryl as well as some heavy metals. These lagoons were used in the past to receive wastewater directly from the shell loading operations. The water was trucked to the lagoons and dumped into the lagoons from concrete spillways. As a result of these operations, the heavily contaminated lagoons (4 and 9) show a high variance in concentration across the lagoon. The highest concentrations (up to 60-70% explosives) are at the spillway while concentrations at the far end of the lagoon are in the low ppm range. The effect of the high concentrations of TNT and RDX is seen in Figure 2. The soil close to the spillway is red in color with sparkling crystals of RDX.

Figure 2. Photograph of Louisiana AAP Lagoon #4
Looking Down on the Lagoon From the Spillway

Both water and core samples were taken from these lagoons at the sites shown in Figure 3. Data for TNT, RDX, HMX, tetryl, lead and chromium levels at the various sampling points in these lagoons are presented in Table II.

C. Letterkenny Army Depot

Letterkenny Army Depot was visited on September 20, 1984. This depot has several pits containing paint scrapings and trichloroethylene from refinishing and degreasing operations. These pits have been filled and the solvents are migrating into the ground water. Several subsurface samples were taken, packed to maintain the samples at 4°C and shipped to ARC's main laboratory for trichloroethylene (TCE) analysis. These samples contained TCE in the 1-2 ppm range. No excavation was performed, therefore, no samples were taken at the 10-20 ft deep levels where the major portions of the contaminants are located.

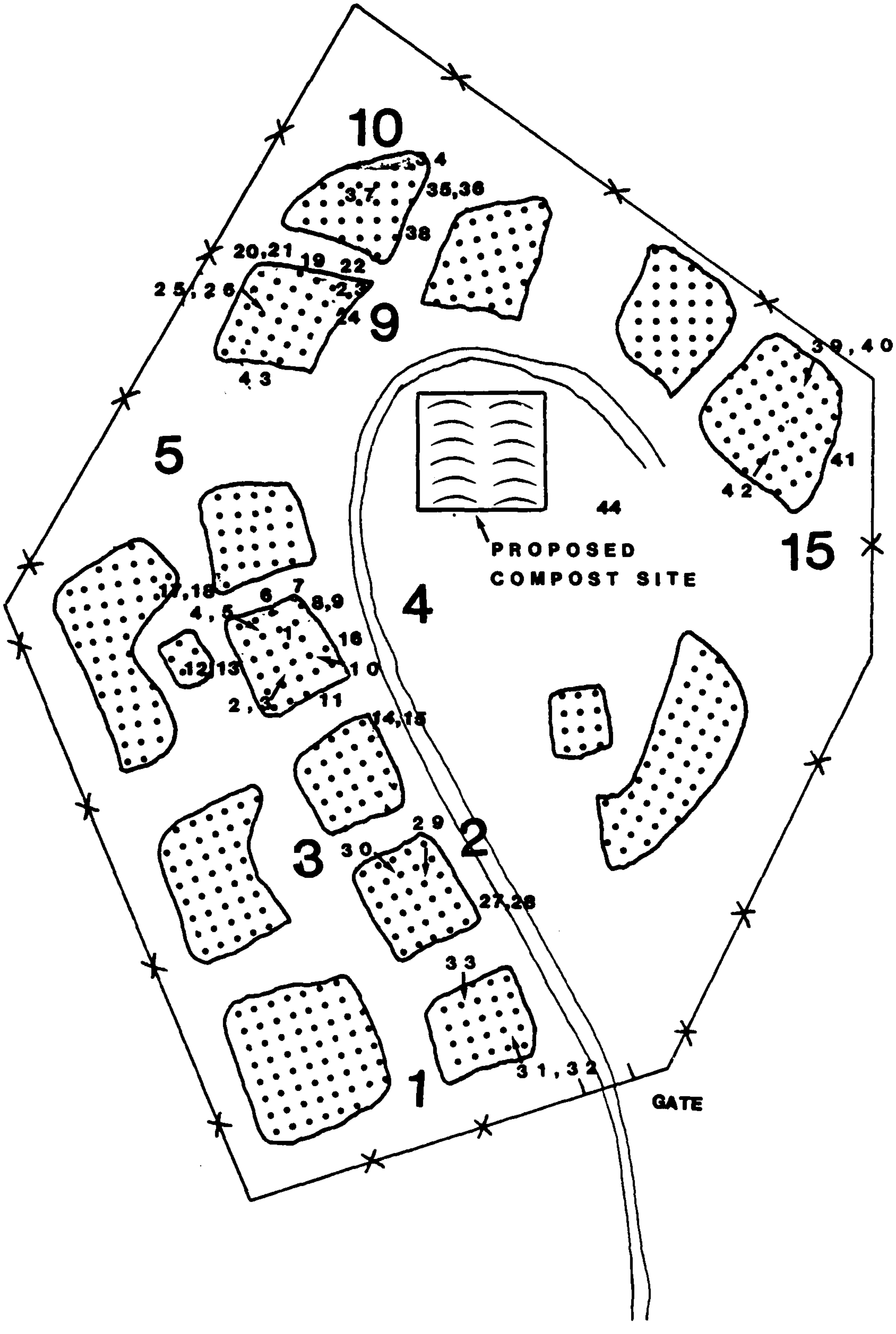

Figure 3. Location of Sample Sites at Louisiana AAP Pink Water Lagoon

Table II. Analysis of Samples from Lagoons at Louisiana AAP**

Sample #	Sample Description	TNT (ppm)	RDX (ppm)	HMX (ppm)	Tetryl (ppm)	Chromium (ppm)	Lead (ppm)
1	Lagoon #4 (Surface sample 16'6" W of spillway)	691,225	68,290	11,917	42,217	< 7.2	53.6
2	Lagoon #4 (Bottom 6" of core, 36' W of spillway)	107,583	32,493	7,363	337	< 7.2	139.6
3	Lagoon #4 (Top 6" of core, 36' W of spillway)	551,691	64,289	13,955	7,113	< 9.8	45.0
4	Lagoon #4 (Botton 6" of core, 36' NW of spillway)	272,138	98,911	11,970	1,547	< 7.2	68.0
5	Lagoon #4 (Top 6" of core, 36' NW of spillway)	614,816	58,630	12,468	193	< 7.2	25.9
6	Lagoon #4 (Bottom 6" of core, 70'N, 36'W of spillway)	790	758	167	<0.3	< 7.2	<7.1
7	Lagoon #4 (Top 6" of core, 70'N, 36'W of spillway)	1,453	1,653	554	<0.3	< 7.2	<7.1
8	Lagoon #4 (Bottom 6" of core approx. half way up the N side of lagoon under 6" water)	< 1.6	18.7	7.8	<0.3	< 7.2	<7.1
9	Lagoon #4 (Top 6" of core approx. half way up the N side of lagoon under 6" water)	10.1	91.2	16.8	<0.3	< 7.2	<7.1
10	Lagoon #4 (Bottom 6" of core, middle of lagoon)	37.9	73.7	14.7	<0.3	< 7.2	<7.1
11	Lagoon #4 (Top 6" of core, middle of lagoon)	40.8	72.6	27.2	<0.3	< 7.2	7.8
12	Lagoon #4 (Bottom 6" of core approx. half way down W side of lagoon)	7.2	21.2	8.6	<0.3	< 7.2	<7.1

** See Appendix E for Analysis Procedures

Table II. Analysis of Samples from Lagoons at Louisiana AAP** (continued)

Sample #	Sample Description	TNT (ppm)	RDX (ppm)	HMX (ppm)	Tetryl (ppm)	Chromium (ppm)	Lead (ppm)
13	Lagoon #4 (Top 6" of core approx. half way down W side of lagoon)	21.8	65.6	12.1	<0.3	<7.2	< 7.1
14	Lagoon #3 (Bottom 6" of core, NE entrance)	44.9	353.4	69.7	< 0.3	<7.2	< 7.1
15	Lagoon #3 (Top 6" of core, NE entrance)	99.5	531.4	1,451	<0.3	<7.2	31.1
16	Lagoon #4 (Water sample E side)	Sample jar broken in shipment					
17	Lagoon #5 (Bottom 6" of core at end of spillway from 4 into 5)	<1.6	26.7	9.7	<0.3	<7.2	< 7.1
18	Lagoon #5 (Top 6" of core at end of spillway from 4 into 5)	<1.6	< 1.4	< 1.4	<0.3	<7.2	<7.1
19	Lagoon #9 (Surface sample 42' W of spillway)	10,812	11,179	4,036	<0.3	8.0	41.8
20	Lagoon #9 (Bottom 6" of core, 100' W of spillway)	118.8	15.0	8.7	<0.3	<7.2	13.1
21	Lagoon #9 (Top 6" of core, 100'W of spillway)	<1.6	9.7	1.4	<0.3	<7.2	10.0
22	Lagoon #9 (Surface sample 66' N of spillway)	12,449	29,871	6,316	<0.3	<7.2	28.3
23	Lagoon #9 (Bottom 6" of core, 70' NE of spillway)	17.1	11.7	9.5	< 0.3	< 7.2	<7.1
24	Lagoon #9 (Top 6" of core, 70' NE of spillway)	1,275	783	98	< 0.3	< 7.2	<7.1
25	Lagoon #9 (Bottom 6" of core, NW side across from spillway)	1,100	461	91.6	< 0.3	< 7.2	< 7.1

Table II. Analysis of Samples from Lagoons at Louisiana AAP** (continued)

Sample #	Sample Description	TNT (ppm)	RDX (ppm)	HMX (ppm)	Tetryl (ppm)	Chromium (ppm)	Lead (ppm)
26	Lagoon #9 (Top 6" of core, NW side across from spillway)	< 1.6	2.6	23.3	< 0.3	<7.2	< 7.1
27	Lagoon #2 (Bottom 6" of core, E side side of lagoon)	124	728	94.4	< 0.3	*	*
28	Lagoon #2 (Top 6" of core, E side of lagoon)	158	646	96.7	< 0.3	*	*
29	Lagoon #2 (Surface sample near spillway under 1' water)	Sample jar broken in shipping					
30	Lagoon #2 (Water sample near spillway)	<0.85pbb	0.21	<0.64ppb	< 5.3ppb		
31	Lagoon #1 (Bottom 6" of core, 12' from spillway)	6,000	3,835	541	< 0.3	<7.2	53.7
32	Lagoon #1 (Top 6" of core, 12' from spillway)	2,056	1,145	198	83	*	*
33	Lagoon #1 (Water sample from near spillway)	0.32	5.8	1.7	< 5.3ppb		
34	Lagoon #10 (Surface sample 10' NW of spillway)	98,034	28,358	7,120	10,039	7.6	26.2
35	Lagoon #10 (Bottom 6" of core, E side of spillway at edge of water)	377	119	25.4	<0.3	< 7.2	11.1
36	Lagoon #10 (Top 6" of core, E side of spillway at edge of water)	747	253	64.4	< 0.3	< 7.2	9.2
37	Lagoon #10 (Water sample near spillway)	117.6	16.4	8.5	1.3	*	*
38	Lagoon #10 (Top 4" of core, 20' from spillway)						

Table II. Analysis of Samples from Lagoons at Louisiana AAP** (continued)

Sample #	Sample Description	TNT (ppm)	RDX (ppm)	HMX (ppm)	Tetryl (ppm)	Chromium (ppm)	Lead (ppm)
39	Lagoon #15 (Bottom 6" of core, 2' W of spillway)	3,661	349	50.7	15.1	< 7.2	< 7.1
40	Lagoon #15 (Top 6" of core, 2' W of spillway)	13,858	376	5	0.3	< 7.2	< 7.1
41	Lagoon #15 (Surface sample, 1' E of spillway)	42,122	11,915	2,676	3,446	11.1	27.1
42	Lagoon #15 (Water sample 3' W of spillway)	6.2	1.8	0.2	< 5.3ppb		
43	Lagoon #9 (Water sample near spillway)	0.12	0.24	1.31	< 5.3ppb		

* Not Analyzed

III. LABORATORY SCALE COMPOSTING TRIALS

A. Materials

1. Composting Materials

Two types of composts were utilized in these studies, and were designated hay-horse feed compost and sewage sludge compost. The hay-horse feed composts were composed of 48% (dry weight basis) chopped alfalfa hay, 48% Purina Sweetena horse feed and 4% seed compost. The seed compost was material from a previously composted mixture of hay and horse feed. The seed was added to provide the microorganisms necessary to initiate rapid composting. The sewage sludge composts were composed of 44% pine wood shavings and 56% sewage sludge from the Arlington Wastewater Treatment Facility in Arlington, Virginia. The sewage sludge was a mixture of primary and secondary sludge with ferric chloride and lime added. The ash content was 35-40%. The sludge itself served as a microorganism seed.

The moisture content of all materials was determined by drying subsamples at 65°C. Materials were sealed in plastic bags to maintain their moisture level. The hay, horse feed, and wood shavings were stored at ambient temperatures; the sewage sludge and hay-horse feed compost seed were maintained at 5°C until used.

2. Contaminated Sediments/Soils

Selected samples from the field survey were pooled for use in the composting trials. Nine samples from Louisiana AAP (sample numbers 1, 2, 3, 4, 5, 19, 22, 34 and 41, Table II, Section II) were selected to obtain the highest possible concentrations of TNT, tetryl, RDX, and HMX. The results of explosives analyses of this pooled sample are presented in Table III.

Table III. Average Levels of Explosives in the Louisiana AAP Sediment Used in the Laboratory Composting Trials.

Explosive	Concentration (ppm) Average	Concentration (ppm) Standard Deviation
TNT	114,600	9,971
RDX	64,205	9,863
HMX	7,043	1,021
Tetryl	5,022	1,271

Four samples of the BAAP soil (sample numbers 2, 4, 12, and 13, Table I, Section II) were pooled to get the highest possible nitrocellulose concentration. Analyses of this pooled soil indicated that nitrocellulose was present at 10,252 ppm (standard deviation 1892 ppm). All samples of soil from Letterkenny AD were pooled to yield one composite sample.

Pooled samples from Louisiana AAP and Badger AAP were air-dried at ambient temperatures by spreading them on aluminum foil in a dark hood. The dried samples were ground and stored in amber bottles. Because of the explosives present in the sediments, oven drying was not considered feasible. Therefore, additions of sediment to composts were based on the air dried weight of the sediments.

3. Compost Seed

A 50:50 mixture of chopped alfalfa hay and Purina Sweetena horse feed was watered to approximately a 60% moisture content, inocculated with fresh horse manure, and allowed to compost for 26 days. The compost was self-sustaining with temperatures ranging from 32-75°C. Samples of this material were stored in sealed plastic bags at 5°C until used to seed the hay-horse feed composts.

4. ^{14}C-Radiolabeled Tracer Compounds

^{14}C-labeled explosives were utilized in all studies as an analytical tool to follow the fate of each explosive in the composts. Uniformly ring-labeled ^{14}C-TNT, uniformly-labeled hexamethylene tetramine, and uniformly ring-labeled N,N-dimethylaniline were obtained from Pathfinder Laboratories. The radiolabeled hexamethylene tetramine was used to synthesize uniformly ring-labeled RDX and HMX (Appendices A and B). Uniformly ring-labeled tetryl was synthesized from the ^{14}C-N,N-dimethylaniline (Appendix C). Uniformly-labeled ^{14}C-cellulose purchased from DuPont NEN Products was utilized to synthesize uniformly-labeled nitrocellulose (Appendix D). The purities and specific activities of these compounds are presented in Table IV. The specific activity of TNT was based on the total activity of the material per unit weight of TNT. The specific activities of the other explosives were calculated as the total activity of the material per unit weight of the material (explosive and impurities).

Table IV. Characteristics of ^{14}C-Labeled Explosives Used in Laboratory Composting Trials

Compound	Labeling Position	Specific Activity (µCi/mg)	Purity	Major Impurity
TNT	Uniform ring	20.96	85.8	-
RDX	Uniform ring	0.381	94.8	HMX (4.0%)
HMX	Uniform ring	0.198	53.4	RDX (40.3%)
Tetryl	Uniform ring	0.076	88.2	-
Nitrocellulose	Uniform	0.158	-	Cellulose (less than 10%)

B. Analytical Methodology

1. Liquid Scintillation Counting

All quantitation of ^{14}C was accomplished using liquid scintillation counting (LSC); however, two methods of quench correction were employed. Routine measurements, used to assay ^{14}C-explosive stock solutions, condensates from composts, acid and base traps, and TLC scraping, were made with the counting window set at 300-655. The lower limit of the window was set to avoid all chemical fluorescence. The H number (used in conjunction with a ^{137}Cs external standard) was utilized to measure quench, and the counting window was automatically adjusted for spectrum shift using the automatic quench control option. A calibration curve to relate the H number to counting efficiency was constructed using sealed quenched and unquenched standards.

The H number was found to be somewhat inaccurate when counting compost extracts or samples from the Tri-Carb oxidizer. The high concentration of organics in the compost extracts and the highly basic ethanolamine in oxidizer samples result in a shift in the Compton distribution which is not completely predicted by the H number. Therefore, all these samples were counted with window settings of 75-655, not using the automatic quench control. A known quantity of ^{14}C-activity was then added to each sample as an internal standard, and the samples were recounted. Increase in the measured activity of each sample was used as the counting efficiency.

All samples, whether using the H number or the internal standard quench correction, were counted in Beckman Ready-Solv MP cocktail until a 2σ error of 2% was obtained or until counting time reached 15 minutes. Calculations for background subtraction, quench, dilution, concentration, and subsampling were done using a program run on an HP-3000 computer or a TI-58 programmable calculator to minimize computational errors.

2. ^{14}C-Product Identification and Quantitation

Product characterization of the compost extracts and purity assays for radiolabeled TNT, RDX, HMX and tetryl were accomplished using thin layer chromatography. Silica gel plates were used for all analyses. The TLC methodology and solvent systems used are presented in Table V. Authentic standards were run adjacent to the samples, and spots were identified by comparing R_f values with those of the standards. Radioactive spots were visualized by autoradiography and were quantified by scraping and liquid scintillation counting.

Table V. Methods of TLC Analysis Used for ^{14}C-Explosive Products Identification

Compound	TLC Analysis Procedure	Solvent Systems
TNT	2-dimensional linear	benzene:hexanes:pentane:acetone (50:40:10:3) petroleum ether:ethyl acetate:hexanes (160:80:25)
RDX	linear	acetonitrile:methylene chloride (25:75)
HMX	linear	acetonitrile:methylene chloride (25:75)
tetryl		
purity check	2-dimensional linear	benzene:hexanes:pentane:acetone (50:40:10:3) petroleum ether:ethyl acetate:hexanes (160:80:25)
compost extracts	circular	benzene:methylene chloride:acetonitrile: hexanes (40:40:5:5)

The following standards were used for TLC product identification:

TNT analysis:	2,4,6-trinitrotoluene (TNT)
	2-amino-4,6-dinitrotoluene (2-amino-DNT)
	4-amino-2,6-dinitrotoluene (4-amino-DNT)
	2,6-diamino-4-nitrotoluene (2,6-diamino-NT)
	2,2',6,6'-tetranitro-4,4'-azoxytoluene (tetra)
RDX and HMX analysis:	hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
	1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
Tetryl analysis:	N-methyl-N,2,4,6-tetranitroaniline (tetryl)

3. Residual ^{14}C-Quantitation

Radioactivity remaining in the compost after solvent extraction (residual ^{14}C) was determined by combusting duplicate subsamples of the dried, ground compost, collecting the resultant CO_2 released, and quantitating the ^{14}C activity associated with the CO_2 using LSC. After solvent extraction all compost samples were freeze dried and then finely ground (0.050 inch screen) in a hammer mill. Subsamples were combusted in a Packard Tri-Carb oxidizer, which automatically collects and dispenses the $^{14}CO_2$ released into a scintillation vial for counting. Oxidizer operation and efficiency were checked by combusting samples spiked with a known quantity of ^{14}C-nitrocellulose. Sample carry over was measured by combusting several blank samples (non-radioactive cellulose) randomly placed in each run. All data were corrected for carry over (background), combustion efficiency, and counting efficiency.

4. Quantitation of ^{14}C Trapped by Activated Carbon

Random subsamples of carbon from the air intake end of the trap were combusted and the ^{14}C released was quantitated by LSC. The combustion procedure used for carbon was the same as employed for residual ^{14}C in the compost (as described in the previous section).

5. Preliminary Extraction Trials

Tests were conducted with all ^{14}C-labeled explosives to evaluate procedures for extracting the explosives out of the different composts. In these tests, sediment spiked with a ^{14}C-explosive was mixed either with composting materials to give a time zero compost or mixed into composted materials to simulate a sample having undergone composting. Both hay-horse feed and sewage sludge composts were utilized. The proposed protocol for the LAAP sediment was to extract three times with acetonitrile. Recovery of TNT with acetonitrile as the extractant was somewhat low (75-85%) for use in a radiolabeled metabolism study. It was determined that extraction with methanol:benzene (1:3) followed by three benzene extractions gave consistently good recovery of TNT. RDX and HMX were adequately extracted with acetonitrile.

Acetonitrile extraction of composts spiked with ^{14}C-tetryl was partially effective in solubilizing the ^{14}C, but analysis of the extracts showed that very little of the ^{14}C was associated with the parent tetryl molecule. Initially, it was believed that tetryl was not stable in compost, and efforts were initiated to isolate and identify the ^{14}C-products in the acetonitrile extracts (Section III-B-6). However, it was discovered that tetryl was not stable in the acetonitrile compost extracts. Benzene, methylene chloride, acetone, and ethanol were tested as extractants. Benzene was found to be an acceptable solvent to extract tetryl out of compost.

Extraction trials using acetone to recover nitrocellulose from compost containing BAAP soil gave mixed results. Recovery of ^{14}C from hay/horse feed compost spiked with ^{14}C-nitrocellulose was 66-69%, but recovery from sewage sludge composts ranged from 19-25%. Acetone is the only known solvent that readily solubilizes nitrocellulose; therefore, other solvents were not tested as extractants.

These studies demonstrated that sonication of samples during extraction increased the extraction efficiency. Therefore, the standard procedure used to extract all explosives was to extract four times @ 35-40^{o}C using sonication.

6. Tetryl Product Characterization

HPLC (Appendix H) and TLC (Section III-B-2, linear one dimensional analysis using benzene:methylene chloride:acetonitrile:hexanes (40:40:5:5) analyses of time zero extracts of LAAP-sediment composts spiked with ring-labeled ^{14}C-tetryl separated 20-30 isolated containing the ^{14}C label. The last of these isolated eluted with retention times of greater than 30 minutes. Initially it was believed that tetryl was not stable in compost and fractions were collected from HPLC separation and characterized by GC-MS. Only one compound produced a GC peak. It was tentatively identified as dimethyldinitrophenylenediamine. During this study, it became apparent that the ^{14}C products in the acetonitrile compost extract were not stable, suggesting that tetryl was not stable in the extract. Extraction trials using benzene demonstrated that tetryl does not degrade immediately in compost materials.

7. Analysis of NaOH Traps

Tests were conducted to confirm that the ^{14}C activity in the NaOH traps predominantly resulted from trapped $^{14}CO_2$. Subsamples of traps containing a substantial amount of ^{14}C were reacted with sufficient $BaCl_2$ to precipitate all $^{14}CO_2$ as Ba $^{14}CO_3$. The solutions were centrifuged, and an aliquot of the clear supernatant was assayed for ^{14}C activity. Results demonstrated that in more than 95% of the samples, all ^{14}C activity resulted from $^{14}CO_2$ evolved from the composts. In no instance was a substantial amount of ^{14}C found in a NaOH trap that could not be attributed to $^{14}CO_2$. Therefore, all ^{14}C activity detected in the NaOH traps is reported as $^{14}CO_2$.

8. Nitrocellulose Analysis

No method currently exists to separate and purify nitrocellulose; therefore, radiochemical quantitation of ^{14}C-nitrocellulose is not possible. A non-specific colorimetric test (Appendix J) that is a modification of the USATHAMA method for soil analysis was used to quantify nitrocellulose.

9. TCE Quantitation

Unlabeled reagent grade TCE was used for all laboratory tests. TCE in soil, compost, or cold traps was extracted into methanol, placed in sealed containers, and maintained at 5°C until analyzed. Quantitation of TCE in the methanol was by gas chromatography using a Hall detector (Appendix K).

C. Laboratory Composting Experiments

1. Experimental Design

Separate experiments were set up to study the fate of individual explosives (TNT, RDX, HMX, and tetryl) in the LAAP sediment and of nitrocellulose in the BAAP sediment during composting. In each experiment, a measured quantity of one of the explosives containing the ^{14}C-label was added to the LAAP or BAAP sediment. This spike did not measurably change the concentration of explosive in the sediment (except for nitrocellulose where the concentration was increased by approximately 20%), but introduced the ^{14}C-label which was utilized to follow breakdown of the single explosive.

Each experiment was setup with three independent variables. The first variable was sampling time. Compost samples were extracted and analyzed for explosives immediately after being setup (time zero) and after a given period of composting. The second variable was compost materials; two types of composts, hay-horse feed and sewage sludge-wood chips, were utilized. The third variable was sediment loading level. The contaminated sediment was added at levels equivalent to 10, 18 and 25% of the total dry weight of the compost.

Duplicates of all compost treatments were setup except the time zero composts which had only one replicate. Data were analyzed in a two way analysis of variance. Time zero data were used to determine the effect of composting on explosive concentration. Differences between means were assessed where appropriate using the Student-Newman-Keuls Multiple range test. No statistical tests were performed to compare explosives remaining after composting to recovery from time zero composts. Rather, an average 30% decrease in explosives attributed to composting was considered a significant reduction in terms of decontaminating the sediment.

2. Experimental Procedures

a. Compost Setup

Individual composts were setup in 1-quart Mason jars. The hay-horse feed composts (50 g dry weight) were watered to obtain a moisture content of approximately 65% (wet weight basis). The sewage sludge composts (70.2 g dry weight) contained 59% moisture and did not require additional water.

Aliquots (5.6 - 23.2 g) of the LAAP or BAAP sediment were weighed into beakers to be individually added to separate composts. Each aliquot was spiked with a ^{14}C-labeled explosive in a small (less than or equal to 0.5 mL) amount of solvent (benzene for TNT and tetryl, acetonitrile for RDX and HMX and acetone for nitrocellulose). The solvent was allowed to evaporate at ambient temperatures for 1-2 hours. Each soil was throughly mixed and then added to a quart jar containing compost materials. Soil remaining in the beaker was washed into the compost with 5 mL of water and the treated composts were thoroughly mixed. Any ^{14}C activity remaining in the beakers was dissolved in 5 mL of acetonitrile for RDX, HMX, TNT and tetryl or acetone for nitrocellulose and was quantitated by LSC. The final compost mixtures had moisture contents of 55 to 60% and contained 10, 18, or 25% sediment (dry weight basis).

A ring of 1/4-inch O.D. polyethylene tubing with holes drilled at 1/4-inch intervals was positioned underneath the compost in each jar, and a thermocouple was inserted into the center of each compost. The jars were stoppered, placed in an incubator held at 60°C and connected with tygon tubing to the aeration system illustrated in Figure 4. Air was continuously drawn through this apparatus to aerate the compost. Air entering the compost was first scrubbed with NaOH to remove CO_2. Offgasses from the compost were scrubbed through 1.8 N H_2SO_4 (to collect volatile amines) and through NaOH (to collect $^{14}CO_2$). The offgasses were then passed through a drying tube ($CaSO_4$) and a tube of activated carbon to trap volatile aromatics.

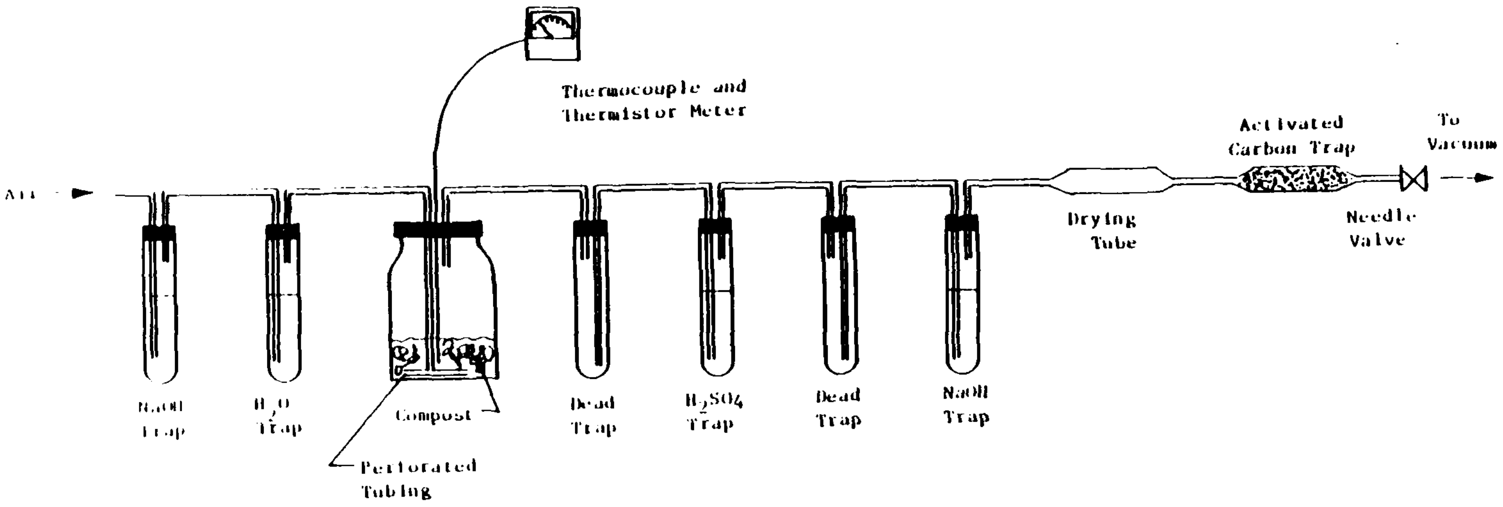

Figure 4. Schematic of ^{14}C Bench-Scale Composting Apparatus

b. Compost Monitoring

Temperatures in each compost and air temperatures in the incubator were checked and recorded daily. Air flow rates through the aeration system were checked and adjusted as needed several times a day. Water was added to the composts on an "as needed basis" to maintain microbial activity.

Water condensate collected in the first dead trap down stream from the compost in the aeration system was removed as needed to prevent spillover into the H_2SO_4 traps. The NaOH traps were replaced as needed to prevent saturation of the traps. The indicator Tropalein-O was added to the NaOH traps to provide a visual warning of when the traps were near the saturation level. Subsamples of both the water condensates and the NaOH traps were assayed for ^{14}C-activity using LSC.

c. Compost Analysis

The analyses for all LAAP and BAAP composts are outlined in Figures 5 and 6. Radioactivity in the water condensates and NaOH traps was quantitated every 1-4 days during composting. Periods of composting varied with the explosive being studied. Composts spiked with ^{14}C-TNT were incubated 36 days, those with ^{14}C-HMX or ^{14}C-RDX were composted 70 days; composts containing ^{14}C-tetryl were composted 44 days and compost containing ^{14}C nitrocellulose were composed 42 days. After the composting period, the composts were removed from the incubator, sealed, and stored at 5°C until extracted. The acid traps were subsampled to quantitate their ^{14}C-content. A number of carbon traps from the hay-horse feed and sewage sludge composts for each explosive were analyzed (combustion and LSC) for the presence of ^{14}C. Radiation levels were at or near background on all carbon traps, tested. ^{14}C-activity was assumed to be negligible for all carbon traps and the remaining traps were not analyzed.

Prior to extraction the composts were removed from cold storage and allowed to warm to ambient temperature. The composts were then extracted in the Mason jar in which they were composted. Sufficient solvent was added to cover the compost materials in the jar. Each compost was extracted four times at 35-40°C using sonication (Ultrasonic generator, Model 2705, used on highest setting). Extracts were vacuum filtered

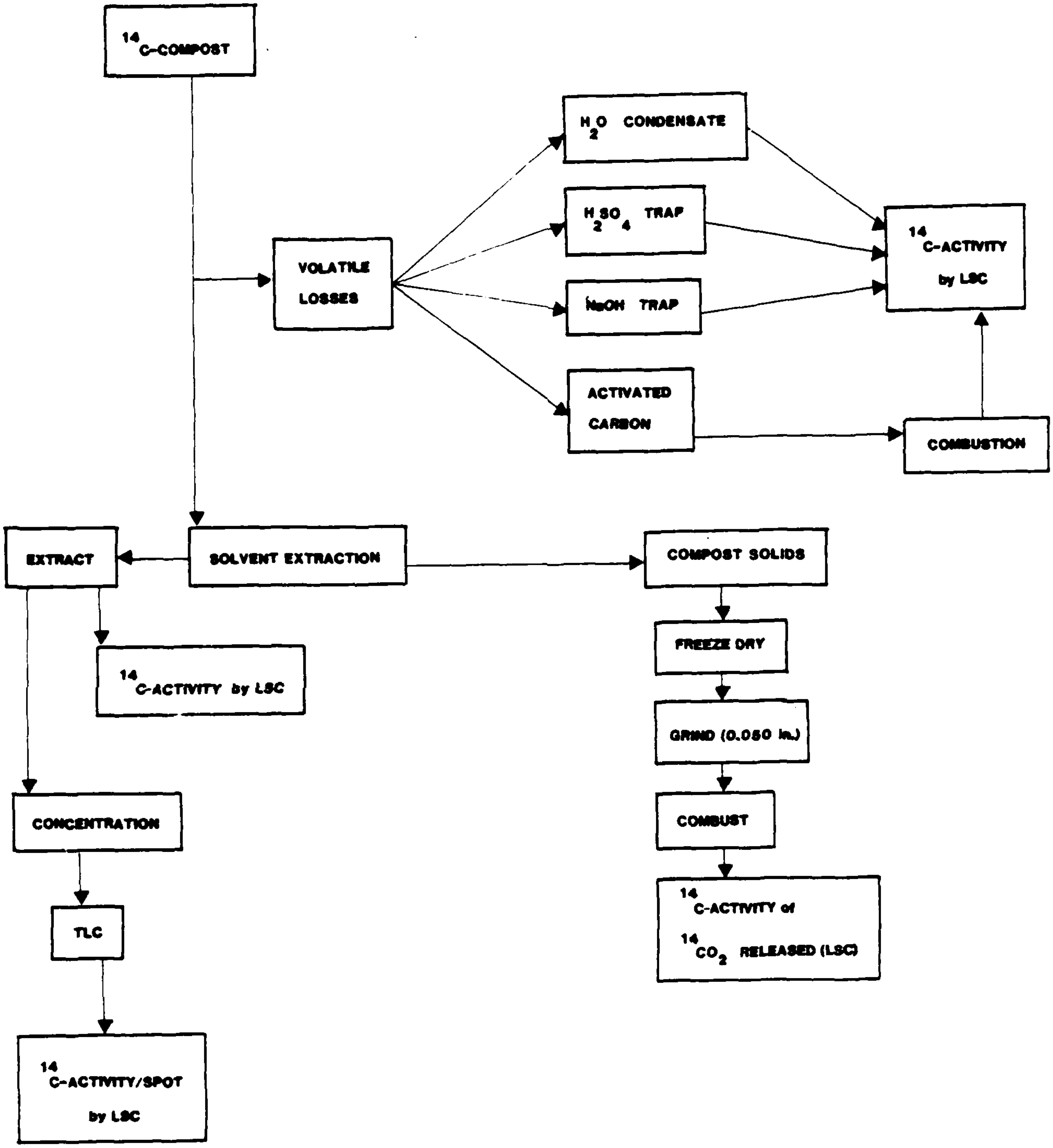

Figure 5. Outline of Louisiana AAP Laboratory Compost Analyses

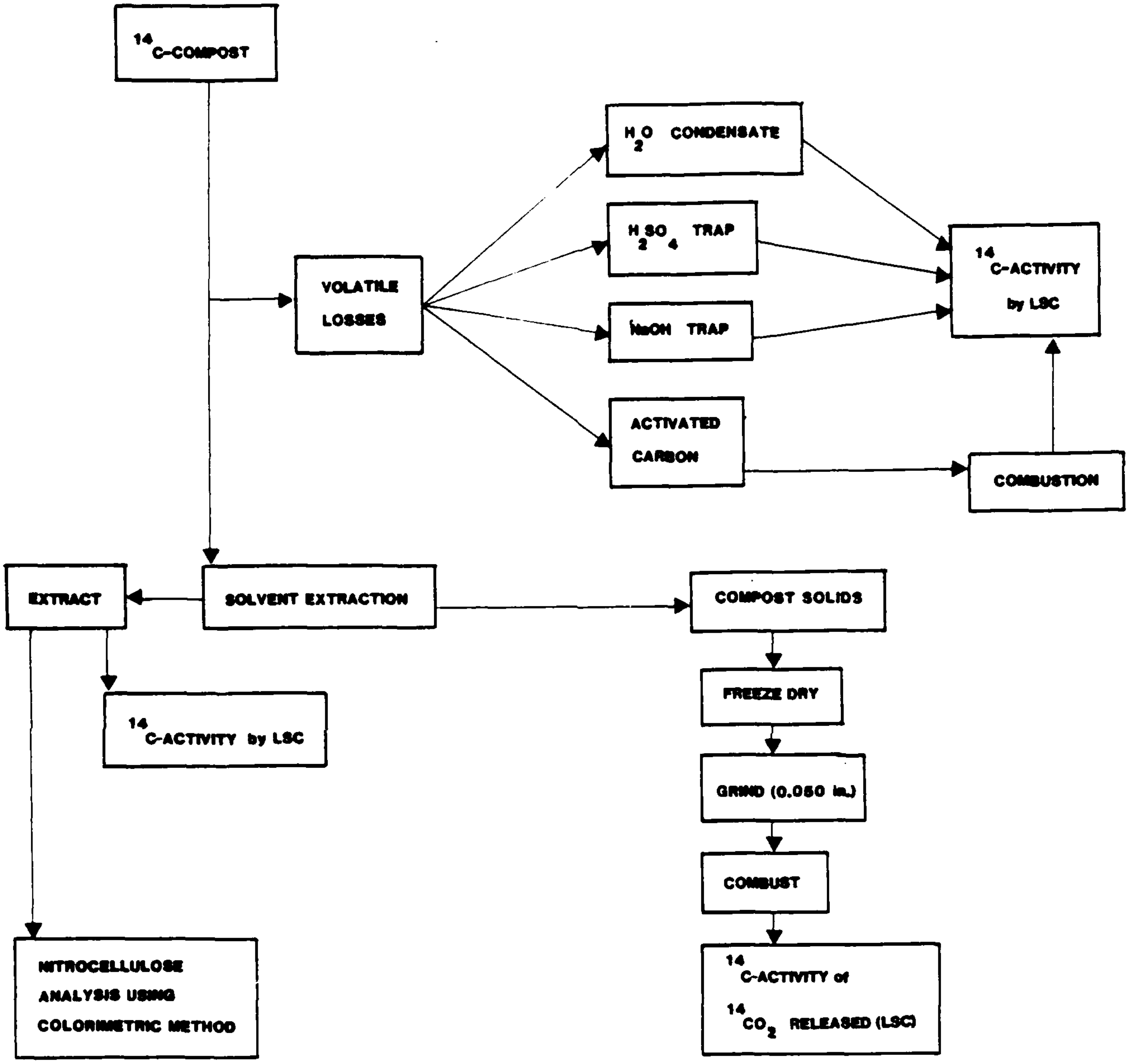

Figure 6. Outline of Badger AAP Laboratory Compost Analyses

through Whatman No. 1 or No. 2 filter paper. With some hard to filter samples a prefilter (Whatman No. 4) was used to speed up the filtration process. The extracts were combined and brought up to a given volume using the extract solvent. The solvents used for each explosive are as follows:

TNT:	Benzene:Methanol (3:1) 1 extraction Benzene 3 extractions
HMX:	Acetonitrile
RDX:	Acetonitrile
Tetryl:	Benzene
Nitrocellulose:	Acetone

The combined extracts for each compost were subsampled to quantitate the ^{14}C-activity in the extract (LSC). An aliquot (50-120 mL) from the extracts was evaporated to dryness at 35-40°C using a nitrogen stream. The residue was washed with an appropriate solvent (benzene for TNT and tetryl, acetonitrile for RDX and HMX,) and the volume of the wash was reduced (evaporation under N_2) to approximately 0.5 mL. These concentrates were analyzed by TLC, using autoradiography and LSC to locate and quantitate all radioactive constituents in the extract. Identification of the parent explosive and selected degradates or impurities was made by comparing R_f values with authentic standards.

The compost solids remaining after extraction were freeze dried, ground in a hammer mill to pass a 0.050-inch screen, and combusted to quantitate the residual ^{14}C-activity.

D. Results of Composting of Louisiana AAP Sediment

1. TNT

Composting of the LAAP sediment composts spiked with ring-labeled ^{14}C-TNT proceeded normally. All composts appeared to have composted. Anaerobic conditions or build up of organic acids were not observed. Throughout the 36 day composting period, compost temperatures remained within 2°C of the incubator temperature.

The distribution of ^{14}C recovered from the composts is summarized in Table VI. The results of the TLC product identification of ^{14}C extracted from the composts are given in Table VII. Losses of TNT due to composting were significant for all treatment combinations. The slowest rates of TNT loss occurred in the hay-horse feed composts amended with LAAP sediment at rates of 18 and 25%. TNT loss in these composts was greater than or equal to 60%. Significantly higher rates of TNT loss were found in the hay-horse feed compost with 10% sediment and in all the sewage sludge composts, where greater than or equal to 99% on the average of TNT was lost within 36 days. Accumulation of TNT transformation productions was minimal. Less than 2% of the ^{14}C was recovered as 2-amino-4,6-dinitrotoluene and less than 1% as 4-amino-2,6-dinitrotoluene. The 2,6-diamino analog of TNT could not be identified on the TLC because of streaking of organics from the origin. An area of the TLC plates encompassing the 2,6-diamino-4-nitrotoluene location was assayed for ^{14}C. Less than 1.4 % of the total activity could be associated with this compound. No other dominant ^{14}C-labeled compound was isolated from the compost extracts.

Loss of TNT was closely correlated to a reduction of the extractable ^{14}C and an increase in the residual ^{14}C. Losses of $^{14}CO_2$ were generally low (less than or equal to 2.4% average value) indicating little to no cleavage of the benzene ring in TNT. Overall loss of ^{14}C as $^{14}CO_2$ was statistically higher in the sewage sludge composts where TNT loss was greatest. Other volatile losses of ^{14}C were inconsequential.

Extraction of time zero samples indicated that the extraction procedures were effective. Overall recovery of ^{14}C average 102.0% with a standard deviation of 6.9%.

2. RDX

LAAP sediment composts spiked with ring-labeled ^{14}C-RDX were initially placed in the bottom of the incubator only a short distance above the heating coils. As a result the composts received excessive heat. During the first six days of the composting temperatures in the compost ranged from 63-75°C (Appendix L), and the composts turned black on the bottom. On day six of the study, the composts were raised off of the bottom of the incubator. Temperatures in the compost thereafter were generally

Table VI. Distribution of ^{14}C in Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-TNT

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of added)					
			$^{14}CO_2$	H_2O Condensate	Acid Trap	Extract	Unextracted Residual	Total
HHF	10%	0	-	-	-	88.2	9.5	97.7
	18%	0	-	-	-	93.0	5.4	98.4
	25%	0	-	-	-	91.8	11.5	103.3
SS	10%	0	-	-	-	89.6	8.5	98.1
	18%	0	-	-	-	89.2	7.4	96.6
	25%	0	-	-	-	98.6	6.9	105.5
HHF	10%	36	0.7b**	0.0a***	0.0a	7.8a	100.3a	108.8
	18%	36	0.3b	0.0a	0.0a	30.8a	75.2a	106.3
	25%	36	0.9b	0.0a	0.0a	21.6a	77.8a	100.3
SS	10%	36	2.4a	0.0a	0.0a	1.4b	94.4a	98.2
	18%	26	1.9a	0.0a	0.0a	1.6b	97.4a	100.9
	25%	36	2.2a	0.0a	0.0a	2.3b	97.6a	102.1

* HHF - hay-horse feed composts; SS - sewage sludge composts

** Values followed by the same letter are not significantly different at the 5% levels of probability according to the Student-Newman-Kuel multiple range test. Data from samples composted zero days were excluded from statistical analyses.

*** Value less than 0.05%

Table VII. TLC Analysis of the Benzene Extracts of Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-TNT

Compost†	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of added)					
			TNT	2-amino* DNT	4-amino* DNT	2,6-diamino* NT	Tetra*	Origin
HHF	10%	0	84.5	ND**	ND	ND	ND	ND
	18%	0	84.0	ND	ND	ND	ND	0.6
	25%	0	90.9***	ND	ND	ND	ND	0.9
SS	10%	0	80.3	ND	ND	ND	ND	1.6
	18%	0	77.0	ND	ND	ND	4.7	1.2
	25%	0	85.2	ND	ND	ND	ND	2.0
HHF	10%	36	1.9b*****	1.1a	0.4a	1.4****	0.4a	4.0
	18%	36	21.9a	1.9a	0.8a	ND	1.4a	2.0
	25%	36	14.9a	1.0a	0.7a	ND	1.6a	1.8
SS	10%	36	NDb	0.1b	0.1b	0.5	NDb	0.6
	18%	36	NDb	0.3b	0.4b	0.4	NDb	0.4
	25%	36	0.3b	0.5b	0.3b	0.6	0.3b	0.2

† HHF - hay-horse feed compost; SS - sewage sludge compost

* 2-amino DNT = 2-amino-4,6-dinitrotoluene
4-amino DNT = 4-amino-2,6-dinitrotoluene
2,6-diamino = 2,6-diamino-4-nitrotoluene
tetra = 2,2',6,6'-tetranitro-4,4'-azoxytoluene

** ND = Not detected

*** Two tailed spot on chromatogram, may include the tetranitroazoxytoluene and an unknown impurity

**** Indistinct spot which may include 2,6-diamino-4-nitrotoluene

***** Values for TNT composted 36 days followed by the same letter are not significantly different at the 5% level of probability according to the Student-Newman-Kuel multiple range test

55-65°C. Individual composts tended to maintain a temperature consistently higher or consistently lower than the incubator temperature. No evidence of anaerobic conditions or organic acid build up was observed.

The results from composting ^{14}C-RDX are presented in Tables VIII and IX. RDX was substantially reduced in all composts. The rate of breakdown appeared to be inhibited by increased rates of sediment addition. This inhibitory effect was readily apparent in the hay-horse feed composts. In the sewage sludge compost, average losses of RDX were decreased somewhat by increased sediment addition, but these differences were not statistically significant (5% level of probability).

In composts where greater than 50% of the RDX was degraded, a substantial quantity of the ^{14}C was evolved as $^{14}CO_2$ (31-64% of the ^{14}C from degraded RDX). The rates of $^{14}CO_2$ evolution are illustrated in Figure 7. The large percentage of ^{14}C evolved as $^{14}CO_2$ demonstrated that breakdown of the RDX molecule was extensive. The radiolabeled carbon from degraded RDX that was not volatilized as $^{14}CO_2$ was largely recovered as residue that could not be extracted from the compost with acetonitrile. ^{14}C found in the water condensate or acid traps was less than or equal to 0.4% of the added carbon for any sample. No substantial quantities of any ^{14}C-degradate were found in the compost extracts. Small quantities (less than 2%) of ^{14}C were found in a single spot in the TLC analysis of the sewage sludge composts. The spot had an R_f of 0.12 indicating the presence of compound(s) more polar than RDX (R_f=0.87). No attempt was made to identify the constituent(s) of this spot.

^{14}C-HMX was present as an impurity in the ^{14}C-RDX used to spike the composts (4.0% HMX by radiochemical analysis). Recoveries of ^{14}C-HMX in the solvent extracts suggested that HMX was degraded during the 70 days of incubation. Losses of HMX were inversely related to rates of sediment addition. In the hay-horse feed composts, approximately 75% reduction of HMX occurred in the 10% sediment composts, but no loss of HMX was observed in the composts with 18 or 25% sediment. HMX losses in the sewage sludge composts amended with 10, 18 and 25% sediment were 65, 52, and 35%, respectively.

Table VIII. Distribution of ^{14}C in Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-RDX

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of added)					
			$^{14}CO_2$	H_2O Condensate	Acid Trap	Extract	Unextracted Residual	Total
HHF	10%	0	-	-	-	94.0	-	-
	18%	0	-	-	-	86.5	12.6	99.1
	25%	0	-	-	-	83.1	14.4	97.5
SS	10%	0	-	-	-	93.1	-	-
	18%	0	-	-	-	89.2	25.0	114.2
	25%	0	-	-	-	98.6	9.6	108.2
HHF	10%	70	46.5a**	0.1b	0.2	5.6b	29.7b	82.1
	18%	70	4.0c	0.1b	0.1	70.9a	24.4b	99.5
	25%	70	0.8c	0.1b	0.0	69.5a	25.8b	96.2
SS	10%	70	37.2a	0.2a	0.2	11.0b	54.6a	103.2
	18%	70	24.2b	0.2a	0.1	15.5b	66.8a	106.8
	25%	70	26.0b	0.2a	0.1	25.4b	46.6a	98.3

* HHF - hay-horse feed compost; SS - sewage sludge composts

** Values followed by the same letter are not significantly different at the 5% level of probability according to the Student-Newman-Kuel multiple range test. Data from samples composted zero days were excluded from statistical analyses.

Table IX. TLC Analysis of the Acetonitrile Extracts of Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-RDX

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of Added)			
			RDX	HMX	Origin	R_f 0.12
HHF	10%	0	90.3	3.6	0.1	ND**
	18%	0	83.5	3.1	0.0***	ND
	25%	0	80.1	3.1	0.0	ND
SS	10%	0	89.7	3.4	0.0	ND
	18%	0	85.8	3.7	0.1	ND
	25%	0	92.3	4.9	0.8	ND
HHF	10%	70	4.6b****	0.8c	0.2a	ND
	18%	70	67.3a	3.3a	0.3a	ND
	25%	70	65.8a	3.3a	0.4a	ND
SS	10%	70	8.7b	1.4bc	0.2a	0.6
	18%	70	12.8b	1.9b	0.4a	0.3
	25%	70	21.2b	2.6a	0.4a	1.2

* HHF - hay-horse feed composts; SS - sewage sludge composts

** ND = Not detected

*** Value less than 0.05%

**** Values followed by the same letter are not significantly different at the 5% level of probability according to the Student-Newman-Kuel multiple range test. Data from samples composted zero days were excluded from statistical analyses.

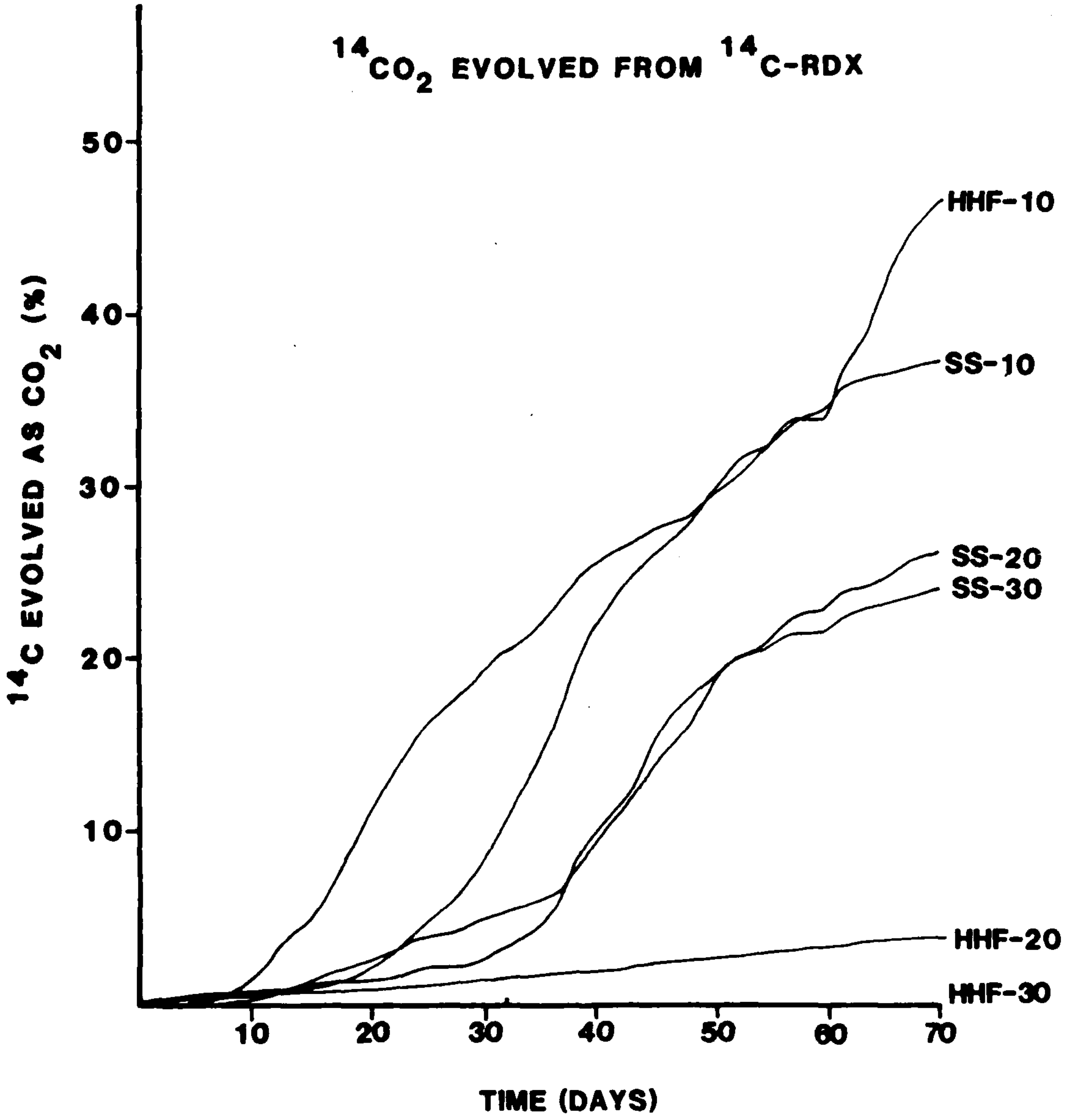

Figure 7. $^{14}CO_2$ Evolved From Louisiana AAP Composts Spiked with Ring-UL ^{14}C-RDX

The average overall recovery of ^{14}C was 99.5% with a standard deviation of 8.8%. The efficiency of the extraction procedure, as determined by time zero extracts, varied somewhat, but was generally high (90.8% average recovery with a standard deviation of 5.6%).

3. HMX

LAAP sediment composts spiked with ^{14}C-HMX composted normally with temperatures ranging from 57 to 64°C. One replicate of the hay/horse feed compost with 18% sediment dried out after 49 days of composting and could not be effectively rehydrated. Anaerobic conditions or the build up of organic acids were not detected.

The interpretation of the data from the composts spiked with ^{14}C-HMX is complicated because HMX accounted for 53.4% of the ^{14}C in the spiking solutions and RDX accounted for 40.3% of the ^{14}C. Summaries of the data are presented in Tables X and XI. Approximately 30-50% of the HMX in the sewage sludge composts was lost during composting. The breakdown of HMX appeared to be related to the rate of sediment addition to the compost, but differences in HMX degradation between sediment loading rates were not significant at the 5% level of probability. No HMX breakdown was observed in the hay-horse feed composts.

RDX degradation in the sewage sludge composts was similar to but somewhat slower than that observed in the composts spiked with ^{14}C-RDX (Table IX). The breakdown of RDX in hay-horse feed composts was slow at all levels of sediment addition (less than 25% breakdown in 70 days).

In this study it was not possible to distinguish between ^{14}C from HMX and RDX after the parent molecule was altered. Breakdown products for both RDX and HMX include $^{14}CO_2$, and residues in the compost that could not be extracted with acetonitrile. No radiolabeled degradates were found in the solvent extracts. Volatile losses of ^{14}C other than as $^{14}CO_2$ were small.

A radiolabeled impurity in the ^{14}C-HMX spiking solution was found at an R_f of 0.44 on the thin layer chromatograph. The impurity was not identified, but it did appear to breakdown during composting.

Table X. Distribution of ^{14}C in Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-HMX

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of added)					
			$^{14}CO_2$	H_2O Condensate	Acid Trap	Extract	Unextracted Residual	Total
HHF	10%	0	-	-	-	83.1	10.5	93.6
	18%	0	-	-	-	87.5	9.4	96.9
	25%	0	-	-	-	79.8	15.8	95.6
SS	10%	0	-	-	-	83.0	18.2	101.2
	18%	0	-	-	-	76.6	15.0	91.6
	25%	0	-	-	-	82.7	12.3	95.0
HHF	10%	70	0.4b**	0.1b	0.0b	76.8a	19.0b	96.3
	18%	70	0.6b	0.1b	0.0b	74.8a	20.6b	96.1
	25%	70	0.6b	0.1b	0.0b	71.6b	27.6b	99.9
SS	10%	70	31.1a	0.6a	0.2a	27.1d	42.8a	101.8
	18%	70	14.6b	0.2a	0.1a	34.2d	50.6a	99.7
	25%	70	13.4b	0.2a	0.1a	43.3c	43.6a	100.6

* HHF - hay-horse feed composts; SS - sewage sludge composts

** Values followed by the same letter are not significantly different at the 5% level of probability according to the Student-Newman-Kuel multiple range test. Data from samples composted zero days were excluded from statistical analyses.

*** Values reported as 0.0% were less than 0.05%

Table XI. TLC Analysis of the Aceonitrile Extracts of Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-HMX

			^{14}C Recovered (% of added)				Normalized Recovery (%)**	
Compost*	Amount of Sediment Added (dry wt%)	Composting Time (days)	HMX	RDX	Origin	R_f 0.44	HMX	RDX
HHF	10%	0	42.2	37.6	0.2	3.7	-	-
	18%	0	45.7	37.6	0.1	4.0	-	-
	25%	0	42.1	33.9	0.1	3.6	-	-
SS	10%	0	45.0	34.3	0.1	3.6	-	-
	18%	0	41.6	31.0	0.1	3.9	-	-
	25%	0	47.3	31.1	0.2	4.2	-	-
HHF	10%	70	44.5a***	30.2a	ND****	2.2	101.1a	83.4a
	18%	70	43.2a	29.4a	0.1	2.2	98.4a	81.2a
	25%	70	41.7a	27.8a	0.2	1.9	94.8a	76.8a
SS	10%	70	21.6b	3.7c	0.5	1.2	49.2b	11.2c
	18%	70	23.4b	9.4b	0.1	1.2	52.8b	29.4b
	25%	70	30.6b	10.4b	0.6	1.7	69.7b	32.5b

* HHF - hay-horse feed composts; SS - sewage sludge composts

** HMX and RDX normalized to average recoveries from time zero composts

*** Values followed by the same letter are not significantly different at the 5% level of probability according to the Student-Newman-Kuel multiple range test. Data from samples composted zero days were excluded from statistical analyses.

**** ND = Not detected

The overall recovery of ^{14}C was very good, with an average recovery of 97.9% and a standard deviation of 3.6%. The extraction procedure gave consistent recoveries of ^{14}C. Average recovery from time zero samples was 82.1% with a standard deviation of 3.7%.

4. Tetryl

Composts containing LAAP sediment spiked with ^{14}C-tetryl composted normally with no indication of organic acid build up and no evidence of anaerobic conditions. Temperatures in the composts (55 -61°C) tended to be somewhat lower than the incubator temperature (59-62°C).

The distribution of ^{14}C from ring-labeled ^{14}C-tetryl breakdown during 44 days of composting is presented in Table XII. Loss of tetryl was substantial and did not vary significantly with composting treatments. Extracted ^{14}C-tetryl accounted for an average of 3.7 and 2.6% of the total added radioactivity in the hay-horse feed and sewage sludge composts, respectively. However, some error in the final tetryl levels resulted from inadequacies of the extraction and TLC methodologies. Extraction efficiency as indicated by ^{14}C extracted from the time zero composts was low (42-68% recovery) in the sewage sludge composts, and tetryl recovery from the 44 day composts may be similarly low. The TLC procedure used for the compost extracts did not separate ^{14}C-tetryl from its primary impurity (unidentified) found in the spiking solution (7.5% of the total activity). Thus, it is not possible to determine if a portion or all of the ^{14}C reported as tetryl was tetryl or an impurity. Despite these errors, 90-100% loss of tetryl was demonstrated.

Loss of ^{14}C-tetryl was accompanied by an increase in the ^{14}C in the unextracted residue. Extractable ^{14}C was primarily composed of ^{14}C-tetryl; only small quantities ($\leq$0.6% of the total ^{14}C) of any individual degradate (unidentified) were solvent extracted. ^{14}C recovered as $^{14}CO_2$ accounted for 1.6-5.0% of the total ^{14}C. Because of the impurities present in the spiking solution (10.4%) it cannot be determined if any $^{14}CO_2$ evolution resulted from tetryl breakdown. The overall recovery of ^{14}C was good. Average recovery was 94.3% with a standard deviation of 6.7%.

Table XII. Distribution of ^{14}C Louisiana AAP Sediment Composts Spiked with Ring-UL ^{14}C-Tetryl

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of Added)						
			$^{14}CO_2$	H_2O Condensate	Acid Traps	Extract	^{14}C-tetryl	Unextracted Residual	Total
HHF	10%	0	-	-	-	84.0	83.7	11.3	95.3
	18%	0	-	-	-	78.4	77.2	8.6	87.0
	25%	0	-	-	-	81.8	81.1	9.4	91.2
SS	10%	0	-	-	-	45.6	41.9	50.4	96.0
	18%	0	-	-	-	56.9	53.1	24.9	81.8
	25%	0	-	-	-	68.1	64.4	21.1	89.2
HHF	10%	44	5.0	0.1	0.0**	0.1	ND***	84.2	89.4
	18%	44	3.0	0.1	0.0	4.6	4.0	89.2	96.9
	25%	44	2.1	0.0	0.0	7.6	7.1	90.1	99.8
SS	10%	44	3.2	0.2	0.0	3.2	2.7	92.6	99.2
	18%	44	3.4	0.1	0.0	4.3	3.8	96.0	103.8
	25%	44	3.9	0.1	0.0	1.8	1.2	96.5	102.3

* HHF - Hay-horse feed compost; SS - sewage sludge compost

** Values less than 0.05%

*** ND = Not detected by TLC analysis

5. Discussion and Conclusions

The laboratory studies demonstrated that composting effectively eliminated the major contaminants in the lagoons at Louisiana AAP. Both the type of composting material and the level of sediment addition significantly influenced the rate at which the explosives were destroyed.

The sediment generally had an adverse effect on degradation of explosives by composting. This effect was most pronounced in the hay-horse feed composts, and may have retarded breakdown to a lesser degree in the sewage sludge composts.

The two primary contaminants in the sediment, TNT and RDX, responded similarly to the composting treatments. Breakdown was most rapid in the hay-horse feed composts amended with sediment at the 10% level and in the sewage sludge composts. Breakdown of these compounds in the hay-horse feed composts was significantly retarded by the 18 and 25% levels of sediment addition. Increased sediment additions may also have slowed TNT and RDX breakdown in the sewage sludge composts; however, data were insufficient to determine if the increased sediment addition slowed the rate of explosives degradation. Losses of tetryl were substantial in all treatments. After composting for 44 days, tetryl levels were reduced by $\geq$92% in every treatment. Data on HMX clearly demonstrated that HMX is susceptible to degradation during composting. However, the rates of breakdown were slow in the sewage sludge compost (30-50% in 77 days), and no loss of HMX was observed in the hay-horse feed composts.

Accumulation of toxic or otherwise undesirable degradation products during the composting of LAAP sediment was not observed. ^{14}C-RDX degradation resulted in extensive metabolism of the molecule as indicated by the substantial release of $^{14}CO_2$. Only one unidentified compound attributed to RDX metabolism was extracted from the composts, and no build up of this compound was evident. Similarly, only one degradate of HMX breakdown was found by TLC analysis. Several unidentified spots appeared in the TLC analyses of extracts from the ^{14}C-TNT study. Also the 2- and 4-amino derivatives, and the tetranitro azoxy analog of TNT were detected.

However, in all cases relatively small percentages of the degradates or transformation products were found. The formation of these products may be incidental to breakdown of the explosives, or, these products may be short lived intermediates in the degradation of the explosives.

Evolution of $^{14}CO_2$ from ring-labeled ^{14}C-TNT and ^{14}C-tetryl was limited. These results indicate that cleavage of the ring may have occurred, but the rate of ring degradation is probably very low. It is hypothesized that the intact ring from TNT or tetryl is incorporated in natural humic substances.

E. Composting of Badger AAP Sediment

1. Results

Composts containing BAAP soil composted extremely well. Temperatures in the composts ranged from 66-77°C (Appendix L), and after 42 days of composting the composts were very dark in color and substantially reduced in volume. Elevated temperatures of this magnitude in composts consisting of either 50 or 70 g of organic materials demonstrated that the BAAP soil or some component(s) of the soil significantly enhanced the composting process. Anaerobic conditions in the composts did not occur but the build up of organic acids may have occurred in one replicate each of the hay-horse feed compost containing 10 and 18% soil. Both of these composts had the smell of lactic acid. At the conclusion of the compost study the acid smell could no longer be detected.

Breakdown of nitrocellulose began within the first week of composting as indicated by the release of $^{14}CO_2$ from UL ^{14}C-nitrocellulose (Figure 8). $^{14}CO_2$ evolution rates were initially very high. After 3-4 weeks, the rates of $^{14}CO_2$ release began to decline. The cumulative losses of ^{14}C from the composts as $^{14}CO_2$ accounted for 43-74% of the total activity added to individual composts. Small quantities of ^{14}C were recovered in the acid traps and in the water condensates from the hay-horse feed composts, and more than 1% of ^{14}C was recovered from the condensates of the sewage sludge composts (Table XIII).

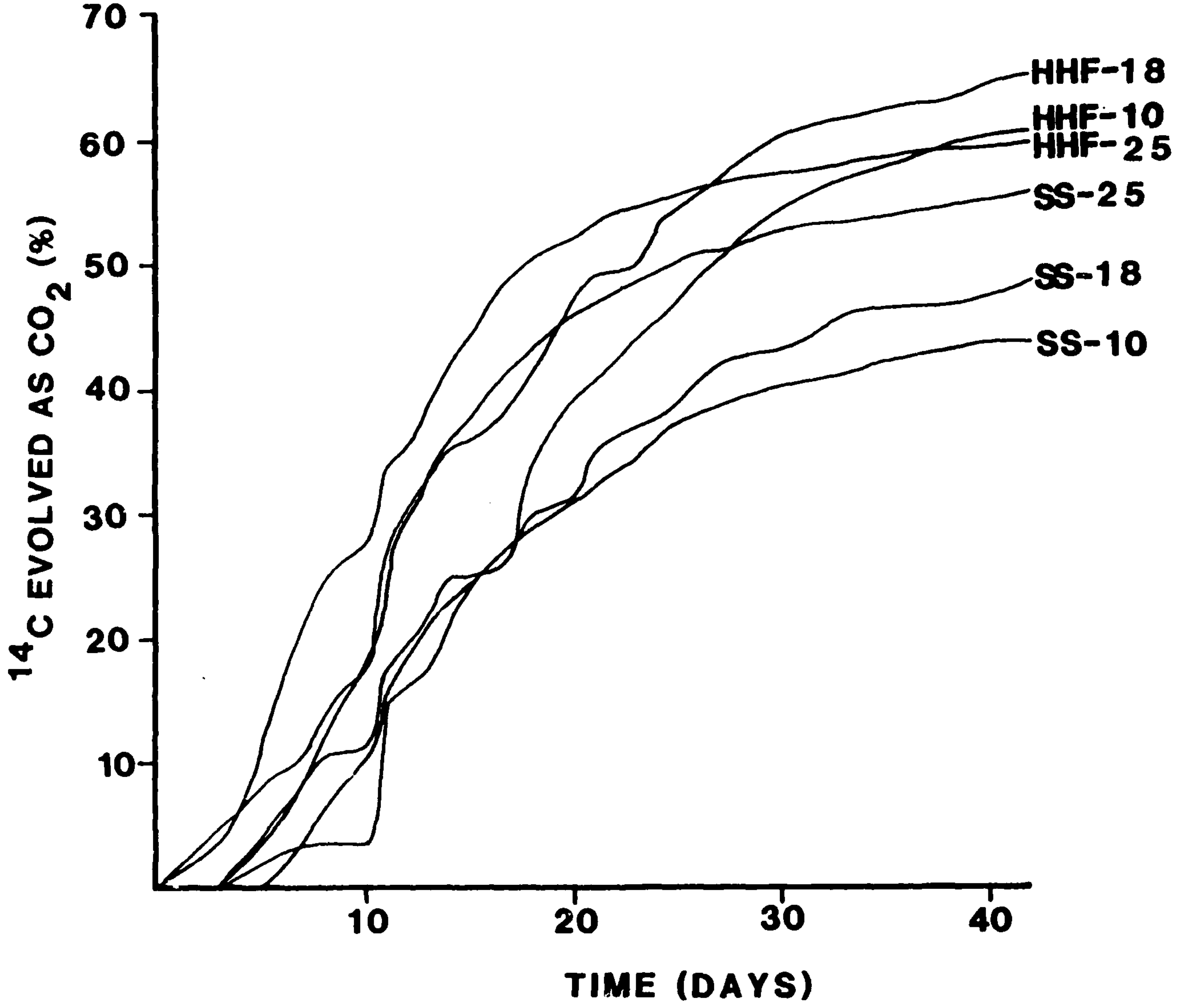

Figure 8. $^{14}CO_2$ Evolved from Badger AAP Composts Spiked with UL ^{14}C-Nitrocellulose

Table XIII. Distribution of ^{14}C Badger AAP Soil Composts Spiked with UL ^{14}C-Nitrocellulose

Compost*	Amount of Sediment Added (dry wt %)	Composting Time (days)	^{14}C Recovered (% of added)					
			$^{14}CO_2$	H_2O Condensate	Acid Trap	Extract	Unextracted Residual	Total
HHF	10%	0	-	-	-	67.2	43.5	110.7
	18%	0	-	-	-	66.4	42.4	108.8
	25%	0	-	-	-	68.8	44.9	113.7
SS	10%	0	-	-	-	22.0	88.8	110.8
	18%	0	-	-	-	24.8	84.0	108.8
	25%	0	-	-	-	19.1	89.9	109.0
HHF	10%	42	60.5a**	0.4b	0.2a	1.8a	25.4b	88.3
	18%	42	65.0a	0.5b	0.2a	2.2a	27.6b	95.5
	25%	42	59.6a	0.5b	0.1a	1.4a	32.6b	94.2
SS	10%	42	44.0b	1.0a	0.4a	2.4a	65.8a	113.6
	18%	42	48.7b	1.2a	0.4a	2.6a	60.0a	112.9
	25%	42	55.6b	1.2a	0.2a	1.6a	62.0a	120.6

* HHF - hay-horse feed composts; SS - sewage sludge composts

** Values followed by the same letter are not significantly different at the 5% level of probability as determined by the analysis of variance

After composting for 43 days very little of the ^{14}C could be extracted into acetone. Essentially all of the ^{14}C activity remained as bound residue in the compost or had been lost as $^{14}CO_2$. Analyses of the extracts for nitrocellulose recovered less than 1.5% of the explosive initially added to the composts.

Overall recovery of ^{14}C averaged 106.2% with a standard deviation of 12.4%. The extraction procedure gave consistent recoveries within each type of compost, but recoveries were low. Average recoveries out of the hay-horse feed composts were 67.5% (standard deviation 1.2%) and 22.0% (standard deviation 2.9%) out of sewage sludge composts.

2. Discussion and Conclusions

The addition of Badger AAP soil to composts enhanced the composting process and resulted in elevated temperatures in the composts throughout the 42 day composting period. Under these highly thermophilic conditions, nitrocellulose was rapidly degraded, releasing a substantial portion of its carbon as CO_2. Such high losses of CO_2 demonstrate that the metabolism of nitrocellulose is extensive; degradates with a structure similar to the parent compound are not expected to occur to any significant extent. The shape of the $^{14}CO_2$ vs time curves indicates that most of the nitrocellulose had degraded within the first 3-4 weeks. Further releases of $^{14}CO_2$ after the fourth week would increasingly result from the secondary metabolism of ^{14}C which had been incorporated into the microbial biomass when the nitrocellulose was metabolized. The extensive breakdown of nitrocellulose was further confirmed by the very low recovery of ^{14}C in the solvent extracts and the low levels of nitrocellulose found in the extract. Even after these values are corrected for extraction efficiency, greater than 90% nitrocellulose degradation is indicated. The nitrocellulose assay is a non-specific colorimetric test with numerous interferences. The compost extracts are highly colored and contain numerous short chain organic acids and nitro groups, which can result in false positive readings in the nitrocellulose assay when nitrocellulose levels are low. Given the dramatic losses of $^{14}CO_2$, the low recovery of ^{14}C in the solvent extracts and the likelihood for false high readings from the nitrocellulose determinations, it appears that nitrocellulose is completely degraded within six weeks of composting.

Nitrocellulose is not toxic, and any degradates resulting from its breakdown are not expected to be toxic or otherwise harmful. Recovery of ^{14}C in the condensates, especially condensates from the sewage sludge composts, suggest that some low molecular weight materials which may have resulted from nitrocellulose degradation are volatilized from the composts. Loss of these materials is not considered to constitute any environmental or health hazards.

F. TCE Volatility Tests (Letterkenny AD Soil)

1. Experimental Procedures

TCE volatility tests were run to determine the rate at which TCE would be lost from a soil-compost material mixture. An apparatus was set up to collect TCE volatilized out of soil and compost samples. A diagram of the apparatus is presented in Figure 9. A one-quart Mason jar was used to hold the sample. The materials to be tested were placed in the jar then 57 µL of TCE was added using a syringe. The opening was immediately stoppered, and hooked up to the aeration system as shown in Figure 9. The jar was placed in an incubator held at 60°C. Air was continuously pulled through the sample jar and then through a liquid nitrogen cold trap to collect the volatilized TCE. After an appropriate period, the cold trap was washed with methanol, and the contents of the jar were extracted twice with methanol. TCE in the methanol washes was quantitated using GC analysis (Appendix K). This system was tested initially by placing TCE in an empty flask. Then TCE losses from soil and compost were tested.

2. Results

All volatilization tests are summarized in Table XIV. Trapping efficiency of the apparatus was good (approximately 96.5%). TCE losses from soil after 1.5 hours of incubation were nearly quantitative. Less than 0.05% of the TCE remained in the soils. Composting materials and soil were mixed together and watered to simulate materials just beginning to compost. Within 3 hours 94.6% of the TCE had volatilized from the compost materials and only 0.1% remained in the compost. This test was repeated using hay and horse feed that had been composted 26 days. No TCE was detected in the compost after 3 hours incubation at 60°C. Recovery of TCE

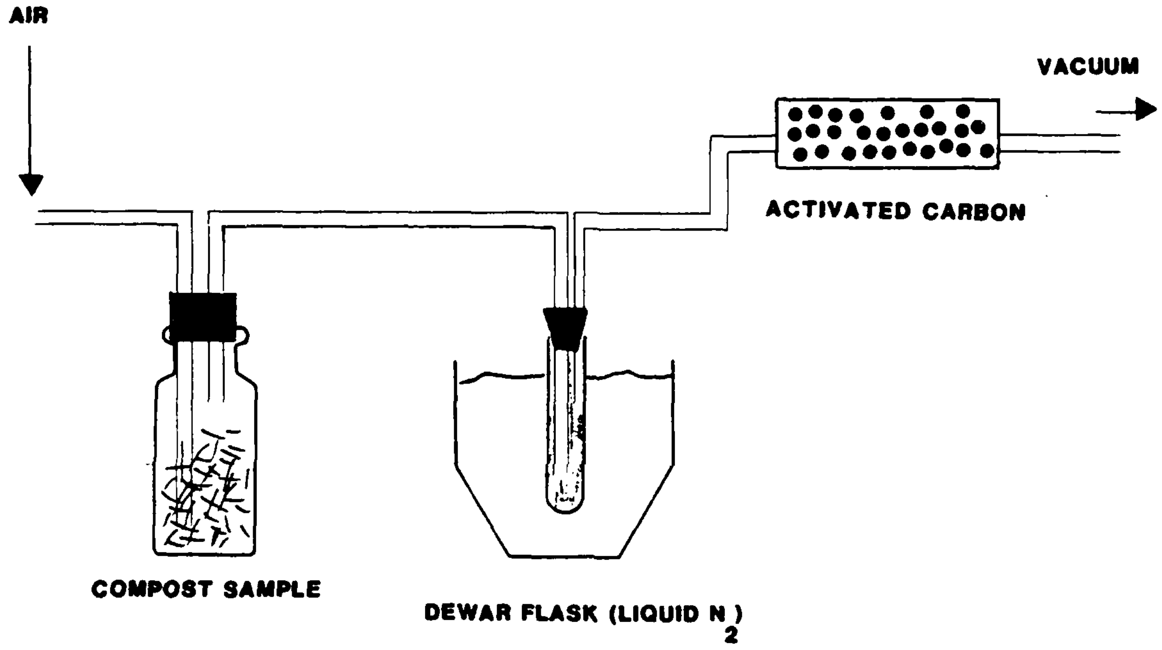

Figure 9. Apparatus Used to Evaluate TCE Volatility From a Soil Compost Mixture

Table XIV. TCE Volatilization From Soil and Compost at 60°C

Materials in Flask	Incubation Time (hours)	TCE Recovery (%)	
		Cold Trap	Flask
Nothing	1.5	96.2	ND
Nothing	1.5	96.9	ND
Lakeland sand (16.7 g)	1.5	99.8	0.05
Soil from Letterkenny (16.7 g)	1.5	94.4	0.05
Hay (25 g) horse feed (25 g) and LAD soil (16.7 g) 100 mL H_2O	3.0	94.6	0.1
Composted hay and horse feed (140.4 g at 64.4% mixture)	3.0	87.8	ND

from the cold trap was 87.8%. The somewhat low overall recovery apparently resulted from water build up in the cold trap. At some point during the test the ice in the cold trap blocked air movement. After that TCE volatilized from the compost was lost through the air intake opening.

3. Discussion and Conclusions

TCE contaminated soil added to composts would result in rapid and near quantitative loss of TCE to the atmosphere as thermophilic temperatures in the composts were reached. Further investigation on the fate of TCE in composts is not warrented.

IV. PILOT SCALE COMPOSTS

A. EPA RCRA Research, Development and Demonstration Permit

The Resource Conservation and Recovery Act designates that waste from munitions manufacturing and loading operations is a K044 hazardous waste. Experiments aimed at treatment of a hazardous waste requires a RCRA permit. The 1984 RCRA amendments provide a distinction between commercial disposal operations and research and development.

In December of 1984, ARC wrote Mr. John Skinner of EPA's Office of Solid Waste asking for guidelines for obtaining a RCRA R&D permit. In January, 1985, ARC representatives met with EPA Office of Solid Waste personnel to discuss composting of LAAP and BAAP sediment. EPA personnel agreed at this time that composting of these sediments would be an appropriate subject for a RCRA R,D&D permit. The actual application for the RCRA R,D&D permit was submitted on 5 February, 1985. EPA completed the initial review of the application on 15 April, 1985 and sent ARC a list of questions. ARC responded to these questions on 19 April, 1985. The permit application and answers to the questions were submitted to an EPA consultant for technical review. Additional questions sought by the consultant were answered by ARC on 25 April, 1985.

The major question then became the reactivity of the sediment. Several tests on the LAAP and BAAP sediments were performed at ARC and the Bureau of Mines. These tests included:

- Gap Test
- DDT Test
- Bureau of Explosives Impact
- Thermal Stability Test
- Electrostatic Discharge Test
- Autoignition Test
- Detonation Test

These tests were run on air dried sediment at the following concentrations of explosives in the sediments (dry weight basis).

LAAP		BAAP	
TNT	11.5%	Nitrocellulose	1.03%
RDX	6.0%		
HMX	0.16%		
Tetryl	0.5%		

All reactivity tests on these sediments were negative. These test results were submitted to EPA on 20 May, 1985.

A draft RCRA R,D&D permit was issued by EPA Region III on 30 May, 1985 and a public hearing announced for 1 July, 1985 (see Appendix M). The public hearing held on 1 July, 1985 and a 30 day comment period produced no comments that warranted hold-up or denial of the permit. Thus, EPA Region III issued ARC the first RCRA R,D&D permit No. VAD 06 112 2156 on 1 August, 1985.

B. Materials

1. Composting Materials

Three composting mixtures were used in these studies, hay-horse feed, sewage sludge-wood chips, and horse manure-hay-saw dust. The hay-horse feed composts were composed of approximately 50% (dry weight basis) chopped alfalfa hay, and 50% Purina Sweetena horse feed. The microbial seed for this compost was provided by soaking the hay in a water slurry of horse manure.

The sewage sludge compost consisted of equal volumes of wood chips (25-35% moisture) and sewage sludge (65-70% moisture). The sewage sludge was obtained from the Arlington Wastewater Treatment Facility in Arlington, Virginia. The sewage sludge was a mixture of primary and secondary sludge with ferric chloride and lime added. The ash content of the sludge was 35-40%. The sludge provided the microbial seed for the sewage sludge-wood chip composts.

The horse manure-hay-saw dust compost was a combination of horse manure from two stables; one using saw dust as bedding, the other using hay as bedding. The relative quantities of saw dust to manure could not be determined. The amount of hay was adjusted by mixing the manure from the two stables to give adequate porosity in the compost. Although no quantitative information on the manure-hay-saw dust compost was obtained, visual inspection indicated that manure comprised greater than 50% of the dry mass. No additional materials were added for seed. The manure from one of the stables was relatively old and did not contain the available nutrients as would be found in fresh manure. As a result of the lack of nutrients, sufficient Purina Sweetena horse feed was added to dilute the compost 19.8% after 19 days of composting.

2. Contaminated Sediments/Soils

Contaminanted sediment was collected from Badger AAP and Louisiana AAP for pilot scale composting. Two drums of Louisiana sediment were taken from lagoon #4 near the spillway. The Badger sediment was taken from the dredgings mound (two drums) and from near the sluice gate at the end of lagoon #1 (one drum). The sediment from the sluice gate was extremely wet due to the heavy rains the night before collection. It was not used in the composting study because the drier soil from the dredgings mound contained sufficient nitrocellulose.

C. Composting Apparatus

Six 488 gallon 304 stainless steel tanks measuring 5 feet in diameter and 4 feet in height were used as composters. These tanks were placed in two 19- x 9-ft Janco greenhouses located within a concrete lined pit and surrounded by a six foot high fence. Three tanks were placed in each greenhouse. Each tank was covered with a plywood cover with galvanized steel inner facing. The outside of each compost tank was covered with fiberglass insulation. A perforated wooden platform was placed on wooden blocks three inches from the bottom of each composter. Woodchips were used to fill the space at the bottom of the tank to provide insulation and to adsorb leachate as it was produced during composting.

As shown in Figure 10, each compost tank had a 2 inch drain near the bottom of the tank which led to a leachate collection system and provided a means of pulling air through the composts. A ball valve in the aeration line to each tank provided individual aeration control. A port was placed in each aeration line so that the air velocity could be measured from each tank and air samples could be taken for analysis. A valve was positioned in each line so that water condensate could be drained from system. The air lines from all six composts tied into a single line from one blower. The blower was intermittently operated by an adjustable timer.

D. Experimental Design

Two sets of pilot scale composting experiments were initially planned. In the first set of experiments, hay-horse feed and sewage sludge-wood chips composts were evaluated with Louisiana soil. Three composters of each compost material were formulated; two of these composters contained the LAAP sediment (Tank 1 and 2 for hay-horse feed and 4 and 5 for sewage sludge-wood chips) and one composter served as a control (Tank 3 for hay-horse feed and 6 for sewage sludge-wood chips). For these studies, the hay-horse feed composts were housed in one greenhouse and the sewage sludge-wood chips in the other greenhouse. Based on the results of the laboratory studies, the hay-horse feed composts were amended with LAAP contaminated soil at the 11% level while the sewage sludge-wood chips composts contained 16% sediment.

In the second set of similar experiments, Badger AAP soil was added to two composts containing hay-horse feed and two sewage sludge-wood chips composts. Both composts types contained 15% by weight of the Badger nitrocellulose contaminated soil.

The first pilot study using LAAP sediment indicated that effective destruction of the explosives occurs in composts that can maintain very high levels of microbial activity (high temperatures) for extended periods (greater than 6 weeks). Sewage sludge-wood chip composts are biologically too stable to provide the environment needed for extended high levels of microbial activity. Therefore, a second LAAP compost study was conducted using horse manure, hay, and saw dust as composting materials.

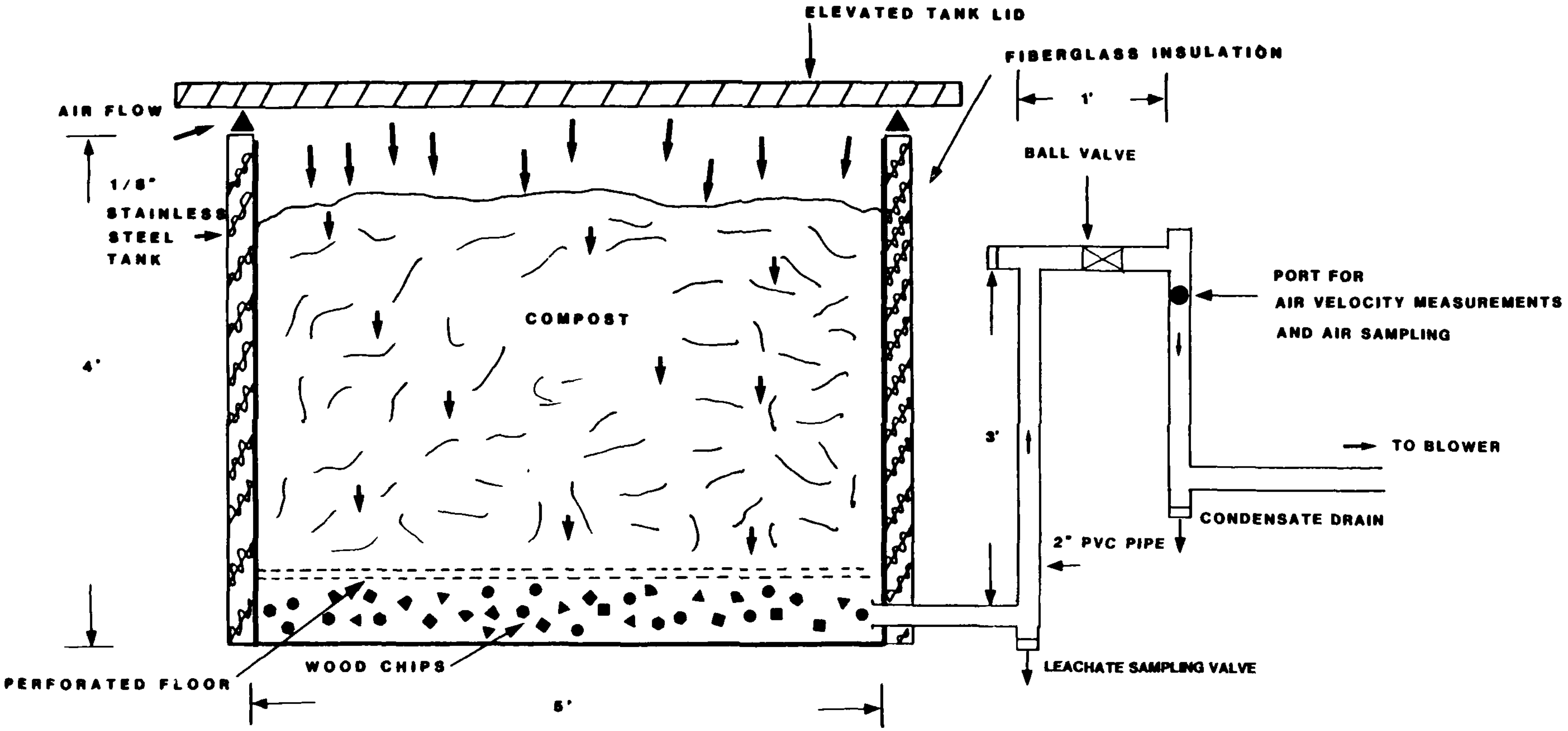

Figure 10. Schematic of Pilot Scale Compost Apparatus

The manure composts were not replicated. One control and one treated compost were set up. LAAP sediment was added to the treated compost at 11.8% by weight; no sediment was added to the control. After composting for 11 days compost temperatures dropped rapidly in the treated compost. This loss of microbial activity was associated with the toxicity of the LAAP sediment. Adjustments in the aeration failed to increase the temperature in the treated compost. Therefore, after 19 days of composting a small amount of compost (34 lb dry weight) was removed from the composts and 90 lb (dry weight) of Purina Sweetena horse feed was added.

E. Compost Set Up

1. Sediment/Soil Preparation

a. Hay-Horse Feed and Sewage Sludge Composts

The volume of sediment/soil needed to setup four composts was too large to allow all the sediment/soil to be homogenized. Therefore, two drums each of the contaminanted sediment from LAAP and BAAP soil were designated as "A" and "B". Each drum was individually mixed by mixing in a 1-cubic yard cement mixer. Approximately half a drum of soil was mixed in the cement mixer and then dumped into a plastic tub. The remaining soil in the drum was mixed in the mixer and also dumped into the tub. Soil in the tub was then mixed by hand. Then the soil was shovelled back into the cement mixer and remixed (in two batches). The partially mixed LAAP sediment in the plastic mixing tub is shown in Figure 11. Subsamples were taken from each drum to quantify the explosives and heavy metals contamination levels, and sediment/soil from only one drum was mixed into any one compost. The analyses of these sediments are presented in Table XV. For the LAAP compost studies, Barrel A was used in Tanks 2 (hay-horse feed) and 5 (sewage sludge-wood chips) and Barrel B in Tank 1 (hay-horse feed) and 4 (sewage sludge-wood chips). In the BAAP composting studies, Barrel A was used in the sewage sludge-wood chip composts and B in the hay-horse feed compost.

b. Manure Composts

LAAP sediment in drums A and B remaining after the hay-horse feed and sewage sludge composts set up was pooled, air dried, and crushed and sieved (2 mm) by hand. The sieved sediment was throughly mixed

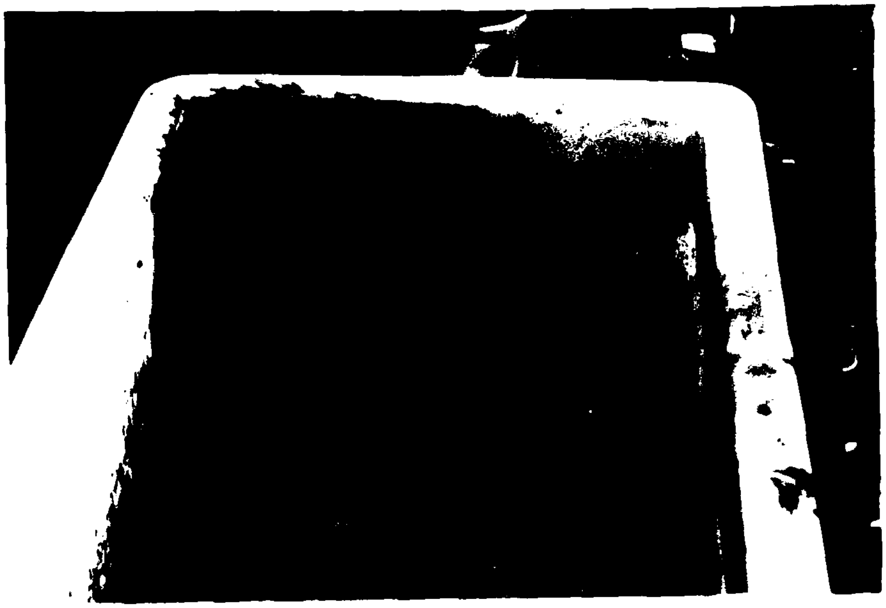

Figure 11. Photograph of Louisiana AAP Soil Used for Composting

Table XV. Analysis of the Louisiana AAP and Badger AAP Sediments

	Concentration µg/g				
	TNT	RDX	HMX	Tetryl	Nitrocellulose
LAAP-A	460,388	62,405	8,450	17,076	
LAAP-B	322,735	67,749	9,001	3,961	
LAAP-C	321,263	52,343	7,500	8,430	
BAAP-A					56,382
BAAP-B					49,950

and then subsampled for explosives analyses. Explosives concentrations (Table XV) were somewhat lower than those previously measured in drums A and B. This discrepancy is believed to result from sieving the pooled sediment. Some particles cemented together by high concentrations of explosives were rejected by the sieving.

2. Mixing Compost Materials and Sediment/Soil

a. Sewage Sludge Compost

The composts were prepared by mixing small preweighed batches of sewage sludge, wood chips and contaminated sediment/soil in a one-cubic yard cement mixer. Approximately equal volumes of sewage sludge and wood chips were weighed out. These weights were corrected for moisture, and the appropriate amount of sediment needed for the batch (corrected for moisture) was calculated. All calculations were handled automatically by using a TI-58 programmable calculator. The calculated weight of sediment soil was then mixed with the sewage sludge and wood chips in the cement mixer. Fourteen to 15 batches were mixed to fill each composter. The composters were filled for the LAAP sediment study. The composters were partially filled for the nitrocellulose (BAAP soil) study (12 batches) to allow for easier handling and sampling of the material.

Delays in initiating the pilot scale studies caused by obtaining the EPA RCRA R,D&D permit forced a very rapid setup of the LAAP composts. As a result, estimates of the moisture levels in the sediment were used when mixing the compost. Small errors in the estimates resulted in adding sediment at slightly higher rates than intended. A summary of the materials used to construct each compost for LAAP and BAAP composts is given in Tables XVI and XVII.

The mixing operation is shown in Figure 12. All operations were carried out on a liner to avoid spills of the contaminated sediment/ soil. Materials in 55 gallon drums were weighed on a balance which was accurate to 0.5 lb. The materials were then dumped into the cement mixer using a fork lift and drum tipper. The cement mixer was started, and the compost materials were mixed for 2-3 minutes. With the mixer still running, the compost was dumped into metal bushel baskets. The baskets

Table XVI. Contents of Louisiana AAP Pilot Scale Composts

	Hay (lb)	Horse feed (lb)	Sediment (lb)	Total Dry Weight (lb)	% Sediment Addition
Tank 1	250	250	45	501	10.8
Tank 2	238	238	53	484	12.1
Tank 3	236	236	55	423	11.6

	Sewage Sludge(lb)	Wood Chips(lb)	Sediment (lb)	Total Dry Weight (lb)	% Sediment Addition
Tank 4	1344	688	128	999	15.5
Tank 5	1348	595	146	1020	15.9
Tank 6	1232	618	176	936	16.9

	Horse Manure (lb)		Sediment (lb)	Total Dry Weight (lb)	% Sediment Addition
Tank 5	1419		64	518	11.8
Tank 6	1181		0	392	0.0

Table XVII. Contents of Badger AAP Pilot Scale Composts

	Hay (lb)*	Horse feed (lb)*	Sediment (lb)*	Total dry weight (lb)*	% Sediment Added
Tank 1	215	216.9	76	508	15
Tank 2	221.3	223.2	78.2	523	15
	Sewage Sludge	Woodchips			
Tank 4	331.5	260.6	109.0	702	15.7
Tank 5	342.6	292.2	107.1	742	14.4

* Dry weights

were placed in the metal feed trough, seen in Figure 12, to contain any materials spilled from the basket. A subsample of material was removed from each batch as shown in Figure 13 and 14. The subsamples were mixed together in a plastic cement pan, and then three subsamples of this composts were taken for later analysis.

The baskets of mixed compost were hand carried and dumped into the composters. The sewage sludge compost tank being filled is shown in Figure 15. The partially exposed bamboo poles and blue thermocouple wire show how the thermocouples were initially positioned in the LAAP sediment composts. Fixed positioned thermocouples were removed from the LAAP sediment composts after four weeks. A thermocouple probe was used in placed of the fixed position thermocouples in the BAAP soil composts. After the composter tanks were filled, a pillow stuffed with styrofoam packing material was laid on the composts to prevent excessive heat loss from the surface.

b. Hay-Horse Feed Composts

The alfalfa hay was coarsely chopped in a hammer mill and weighed into 55 gallon drums. Before being mixed into the compost the hay was soaked for 2-14 hours in a dilute water slurry of horse manure.

The hay-horse feed, and sediment/soil were mixed in small batches in a cement mixer as previously described for mixing the sewage sludge composts. The manure soaking solution was drained from the hay immediately before the hay was used. An amount of horse feed was weighed out to equal the weight of hay in a drum (dry weight basis). Based on these weights, an appropriate amount of sediment/soil was weighed out. Depending on the weight of hay in a drum, either 1/2 or 1/3 of the volume of each component was dumped into the mixer and mixed. Each drum of hay and the corresponding amounts of horse feed and sediment/soil were considered a batch in the mixing process. Nine batches of hay were mixed to fill each composter.

Calculations to determine the weights of sediment/soil to add to each were performed in a TI-58 programmable calculator to avoid computational errors. Sampling was as described for the sewage sludge composts. The loading of one of the hay-horse feed compost in the composter tank is shown in Figure 16.

Figure 12. Compost Mixing Operation

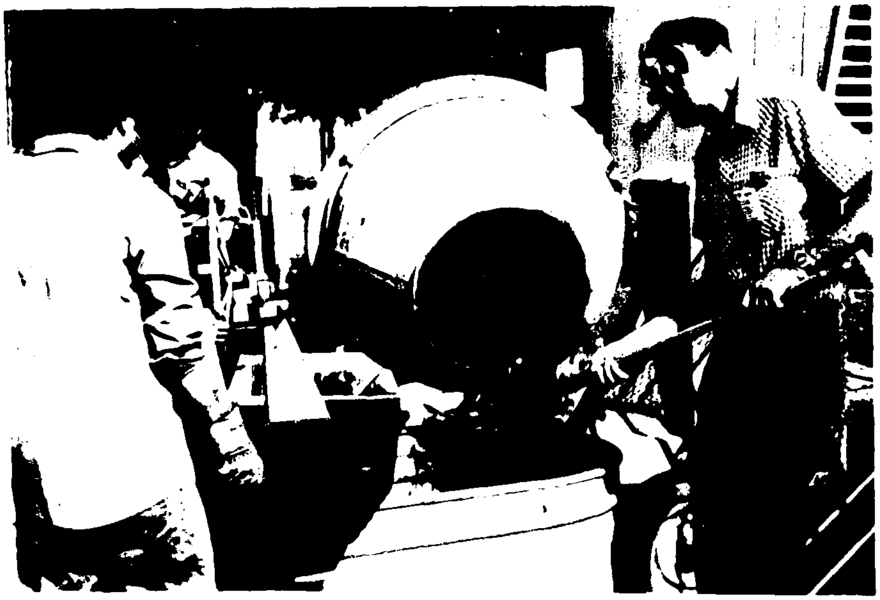

Figure 13. Subsampling Sewage Sludge-Wood Chips Compost During Mixing

Figure 14. Subsampling Hay-Horse Feed Compost During Mixing

Figure 15. Composter Being Filled with Sewage Sludge-Wood Chips Compost

Figure 16. Composter Being Filled With Hay-Horse Feed Compost

c. Horse Manure Compost

Horse manure from the two stables was mixed by hand with pitch forks and rakes in proportions that gave a mixture with sufficient hay to provide adequate porosity for aeration of the compost. Weighed aliquots of this mixture were blended with the previously processed LAAP sediment in a cement mixer. The final compost contained 11.8% LAAP sediment by weight. Subsampling the compost from the cement mixer was as previously described. Thirteen batches of soil amended compost were required to fill Tank 5. No sediment was added to the Control compost which was placed in Tank 6. A polyethylene liner was placed over the compost in each tank. Numerous slits in each liner allowed for uniform aeration. The liner also helped to prevent rapid moisture losses from the top of the composters.

F. Analytical Methodology

All analyses for the pilot scale composting conform to USATHAMA's quality assurance program. These analyses are described below and in the appropriate appendices.

1. TNT, RDX, HMX, and Tetryl

Triplicate LAAP compost samples were weighed into tared Mason jars and stored at 5°C until extracted. Samples were extracted and analyzed for TNT, RDX, HMX, and TNT transformation products according to the method in Appendix H. Replicate samples were also taken for moisture determination (dried 48 hours at 65°C). Additional triplicate sets of samples from week seven hay-horse feed and sewage sludge composts and from week 0-8 horse manure composts were collected for tetryl analysis. These samples were stored at 5°C, and then extracted and analyzed according to the method in Appendix I.

Leachate samples pooled over each week of composting were subsampled and stored at 5°C until analyzed (Appendix N).

2. Nitrocellulose

Replicate samples of compost were weighed into tared Mason jars and dried for 72 hours at 55°C. Percent moisture was calculated from the weight loss. The dried samples were extracted as described in Appendix J to quantitate nitrocellulose.

3. Gas Analysis

Gas samples were obtained from the headspace above each compost and from a port placed in the aeration system to allow for sampling of the atmosphere pulled through the compost. These samples were monitored for CO_2, N_2, NH_3, and CH_4 by GC (Appendix R). Gas samples were not taken from the manure composts.

4. Heavy Metal and Pesticide Analyses

Samples of compost materials and compost from the hay-horse feed and sewage sludge composts were dried (48 hours at 65°C) and finely ground in a micro-mill. Subsamples were analyzed for heavy metals (Appendix O). Selected subsamples were also analyzed for pesticides (Appendix Q). Leachate samples pooled by week were subsampled, acidified (pH less than or equal to 2.0), and stored (5°C) until analyzed. Heavy metals were quantitated in these samples according to the methods presented in Appendix P.

G. Composting Louisiana AAP Sediment

1. Monitoring and Sampling Procedures

a. Hay-Horse Feed and Sewage Sludge-Wood Chip Composts

Daily compost inspection sheets were filled in to conform to the EPA RCRA R,D&D permit. Included in this inspection were the security of the facility, the integrity of the composters, composter aeration plumbing, greenhouse liners, hazardous waste storage facilities, the availability of safety equipment, and volumes of leachate and condensate. Temperatures throughout each compost were measured and recorded on a daily basis. The air flow through each compost was measured every day and adjusted, if needed. If all composts needed substantial increases or decreases in aeration, the length of time the blower ran during a 15 minute cycle was altered appropriately. A ball valve on each aeration line was utilized to individually adjust the air flow through each compost.

Air samples were collected weekly. Samples were taken from the headspace above each compost when the blower was not on to determine the composition of gases released by the compost. Samples were also taken from the aeration plumbing (Figure 10) when the blower was running.

Compost samples were collected from each compost on a weekly basis. Fifteen to twenty grab samples were removed from each compost using a sampling device that consisted of six-inch double hinged wing on the end of a 5-foot shaft. Samples were taken from throughout the pile at varying depths and were pooled. Two subsamples for explosives analysis and three subsamples for moisture determination were removed from the pooled sample and the remaining compost was returned to the compost tank.

Initially the composts were not mixed because spacial sampling of the compost was planned after three weeks of composting. After two weeks, it became apparent that the compost had to be watered and mixed every 1-2 weeks to maintain desired levels of microbial activity. Composts were mixed and watered by hand. At times, a portion of the compost was removed to facilitate proper mixing.

b. Manure-Hay-Saw Dust Compost

Monitoring, leachate collection, temperature measurements, and aeration adjustments were as described for the hay-horse feed and sewage sludge composts. Composting conditions were adjusted to obtain the highest possible compost temperatures in the treated compost. Aeration in the control compost was utilized to hold its temperature range near to the temperatures in the treated compost. No air samples were collected.

Compost samples were collected after 0, 10, 19, 31, 42 and 56 days. The 0- and 56-day samples were collected while loading and unloading the compost. On the other sampling days, a cross sectional area of the compost was sampled by digging a trench across each compost through the center of the pile. The removed compost was homogenized and then subsampled for individual analyses.

Composts were mixed and watered if needed after 7, 19, 34, and 42 days of composting. After the 7-day mixing the treated compost (Tank 5) began to cool. Attempts to increase the temperature by adjusting the aeration were unsuccessful; temperatures were less than 40°C by day 11 (Appendix T). To revive Tank 5, 38 lb (dry wt) of compost were removed and 90 lb of Purina Sweetena horse feed and 8 lb (dry wt) of fresh horse manure were mixed into the compost on day 12 of the study. Compost temperatures recovered within a few days.

2. Results

a. Hay-Horse Feed Composts

Materials in the hay-horse feed composts were well composted during the 7-week trial. Variations between tanks appeared to be random with no indications of the contaminated sediments inhibiting the composting process, relative to the control tank. Mass reductions attributed to composting were 66.1% in Tank 1, 46.5% in Tank 2, and 56.8% in Tank 3 (control).

During the first three weeks of composting, average compost temperatures (see Appendix T)were in the moderate range (38-55°C) and variation in temperature within each tank was extreme. This variation was found to result from two parameters which were not properly controlled, the rate of air flow and the distribution of air flow. The air flow rate was initially set based upon previous experience with composting organic wastes to stabilize and reduce their mass. After the first week of composting the air flow rates were significantly reduced. A small increase in the average temperature of the composts resulted, with average estimated temperatures ranging from 45-55°C (see Appendix T, Table T-1).

The uneven air flow seriously impaired the performance of the composts. The insulating pillow channelled most of the air flow into a limited portion of each compost. The presence of the thermocouple wires and the bamboo stakes to which the thermocouples were attached tended to push the pillow up and away from the compost. As a result, much of the air flow was directed into the compost in the area around the thermocouples. The excessive air flow in these regions rapidly dried the compost material, which in turn reduced microbial activity and resulted in decreased temperatures. These "cold spots" would slowly expand into the surrounding compost. Typically 30% of each tank was cold and inactive most of the time during the first three to four weeks of composting. It should be pointed out that this cooling phenomena largely centered around the area where the thermocouples were positioned, thus average compost temperatures during the first four weeks of composting may be underestimated.

The compost drying and cooling problem caused by uneven air flow initially was too slight to notice (first week of composting), but progressively worsened. When the cause of the problem was identified, the insulating pillows were removed (after 3 1/2 weeks of composting). Temperature and moisture variability within each tank was substantially reduced by removing the pillows; however, compost along one edge of each tank still tended to dry rapidly and be somewhat cooler than the bulk of the material in the tank. When the tanks were emptied at the conclusion of the experiment, the cool edge was found to largely occur over an unsealed seam in the aeration board at the bottom of the tank.

In the original experimental design, detailed sampling around each of nine carefully positioned thermocouples was to occur after three weeks of composting to provide information on explosives breakdown relative to compost temperature and position in the compost pile. Drying and cooling of the composts negated any results obtained from this type of sampling. Also, settling and shifting within each compost moved the thermocouples substantially within short time intervals. Therefore, after four weeks of composting the thermocouples were removed and temperature readings were obtained with a removable probe. Given that the composts had to be watered and mixed frequently to counter the effects of the cool spot, the use of a probe was much more practical than attempting to maintain fixed thermocouples in each compost tank.

Numerous probe readings were taken in each tank on a daily basis to determine compost temperature. Three readings were taken in the center of each tank: one reading near the bottom of the compost, one at the center of the compost, and one between the center and the upper surface. Four to eight readings were taken at mid-depth approximately mid-way between the center and the outer edge of the tank. One or more readings were taken mid-depth about six inches from the edge of the tank.

Average compost temperatures obtained using the probe after the insulation pillows had been removed generally ranged from 52-62°C. After seven weeks, all hay-horse feed composts were highly active and capable of continued composting.

The loss of explosives during seven weeks of composting is summarized in Table XVIII. Concentrations are corrected for loss of compost mass. Mass loss was assumed to be linear with time, with losses beginning after one week of composting. Variation in the data is somewhat high due to the difficulty in obtaining representative samples; however, losses of TNT, RDX, and HMX conform to first order kinetics. The log of TNT loss with time in Tank 1 is illustrated in Figure 17. The first order rate constants and half-lives for TNT, RDX, and HMX losses are given below:

Compost	Explosive	K*	Half-life (wk)
Tank 1	TNT	0.393	1.8
	RDX	0.246	2.8
	HMX	0.151	4.6
Tank 2	TNT	0.495	1.4
	RDX	0.211	3.3
	HMX	0.146	4.8

* Values calculated using least squares regression.

Table XVIII. Concentrations of Explosives in Pilot Scale Hay-Horse Feed Composts Amended with 11% Louisiana AAP Sediment

		Explosive Concentration (ppm)*				
Tank	Composting Time (wk)	TNT	2-amino** DNT	4-amino*** DNT	RDX	HMX
1	0	39337	-	-	3483	510
	1	19986	172	74	2698	347
	2	13521	328	143	2261	413
	3	8841	265	129	2044	391
	4	8627	246	70	2011	437
	5	NA	184	34	1250	479
	6	4760	-	-	435	86
	7	2535	107	10	637	163
2	0	53019	27	28	3677	640
	1	25083	353	162	3504	496
	2	37075	429	190	3049	528
	3	4933	436	217	2099	474
	4	17830	534	151	2736	676
	5	NA	509	135	NA	NA
	6	4088	20	23	992	246
	7	897	199	25	563	160

* Adjusted for loss of compost weight

** 2-amino-4,6-dinitrotoluene

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

NA - Not Analyzed

\- Not Detected

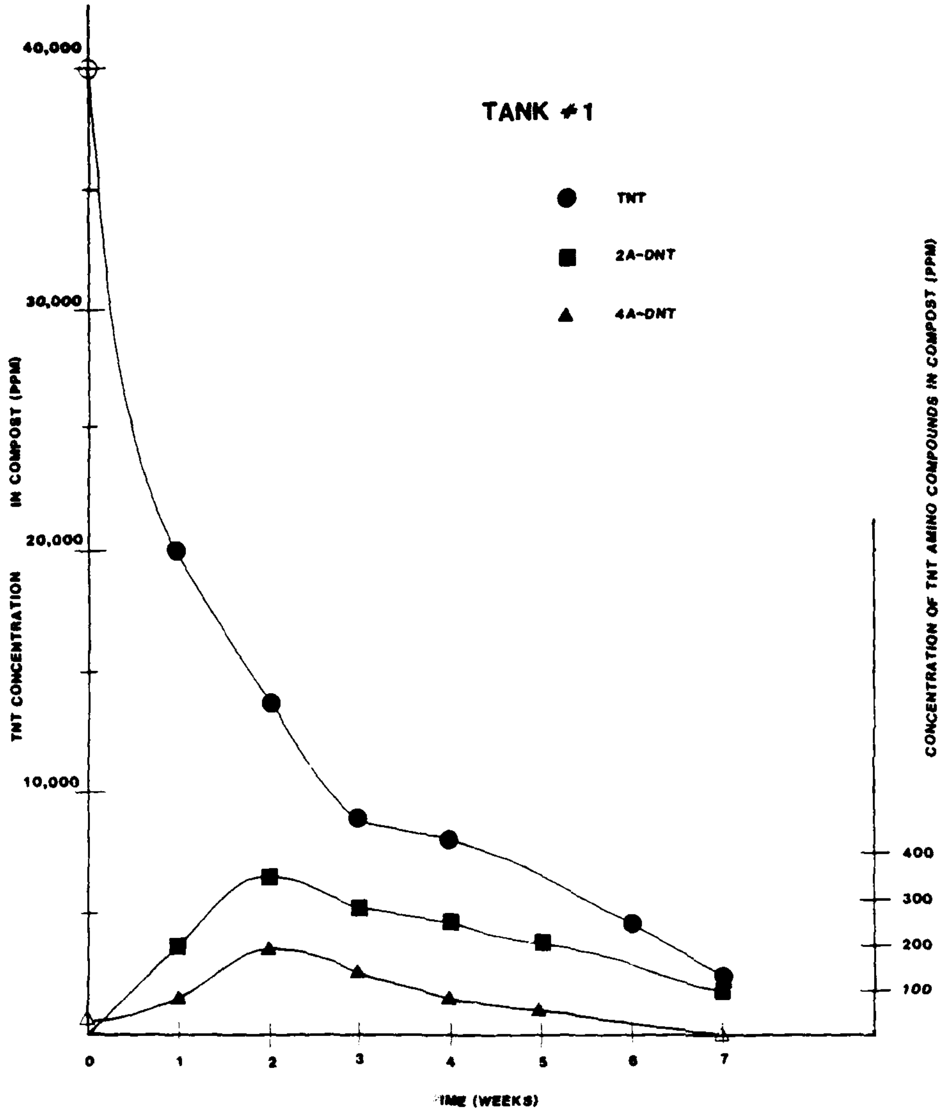

Figure 18. TNT, 2A-DNT, 4A-DNT Concentrations in Hay Horse Feed Comost as a Functi of Composting Time

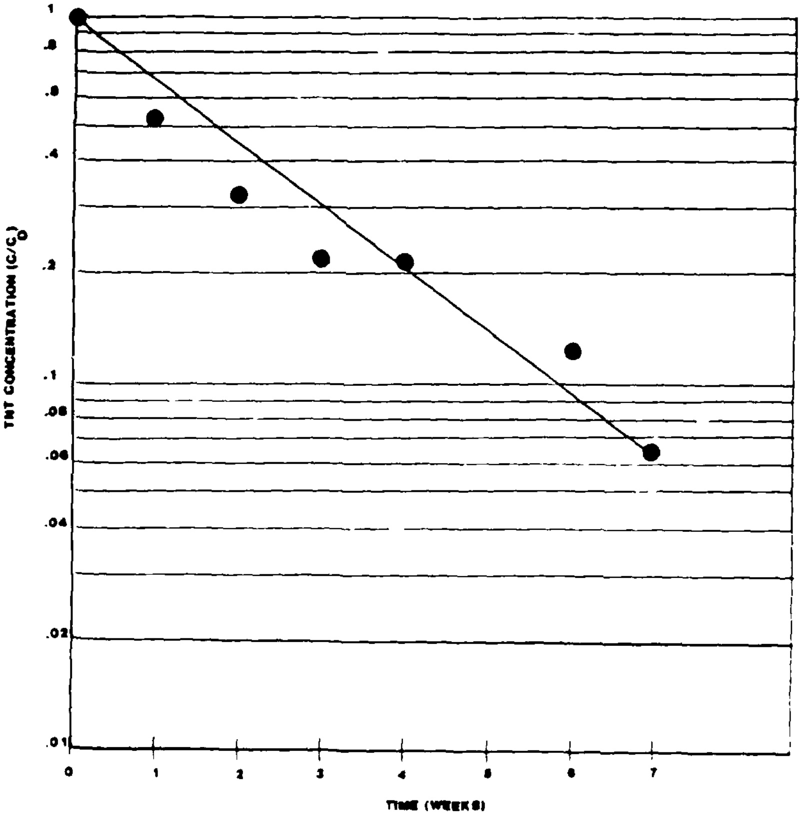

Figure 17. Loss of TNT with Time Hay-Horse Feed Compost)

TNT degraded most rapidly with an average half-life of 1.6 weeks. Losses of TNT were initially accompanied by small increases in the 2- and 4-amino dinitrotoluene derivatives (Figure 18). Neither the 2- or 4-amino dinitrotoluene accumulated in the compost, and both compounds decreased with increased length of composting. RDX degraded with an average half-life of three weeks. HMX breakdown was slowest with an average half-life of 4.7 weeks. Tetryl was quantitated only in the 7-week samples; therefore, rates of tetryl breakdown are not available. After 7 weeks of composting, tetryl levels were reduced $\geq$93% as shown below:

	Tetryl (ppm)		
Compost	0 Week*	7 Weeks	% Reduction
Tank 1	360	18	95.0
Tank 2	1895	125	93.4

* Values based on concentrations in the sediment.

Loss of explosives in compost leachate was very low as expected, given the low solubility of explosives in water. Concentrations of explosives in the leachate are given in Appendix T (Tables T-9 and T-10). HPLC analysis of the leachate produced a multitude of peaks. Some of the natural organic products in the leachate have retention times on the HPLC chromatograph that cannot be distinguished from those of the explosives. Reported concentrations of explosives in the leachate may therefore be overestimated.

Metal analyses of the LAAP sediment and the composting materials are presented in Appendix T. Metal concentrations in the hay and horse feed were as expected for natural uncontaminated materials. The sediment had elevated levels of lead and chromium, and low levels of mercury were detected. Leachate during the first week of composting was slightly acidic and the concentration of zinc in the leachate was relatively high, eg. 618 ppm (Appendix T, Tables T-3 through T-5). All subsequent leachates were alkaline and loss of metals from the compost via leachate was low. Small losses of copper and zinc were observed throughout the study. The formation of soluble ammonium complexes with copper and zinc is thought to be responsible for these losses.

b. Sewage Sludge-Wood Chips Composts

Good composting conditions for explosives degradation were not achieved in the sewage sludge composts. As discussed in the previous section, the initial use of high aeration rates and problems in getting uniform air flow through the composts decreased the overall microbial activity and prevented the composts from reaching temperatures substantially above 50°C during the first 3 1/2 weeks of composting (Appendix T, Table T-1). This problem was intensified by the contaminated sediment which appeared to inhibit the composting process. During the first several weeks of composting, the aeration rate in the control tank had to be increased to hold its temperature down in the same range as the tanks amended with the contaminated sediment.

Removal of the insulation pillows reduced the localized drying and cooling problems within each compost tank. However, the average temperatures of the sewage sludge composts did not increase after the pillows were removed. Apparently, readily useable energy sources in the compost had been largely exhausted by that time. Solutions of sugar and then molasses were mixed into the tanks several times during the sixth week of composting in an attempt to increase microbial activity, but no increases in temperature resulted. The mass losses for Tanks 4, 5, and 6 (control) during seven weeks of composting were 17.3, 14.6, and 32.8%, respectively.

No loss of explosives during composting could be confirmed. Sampling the sewage sludge composts was difficult, and variation between subsamples was high. Concentrations of explosives showed both increases and decreases during the 7-week composting period (Table XIX). No significant differences in the explosives concentration among weeks could be detected by analysis of variance testing at the 5% level of probability. The concentrations of explosives reported in Table XIX are corrected for compost mass loss assuming a constant rate of loss beginning after two weeks of composting.

Table XIX. Concentrations of Explosives in Pilot Scale Sewage Sludge Composts Amended with 16% Louisiana AAP Sediment

		Explosive Concentration (ppm)*					
Tank	Composting Time (wk)	TNT	2-amino** DNT	4-amino*** DNT	RDX	HMX	Tetryl
4	0	28928	698	395	6819	1008	522
	1	20740	1790	792	6177	587	-
	2	30654	538	217	8339	1473	-
	3	15200	799	308	7028	1265	-
	4	28675	743	277	6449	1181	-
	5	16574	889	325	7552	1318	-
	6	11766	389	121	4891	888	-
	7	9860	798	263	4428	1313	43
5	0	49544	923	289	6577	915	2493
	1	53846	1809	832	8464	1154	-
	2	78412	640	236	10239	1852	-
	3	47121	692	321	7252	1219	-
	4	42650	971	377	8001	1448	-
	5	48032	814	276	7849	1263	-
	6	45615	756	272	8603	1369	-
	7	11032	749	200	513	515	240

* Adjusted for loss of compost weight

** 2-amino-4,6-dinitrotoluene

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

Leachate from the composts contained apparent low levels of explosives throughout the experiment (see Appendix T, Tables T-11 and T-12). As previously discussed (in the previous section) the organics from the compost materials leached from the compost produced a large number of peaks during HPLC analysis. Analysis of the control compost demonstrated that some of these peaks coincided with peaks attributed to the explosives. Therefore, explosives concentrations in the leachate may be overestimated.

Metal analyses of the sewage sludge indicated that the sludge was relatively clean (Appendix T, Tables T-16 through T-8). Compost from this operation would not have metal concentrations that would restrict the use of this material as a soil amendment. Leachate from the sewage sludge composts was alkaline (pH 8.4 - 8.9) and solubilization of metals in the leachate was limited to relatively low concentrations of copper and zinc. These metals readily complex with ammonia to form water soluble species under basic conditions.

c. Manure Composts

Initially the LAAP sediment inhibited composting and the treated compost only marginally maintained thermophilic temperatures. Mixing after 10 days upset the compost and horse feed had to be added to affect compost recovery. After recovery (21 days) the treated compost produced thermophilic temperatures until the termination of the study (56 days). The mass reduction resulting from composting was 31.5%. The control compost was very well composted. Readily available energy sources were exhausted in the control and temperatures dropped below 50^{o}C after 43 days of composting. The total mass reduction was 48.7%.

Explosives levels in the compost decreased rapidly, approximating first order kinetics. TNT, tetryl, and HMX concentrations decreased substantially within the first 10 days of composting (Figures 19 and 20). RDX levels remained relatively constant during the first 19 days of composting and then dropped dramatically as the microbial activity in the compost increased after day 21 of the experiment. Listed below are the first order rate constants and half-lives of each explosive.

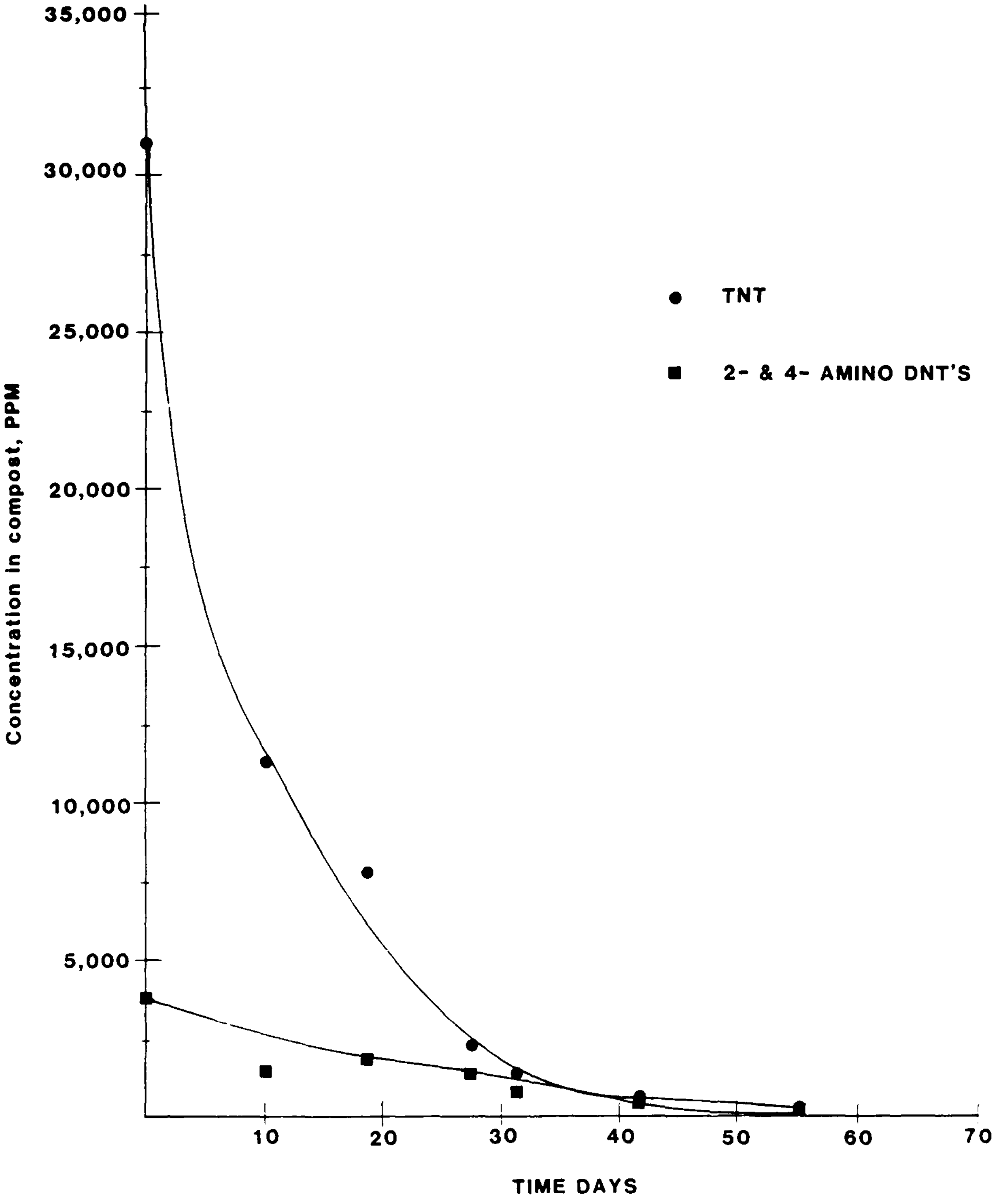

Figure 19. TNT and Amino Compounds Concentrations in Manure Compost as a Function of Composting Time

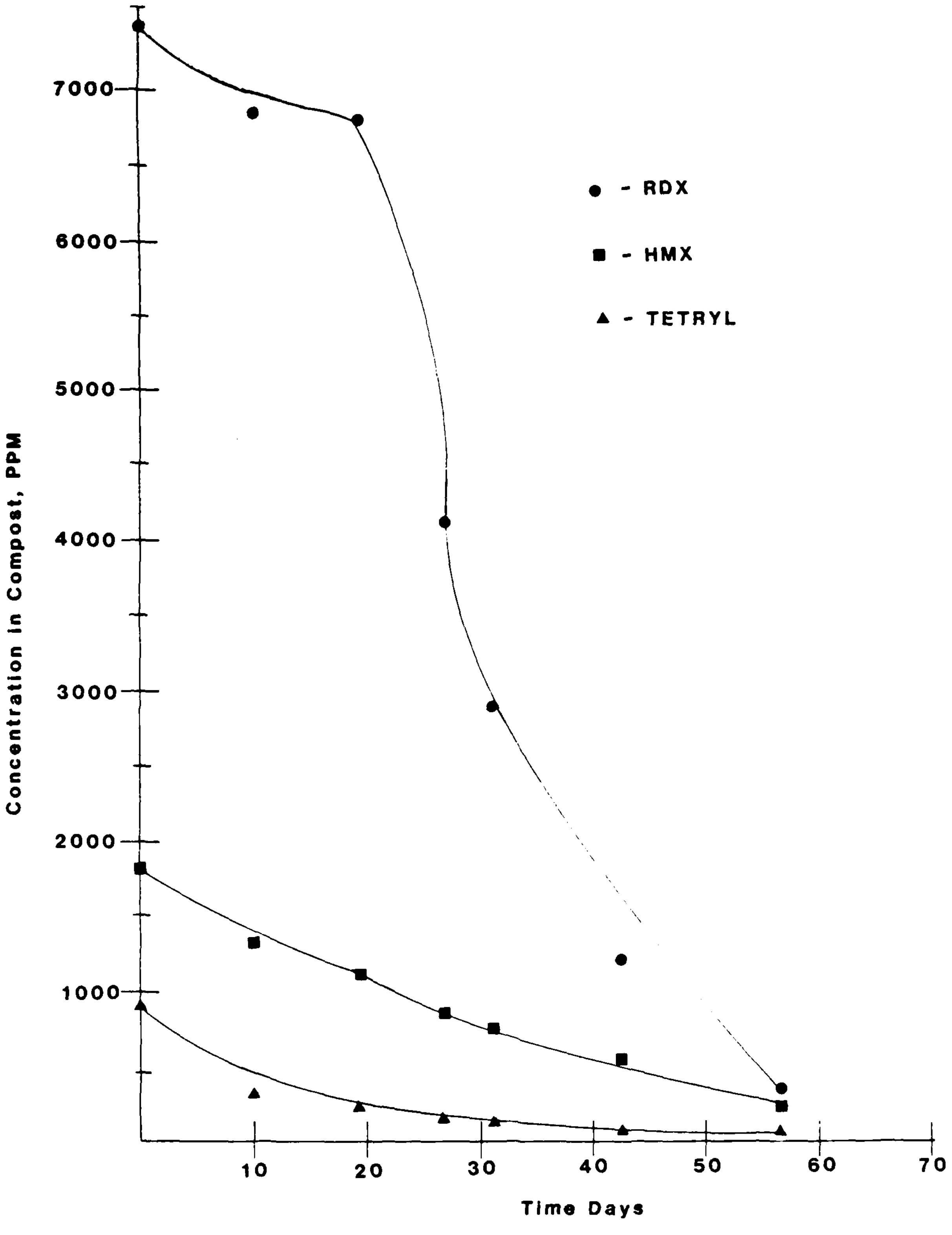

Figure 20. RDX, HMX and Tetryl Concentrations in Manure Compost as a Function of Composting Time

Explosive	K*	Half-life (wks)
TNT	0.678	1.0
RDX	0.280	2.5
HMX	0.211	3.3
Tetryl	0.576	1.2

* Values calculated using least square regression.

The relative rates of loss of the explosives were similar to those found in the hay-horse feed composts, but overall decontamination was noticeably faster in the manure compost. After 56 days of composting, RDX levels were reduced 94% and HMX was decreased by 81%. Tetryl levels were reduced 91% within 27 days, and after 31 days, concentrations were too low to be quantitated. The TNT concentration was reduced from 31021 ppm in the compost at time zero to 138 ppm in 56 days, a 99.6% reduction. The 2- and 4-amino-dinitrotoluenes did not accumulate in the compost as the result of TNT loss. Collectively these amino derivatives decreased 60% during the first 10 days of composting, remained constant through the 27th day of composting, and then decreased to 124 ppm (97% overall decrease) by the 56th day of composting.

Mobilization of explosives via the compost leachates was negligible. No explosives were detected in leachate collected during the first two weeks of the study. Between 0.2 and 2 ppm of TNT, RDX, and HMX was found in the week 3 leachate, but only 200 mL of leachate was produced. With increased length of composting, increased numbers and concentrations of organics from the manure were found in the leachate. These compounds interfered with explosives quantitation and resulted in falsely inflated explosives levels. Some RDX and/or HMX may have been in week 4-8 leachate, but interferences in the leachate did not permit accurate quantititation (Appendix T, Table T-13).

3. Discussion and Conclusions

Composting conditions in hay-horse feed composts were sub-optimal during the first 3 1/2 weeks of the 7-week trial due to improper aeration. Despite these conditions TNT, RDX, and HMX degraded rapidly. Rates of breakdown could be adequately represented by first order kinetic and half-lives of 1.6, 3.0, and 4.7 weeks were found for TNT, RDX, and HMX, respectively. Insufficient data was collected to model tetryl breakdown, but tetryl concentrations decreased 94% during 7 weeks of composting. Rates of loss were as good as or better than those indicated by the laboratory studies. Previous work in this area (Isbister et al., 1982) had also indicated that artifically maintained laboratory scale composts tend to underestimate the rate of explosives loss in larger self-sustained composts.

Losses of explosives during 7 weeks of composting in sewage sludge composts were erratic and generally low. Such results are completely contrary to the findings from the laboratory studies where explosives breakdown were generally faster in the sewage sludge compared to the hay-horse feed compost. In the laboratory studies, all composts were externally heated to maintain a temperature at or near 60°C. In the pilot scale composts, temperatures resided largely in the 48-52°C range. This temperature difference appears to be the key element in the susceptability of the explosives to degradation via composting. The high temperatures in the laboratory studies may reflect higher levels of microbial activity needed to effectively degrade the explosive. The high temperature could be necessary for the proliferation of specific thermophilic organism involved in the breakdown of these explosives. Increased temperatures may be necessary to alter the physical state of the explosives to render them susceptable to chemical or microbial attack. Another alternative is that at elevated temperatures the explosives are not chemically stable in the compost environment. Mechanisms of explosive loss are not sufficiently understood to evaluate the means by which the temperatures regulate the breakdown of explosives.

The manure compost was highly successful in decontaminating the LAAP sediment; RDX, HMX, and TNT levels were reduced 94.4, 81.2, and 99.6%, respectively, within 8 weeks of composting. Tetryl concentrations were reduced below the detection limit (80 ppm) after 27 days of composting. The manure compost was initially inhibited by the high concentrations of explosives and was completely upset after 10 days of composting. Undoubtedly if this inhibitory effect is lessened or eliminated, the rates of decontamination will be improved.

The build up of TNT transformation products (amino derivatives of TNT) was not observed in any of the composts. In the hay-horse feed composts, a small increase in the 2- and 4-aminodinitrotoluene levels coincided with large decreases in TNT. However, the concentration of amino derivatives decreased with increasing the length of composting. Sufficient data are not available to determine if the formation of amino derivatives is an intermediate step in the loss of TNT or if this is an ancillary reaction. In either case, the amino compounds appear to be short lived in the compost.

Leaching of hazardous materials from the composts does not appear to present any significant environmental hazard. The volume of leachate from well managed composts is relatively small, and could easily be collected and added back to the compost. Explosives concentrations in the leachate were either low or not detected. The high levels of natural humic materials leached from the composts interfered with the analyses and could have resulted in falsely inflated explosives levels. It is not certain that explosives were actually present in the leachate. Leachates for the most part were alkaline; thus losses of heavy metals were minimal. Small amounts of copper and zinc were solubilized as ammonium complexes. Leachates also contained moderate concentrations of iron, apparently present as organic chelates.

Sewage sludge and wood chips were not adequate materials to compost the LAAP sediment. Sewage sludge has a high ash content and its organic fraction is partially stabilized at the waste water treatment facility. As a result, sewage sludge composts have relatively low energy reserves available for microbial activity when compared to hay-horse feed or manure -hay composts. The lower energy reserves result in shorter intervals during which composts can be maintained at temperatures above 60°C. In addition, the decreased availability of energy makes the sewage sludge composts more susceptable to the inhibitory effects of LAAP sediment and hinders the recovery of composts when the microbial activity is temporarily upset.

The use of hay and horse feed as composting materials was successful in decontaminating LAAP sediments, and the subsequent use of manure mixed with bedding materials gave significantly improved results. All of these materials are well suited for composting. They are easily aerated and have large reserves of readily available energy with a favorable nitrogen to carbon ratio for maintaining high microbial activity over extended periods of composting (greater than two months). Although the hay-horse feed compost probably provides a better microbial nutrition source, the cost of hay and horse feed for large scale composting is prohibitive. The improved performance of the manure compost over the hay-horse feed compost was largely related to better compost mixing and better daily management. Additional experience in the operation of composts for explosives destruction should lead to further substantial increases in decontamination rates. Improvements in compost formulation and mixing should reduce or eliminate the inhibitory effects of the explosives on microbial activity.

H. Composting Badger AAP Sediment

1. Monitoring and Sampling Procedures

Daily inspections to conform to the requirements of the permit were as described in Section IV.G. 1. Temperature readings were taken daily using a thermocouple probe. Several temperature readings were taken to estimate the overall condition of the compost. Three readings were taken in the center of each compost, near the bottom, midway up the pile and between the middle reading and surface of the compost. Four to eight mid-depth readings were taken midway between the center and the outer edge of the composts, and at least one mid-depth reading was taken 6 inches in from the outer edge at a point adjacent to where temperatures in the compost were near the average. The air flow was adjusted as needed to maintain appropriate compost temperatures.

Air samples were collected weekly. Samples were taken from the headspace above each compost when the blower was not on to determine the composition of gases released by the compost. Samples were also taken from the aeration plumbing (Figure 10) when the blower was running.

Composts were sampled at time zero and then after three and four weeks of composting. Preliminary tests indicated that large quantities of substances which interfere with the nitrocellulose analysis are produced in the compost during the initial composting process (first two weeks). Therefore, no compost samples were taken after one and two weeks of composting. The sampling procedure consisted of digging a narrow (8-inch) trench across the compost to the bottom of the composter. Then a 1- to 2-inch layer was removed from the wall of the trench. This material was thoroughly mixed, and three subsamples were removed for nitrocellulose and moisture analysis. The remaining compost was returned to the composter.

2. Results

a. Hay-Horse Feed Composts

The hay-horse feed composts amended with Badger AAP sediment composted extremely well. The earlier problems associated with cool spots due to the pillows, thermocouples and air diffuser board were fixed by eliminating the pillows and fixed thermocouples, and sealing the crack in the diffuser boards. Temperatures in these composts were significantly higher than those observed in the LAAP composts. Once thermophilic temperatures were reached, Tank 1 ranged from 65-83°C and Tank 2 ranged from 62-86°C (see Appendix U, Table U-1). The air flow rates in the hay-horse feed composts were maintained between 700 and 1200 linear feet per minute (about six times that utilized for the LAAP composts). This high air flow was necessary to maintain the temperature low enough so that the compost would not catch on fire. As a result of the high temperatures and air flow, the composts tended to dry out very quickly. Therefore, to maintain microbial activity, each hay-horse feed compost was watered and mixed twice weekly.

The nitrocellulose concentrations in the compost as a function of composting time are presented in Table XX. The initial concentration of nitrocellulose in the compost was 6655 to 8176 ppm. After three weeks of composting, the nitrocellulose levels in both of the hay-horse feed composts were below the detection limit of 25 µg/g.

The pH of all leachates was in the basic range from 8.1 to 9.0. The only metals detected in the leachate were small amounts of iron, copper and zinc (Appendix U, Tables U-3 and U-4).

b. Sewage Sludge-Wood Chips Composts

The sewage sludge-wood chip composts amended with the BAAP sediment composted better than that observed for the LAAP sediment amended composts. Temperatures in the BAAP sewage sludge-wood chips compost were generally in the 60+°C range (see Appendix U, Table U-1). Air flow was only 100-200 linear feet per minute and the compost did not generate the extremely high temperatures that were observed in the hay-horse feed BAAP compost.

The nitrocellulose in the zero time composts were 1449 to 5687 µg/g. The 1449 number is way below the calculated amount of nitrocellulose mixed in the compost. This low time zero nitrocellulose value is due to problems with the analysis. The "A" barrel BAAP soil contained another substance which tended to gel during the nitrocellulose hydrolysis and give artificially low nitrocellulose values. Composting for three weeks led to a decrease in the nitrocellulose concentration to below the 100 µg/g detection limit in one case. Small increases in the nitrocellulose concentrations were observed after four weeks of composting. These increases could be due to organic acids which react with the color reagent. In any case, a significant decrease in the nitrocellulose concentration was observed in both sewage sludge-wood chips composts. Only small quantities of zinc, iron and copper were found in the sewage sludge leachates in spite of relatively high levels of Pb, Ba and Cr in the original compost material.

Table XX. Concentration of Nitrocellulose in Compost Amended with Badger AAP Soil as a Function of Composting Time

		Nitrocellulose in μg/g*			
	Tank #	Calculated NC Input	Analysis of Time Zero Compost	Analysis of Week 3 Compost	Analysis of Week 4 Compost
HHF	1	8435	8176	<25	NA
HHF	2	8430	6655	<25	NA
SS	4	7350	1449	100	117.2
SS	5	6779	5687	184.9	202.7

* Detection limit for NC in hay-horse feed composts is 25 μg/g.
Detection limit for NC in sewage sludge-wood chips compost is 100 μg/g.

3. Discussion and Conclusions

Nitrocellulose is rapidly degraded in both hay-horse feed and sewage sludge-wood chips composts. In fact, the addition of the BAAP soil containing the nitrocellulose enhances the composting process over that observed for controls (no explosives contamination). Leachates were not analyzed for nitrocellulose because of the insolubility of nitrocellulose in water. Only small quantities of iron, copper, and zinc were found in the leachate.

Several studies have been conducted to determine the ability of mesophilic microorganisms to breakdown esterified celluloses. In essence, the literature indicates that these microbes cannot attack esterified cellulose. This information led to the question as to whether the thermophilic compost microbes were actually attacking the nitrocellulose or if the observed degradation was simply due to thermal decomposition. To resolve this question, literature was gathered on the thermal degradation of nitrocellulose. Thermal degradation studies on nitrocellulose were conducted by Leider and Pane (1981). They measured both the $-ONO_2$ ester content and molecular weight of nitrocellulose after thermal aging at various temperatures for various times. The loss of $-ONO_2$ ($2(-ONO_2) \rightarrow$ product) is described by the second order rate constant.

$$k_2t = \frac{1}{C(0)} \left[\frac{C(0) - C(t)}{C(t)} \right]$$

where k_2 is the second order rate constant

$C(0)$ is the initial concentration

$C(t)$ is the concentration after time, t.

The molecular weight degradation is first order and described by the following equation:

$$\frac{1}{M_n(t)} = \frac{1}{M_n(0)} + k_1t$$

k_1 is the first order rate constant where $M_n(0)$ is the initial number average molecular weight

$M_n(t)$ is the number average molecular weight at time, t.

Rate constants calculated from their data are as follows:

T°C	$k_2(T)$ (nitrate ester loss)/day	$k_1(T)$ (molecular wt degradation)/day
80	1.3×10^{-4}	4.8×10^{-7}
90	5.8×10^{-4}	8.9×10^{-7}
100	2.2×10^{-3}	3.5×10^{-6}

Using the rate constants for nitrate ester loss, the degradation predicted from thermal decomposition was compared to the compost degradation after three weeks. The compost degradation was 108 and 24 times faster than that due to thermal effects alone at 80°C and 90°C, respectively.

After comparing the thermal degradation rates with the composting degradation rates of nitrocellulose, it was concluded that the degradation observed during the composting process is due to the action of thermophilic microorganisms in the compost and not due to thermal aging.

The major problem associated with using composting as a method to clean up nitrocellulose contaminated soils and sediments is in analysis. No specific method to identify and quantitate nitrocellulose exists. Methods in current use require that the nitrocellulose be hydrolyzed to produce nitrite which can be determined with color reagents or specific ion electrodes. The Badger soil contains some other substance which produces a gel during the hydrolysis reaction yielding false low concentrations for nitrocellulose. The color reagents also react with organic acids and formaldehyde, which are often produced during the composting process, giving a false high concentration for nitrocellulose. Nitrite specific ion electrode was evaluated for quantitation of the nitrite produced from hydrolysis of nitrocellulose during this study. However, use of an electrode to determine nitrite is also subject to several interferences from compost breakdown products. These problems combined with the different solubility characteristics of the various grades of nitrocellulose make this explosive an analytical nightmare.

New research in the forensic identification of nitrocellulose has been based on size exclusion chromatography. Lloyd (1984) used reductive mode electrochemical detection with a pendant mercury drop electrode coupled with size-exclusion chromatography to detect as little as 100µg of nitrocellulose. Some effort was expended during this contract to use size-exclusion chromatography, coupled with electrochemical detection for nitrocellulose with some success. However, funds were not available for analytical methods development during this contract and the effort had to be abandoned before it could be perfected. It is recommended that further investigation be conducted to develop a method that is specific for nitrocellulose.

The BAAP sediment composted extremely well in both the laboratory scale and pilot scale tests. The composting technique for nitrocellulose is ready for a field demonstration at Badger AAP. A demonstration and costs for the demonstration and full-scale treatment facility are presented in Section VI. Composting is an economical method of decontaminating BAAP soil and could be used to process wastewater should the facility be put back into operation. Local materials, eg. corn stalks and cow manure, would make excellent compost materials for inexpensively decontaminating this soil.

I. EP Toxicity - Ames Assay of Compost Leachates

1. Sample Preparation

Water extracts (leachates) of the pilot-scale composts were prepared as described in the EP Toxicity Test Procedures in 40 CFR 261 App. II (one hay-horse feed, one sewage sludge-wood chips and one manure-hay-saw dust compost). A water extract (leachate) was also prepared from control composts which were not contaminated with explosives. These extracts served as control compost leachates for the Ames tests. All extracts were centrifuged to remove large particulate matter, filtered through Whatman No. 50 filters, 0.45 μ and 0.2 μ Gelman sterile membrane filters. Sterility of the extracts was monitored by spotting filtered extracts on nutrient agar plates.

2. Toxicity Testing and Ames Assay

Five Salmonella tester strains, TA1535, TA1537, TA1538, TA98 and TA100, were obtained from B. Ames, Department of Biology, University of California, Berkeley. The liver homogenate (Aroclor-induced S-9) was obtained from Litton Bionetics, Inc., Kensington, Maryland. The mutagen assay was performed as described by Ames et al. (1975). Replicate extracts were tested in duplicate. Control mutagens not requiring S-9 activation were 2-nitrofluorene (obtained from Aldrich Chemical Company and with TA1538 and TA78), and N-methyl-N'-nitrosoguanidine (MNNG) (obtained from Sigma Chemical Company and used with TA1535 and TA100). 2-Aminoanthracene (Aldrich Chemical Company) was used as the positive control mutagen for metabolic activation with S-9.

Preliminary testing of the compost extracts indicated that the sewage sludge-wood chips compost extracts were toxic to the tester strains at 100 μL/plate. Extracts of the hay-horse feed and manure-hay-saw dust composts inhibited the growth of the tester strain lawns at 100 μL/plate. Based on these observations, each of the leachates was tested at 50 μL/plate in the incorporation assay. Spot testing of the leachates at 50 μL on each of the tester strains showed slight inhibition of the bacterial lawns but no increased colony formation around any of the 'spots' on the tester strain lawns.

3. Results and Discussion

Data from Ames testing of duplicate extracts of each compost are presented in Tables XXI through XXIII. Each leachate was tested in the incorporation assay at 50 µL/plate. One set of tests was run without addition of the rat liver metabolic activating enzymes, with a second set of identical tests performed with the addition of the metabolic activation system (S-9 fraction of an Aroclor induced rat liver). As shown in the Tables, the compost extracts tested at 50 µL per plate gave no mutagenic responses in the plate incorporation assays with tester strains capable of detecting frame-shift mutations (TA1537, TA1538, TA98) or tester strains detecting base-pair substitutions (TA1535 and TA100).

Mutagens used to demonstrate positive reactions in the tester strains included MNNG (N-methyl-N'-nitro-nitrosoguanidine), 2AA (2-amino-anthracene), 2NF (2-nitrofluorene) and 9AA (9-aminoacridine).

In general, a negative result in the Ames test is defined as the absence of a reproducible increase at least equal to, or greater than, twice the number of spontaneous revertant histidine-independent colonies. These compost extracts did not elicit a mutagenic response in any of the tester strains indicating that neither mutagenic substances nor promutagens (substances mutagenic only when metabolized) detected by this assay are present in the water extracts of composts used to decontaminate sediments from LAAP and BAAP.

Table XXI. Ames Assay Data on Extracts From Hay-Horse Feed and Sewage Sludge Composts Amended with Louisiana AAP Sediment

	TA1535		TA1537		TA1538		TA98		TA100	
	No S-9	S-9	No S-9	S-9	No S-9	S-9	No S-9	S-9	No S-9	S-9
Tank 1	16, 12	30	16	8	28	30	30	53	118	139
LAAP	10	43	12	10	36	30	28	87	107	157
HHF	13	45	11		40		52	85	110	163
			10		38		40		93	
					42					
Tank 2	9, 9, 9	29	8	12	50	31	30	37	115	154
LAAP	13	29	10	8	40	35	27	41	135	100
HHF			9		37		28		110	181
			10		49		25			175
Tank 3	9, 13	19	7	10	50	30	23	81	91	190
Control	17, 15	35	6	12	53	26	28	73	90	143
HHF			7		28		24	71	90	137
			5		30		30		83	
Tank 4	16, 10	49	6	9	14	23	21	89	90	103
LAAP	14, 12	31	5	10	12	27	18	70	81	131
SS/WC		23	10	11	5	26	16	78		121
			12	11	30	17	83			168
Tank 5	14, 13	21	6	7	17	25	19	89	84	127
LAAP	13, 16	25	7	6	18	21	17	80	95	135
SS/WC		29	4	6	15	23	17	82	102	160
			4		16	25	15	79	95	124
Tank 6	13, 16	34	8	7	9	29	24	60	96	155
Control	15, 12	29	7	6	11	23	25	57	80	128
SS/WC		27		9	35	21	23	59	75	113
		25			27	23	31	61	86	146
Reversion	13	25	6	10	27	32	31	44	83	110
Positive Control	10 μg MNNG 250	-	-	2.5 μg 2AA 228	10 μg 2NF 391	2.5 μg 2AA 958	10 μg 2NF 557	2.5 μg 2AA 1055	10 μg MNNG 1008	2.5 μg 2AA TNTC

Table XXII. Ames Assay Data on Extracts From Manure-Hay Composts Amended with Louisiana AAP Sediment

	TA1535		TA1537		TA1538		TA100	
	No S-9	S-9	No S-9	S-9	No S-9	S-9	No S-9	S-9
Tank 5	16	17	12	34	27	81	160	158
LAAP	11	18	10	30	20	77	134	183
Manure-	15	15	8	24	18	65	139	183
Hay	15	20	7	-	21	52	142	201
Tank 6	13	28	9	31	21	64	131	189
Manure-	13	25	10	32	20	63	150	179
Hay	18	15	8	25	16	76	123	192
Control	19	24	10	25	22	80	132	163
Reversion	18	20	12	27	18	52	120	173
Positive Control	10 μg	-	50 μg	10 μg	10 μg	10 μg	10 μg	2.5 μg
	MNNG	-	9AA	2AF	2NF	2AF	MNNG	2AA
	65	-	800	95	TNTC	TNTC	515	530

Table XXIII. Ames Assay Data on Extracts From Composts Amended with Badger AAP Soil

	TA1535		TA1537		TA1538		TA98		TA100	
	No S-9	S-9	No S-9	S-9	No S-9	S-9	No S-9	S-9	No S-9	S-9
Tank 7	12 20	25	6	7	26	25	33	38	68	133
BAAP	17, 11	25	5	7	41	19	31	25	80	153
HHF		29	7	13	10	35	40	25	78	134
		35	7		13	39				156
Tank 8	16, 16	44	8	15	42	41	40	25	60	116
BAAP	14, 16	53	11	12	44	45	41	34	62	136
HHF		45	9		18	28	21	21	68	110
			10		26	33	23	19		115
Tank 9	12, 11	33	5	10	6	31	10	25	68	123
BAAP	14, 14	34	5	13	8	29	12	41	71	136
SS/WC		30	6	7	12	25		44	74	107
		27	5		8	23		43		107
Tank 10	7, 6	15	8	8	17	21	15	49	76	124
BAAP	8, 7	19	11	5	29	25	12	55	78	136
SS/WC		18	8	6	8	19	12	80	70	112
		21	10	5	10	17	14	74	90	118
Reversion	13	25	6	10	27	32	31	44	83	110
Positive Control	10 µg MNNG 250	-	-	2.5 µg 2AA 228	10 µg 2NF 391	2.5 µg 2AA 958	10 µg 2NF 557	2.5 µg 2AA 1055	10 µg MNNG 1008	2.5 µg 2AA TNTC

V. CONCLUSIONS AND RECOMMENDATIONS

Breakdown of both the LAAP (TNT, RDX, HMX, and tetryl) explosives in hay-horse feed and manure composts was highly successful. Explosives degradation was relatively fast, and no toxic or objectionable degradates accumulated in the composted materials. The materials in the hay-horse feed compost are expensive; however, horse manure and other similar materials can be obtained at minimal or no cost in the locality of military installations.

Nitrocellulose in BAAP soil enhances the hay-horse feed composting significantly. Thus, any fairly high energy material, such as cattle manure, would be effective as a composting material to degrade nitrocellulose.

No breakdown of explosives in the LAAP sediment-sewage sludge composts was observed in these studies and loss of nitrocellulose in sewage sludge composts was somewhat slower than in hay-horse feed compost. The energy content of sewage sludge is not adequate to sustain high temperature composting conditions for extended periods of time. Sewage sludge composts also do not recover well if upset by toxic materials or unfavorable composting conditions. Further work with sewage sludge compost as a means to degrade explosives is not warranted.

In summary, it is recommended the following activities be pursued to bring composting to a full-scale treatment process for contaminated lagoon sediment:

- No additional work using sewage sludge-wood chips should be pursued for degradation of hazardous materials because of the limited energy source available in these materials.
- Composting LAAP sediment with hay and horse feed or manure and bedding materials (hay, saw dust, etc.) has been shown to be an effective decontamination procedure. It is recommended that composting be tested in a full scale demonstration at LAAP.

- TNT is the major contaminant in the LAAP sediments. Although TNT is rapidly lost during composting, the mechanism of TNT loss and the fate of the TNT molecule are not known. It is recommended that laboratory studies be conducted (prior to or concurrent with the LAAP demonstration) to investigate the mechanism(s) of TNT loss, to identify degradates, and to determine the fate of these degradates in the environment. This information will be helpful, if not essential, in obtaining a delisting of composted explosives.
- Nitrocellulose in the BAAP soil increases the rate of composting. Thus, composting should provide an inexpensive method for decontamination of BAAP soil. It is recommended that composting of nitrocellulose proceed to a demonstration at BAAP.

VI. COMPOST DEMONSTRATION AND PRELIMINARY FULL SCALE COMPOST DESIGN AND ECONOMICS

A. Composting Demonstration at Badger AAP

A time-task chart for a field demonstration of composting as a means of degrading nitrocellulose from BAAP soil is presented in Figure 21. The BAAP demonstration program will take approximately 15 months to complete including the following:

- obtain EPA RCRA R,D&D permit
- develop a better method to quantitate nitrocellulose in soil and compost
- site preparation
- evaluation of three full scale composting scenarios
- evaluation of compost curing methods
- final design and economics for full scale clean-up of BAAP soil by composting.

The total cost of this field demonstration program is $298,892 including $57,791 for materials of construction.

B. Composting Demonstration at Louisiana AAP

Demonstration of the composting technique for decontamination of TNT, RDX, HMX and tetryl from LAAP sediment will require 14 months to complete as shown in Figure 22. The demonstration program should include the following tasks:

- obtain Superfund approval
- identify and determine the fate of composted TNT
- site preparation
- evaluate three rates of sediment loading during full scale composting operations
- evaluate the need for benefits of recomposting (optional)
- evaluate compost curing methods

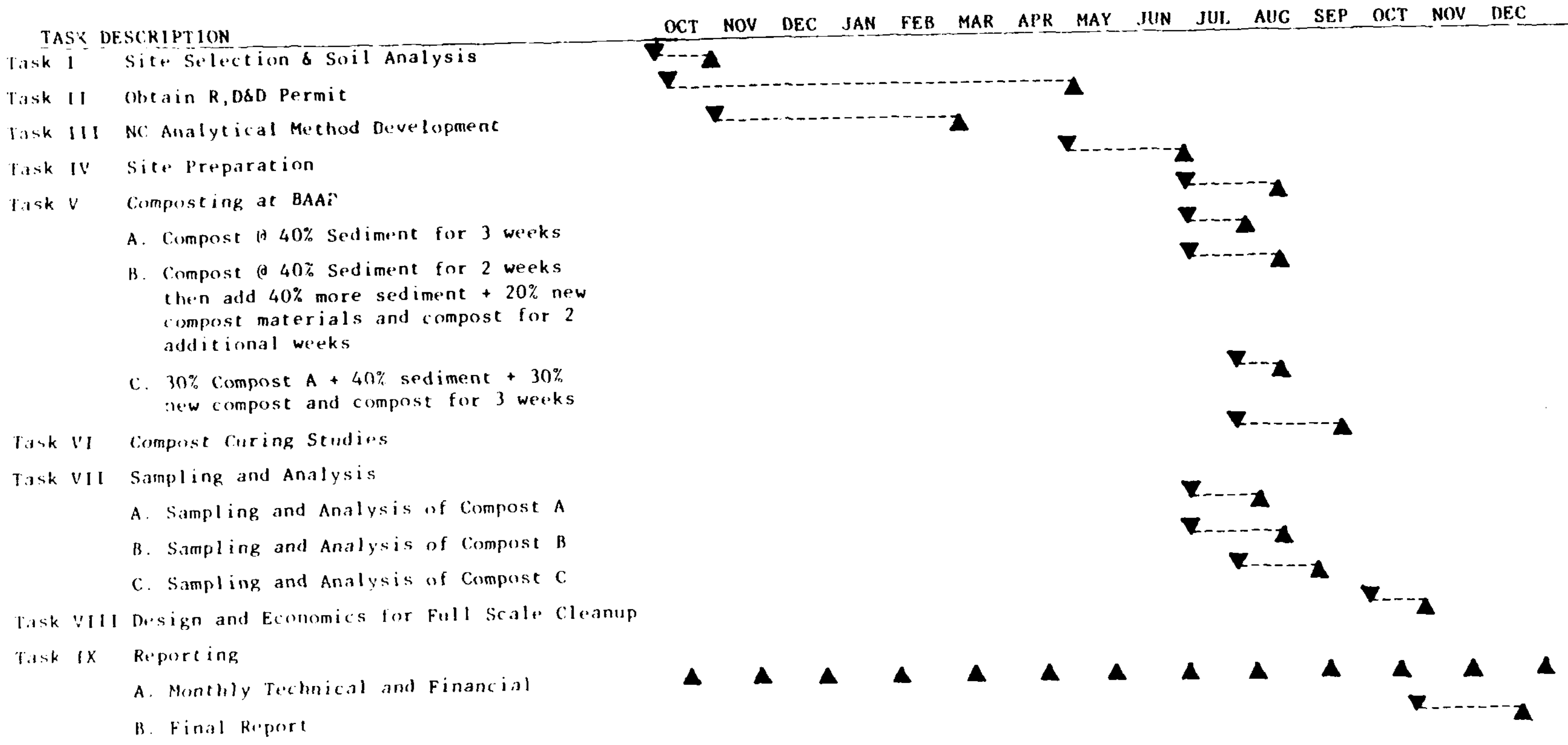

Figure 21. Time Task Chart for Composting of Badger AAP Soil

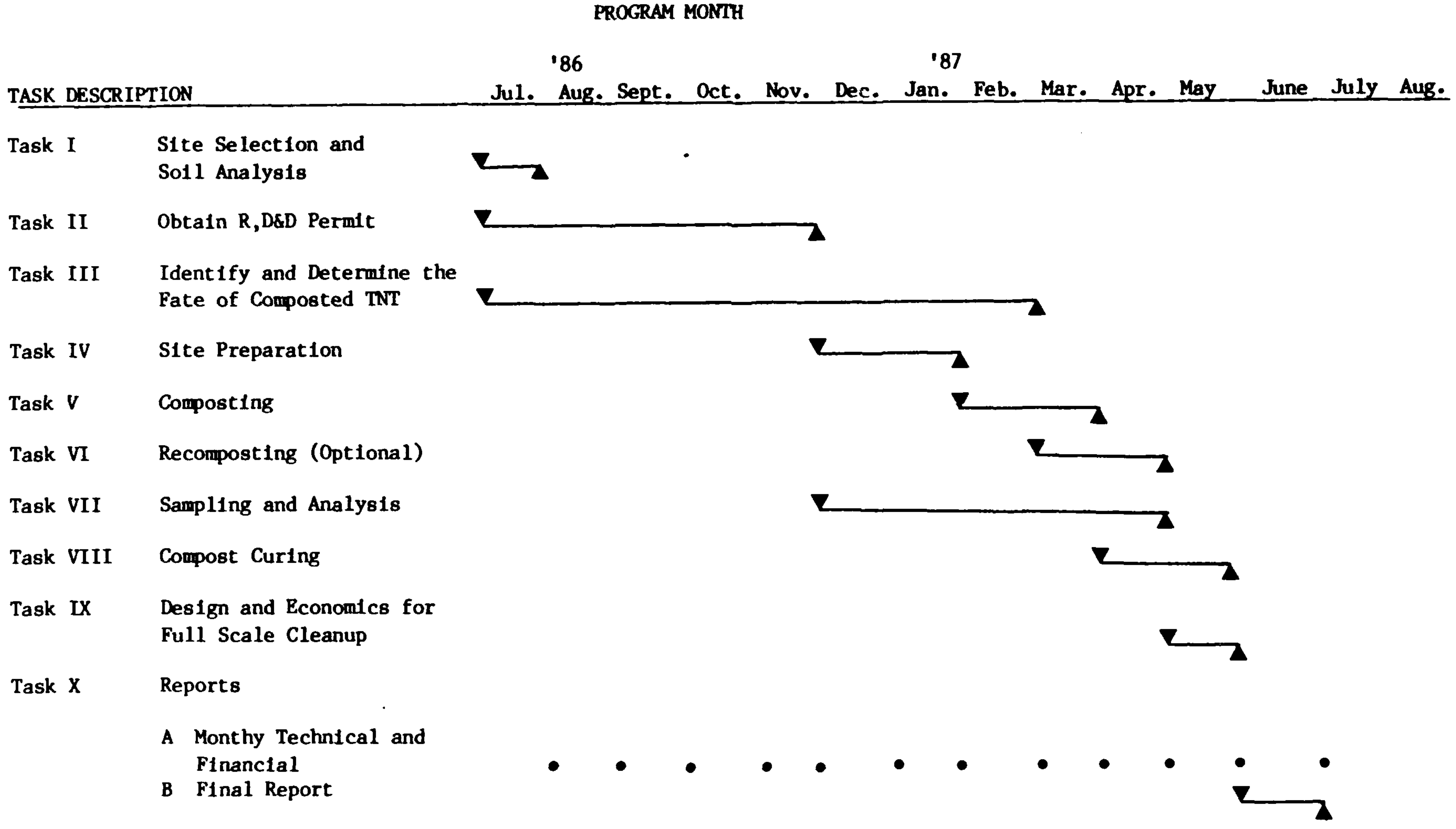

Figure 22. Time Task Chart for Composting of Louisiana AAP Soil

- final design and economics for full scale and curing methods clean-up of LAAP sediment by composting.

The total cost for this field demonstration program is $458,424 including $72,786 for materials of construction.

C. Preliminary Design and Economics for a Full Scale Cleanup of BAAP Soil by Composting

In performing this preliminary design and economics for a full scale cleanup of BAAP soil by composting, several assumptions were made. These assumptions are listed below:

- twenty-five acres of soil contaminated to a depth of 2 feet must be cleaned up (80,700 cubic yards)
- density of the soil is approximately 2500 lb/cubic yard
- soil moisture at field saturation level is approximately 44%
- compost materials consist of manure, hay or other bedding material obtained locally at little or no cost
- density of the composting materials is approximately 200 lb/cubic yard
- compost materials moisture is approximately 65%
- soil amended to compost at a level of 40%
- compost moisture maintained at 50-65%
- composting time for complete degradation of nitrocellulose is 2 to 3 weeks
- composting will be conducted on a concrete pad with a roof to minimize leachate problems
- windrow-forced aeration composting is used with the material turned 2 times per week

The layout of a full scale compost system is shown in Figure 23. This system consists of 24 windrow composts. Each windrow is 6 feet in height, 14 feet at the base, 3 feet across the top and 140 feet in length and contains 264 cubic yards of material. At 40% sediment addition, each compost will initially contain 17.8 tons of soil (dry wt basis) and 26.7 tons of compost materials. After composting for two weeks, the compost will be remixed with the addition of 17.8 tons of soil and 5.3 tons of new

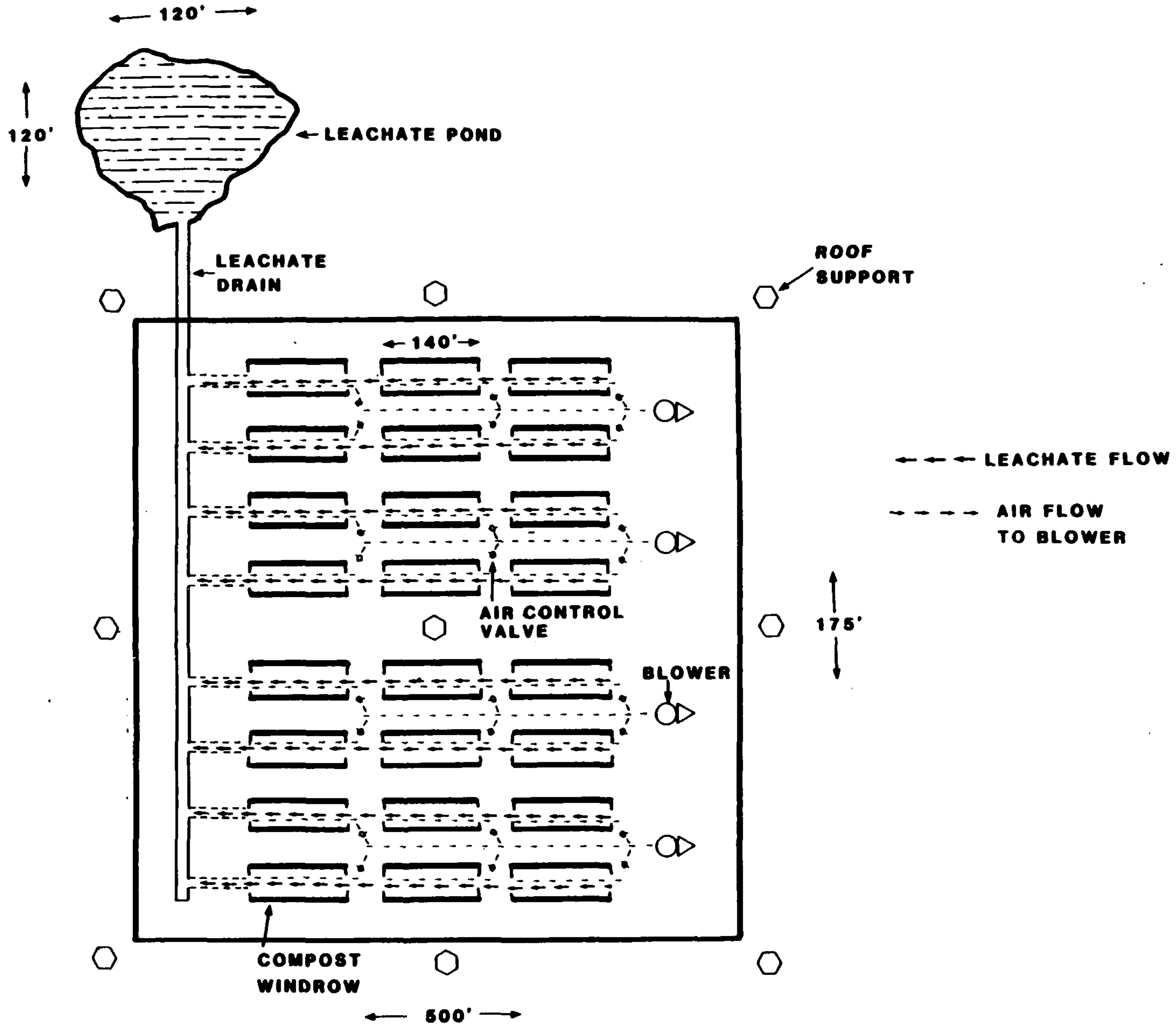

Figure 23. Layout of Full Scale Composting Plant at Badger AAP

compost materials. With 24 windrows in simultaneous operation, cleanup of the 80,700 cubic yards of BAAP soil will required 9 years. The capital cost for the system are presented in Table XXIV. Operating costs are presented in Table XXV. Costs for decontamination of 80,700 cubic yards of BAAP soil are estimated to be $2,620,200 compared to $8,070,000 to $16,140,000 for fuel for incineration (no capital or labor).

D. Preliminary Design and Economics for a Full Scale Cleanup of Louisiana AAP Sediment by Composting

Several assumptions were made in completing the design and economics for a full scale cleanup of explosive contaminated lagoons at LAAP by composting. These assumptions are as follows:

- a total of 5 acres of land (lagoon bottom sediments) contaminated to a depth of 2 feet must be cleaned up (16,133 cubic yards)
- density of the sediment (dry) is 2500 lb/cubic yard
- Moisture levels in the sediment are approximately 50% at saturation
- composting materials (manure, straw, hay and other bedding materials) can be obtained locally at little or no cost
- density of the composting materials is approximately 200 lb/cubic yard
- moisture in the composting materials is 60-65%
- contaminated sediment will comprise 20% of the initial compost dry weight
- composting time for the complete destruction of explosives is 6 weeks
- composting will be conducted on a concrete pad with a roof to minimize leachate problems
- window-forced aeration composting is used with materials being turned and mixed 1 to 2 times per week.

The full scale composting facility for treatment of the LAAP sediment will be identical in design but twice as large as the BAAP site (Figure 23). Windrow size will also be the same and each compost will contain 264 cubic yards of composting materials (267 dry tons). Contaminated sediment will comprise 20% of the initial dry mass of each compost (11.4 tons of

Table XXIV. Capital Costs for Badger AAP Composting Plant

Leveling, grading of land	3,200
Concrete pad with 1-ft x 1-ft troughs	300,000
Roof and support trusses	106,000
Aeration system	
Piping	13,500
Valves	4,900
Blower	18,000
Metal for aeration system cover	19,300
Liner for leachate pond	33,400
SCARAB composter	35,000
Front end loader	20,000
	$535,300

Table XXV. Operating Costs for Full Scale Composting of Badger AAP Soil

Operators	
3 people for 9 years @ 20,000	540,000
Benefits @ 25% salaries	135,000
Analysis	
1 person for 9 years @ 20,000	180,000
Benefits @ 25% salaries	45,000
Maintenance @ 3,000/year	27,000
Electricity @ 2,000/year	18,000
Miscellaneous @ 3,000/year	27,000
Compost materials 890,200 cubic yards @ 50/40 cubic yards	1,112,900
	$2,084,900

sediment/compost). Destruction of the explosives will be complete with 6 weeks and the compost will be removed from the concrete pad and transported to an adjacent area for final curing. Continuous operation of 48 windows will decontaminate the estimated 16,133 cubic yards of sediment in 4.2 years. Capital costs and operating costs for this facility are presented in Tables XXVI and XXVII. The estimated total costs for sediment decontamination by composting (16,133 cubic yards) are $2,431,150, compared to fuel costs of $1,613,300 to $3,226,600 for incineration (capital and labor costs for incineration not included).

Table XXVI. Capital Costs for Louisiana AAP Composting Plant

Leveling, grading of land	6,400
Concrete pad with [illegible]-ft x 1-ft troughs	500,000
Roof and support trusses	212,000
Aeration system	
Piping	27,000
Valves	9,800
Blower	36,000
Metal for aeration system cover	38,600
Liner for leachate pond	66,800
SCARAB composter	35,000
Front end loader	20,000
	$951,600

Table XXVII. Operating Costs for Full Scale Composting of Louisiana AAP Sediment

Operators	
3 people for 4.3 years @ 20,000	258,000
Benefits @ 25% of salaries	64,500
Analysis	
1 person for 4.3 years @ 20,000	86,000
Benefits @ 25% of salary	21,500
Maintenance @ 6,000/year	25,800
Electricity @ 3,000/year	12,900
Miscellaneous @ 6,000/year	25,800
Compost materials 788,040 cubic yards @ 50/40 cubic yards	985,050
	$1,479,550

VII. REFERENCES

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40 CFR 261 App. II, EP Toxicity Test Procedures.

Isbister, J.D.; R.C. Doyle and J.F. Kitchens (1982) "Engineering and Development Support of General Decon Technology for the U.S. Army's Installation Restoration Program, Task II, Composting of Explosives" USATHAMA Contract DAAK11-80-C-0027.

Leider, H.R. and A.J. Pane (1981) "Degradation of the Molecular Weight and Nitrate Ester Content of Cellulose Nitrate on Thermal Aging", NTIS DE81 029875.

Lloyd, J.F.D. (1984) "Detection of Differentiation of Nitrocellulose Traces of Forensic Science Interest with Reductive Mode Electrochemical Detection at a Pendant Mercury Drop Electrode Coupled with Size-Exclusion Chromatography," Anal. Chem., 57, 1907-1912.

Appendix A

Synthesis of ^{14}C-RDX

^{14}C-RDX was synthesized via the processes outlined by Bachmann et al. (1951). This process is carried out by nitrating hexamethylenetetramine dinitrate with 98% nitric acid in the presence of acetic anhydride and ammonium nitrate.

The experimental setup used for this synthesis is shown in Figure A-1. The reaction vessel was a 25 mL, 3-necked, round-bottomed flask. The flask was supported in a water bath. Stirring was provided with a 1/4- x 1/16-inch magnetic stir bar and a water driven, magnetic stirrer located in the water bath under the flask. Reagents were added via two 2 mL disposable pipets which were mounted in teflon sleeves in the right and left side necks. The flow of liquid was controlled by a bulb on each pipet. Solid reagents were added via a small funnel located in the center neck.

The ^{14}C-hexamethylenetetramine dinitrate was made as follows. 0.43 g of reagent grade hexamethylenetetramine was placed in a 10 mL beaker. Approximately 0.5 mCi of ^{14}C-hexamethylenetetramine (purchased from Pathfinder Laboratories) in 0.75 mL of methylene chloride was added to the beaker and the solvent evaporated. The beaker was placed in an ice salt bath on a water driven, magnetic stirrer. Then 0.47 mL of 70% nitric acid was added to the hexamethylenetetramine in the beaker with stirring. The mixture was maintained at 5°C for 15 minutes. The precipitated ^{14}C-hexamethylenetetramine dinitrate was collected by vacuum filtration and dried in a vacuum oven at 20°C.

To prepare the RDX, 0.5 g ammonium nitrate and 0.3 mL of acetic acid were added to the three-necked, round bottomed flask and warmed to 75°C. To this mixture, 0.3 mL of acetic anhydride and 0.16 g of ^{14}C-hexamethylenetetramine dinitrate were added. 1.45 mL of acetic anhydride, 0.26 mL of 98% nitric acid and 0.49 g hexamethylenetetramine dinitrate were alternately added to the flask via the pipets or funnel over a 15 minute time period while maintaining vigorous stirring. The mixture was allowed to remain at 75°C for an additional 15 minutes. The mixture was then cooled

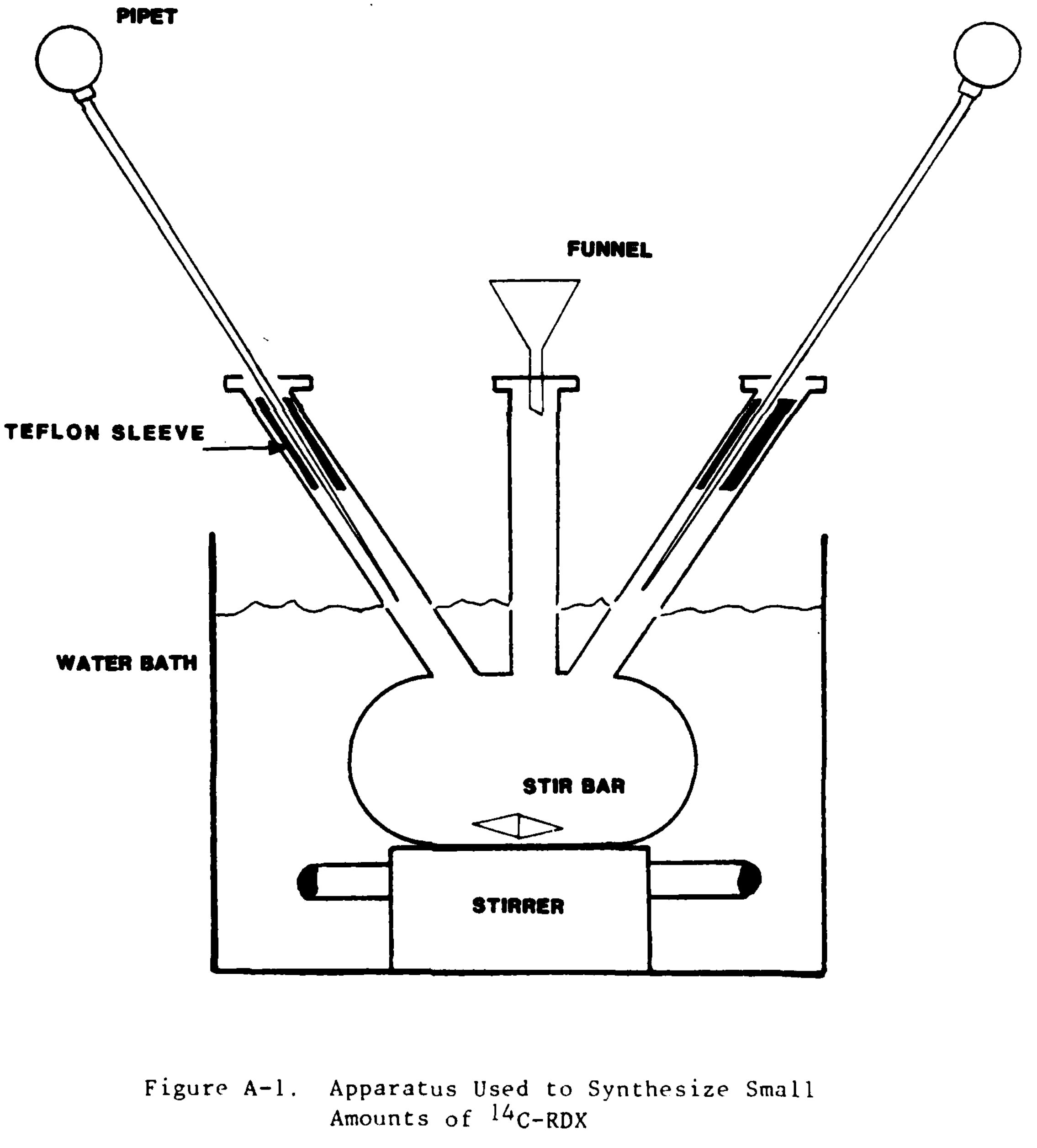

Figure A-1. Apparatus Used to Synthesize Small Amounts of ^{14}C-RDX to 60°C, and the precipitated RDX was filtered from the hot mixture with a Millipore filter and 0.45 μ teflon filter disc. The crude ^{14}C-RDX was recrystallized from acetone. The resultant product had a purity of 90% and a specific activity of 0.38 μCi/mg.

Reference

Bachmann, W.E.; W.J. Horton; E.L. Jenner; N.W. MacNaughton and L.B. Scott (1951), "Cyclic and Linear Nitramines Formed by Nitrolysis of Hexamine," *J. Amer. Chem. Soc.*, *73*, 2769-2773.

Appendix B

Synthesis of ^{14}C-HMX

^{14}C-HMX was synthesized according to the method of Solomon and Silverman (1978). The reaction vessel shown in Figure A-1 was used in the synthesis except the funnel was replaced with a third pipet.

Three mixtures were prepared as follows:

1. Heel: 0.04 g ^{14}C/reagent grade hexamethylenetetramine was dissolved in 5.1 g glacial acetic acid and 0.04 g acetic anhydride.
2. 38% hexamethylenetetramine solution: 0.2 g ^{14}C/reagent grade hexamethylenetetramine was dissolved in 3.3 g glacial acetic acid.
3. 56.4% HNO_3/NH_4NO_3 solution: 0.37 g NH_4NO_3 was dissolved in 0.48 g HNO_3.

The reaction was initiated by placing the "heel" solution in the reaction vessel and heating to 45°C in a water bath. The hexamethylenetetramine solution, 0.33 g of the 56.4% HNO_3/NH_4NO_3 solution and 1.1 g acetic anhydride were then alternately added to the heel over a period of approximately 20 minutes. The reaction mixture was allowed to age for 6 minutes. The remainder of the 56.4% HNO_3/NH_4NO_3 solution was then added alternately with 1.6 g of acetic anhydride over a period of 15 minutes. The mixture was then aged for 30 minutes. After the aging process, the mixture was added to 3.5 mL of water and heated to 110°C for one hour to decompose linear nitramine by-products. The mixture was then cooled, filtered through a 0.45 μ teflon filter and air dried. The resultant product had a specific activity of 0.198 μ Ci/mg.

Reference

Solomon, I.J. and L.B. Siverman (1978), "Process for Preparing Cyclotetramethylenetetramine," U.S. Patent No. 4,086,228.

Appendix C

Synthesis of ^{14}C-Tetryl

Tetryl was synthesized according to the procedures outlined by Clarkson et al. (1950). The reaction was carried out in a 25 mL, three-necked, round bottomed flask fitted with a thermometer in the left neck and a 2 mL pipet in the right neck. The flask arrangement was placed on a water driven magnetic stirrer in a water or ice bath. Stirring was accomplished with a 1/4-in x 1/16-in magnetic stir bar.

Two solutions were prepared - a solution of dimethylaniline in concentrated sulfuric acid (D.M.A.S.), and the nitrating acid. D.M.A.S. was prepared by first mixing 0.6 mL of reagent grade N,N-dimethylaniline with 100μ Ci of ^{14}C-N,N-dimethylaniline (from Pathfinder Laboratories, Inc.). The ^{14}C-N,N-dimethylaniline mixture was then added to 1.8 mL 96% sulfuric acid in a 5 mL beaker while stirring and cooling. The nitrating acid was prepared by adding 3.6 mL of 99% sulfuric acid to 2.5 g crushed ice in a 20 mL beaker. The mixture was stirred and cooled in an ice bath. Then 2.3 mL of 98% nitric acid were added to the sulfuric acid/water mixture.

The nitration reaction was carried out by placing 2.4 mL of the nitrating acid in the 25 mL flask. The D.M.A.S. solution was slowly dripped into the nitrating acid from the pipet while maintaining the temperature of the reaction between 30 and 35°C. A red color was produced with the addition of the D.M.A.S. When the addition was complete, the reaction vessel was placed in a 90°C water bath until the temperature reached 60°C. The temperature continued to rise on its own but was not allowed to exceed 100°C (cool with an ice bath). The solution turned purple followed by separation of tetryl and violent evolution of brown fumes. After completion of the fume-off, the mixture was heated in a water bath (70°C) for 30 minutes to clarify the solution and expel the residual oxides of nitrogen. The mixture was then cooled and the precipitated tetryl collected with a Millipore filter and a 0.45 μ teflon filter disc.

The tetryl was purified by suspending it in 20 mL of water and blowing steam through the water for 30 minutes. After cooling, the tetryl was filtered and vacuum dried. The resultant ^{14}C-tetryl had a specific activity of 0.0755 μ Ci/mg.

References

Clarkson, C.E.; I.G. Holden and T. Malkin (1950), "The Nitration of Dimethylaniline to Tetryl, 2:4:6: N-Tetranitromethylaniline. The Course of the Reaction," J. Chem. Soc., 1556-1562.

Appendix D
Synthesis of ^{14}C-Nitrocellulose

^{14}C-Nitrocellulose was prepared by a method similar to the laboratory procedure used at Picatinny Arsenal (1966) using the mixed acid method for 13.4% N nitrocellulose.

.25 mCi of ^{14}C-cellulose (10.3 mg) from tobacco was mixed with 718 mg of microcrystalline cellulose. The cellulose was placed in a 50 mL beaker and the nitrating acid mixture was added. This mixture consisted of 13.7 mL 98% H_2SO_4, 5.7 mL 90% HNO_3 and 3.3 mL of distilled water. The nitration mixture was placed in a water bath and the temperature was maintained at 34°C for 30 minutes.

The reaction was then quenched by dumping the nitrocellulose and mixed acids into 100 mL of distilled water and filtering with a Buchner funnel using a 1.6 μ pore glass fiber filter. The filtrate was then washed with 3 x 100 mL portions of distilled water. After washing, 0.5 mL of H_2SO_4 and the filtrate were added to 100 mL water. This solution was placed in a 90°C water bath for approximately 60 hours. When the acid boil was completed, the nitrocellulose was filtered again and the 100 mL of fresh distilled water was added. The pH was adjusted to 8.5 with Na_2CO_3 and the nitrocellulose mixture was placed back on the 90°C water bath for an additional hour. At this point the nitrocellulose was filtered, washed with 2 x 100 mL portions of distilled water, and dried in a vacuum oven at 40°C. Yield was 1.09 g nitrocellulose. The specific activity was 0.158 μ Ci/mg.

Reference

Encyclopedia of Explosives and Related Items (1966), Vol. 2. C102.

Appendix E
Analysis of TNT, RDX, HMX and Tetryl in Sediment

TNT, RDX, HMX, and tetryl were extracted from the Louisiana sediment by the following procedure. Fifteen mL of HPLC grade acetonitrile were added to a weighed amount (approximately 1 g) of air dried sediment. The mixture was thoroughly shaken to extract the explosives from the soil and then centrifuged. The liquid was carefully removed from the soil with a Pasteur pipet and placed in a 100 mL volumetric flask. The sediment was extracted three additional times with acetonitrile and the extracts combined in the 100 mL volumetric. If high levels of the explosives were expected, the extract was brought up to 100 mL with acetronitrile. If low levels of explosives were expected, the extract was brought up to 100 mL with distilled water.

Analysis of the samples were accomplished using a HPLC consisting of a LKB 2150 HPLC pump, a Perkin-Elmer LC55 UV detector and a HP 5880 GC data system, computer controller and integrator. To accomplish the separation of the explosives, 4.6 mm I.D. x 25 cm 5 μm spherical ODS column purchased from Alltech was used with a mobile phase consisting of 51% methanol, 2% dioxane, 0.5% acetonitrile and 46.5% high purity water which was 0.004 M in n-hexylamine and 0.004 M in tetramethylammonium hydroxide buffered to pH 7.5 with phosphoric acid. The mobile phase flow rate was 1.3 mL/min. The injection volume was 20 μL using a Valco 6-port valve with 20 μL loop. Detection of the explosives was a 232 nm. Under these conditions the retention time and detection limit were as follows:

Explosive	Retention Time (min)	Detection Limit (ppm in soil)
HMX	3.5	24.77
RDX	4.7	31.60
Tetryl	6.9	26.79
TNT	7.8	29.99
2A-DNT	8.5	30.89
4A-DNT	9.2	26.26

Solutions containing high levels of explosives were diluted with distilled water or distilled water/acetonitrile so as to maintain a 1:1 acetonitrile:water mixture.

The explosives were quantitated by comparing the HPLC peak areas to that obtained for solutions containing known amounts of the explosives. The concentration of the explosives in the sediment is calculated as follows:

$$\text{Expl. in soil } (\mu g/g) = \frac{\text{ppm in soln x 100 x dilution factor}}{\text{wt of soil, g}}$$

Appendix F

Analysis of Nitrocellulose in Soil

A sample of dried soil (approximately 1 g) was weighed into a 20 mL tube. The soil was extracted twice with 2 mL of methanol whereby the methanol was added to the soil and shaken. The slurry was centrifuged and the liquid carefully drawn off the top of the soil with a Pasteur pipet. This procedure removes free nitrates and nitrites which will interfere with the nitrocellulose test from the soil.

The washed soil was then extracted with 3 mL of acetone. The soil was shaken with the acetone, centrifuged and the acetone withdrawn with a Pasteur pipet and placed in a graduated centrifuge tube. The extraction was repeated twice and the extracts combined. Three mL of 1 N sodium hydroxide were added to the combined extracts. The tubes were placed in a 30°C water bath under a stream of nitrogen until the volume was less than 3 mL (but not less than 2 mL). A blank and standards containing from 20 to 160 µg nitrocellulose were also prepared at this time.

Approximately, 2-3 mL of nitrate/nitrite free distilled water were added to each tube. The solutions were acidified to pH 2.2 with 3 N H_2SO_4 and brought to 25 mL with nitrate/nitrite free distilled water. If high levels of nitrocellulose are expected, dilution using nitrate/nitrite free distilled water was done at this time. The samples were analyzed within 15-30 minutes after acidification.

A Hach Nitraver 3 powder pillow was then added to the blank and each standard. The color development was allowed to proceed for 10 minutes, but not more than 15 minutes. The spectrophotometer wavelength was set at 540 nm and the Hach DR2 spectrophotometer set to 100%T with the blank. The absorbance of the standards was then measured and a standard curve of nitrocellulose vs absorbance was constructed.

The powder pillows were then added to the samples. After the appropriate time, the absorbances of the standards were measured. The μg of nitrocellulose in each sample was determined from the standard curve. The μg/g of nitrocellulose in the soil were calculated from the following formula:

$$\frac{\mu g \text{ Nitrocellulose}}{g \text{ soil}} = \frac{\mu g \text{ Nitrocellulose x dilution factor}}{\text{dry weight of soil, g}}$$

This method differs from the USATHAMA standard method using acidifed sulfanilamide in that solutions are acidified to pH 2.2 with H_2SO_4. This acidification allows better control of the pH for the color reaction than the neutralization which depends on the acid in the reagent. If the color reaction is carried out immediately after neutralization, addition of Cd to reduce nitrate to nitrite is not necessary. Dilution before the addition of the color reagent is preferable. However, equally good results can be obtained if dilution is made after the addition of the color reagent using a blank to which no color reagent has been added. This method eliminates the problems of dilution that were experienced with the acidic sulfanilamide color reagent.

Appendix G
Analysis of Trichloroethylene (TCE) in Soil

Approximately 5 g of contaminated soil were weighed into 20 mL screw-cap vials and extracted with 2 x 10 mL portions of methanol. The combined extracts were brought to 25 mL with methanol. The samples were then analyzed by gas chromatography-mass spectrometry.

The HP 5992 GC-MS system was set up using a packed column and a molecular jet separator between the column effluent and MS ionization source. The GC parameters were as follows:

Column: 8 ft x 2 mm I.D. glass packed with 1% SP-1000 on 60/80 mesh Carbopack B with the first 4 in packed with 10% SP-1000 on 80/100 mesh Supelcoport.

Oven: Initial temperature 70°C. Hold for 2 minutes, then 10°C/min to 220°C and hold.

Injection Port Temp: 230°C

Solvent Time Out: 3.5 minutes

Carrier Gas: Helium at 30 mL/min

The sample injection size was 8 µl. The mass spectrometer was scanned from 29 to 400 amu in the electron impact mode. Quantitation was accomplished by comparing peak height of the total ion-currnt chromatogram vs standards.

Appendix H

Analysis of TNT, RDX, HMX, 2A-DNT and 4A-DNT in Compost

The composts (either hay-horse feed, hay-manure, or sewage sludge-wood chips) were extracted by placing 280 mL of acetonitrile in the Mason jar with the preweighed, wet compost materials. The mixture was carefully stirred and placed in an ultrasonic bath (maintained at 35°C) at full intensity for 30 minutes. The solvent was then decanted from the solids and filtered using Whatman #2 filter paper on a Buchner funnel and vacuum. The compost solids were then extracted three additional times using this same procedure except that only 200 mL of acetonitrile were used in the second, third and fourth extractions. The extracts were combined in a one liter volumetric and brought up to volume with acetonitrile.

Clean-up of the extract for HPLC analysis was accomplsihed in the following manner. Thirty mL of the extract were placed in a 50 mL (25 mm x 150 mm) culture tube. The extract was then blown to dryness under a stream of nitrogen while maintaining the temperature at 35-40°C using a water bath. The residue was then sonicated for 30 minutes with 4 mL of acetonitrile. The liquid was withdrawn from the tube and run through a Pasteur pipet packed with 50 mm x 6 mm of activated florisil. The eluate was collected in a 10 mL volumetric flask. The culture tube was rinsed with 1 mL of acetonitrile and this liquid was run through the florisil column. The florisil column was rinsed with an additional 10 mL of acetonitrile. The volume of the eluate was brought up to 10 mL with high purity water. The solution turns cloudy at this point and is clarified by running through a 0.2 μ nylon 66 disposable filter which fits a 10 mL syringe. The clarified solution was placed in a 16 mm x 125 mm culture tube. The hay-horse feed and hay-manure extracts were analyzed by HPLC at this point.

The sewage sludge-wood chip extracts required further cleansing to remove interferences. The sample was run through a C-18 sep pak under 5 inches Hg vacuum and the C-18 cartridge was then rinse with 1 mL of 65/35 acetonitrile/water.

All samples were analyzed by HPLC using a LKB 2150 HPLC Pump, a Perkin-Elmer LC55 UV detector and a Vista CDS 402 computer controller and integrator. Separation was accomplished on a 4.6 mm I.D. x 25 cm Alltech Econosphere 5 C-18 column using a mobile phase which consisted of 51% methanol, 2% dioxane, 0.5% acetonitrile and 46.5% high purity water which was 0.004 M in n-hexylamine and 0.004 M in the tetramethylammomium hydroxide buffered to pH 7.5 with phosphoric acid. The mobile phase flow rate was 1.3 mL/min. UV detection was at 232 nm. Under these conditions, the explosives had the following retention times and detection limits:

Explosive	Retention Time (min)	Detection Limit (ppm in Compost) H/HF or H/M	SS/WC
HMX	3.5	4.8	7.1
RDX	4.7	4.0	8.0
TNT	7.8	3.7	8.2
2A-DNT	8.5	9.3	7.7
4A-DNT	9.2	3.3	7.7

The explosives were quantitated by comparing the HPLC peak areas of the extracts to those of standard explosive solutions. The ppm of the explosives in the composts were calculated as follows:

H/HF or H/M Conc. in compost (dry wt. basis) µg/g =

$$\frac{\text{\# ng explosive x 1000 mL extraction volume x DF}}{\text{3 (conc. factor) x dry wt of compost*}}$$

SS/WC Conc. in compost (dry wt. basis) µg/g -

$$\frac{\text{\# ng explosive x 1000 mL extraction volume x DF}}{\text{2.727 (conc. factor) x dry wt of compost*}}$$

* Dry wt. determined from % moisture calculations.

Appendix I
Analysis of Tetryl in Compost

The composts (either hay-horse feed, hay-manure, or sewage sludge-wood chip) were extracted four time with 200 mL of benzene. The extractions were accomplished by placing the preweighed, wet compost materials in a Mason jar with 200 mL of benzene. The mixture was carefully stirred and placed in an ultrasonic bath (maintained at 35°C) at full intensity for 30 minutes. The solvent was then decanted from the solids and filtered using a Whatman #2 filter paper on a Buchner funnel with vacuum. The procedure was repeated three additional times and the extracts combined and brought up to 800 mL.

Tetryl was quantitated on a HP-5880 gas chromatograph using a nitrogen-phosphorus detector. A 4 ft x 22 mm I.D. column packed with 3% OV17 on 80/100 ANAKROM was used to affect separation with a nitrogen carrier gas at a flow rate of 30 mL/min. A 4 μL injection volume was used. The oven was programmed as follows: initial temperature was 190°C, held for 1 minute then 5°C/min to 210°C and hold for 0.5 min then 10°C/min to 240°C and hold. The injection port was maintained at 215°C and the detector at 310°C. Retention time for tetryl was 8.1 min with a detection limit of 128 ppm in the compost.

Tetryl was quantitated by comparing the GC peak areas of these extracts to those of standard explosive solutions. The ppm of the explosives in the compost were calculated as follows:

$$\text{Tetryl Conc. in compost (dry wt basis*)}\ \mu g/g = \frac{\#\ \mu g \text{ explosive x 800 mL extraction vol}}{\text{dry wt of compost}}$$

* Dry wt determined from % moisture calculations

Appendix J

Analysis of Nitrocellulose in Compost

Dried samples were weighed into quart Mason jars and then extracted with acetone. For this extraction, 250 mL of acetone were added to each Mason jar and the jars placed in an ultrasonic bath at 35°C for 30 minutes. The mixture was then filtered through a Whatman No. 1 filter paper with a Buchner vacuum filter apparatus and the filtrate saved. The compost material was then extracted three more times with 250 mL of acetone. All the extracts were combined and the final volume brought to 1000 mL.

Twenty mL of each extract were placed in a 50 mL graduated centrifuge tube and were blown to dryness under a steam of nitrogen at 35°C. Two mL of a 90:10 methanol-water mixture were added to each tube, shaken, and centrifuged. The liquid was then carefully withdrawn with a Pasteur pipet and the procedure repeated. This washing procedure removed nitrate and nitrite from the sample which interfere with the analysis.

The washed sample, containing the nitrocellulose, was then reacted with base to hydrolyze the nitrocellulose to cellulose and nitrite. To perform this reaction, 9 mL of acetone and 3 mL of 1 $\underline{N}$ sodium hydroxide were added to each tube. The tubes were placed in a water bath at 30°C under a stream of nitrogen. The reaction was allowed to proceed until the volume was less than three mL (but not less than 2 mL). A blank (containing reagents only) and standards ranging from 20 µg to 100 µg of nitrocellulose were also reacted in the same manner at this time.

Two to three mL of nitrite/nitrate free distilled water were added to each tube. The solutions were then acidified to pH approximately 2.2 with 3 $\underline{N}$ H_2SO_4. After acidification, the volume was brought to 25 mL with nitrate/nitrite free distilled water. If the mixture was cloudy, it was filtered through a nylon 66 0.45 µm filter using a 25 mL leuer-lock syringe. If high levels of nitrocellulose were expected, appropriate dilutions were made at this point using nitrate/nitrite free distilled water. The remainder of the analysis proceeded immediately after the acidification.

The nitrite from the nitrocellulose hydrolysis was determined colorimetrically using Hach Nitraver 3 powder pillows. One powder pillow was added to each 25 mL standard or blank sample. The color was allowed to develop for 10 minutes (but no longer than 15 minutes). The Hach DR2 spectrophotometer was set to 540 nm and the 100% T set with the blank. The standards were then run and the absorbance of the standards determined. A standard curve of μg nitrocellulose vs absorbance was then constructed. Powder pillows were then added to the samples (no more than 6 at a time). After the appropriate reaction time, the absorbance of the samples were determined. The number of μg of nitrocellulose in the samples was then determined from the standard curve. The μg/g of nitrocellulose in the compost was determined by the following formula:

$$\mu\text{g NC/g in compost} = \frac{\mu\text{g NC} \times 1000\text{ mL extraction volume} \times \text{dilution factor}}{\text{dry weight of compost in g}}$$

Appendix K

Analysis of Trichloroethylene (TCE) in Methanol

Methanol samples from cold trap washings were analyzed using a Varian 6000 gas-chromatograph and a Hall Electrolytic Conductivity Detector (HECD) operated in the halogen mode. The GC and HECD operating parameters are given below:

Column: 8 ft x 2 mm I.D. I.D. glass pack with 1% SP-1000 on 60/80 mesh Carbopack B with the first 4 in packed with 10% SP-1000 on 80/100 mesh Supelcoport.

Column Oven: 150°C Isothermal

Injection Port: 230°C

Detector Base: 300°C

HECD Reaction Tube: 800°C

Reaction Gas: Hydrogen at 35 mL/min

Carrier Gas: Helium at 32 mL/min

Solvent: n-propanol at .5 mL/min

Injection Size: 4μl

Appendix L

Temperature Records for Laboratory Composts

^{14}C-TNT Compost Temperatures (°C)

DATE	Hay-Horsefeed Compost 10A*	10B	18A	18B	25A	25B	Sewage Sludge Compost 10A	10B	18A	18B	25A	25B	INCUBATOR
11/28/84	60	60	60	60	59	-	59	59	59	59	59	59	59
11/29/84	60	60	60	60	59	-	60	59	59	59	59	59	59
11/30/84	61	60	60	60	60	-	60	60	59	60	60	60	60
12/02/84	60	60	60	59	59	-	60	59	58	58	59	59	59
12/03/84	60	60	60	59	59	-	60	59	58	58	59	59	59
12/04/84	61	61	61	61	60	-	61	60	59	59	60	60	60
12/05/84	62	62	61	61	61	-	61	61	60	60	61	61	61
12/06/84	63	63	62	62	62	-	62	62	60	61	61	61	61
12/07/84	62	62	62	62	61	-	62	61	60	61	61	61	61
12/08/84	62	62	62	62	62	-	62	62	61	61	62	62	61
12/10/84	63	63	62	62	62	-	62	62	61	62	62	62	62
12/12/84	62	62	61	61	61	-	62	61	60	61	61	61	61
12/13/84	61	61	61	61	60	-	61	60	59	60	60	60	59
12/14/84	62	62	61	61	61	-	61	61	60	60	61	61	61
12/18/84	62	62	61	61	61	-	62	61	60	60	61	61	61
12/19/84	62	61	61	60	60	-	61	61	60	59	60	60	60
12/20/84	62	62	62	61	61	-	62	62	60	61	61	61	61
12/21/84	61	62	61	61	61	-	61	60	60	60	61	61	61
12/22/84	59	60	60	60	60	-	61	60	59	59	60	60	59
12/23/84	59	61	60	60	60	-	61	60	59	59	60	60	59
12/24/84	61	61	60	60	60	-	60	60	59	59	60	60	59
12/25/84	61	61	60	60	60	-	61	60	59	59	60	60	60
12/26/84	61	61	60	60	60	-	60	60	59	59	60	60	59
12/27/84	61	61	61	60	60	-	61	61	59	60	60	60	60
12/28/84	61	61	60	60	60	-	60	60	59	59	60	60	59
12/29/84	61	61	60	60	60	-	61	60	59	59	60	60	59
12/30/84	61	61	60	60	60	-	61	60	59	59	60	60	59
12/31/84	61	61	60	60	60	-	61	60	59	59	60	60	59
01/01/85	61	61	60	60	60	-	61	60	59	59	60	60	60

* 10A = first replicate of compost with 10% sediment addition

\- = thermocouple malfunction

RDX Compost Temperature (°C)

DATE	Hay-Horsefeed Compost 10A*	10B	18A	18B	25A	25B	Sewage Sludge Compost 10A	10B	18A	18B	25A	25B	INCUBATOR
12/22/84	67	70	-	64	72	68	71	70	70	74	63	68	59
12/23/84	67	70	72	70	72	67	71	70	70	74	63	68	59
12/24/84	67	70	72	72	72	68	71	70	70	74	63	68	59
12/25/84	67	70	72	71	72	67	71	70	70	75	64	68	60
12/26/84	66	70	72	71	72	68	71	70	70	74	63	67	59
12/27/84	66	70	72	72	72	67	72	70	70	74	63	68	60
12/28/84	58	60	61	62	64	59	61	61	61	63	58	59	59
12/29/84	59	61	61	62	64	59	61	61	61	63	58	59	59
12/30/84	58	60	61	61	64	59	61	61	61	63	58	59	59
12/31/84	60	61	61	62	64	61	61	61	62	63	58	59	59
01/01/85	59	61	61	62	64	59	61	61	62	63	58	59	60
01/02/85	59	61	62	62	64	60	61	62	62	64	58	59	60
01/03/85	59	61	62	61	65	60	61	62	62	64	58	60	60
01/04/85	58	61	62	61	65	59	60	61	62	64	59	60	60
01/05/85	58	61	61	61	66	59	61	62	62	64	58	60	60
01/07/85	59	61	62	61	65	59	61	62	62	64	58	59	60
01/08/85	59	61	61	61	65	59	61	61	62	64	58	59	60
01/09/85	58	60	61	60	65	58	60	61	62	63	58	59	59
01/10/85	59	61	61	60	65	58	60	61	62	63	58	59	60
01/11/85	59	61	62	61	65	59	61	62	62	64	59	60	61
01/14/85	59	61	61	60	65	59	61	61	62	63	58	59	60
01/15/85	59	61	62	61	65	59	60	61	62	63	58	59	60
01/16/85	59	62	62	61	65	59	61	62	63	64	59	60	60
01/17/85	59	62	62	61	65	59	61	62	63	64	59	60	60
01/18/85	59	61	61	60	65	59	60	61	62	63	58	59	60
01/21/85	60	63	63	62	66	60	62	62	63	64	58	59	61
01/22/85	58	61	61	60	64	58	60	61	62	63	58	59	60
01/23/85	59	62	62	61	65	59	61	62	62	64	58	60	61
01/24/85	59	61	62	60	65	59	61	62	62	64	58	59	62
01/25/85	61	64	64	62	67	61	63	63	64	65	59	61	64
01/28/85	61	63	64	62	67	61	63	64	64	66	60	61	64
01/29/85	59	61	62	60	64	59	61	62	62	63	58	59	61
01/30/85	61	63	64	62	66	61	63	64	64	66	60	61	64

RDX Compost Temperature (^{o}C) (continued)

02/01/85	59	62	62	61	64	59	61	62	62	64	58	60	62
02/04/85	56	58	58	57	61	56	57	58	59	60	54	56	59
02/05/85	55	57	57	56	59	55	57	57	58	59	54	56	60
02/06/85	57	58	59	58	61	57	58	59	59	61	55	57	60
02/07/85	57	58	59	58	61	57	58	59	59	61	55	57	60
02/08/85	57	58	59	58	61	57	58	59	59	61	56	57	61
02/11/85	55	57	58	56	60	55	57	58	58	60	54	56	60
02/12/85	57	59	59	58	62	57	58	59	59	61	56	56	60
02/13/85	55	57	58	56	60	55	57	58	58	60	54	56	60
02/14/85	55	57	57	56	60	55	57	57	58	60	54	56	59
02/15/85	56	58	59	57	61	56	58	59	59	61	55	57	61
02/18/85	56	58	59	58	61	56	58	58	59	61	55	57	59
02/19/85	57	58	59	58	61	57	58	59	59	61	55	57	59
02/20/85	57	58	59	58	61	57	58	59	59	61	56	57	61
02/21/85	56	58	59	58	61	57	58	58	59	60	55	57	59
02/22/85	57	59	59	58	62	57	59	59	59	61	56	57	-
02/25/85	58	59	60	59	63	58	56	60	61	62	57	58	61
02/26/85	58	60	61	60	63	59	60	61	61	63	57	59	62
02/27/85	57	59	60	59	62	57	58	60	60	62	56	58	61
02/28/85	57	58	59	58	62	57	57	59	60	61	55	58	61
03/01/85	57	58	59	59	62	57	58	59	60	61	56	58	61

*10A = first replicate of compost with 10% sediment addition

HMX-Compost Temperature (^{o}C)

DATE	Hay-Horsefeed Compost 10A*	10B	18A	18B	25A	25B	Sewage Sludge Compost 10A	10B	18A	18B	25A	25B	INCUBATOR
12/18/84	59	60	61	62	63	60	61	62	63	65	60	61	61
12/19/84	59	58	59	60	61	57	57	59	61	63	59	60	60
12/20/84	60	60	62	61	62	59	60	61	63	65	60	61	61
12/21/84	59	60	61	60	60	56	57	59	60	62	58	59	61
12/22/84	58	59	60	61	62	58	60	61	62	64	60	61	59
12/23/84	58	59	60	62	63	59	60	61	62	64	60	60	59
12/24/84	58	59	60	61	63	59	60	61	62	64	60	60	59
12/25/84	59	59	60	62	63	59	60	61	62	64	60	61	60
12/26/84	58	59	60	61	63	59	60	61	62	64	60	60	59
12/27/84	58	59	60	61	63	59	60	61	62	64	60	60	59
12/28/84	59	59	58	59	61	58	58	59	60	62	58	59	59
12/29/84	57	57	58	58	60	57	58	59	60	62	59	59	59
12/30/84	57	57	58	58	59	56	57	58	59	61	58	58	59
12/31/84	57	57	58	59	61	58	59	60	60	62	59	60	59
01/01/85	58	58	59	60	61	58	59	60	61	62	59	60	60
01/02/85	58	58	59	61	61	58	59	60	61	62	59	60	60
01/03/85	58	58	59	60	61	58	59	60	60	62	59	59	60
01/04/85	57	58	59	59	60	57	58	59	60	62	59	59	60
01/05/85	57	58	59	59	59	56	58	59	60	61	58	59	60
01/07/85	57	57	59	59	60	57	59	60	60	61	58	60	60
01/08/85	57	57	58	59	59	56	58	59	60	60	58	60	59
01/09/85	57	56	58	58	59	53	57	58	59	60	58	59	59
01/10/85	57	57	59	59	59	54	58	59	60	61	59	60	60
01/11/85	58	57	59	59	59	52	57	59	60	61	58	60	61
01/14/85	57	57	59	59	59	56	57	59	59	61	58	59	60
01/15/85	57	57	59	60	60	57	59	60	60	61	59	60	60
01/16/85	57	58	59	59	60	57	58	59	60	61	59	60	60
01/17/85	57	57	59	59	60	57	59	59	60	62	60	60	60
01/18/85	57	58	59	59	60	57	58	59	59	61	58	59	61
01/21/85	57	57	59	59	60	58	60	61	61	63	60	60	61
01/22/85	57	57	59	58	58	55	56	58	58	60	57	58	60
01/23/85	57	57	59	59	59	56	58	59	59	61	59	59	61
01/24/85	57	57	59	58	59	56	57	59	59	61	58	59	62
01/25/85	58	58	60	61	63	59	61	62	62	64	61	62	64
01/28/85	59	59	60	60	61	58	59	61	62	63	60	62	64
01/29/85	57	57	59	59	59	57	58	59	60	61	59	60	61
01/30/85	58	59	60	60	61	58	60	61	62	62	60	61	64

HMX-Compost Temperature (^{o}C) (continued)

02/01/85	58	58	59	59	59	58	59	60	61	61	59	60	62
02/04/85	55	55	56	56	57	55	56	57	58	59	56	57	59
02/05/85	54	55	56	56	57	54	55	56	57	56	55	56	60
02/06/85	55	55	56	57	57	56	57	58	58	58	56	57	60
02/07/85	55	55	57	56	56	54	55	57	58	57	56	57	60
02/08/85	55	56	57	56	57	54	55	56	57	55	56	57	61
02/11/85	55	55	57	57	57	55	56	57	57	58	56	56	60
02/12/85	55	55	56	57	57	55	57	57	58	59	57	57	60
02/13/85	55	55	56	56	56	54	55	56	57	57	55	56	60
02/14/85	55	55	56	57	56	55	55	56	57	57	55	56	59
02/15/85	55	56	57	58	58	56	57	57	58	58	56	57	61
02/18/85	55	55	56	57	56	52	56	57	58	59	56	57	59
02/19/85	55	55	57	57	57	52	56	57	58	59	57	57	59
02/20/85	55	56	57	57	57	53	57	58	58	59	57	57	61
02/21/85	55	56	57	56	56	54	55	56	58	59	56	57	59
02/22/85	56	56	57	57	57	55	56	57	58	59	57	57	-

* 10A = first replicate of compost with 10% sediment addition

Tetryl Compost Temperatures (^oC)

DATE	Hay-Horsefeed Compost						Sewage Sludge Compost						INCUBATOR
	10A*	10B	18A	18B	25A	25B	10A	10B	18A	18B	25A	25B	
4/18/85	56	56	58	58	60	56	57	58	59	59	56	57	60
4/19/85	56	57	58	59	60	56	57	58	59	59	56	57	60
4/21/85	57	58	59	59	61	57	53	58	60	59	57	57	61
4/22/85	57	57	59	59	61	57	50	58	60	59	57	57	61
4/23/85	57	58	59	60	61	57	57	59	60	59	57	58	61
4/24/85	57	58	59	59	61	57	57	58	60	59	57	57	60
4/25/85	57	57	58	59	60	56	57	58	60	59	57	57	60
4/26/85	56	57	59	59	60	56	57	58	59	59	56	57	60
4/28/85	57	58	59	59	60	56	57	58	60	59	57	57	61
4/29/85	57	58	58	59	60	56	57	58	59	59	56	57	60
4/30/85	57	58	59	59	60	56	57	58	59	59	57	57	60
5/01/85	57	58	59	59	60	57	57	58	59	59	57	57	60
5/02/85	57	58	59	59	60	57	57	58	60	59	57	57	60
5/03/85	57	58	59	59	60	57	58	58	59	59	57	57	60
5/05/85	58	58	59	59	60	57	58	58	59	59	57	58	60
5/06/85	57	58	58	58	60	56	57	57	59	59	56	57	60
5/07/85	57	57	58	58	60	56	57	57	59	58	57	57	60
5/08/85	58	58	58	58	60	56	57	58	59	59	57	57	60
5/09/85	58	58	58	58	60	56	57	57	59	58	57	57	62
5/10/85	57	57	58	58	59	56	57	57	58	58	56	57	59
5/13/85	56	57	58	59	60	57	57	57	59	59	57	57	59
5/14/85	55	56	58	58	59	55	56	57	59	58	56	57	59
5/15/85	56	57	58	58	59	56	56	57	59	58	56	57	59
5/16/85	56	57	58	59	59	56	57	57	59	58	56	57	60
5/17/85	55	56	58	58	59	55	56	57	59	58	56	57	60
5/20/85	58	58	60	60	61	57	58	59	61	60	58	58	61
5/21/85	56	57	59	59	60	56	57	57	59	58	56	57	60
5/22/85	57	57	59	59	60	56	57	58	60	59	57	57	60
5/23/85	57	57	58	58	60	57	57	58	60	59	57	58	60
5/24/85	57	57	58	58	59	56	57	58	59	59	57	58	60
5/28/85	57	57	58	58	60	57	57	58	60	59	57	57	60
5/29/85	57	57	58	59	60	57	57	58	59	59	57	57	60
5/30/85	57	57	58	59	60	56	57	58	59	59	56	57	60
5/31/85	57	58	59	59	60	57	57	58	60	59	56	57	61

* 10A = first replicate of compost with 10% sediment addition

Control Compost Temperatures

	Hay-Horsefeed Compost				Sewage Sludge Compost				
DATE	0	10	18	25	0	10	18	25	INCUBATOR
12/29/84	59	59	59	60	59	59	59	59	61
12/30/84	61	61	61	62	61	61	61	61	64
01/01/85	60	59	60	60	60	59	59	60	62
01/04/85	58	57	57	58	57	57	57	57	59
01/05/85	57	58	58	58	57	57	57	57	60
01/06/85	57	57	57	58	57	57	56	57	60
01/07/85	58	57	58	58	58	57	57	57	60
01/08/85	57	58	57	58	58	57	57	57	61
01/11/85	57	58	57	58	58	57	57	57	60
01/12/85	57	58	58	58	58	57	57	57	60
01/13/85	57	58	58	58	57	57	57	57	60
01/14/85	57	57	58	58	57	57	57	57	59
01/15/85	58	58	59	59	58	58	58	58	61
01/18/85	57	57	57	57	58	57	58	58	59
01/19/85	58	57	58	58	58	58	57	57	59
01/20/85	58	57	58	58	58	58	57	58	61
01/21/85	58	57	58	58	58	58	57	57	59
01/22/85	58	58	58	59	59	58	58	58	-
01/25/85	59	58	58	59	59	59	58	58	61
01/26/85	59	58	59	59	59	59	58	58	62
01/27/85	59	58	58	59	59	59	58	58	61
01/28/85	59	57	57	58	59	59	57	58	61

Nitrocellulose Compost Temperatures

DATE	Hay-Horsefeed Compost						Sewage Sludge Compost						INCUBATOR
	10A*	10B	18A	18B	25A	25B	10A	10B	18A	18B	25A	25B	
4/01/85	68	68	69	71	70	70	70	70	70	70	69	69	60
4/02/85	71	71	73	73	70	70	70	70	70	70	69	69	60
4/03/85	67	68	68	69	69	67	67	67	68	67	67	67	60
4/04/85	74	75	75	75	76	74	74	74	74	74	74	74	65
4/05/85	67	67	67	67	67	68	66	66	66	66	66	65	60
4/06/85	68	67	68	67	67	66	66	66	66	66	66	66	60
4/07/85	66	66	66	66	66	65	65	65	66	66	66	66	60
4/08/85	68	68	68	67	67	67	67	67	67	68	68	68	60
4/09/85	69	69	69	69	69	69	68	68	69	69	68	68	60
4/10/85	67	68	68	68	69	68	68	68	69	68	68	69	60
4/11/85	70	71	72	71	71	71	71	71	71	71	71	71	59
4/12/85	69	69	69	70	69	70	69	69	70	70	69	69	59
4/13/85	65	65	66	66	66	66	66	66	66	66	66	66	59
4/14/85	66	66	66	67	67	66	66	66	67	67	66	66	60
4/15/85	68	68	68	68	68	68	68	68	68	68	68	68	60
4/16/85	68	69	68	68	68	68	68	68	68	68	68	68	60
4/17/85	68	67	68	68	68	67	67	68	68	68	67	68	60
4/18/85	64	65	65	65	65	65	65	65	65	66	64	64	60
4/19/85	69	70	70	70	70	70	70	71	71	72	71	71	60
4/21/85	67	67	67	67	67	67	67	67	67	68	67	68	61
4/22/85	69	69	69	69	69	68	69	69	70	70	69	70	61
4/23/85	65	66	67	66	67	66	66	66	67	67	66	67	61
4/24/85	67	67	67	68	68	67	67	68	68	68	67	68	60
4/25/85	68	68	69	69	69	68	68	68	69	69	68	68	60
4/26/85	67	68	69	69	69	68	68	68	69	69	69	68	60
4/28/85	66	67	67	67	67	67	67	67	67	68	67	67	60
4/29/85	67	67	67	68	68	67	67	67	68	68	67	68	60
4/30/85	66	66	66	67	67	66	66	66	66	67	66	66	60
5/01/85	68	68	68	68	68	68	68	68	68	68	68	68	60
5/02/85	71	72	72	72	72	72	72	72	72	73	72	72	60
5/03/85	68	69	69	69	69	69	69	69	69	69	69	69	60
5/05/85	66	66	66	66	66	66	66	66	67	67	66	67	60
5/06/85	66	67	67	67	67	67	67	67	68	68	67	67	60
5/07/85	69	69	69	69	70	69	69	69	70	70	69	69	60
5/08/85	69	67	67	67	67	67	67	67	67	68	67	67	60
5/09/85	65	66	66	66	66	66	66	66	66	67	66	66	62
5/10/85	66	66	66	66	67	66	66	66	67	67	66	66	59

* 10A = first replicate of compost with 10% sediment addition

Appendix M

RCRA R,D&D Permit Public Announcement and Fact Sheet

Notice of Preparation of a Hazardous Waste Research, Development and Demonstration Permit and Public Hearing Resource Conservation and Recovery Act

United States Environmental Protection Agency
Region III
841 Chestnut Building
Philadelphia, Pennsylvania 19107

Notice Date: May 30, 1986

The United States Environmental Protection Agency (EPA) has reviewed a Research, Development, and Demonstration (RD & D) permit application from Atlantic Research Corporation, 5390 Cherokee Road, Alexandria, Virginia 22312, EPA Identification Number VAD 08 112 2158. The review was conducted pursuant to the Resource Conservation and Recovery Act (RCRA) as amended by the Hazardous and Solid Waste Amendments of 1984 (HSWA). Atlantic Research Corporation proposes to conduct research and development on a composting technique for treatment of hazardous waste. EPA has made a tentative determination to issue a permit under the authority of RCRA.

HSWA imposes additional requirements which will be administered and enforced by EPA until the Commonwealth of Virginia receives additional authorization for those requirements. Since Virginia is currently not authorized to issue RD & D permits, EPA will determine whether to issue a permit to Atlantic Research Corporation.

Project Description

Atlantic Research Corporation proposes to conduct a pilot-scale research project at its Headquarters and Principle Laboratories located in Alexandria, Virginia. This project is to test composting as a waste treatment process to decontaminate soils and sediments that are contaminated with explosives. Composting is an accelerated process normally used to convert waste products such as sewage sludge or leaves into a humus to be used as a soil conditioner. In composting the waste product is mixed with a bulking agent to form a compost "pile" and allowed to decompose aerobically for a period of time.

EPA proposes to issue a permit for an RD & D project. The sediments will be obtained from waste lagoons located as US Army Ammunition Plants in Louisiana and Wisconsin. The sediment in plastic lined steel drums will be stored in a concrete tank at Atlantic Research. Composting experiments will be conducted in four stainless steel 488 gallon tanks located in a secured greenhouse. The maximum amount of hazardous waste to be received and treated under this permit will not exceed 500 gallons.

Tentative Decision

EPA proposes to issue the permit with the following requirements:

1) The permit includes "boilerplate" requirements which are in all EPA operating permits per regulations 40 C.F.R. Section 270.30.
2) The permit will expire one year following date of issuance.
3) Experimental procedures to follow in conducting the research are identified in the permit.

Public Participation

Persons wishing to comment on EPA's draft permit should submit their comments in writing to:

Environmental Protection Agency, Region III
841 Chestnut Building
Philadelphia, PA 19107
ATTN: Harry Harbold
VA/WV Section (3HW61)

All persons, including the applicant, who believe any conditions of the draft permit is inappropriate or that EPA's tentative decision to prepare a draft permit is inappropriate, must raise all reasonably ascertainable issues and submit all reasonably available arguments and factual grounds supporting their position, including all supporting materials, by the close of the public comment period. All supporting materials shall be included in full and may not be incorporated by reference, unless they are already part of the administrative record in this permit action or consist of State or Federal statutes and regulations, EPA documents of general applicability, or other generally available reference materials. Commenters shall make supporting material not already included in the administrative record available to EPA. All comments must be received within the 45-day period ending July 15, 1986.

A public hearing to consider the draft permit conditions and permit issuance will be held at the time and place stated below:

July 1, 1986
7:00 p.m.
Atlantic Research Corporation
5390 Cherokee Road
Alexandria, Virginia

Any person may submit oral or written statements or data concerning EPA's draft permit at this time. If presenting an oral statement, a written statement is requested so the EPA can adequately respond to all issues raised.

EPA's administrative record, including all data submitted by the applicant, the fact sheet, the draft permit, maps showing the exact facility location, and comments received, may be reviewed and copied at EPA, Region III, 841 Chestnut Building, 6th floor, Philadelphia, Pennsylvania 19107, between the hours of 8:30 a.m. and 4:30 p.m., Monday through Friday. A copying machine will be provided for public use at a charge per page. Any person desiring further information, copies of portions of the administrative record, or an appointment to review the record should contact Joan Henry at the above address or call (215) 597-7269.

Any relevent comments received within forty-five days of the date of this public notice will be considered in the formulation of final determinations regarding EPA's permit. After consideration of all written comments and the requirements and policies in RCRA and HSWA, EPA will make a final decision to either issue, modify or deny the permit. At that time, EPA will notify the applicant and each person who has submitted written comments or requested notice of the final permit decision. The final permit decision will become effective thirty (30) days after the service of notice of the decision unless a later date is specified or review is requested under regulation 40 CFR Section 124.19. If no comments requested a change in the draft permit, the final permit will become effective immediately upon issuance.

May 30, 1986

5R3001000

- FACT SHEET -

FOR DRAFT RESEARCH, DEVELOPMENT AND DEMONSTRATION PERMIT

Atlantic Research Corporation, Alexandria, Virginia

This fact sheet has been developed for the draft permit under Section 3005(g) of the Resource Conservation and Recovery Act (RCRA) (42 U.S.C. §6925(g)) for research, development and demonstration (RD & D) of a hazardous waste treatment process which EPA intends to issue to Atlantic Research Corporation, Alexandria, Virginia (Permittee). This fact sheet was prepared in accordance with the general permitting requirements of Section 124.8 of Title 40 of the Code of Federal Regfulations (40 CFR).

A. PURPOSE OF THE PERMITTING PROCESS

The purpose of the permitting process is to afford the Environmental Protection Agency (EPA), interested citizens and other governmental agencies the opportunity to evaluate the ability of the Permittee to comply with the relevant and applicable hazardous waste research requirements promulgated under the Solid Waste Disposal Act (commonly referred to as the Resource Conservation and Recovery Act or "RCRA"). EPA is required to prepare a draft permit which sets forth in one concise document all the relevant and applicable requirements the Agency has established for the Permittee during the one-year duration of the permit. The public is given forty-five days to review the application and comment on the draft permit conditions prior to EPA taking any final action on the application for a hazardous waste research permit.

B. PROCEDURES FOR REACHING A FINAL DECISION

Section 7004(b) of RCRA and 40 CFR §124.10 require that the public be given forty-five (45) days to comment on each draft permit prepared under the Resource Conservation and Recovery Act. The comment period for this draft permit will begin on May 30, 1985 and will end on July 15, 1985. Any person interested in commenting on the application or draft permit must do so within this forty-five (45) day comment period.

All persons wishing to comment on any of the permit conditions or the permit application should submit the comments in writing to the Environmental Protection Agency (EPA), Region III, 841 Chestnut Street, Philadelphia, Pennsylvania 19107, Attention: Mr. Roland Schrecongost, Acting Director, Hazardous Waste Management Division (3HW00). Comments should include all reasonably available references, factual grounds and supporting material.

EPA has scheduled a public hearing on the draft permit for July 1, 1985 at the following location:

Atlantic Research Corporation
5390 Cherokee Avenue
Alexandria, Virginia 22312

Time: 7:00 p.m.

Any written comments should be addressed to Mr. Roland Schrecongost, Acting Director, Hazardous Waste Management Division, Environmental Protection Agency, Region III, 841 Chestnut Street, Philadelphia, Pennsylvania 19107.

When making a determination regarding the issuance of a hazardous waste permit to Atlantic Research Corporation, EPA will consider all written comments received during the comment period, any oral or written statements received during the public hearing, any relevant provisions of the hazardous waste regulations of 40 CFR Parts 124, 260-264, and 270, 42 U.S.C. §6925(g) and the Agency's permitting policies.

When EPA makes a final permit decision to either issue, deny or modify the permit, notice will be given to the applicant and each person who has submitted written comments or requested notice of the final decision. If no comments requested a change in the draft permit, the final permit shall become effective immediately upon issuance, in accordance with 40 CFR §124.15(b)(3).

The contact person for the Atlantic Research Corporation draft permit is:

Mr. Harry Harbold
U.S. Environmental Protection Agency
Region III
841 Chestnut Street
Philadelphia, Pennsylvania 19107
(215) 597-9287

C. AUTHORITY FOR ISSUING RD&D PERMIT

On November 8, 1984, the President signed into law the Hazardous and Solid Waste Amendments of 1984 (HSWA), which amended the Resource Conservation and Recovery Act (RCRA). RCRA, as amended by HSWA, under Section 3005(g), (42 U.S.C. §6925(g))authorizes the Agency to permit research, development, and demonstration facilities for the treatment of hazardous waste without having substantive regulations in effect under 40 CFR Part 264. Many requirements of 40 CFR §264 can be and are applied to the facility through this permit, as they are necessary to protect human health and the environment.

The new Section 3005(g) provides that:

- EPA may issue RD&D permits, without promulgation of permitting regulations under Part 264, for technologies or processes that treat hazardous waste in an innovative and experimental manner.
- An RD&D permit shall provide for the receipt and treatment of only those types and quantities of hazardous waste that are necessary for determination of the efficacy and performance capabilities of the technology or process being researched and tested and its effects on human health and the environment.
- RD&D permits shall include any conditions that the Agency believes are necessary for protection of human health and the environment.
- For RD&D permits, the Agency may modify or waive the permit application and permit issuance requirements applicable to hazardous waste management facilities, except that financial responsibility requirements and the public participation requirements may not be modified or waived.
- An RD&D permit shall provide for the construction of the RD&D facility if necessary and for its operation for a period not exceeding 365 operating days. Permits may be renewed up to three times, with each renewal not to exceed 365 operating days.
- The Agency may terminate an RD&D permit at any time necessary for protection of human health or the environment.

D. FACILITY DESCRIPTION

Atlantic Research Corporation, Inc. (ARC), proposes to conduct research on composting techniques for degrading explosives, propellents and explosives related compounds from lagoon sediments at two Army ammunition plants, the Louisiana Army Ammunition Plant in Doyline, Louisiana and the Badger Army

Ammunition Plant in Baraboo, Wisconsin. The sediments from the Louisiana plant contain TNT, RDX, HMX and tetryl as the main explosive contaminants; the sediments from the Badger plant contain nitrocellulose as the main explosive contaminant. Composting is an accelerated aerobic microbial digestion of organic materials at elevated temperatures.

The purpose of this research is to develop a safe and economical process for degradation of explosives, propellents and explosives related compounds in sediments of waste water settling lagoons which pose environmental problems at various Army ammunition plants. The proposed research, to be performed by ARC under contract with the U.S. Army Toxic and Hazardous Materials Agency, would attempt to demonstrate the viability of two composting techniques for reducing the explosives content of the actual explosive contaminated sediments from the two above-mentioned Army ammunition plants on a pilot-project scale.

In conducting the research, sediments from the two plants will be mixed with bulking materials and nutrients required for efficient composting. Six composts will be set up for each sediment, three using hay-horse feed and three using sewage sludge-wood chips as bulking materials/nutrients. The compost will be moistened to approximately 60% moisture content.

The contaminated sediments and bulking materials/nutrients will be manually loaded and mixed in four 488-gallon stainless steel tanks used as composting vessels. To serve as a control, two additional 488-gallon stainless steel tanks will be set up using uncontaminated soil from areas near the two contaminated sites.

The tanks will be maintained within two fully enclosed greenhouses located within the ARC complex during the entire duration of the composting experiment, with three tanks in each greenhouse. Each greenhouse is underlined with a concrete lined pit filled with gravel. The gravel is covered with a 3.5 mm polyethylene liner to contain any spilled wastes. At the end of each compost experiment, all composted material, spilled material and associated cleaning materials will be placed in polyethylene lined 55 gallon drums and hauled off-site by a licensed commercial hazardous waste hauler for disposal in a qualifying RCRA hazardous waste landfill. Removal of the composted material will occur within two weeks of the completion of each experiment.

The duration of each composting experiment is expected to be approximately six weeks. Since there will be two sets of experiments, the total duration of the research, development and demonstration is expected to be approximately twelve weeks.

Contaminated sediments from the two Army ammunition plants will be stored in a fully enclosed building located within the ARC complex. The contaminated sediments will be contained within polyethylene-lined 55 gallon drums which will remain closed and secured within the storage building at all times during storage. The drums will be kept within a concrete pit inside the shed for secondary containment of spilled or leaked wastes and will be elevated at all times to minimize contact with any spilled or leaked wastes contained within the pit. A maximum of seven drums with a total of 385 gallons of contaminated soil will be stored at the facility at any time during the permit period.

ARC conducted tests on samples of wastes from both Army Ammunition Plants to determine their reactivity at the maximum explosive concentrations to be used in the RD&D project under this proposed permit. All tests were negative, indicating that the wastes to be used in the research activity are not reactive. This testing, along with sampling in the Waste Analysis Plan, are considered adequate to ensure safe handling of the waste. These tests were conducted under the direction of EPA and have been approved by the Agency.

The total amount of wastes that the Permittee plans to receive and treat under this permit are as follows:

Hazardous Waste No.	Description	Annual Amount
K044	Waste water treatment sludges from the manufacturing and processing of explosives	500 gallons

The application from Atlantic Research Corporation indicates the site is located 25 feet above the 100-year flood plain.

The final project report will be public record and forwarded to EPA when completed. The permittee shall provide EPA with experimental data upon project completion after receiving approval by the U.S. Army. EPA will notify the Army in writing of this requirement. This approval is necessary to satisfy contractural requirements between Atlantic Research and the U.S. Army. Records of data used to complete the permit application will be retained by the permittee for a period of one year.

E. PERMIT ORGANIZATION

The permit is divided into five sections as outlined below.

Section	Topic
Part I	Standard Conditions
Part II	General Facility Conditions
Part III	Storage In Containers
Part IV	Storage And Treatment In Tanks
Part V	Special Conditions

Parts I and II contain conditions which generally apply to all hazardous waste facilities, although certain conditions, described in the following summary, have been deleted, added or revised to fit the unique RD & D project. Part(s) III and IV pertain specifically to the hazardous waste RD&D facilities at the ARC facility in Alexandria, Virginia. Part V identifies the total amount of sediment to be received and treated during the project.

F. SUMMARY OF THE PERMIT CONDITIONS

This section of the fact sheet provides a summary of the standard conditions in the draft permit. The column titled "Regulation" provides the regulatory authority for the permit condition specified in the column titled "Permit Condition." For convenience in reviewing the permit application, the column headed "Location in Application" is provided. The permit application cited in this section is the February 5, 1985 permit application, as amended on April 19, 1985, April 25, 1985, and May 20, 1985.

PART I

STANDARD CONDITIONS

Part I of the permit sets forth the standard procedural conditions that are applicable to the hazardous waste management facility. All citations of the regulations refer to the regulations as codified in Title 40 of the Code of Federal Regulations (40 CFR).

Permit Condition	Subject	Regulation (40 CFR)
I.A	Effect of Permit	§270.4 §270.30(g)
I.B	Permit Actions	§270.30(f) §270.41 §270.42 §270.43
I.C	Severability	§124.16(a)
I.D	Definitions	Part 264 Part 260 Part 270
I.E	Reports, Notifications, and Submissions to the Regional Administrator	
I.F	Signatory Requirement	§270.11 §270.30(k)
I.G	Documents to be Maintained at Facility Site	§264.13 §264.16(d) §264.53(a) §264.112(a) §264.73 §264.15(b) §264.142(d)
I.H	Duties and Requirements	
I.H.1	Duty to Comply	§270.30(a)

Permit Condition	Subject	Regulation (40 CFR)*
I.H.2	Need to Halt or Reduce Activity Not a Defense	§270.30(c)
I.H.3	Duty to Mitigate	§270.30(d)
I.H.4	Proper Operation and Maintenance	§270.30(e)
I.H.5	Property Rights	§270.30(g)
I.H.6	Duty to Provide Information	§270.30(h) §264.74(a)
I.H.7	Inspection and Entry	§270.30(i)
I.H.8	Monitoring and Records	§270.30(j)
I.H.9	Planned Changes	§270.30(l)(1)
I.H.10	Anticipated Non-compliance	§270.30(l)(2)
I.H.11	Twenty-Four Hour Reporting	§270.30(l)(6) §264.56(d)(1) and (j)
I.H.12	Manifest Discrepancy Report	§264.72
I.H.13	Unmanifested Waste Report	§264.76
I.H.14	Other Noncompliance	§270.30)l)(10)
I.H.15	Other Information	§270.30(l)(11)
I.H.16	Protection of Human Health and the Environment	§42 U.S.C. §6925(g)

A. WAIVERS

No waivers from the standard conditions applicable to all hazardous waste management facilities have been given, except those related to requiring Notice to Generators, permit renewal and transferability.

* Title 40 of the Code of Federal Regulations

1. Required Notice to Generator:

The permittee will be responsible for collecting sediment samples and shipping the waste to its facility in Alexandria, VA. Since the permittee will be involved in preparing samples for shipment with the Army (generator), the notice to generator requirements under C.F.R. §264.12(b) have not been included in the permit.

2. Permit Renewal:

Pursuant to Section 3005(g)(4) of RCRA, an RD & D permit may be renewed, for a period not to exceed one year, for a maximum of three times. However, due to the anticipated short duration of the RD & D activities under this proposed permit (approximately 12 weeks), EPA has not included a standard condition providing for renewal. The time provided in the proposed permit, one year from the date of issuance, should be sufficient for completion of all RD & D activities and the closure plan provided in this permit.

3. Transfer of Permit:

Due to the short period of RD & D activities under this proposed permit, the requirements of Sections 270.40 and 270.30(1)(3), which provide procedures for transfer of permits, are not incorporated in the proposed permit. Therefore, this permit may not be transferred.

B. ADDITIONAL CONDITIONS

1. Protection of Human Health and the Environment:

Under Section 3005(g) of RCRA, 42 U.S.C. §6925(g), EPA may order an immediate termination of all operations at an RD & D facility at any time it determines that termination is necessary to protect human health and the environment. This provision is included in Section I.H.14 of the standard conditions of the proposed permit.

PART II

GENERAL FACILITY CONDITIONS

Part II of the permit sets forth general conditions for this facility with which the Permittee must comply. All citations of the regulations refer to the regulations as codified in Title 40 of the Code of Federal Regulations (40 CFR). <u>NOTE</u>: N.C. = NOT COVERED - the application is not required to cover this topic.

Permit Condition	Subject	Regulation (40 CFR)	Location in Application*
II.A	Design and Operation of the Facility	§264.31	(1): pp. 7-22; (3): pp. 4-10.
II.B	General Waste Analysis	§264.13	(3): pp. 1-11; (4): pp. 7-8.
II.C	General Inspection Requirements	§264.15	(3): pp. 12, 14, 15; (4): pp. 9-12.
II.D	Personnel Training	§264.16	(3): pp. 13, 16, 17, 18; (4): p. 16.
II.E	Preparedness and Prevention		
II.E.1	Required Equipment	§264.32	(3): pp. 29, 32; (4): p. 15.
II.E.2	Testing and Maintenance of Equipment	§264.33	(4): p. 13.
II.E.3	Access to Communications or Alarm System	§264.34	(3): pp. 29, 32.
II.E.4	Required Aisle Space	§264.35	(4): p. 14.
II.E.5	Arrangements with Local Authorities	§264.37	(3): pp. 36-39.
II.F	Contingency Plan		

Permit Condition	Subject	Regulation (40 CFR)	Location in Application*
II.F.1	Implementation of Contingency Plan	§264.51	(3): p. 30.
II.F.2	Copies of Plan	§264.53	N.C.
II.F.3	Amendment of Contingency Plan	§264.54	N.C.
II.F.4	Emergency Coordinator	§264.55	(3): p. 30; (4): p. 13.
II.F.5	Emergency Procedures	§264.56	(3): pp. 30, 34, 35.
II.G	Recordkeeping and Reporting		
II.G.1	Operating Record	§264.73	N.C.
II.G.~~1~~2	Required Reports	§264.113 §264.115 §264.143 §264.148 §264.56(d) (1) & (j)	N.C.
II.H	Closure		
II.H.1	Closure Performance Standard	§264.111	(3): p. 41.
II.H.2	Amendment to Closure Plan	§264.112	N.C.
II.H.3	Notification of Closure	§264.113	(3): p. 42.
II.H.4	Time Allowed for Closure	§264.113	(3): p. 42.
II.H.5	Disposal or Decontamination of Equipment	§264.114	(3): pp. 41-42; (4): p. 17.
II.H.6	Certification of Closure	§264.115	(3): p. 42.
II.I	Cost Estimate for Facility Closure		(3): p. 42; (4): p. 17.
II.I.1	Adjustment for Changed Conditions	§264.142(c)	N.C.
II.I.2	Availability	§264.142(d)	N.C.

Permit Condition	Subject	Regulation (40 CFR)	Location in Application*
II.J	Incapacity of Owners/Operators, Guarantors or Financial Institutions	§264.148	N.C.
II.K	Manifest System	§264.71 §264.72 §264.76	(3): p. 40.
II.L	Financial Assurance for Facility Closure	§264.143 §264.151	(2): pp. 15, 16; (3): pp. 46-48.
II.M	Liability Requirements	§264.147	(2): pp. 15-17; (3): pp. 55-57.
II.N	Security	§264.14	(3): p. 12.
II.O	Experimental Procedures	N.A.	(1): pp. 7-22; (3): pp. 4-10.

* Key to Application References:

(1) Application for a Research, Development and Demonstration Permit as specified in "The Hazardous and Solid Waste Amendments of 1984," dated February 5, 1985.

(2) Response to EPA's Questions on ARC Research, Development and Demonstration Permit Application, undated, provided to EPA at April 19, 1985 site visit.

(3) Revision #2, Application for a Research, Development and Demonstration permit as specified in "The Hazardous and Solid Waste Amendments of 1984," dated April 25, 1985.

(4) Revision #3, Application for a Research, Development and Demonstration Permit as specified in "The Hazardous and Solid Waste Amendments of 1984," dated May 20, 1985.

A. WAIVERS

Due to the short period of experimental activities under this permit, the requirements of §264.75 for biennial reports is waived.

B. ADDITIONAL CONDITIONS

An additional condition, II.P (Experimental Procedures), has been included to assure that the scope of activities conducted under this permit are limited to RD&D activities. This additional condition also requires that all leachate produced during the experiment shall be analyzed, collected and stored or recycled for use in the experiment and that the quantity of leachate produced shall be measured and recorded.

PART III

STORAGE IN CONTAINERS

Part III of the permit sets forth conditions for storage in containers with which the Permittee must comply. All citations of the regulations refer to the regulations as codified in Title 40 of the Code of Federal Regulations (40 CFR).

Permit Condition	Subject	Regulation (40 CFR)	Location in Application*
III.A	Waste Identification	§264.13	(4): p. 4.
III.B	Condition of Containers	§264.171	(4): p. 2.
III.C	Placement Requirements	§264.176 §264.177	(3): pp. 21, 26, 27; (4): p. 3.
III.D	Compatibility of Wastes With Containers	§264.172	(3): p. 2.
III.E	Management of Containers	§264.173	(3): p. 2.
III.F	Containment	§264.175	(3): pp. 21, 26, 27; (4): p. 3.

* See key to Application References in Part II of this fact sheet.

PART IV

STORAGE AND TREATMENT IN TANKS

Part IV of the permit sets forth conditions for storage and treatment in tanks with which the Permittee must comply. All citations of the regulations refer to the regulations as codified in Title 40 of the Code of Federal Regulations (40 CFR).

Permit Condition	Subject	Regulation (40 CFR)	Location in Application*
IV.A	Waste Identification	§264.13	(4): p. 4.
IV.B	Design and Construction of Tanks	§264.191	(3): pp. 21-25, 43-44.
IV.C	Protection from Overfilling	§264.192(b)	(1): pp. 16-18.
IV.D	Secondary Containment	N.A.	(3): p. 21; (4): p. 8.

* See key to Application References in Part II of this fact sheet.

A. WAIVERS

Given that the tanks are new and made of corrosion resistant materials, that the hazardous wastes are compatible with the tank materials, that the duration of the experiment is less than one year, and that the lined secondary containment system is in place to contain any leaks or spills, EPA expects that the minimum shell thickness that might result during the experiment will ensure sufficient shell strength.

B. ADDITIONAL CONDITIONS

Permit condition IV.D requires that the Permittee maintain, at all times, a liner beneath the tanks capable of containing all materials spilled or leaked from tanks during loading, unloading or experimental operations.

PART V

SPECIAL CONDITION

Part V of the permit limits the maximum amount of hazardous waste to be received and treated by the facility during the term of this permit to no more than 500 gallons.

Appendix N

Analysis of TNT, RDX, Tetryl, HMX, 2A-DNT and 4A-DNT in Compost Leachates

The water leachate was filtered through a 0.45 millipore filter in preparation for direct injection into the HPLC for quantitation. A LKB 2150 HPLC pump, a Perkin-Elmer LC55 UV detector and a Vista CDS 402 computer controller and integrator were used. Separation was accomplished on a 4.6 mm I.D. x 25 cm Alltech Econosphere 5 μ C-18 column using a mobile phase consisting of 51% methanol, 2% dioxane, 0.5% acetonitrile and 46.5% high purity water which was 0.004 M in n-hexylamine and 0.004 M in tetramethylammonium hydroxide buffered to pH 7.5 with phosphoric acid. The mobile phase flow rate was 1.3 mL/min. UV detection was at 232 nm. Under these conditions, the explosives had the following retention times and detection limits.

Explosive	Retention Time (min)	Detection Limit (mg/L in Leachate)
HMX	3.5	0.248
RDX	4.7	0.303
Tetryl	6.9	0.267
TNT	7.8	0.285
2A-DNT	8.5	0.309
4A-DNT	9.2	0.263

Appendix O

Metal Analysis in Compost Materials and Soils

Barium, Cadmium, Chromium, Copper, Iron, Lead, and Zinc were analyzed according to EPA Methodology using direct aspiration flame Atomic Absorption. Mercury was determine using the Cold Vapor technique according to EPA Method #7471.

Samples were digested for Ba, Cd, Cr, Cu, Fe, Pb, and Zn analysis by EPA Method #3050. Approximately 1 g of ground soil or compost material was weighed into 125 mL Erlenmyer flask. Ten mL of 50:50 HNO_3-H_2O were added and each flask was covered with a small watch glass. The HNO_3-H_2O digest was then boiled on a hot plate for ten minutes. Then 10 mL of concentrated HNO_3 were added and boiling was continued for another 30 minutes. At this point, the flasks were cooled and 2 mL of H_2O and 3 mL of 35% H_2O_2 were added. The flasks were then gently warmed until effervescence started. Then more H_2O_2 was added in 2 mL aliquots until a total 10 mL of H_2O_2 had been added. When the efferverscence subsided, 10 mL of H_2O and 5 mL of 50:50 HCl-H_2O were added. The mixture was refluxed for an additional 10 minutes and cooled. The digest was then filtered through a Whatman #42 ashless filter paper and brought to 100 mL with H_2O in a volumetric flask.

A Varian AA-775 Atomic Absorption Spectrometer with a variable uptake nubulizer and D_2 Background Correction Lamp was used. Uptake rate was set at approximately .7 mL/min. Operating parameters were taken from EPA methods for the individual elements and summaried below:

Barium - λ = 553.6 nm, slit width = .2 nm. A nitrous oxide-acetylene flame in the reducing mode was used. A 10% potassium (as KNO_3) stock was spiked into samples and standards such that all solutions contained, 2000 ppm K. Lamp Current = 10 milliamps. Since a D_2 lamp has insufficient emmision at 553.6 nm wavelength to match the Hollow Cathode lamp, no background correction was used. (EPA Method #7080).

Cadmium - $\lambda = 228.8$ nm, slit width = .5 nm. An air-acetylene flame was used in the oxidizing mode. Background correction was used. Hollow Cathode lamp current was 5 milliamps. (EPA Method #7130)

Chromium - $\lambda = 357.9$ nm, slit width = .2 nm. A nitrous oxide - acetylene flame was used in the reducing mode. 10% potassium (as KNO_3) solution was spiked into all standards and samples such that all solutions contained 2000 ppm K. Since the D_2 lamp emission was insufficient, no background correction was used. (EPA Method #7190)

Copper - $\lambda = 324.7$, slit width = .5 nm. Air-acetylene flame in the oxidizing mode was used. Background correction was used. Hollow Cathode lamp current = 3.5 ma. (EPA Method #7380)

Lead - $\lambda = 283.3$, slit width = .5 nm. Air-acetylene flame in the oxidizing mode was used. Background correction was used. Hollow cathode lamp current = 5 ma. (EPA Method #7420)

Zinc - $\lambda = 213.9$ nm, slit width = 1.0 nm. Air-acetylene flame in the oxidizing mode was used. Background correction was used. Hollow cathode lamp current = 5 ma. 12% strontium (as $Sr(NO_3)_2$) was spiked into all standards and samples such that all solutions contained 1500 ppm Sr. (EPA Method #7950)

Mercury Analysis

Compost samples were screened for mercury using the cold vapor generation technique according to EPA Method #7471. Samples (0.2 grams) were weighed into BOD bottles. Five mL of Aqua Regia and 5 mL of distilled water were added to the bottle and it was placed on a boiling water bath for 2 minutes. The bottle was cooled and 15 mL of 5% potassium permanganate was added before placing the bottle back in the boiling water bath for an additional 30 minutes. After cooling, 55 mL of distilled water and 6 mL of sodium chloride-hydroxylamine sulfate solution were added. This solution contained 12% of each component dissolved in distilled water. At this point, 5 mL of a 10% suspension of stannous sulfate in 0.5 N sulfuric acid were added and the bottle attached to the aeration system.

The aeration system was assembled according to Figure O-1 with the absorption cell placed in the light path of a Varian AA-775 Atomic Absorption Spectrometer. The desiccant used was magnesium perchlorate. A Masterflex peristalltic pump was used and the flow rate was maintained at 1 liter per minute. The flow rate was monitored with a flowmeter inserted between the desiccant tube and the absorption cell.

A mercury Hollow Cathode lamp was used with a 5 ma lamp current. The wavelength was 253.7 nm with a band pass of .5 nm. Absorbance was read directly from the spectrometer readout once it had reached a maximum value.

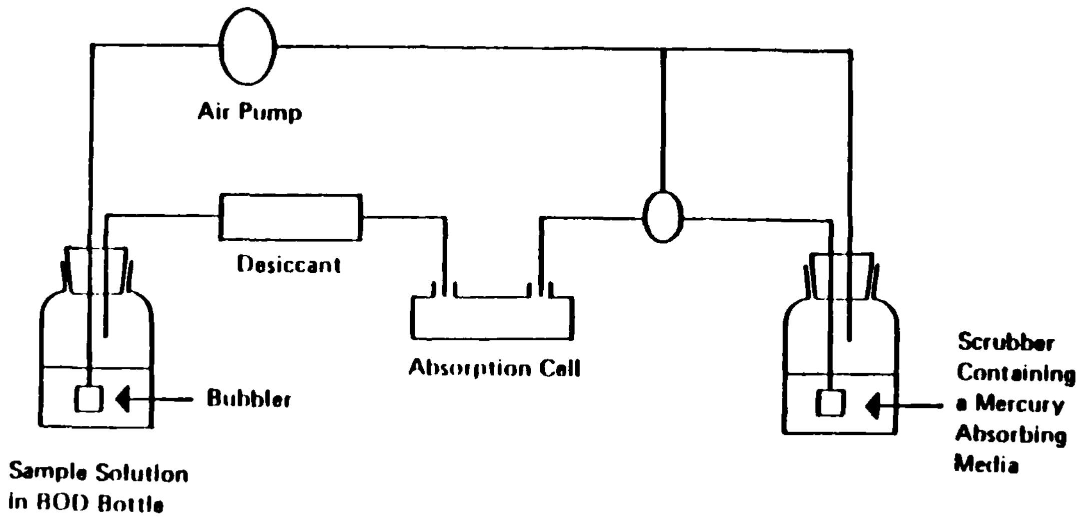

Figure O-1. Apparatus for Flameless Mercury Determination

Appendix P

Metal Analysis in Leachate Samples

Aqueous leachate samples were prepared for metal analysis by EPA Method #3010. Three mL of conc HNO_3 were added to 20 mL of the leachate in a beaker and the beaker covered. The solution was heated (without boiling) to near dryness on a hot plate. The solution was cooled and an additional 3 mL of conc HNO_3 added. The sample was covered and refluxed until it became clear and light colored after which it was evaporated to near dryness. One mL of 1:1 HCl was added to the beaker and warmed to dissolve any precipitate. The solution was transferred to a 10 mL volumetric, and the beaker rinsed with high purity water and added to the solution in the volumetric. High purity water was added to bring the volume to 10 mL. Analysis for the individual metals was as described in Appendix O.

Appendix Q
Pesticide Analysis

Compost samples were screened for pesticides using the EPA contract laboratory procedure for soil samples. This method was taken from an RFP released in January 1985. This procedure was used because EPA method #608 (Federal Register Vol. 48, #209, p. 43321) does not specifically address the preparation of solid samples. Additionally, the method was scaled down by a factor of three to accommodate the limited sample size.

Approximately 10 g of compost sample and 15 grams of anhydrous sodium sulfate were shaken in a 50 mL test tube. The sample was then extracted with 3 x 30 mL portions of 1:1 acetone-methylene chloride using an ultrasonic bath and occasional shaking. The extracts were collected in a 125 mL Erlynmyer flask and the volume reduced to approximately 1 mL under a gentle stream of nitrogen. The flask was washed with 10 mL of hexane and the solution transferred to a 15 mL test tube. The volume was again reduced to 1 mL using nitrogen blowdown. The sample was then quantitatively transferred and brought to volume in a 10 mL volumetric flask.

At this point, an alumina sample clean-up was used. Activity III alumina was prepared baking 100 g of neutral alumina (Fisher) at 400°C for 24 hours in a muffle furnace and, after cooling, adding 7 mL of distilled water. The alumina was then tumbled overnight using a laboratory shaker. Three grams of activity III alumina were placed in a 5 mL serological pipet, fitted with a glass wool plug. 1.9 mL of the hexane extract was then added to the top of the dry column and hexane was used to elute 10 mL in a volumetric flask. This solution was analyzed using gas chromatography and an electron capture detector. The analytical parameters were as follows:

Column:	10 ft x 2 mm I.D. packed with 1.5% OV-17/1.95% OV-210 of 80/100 mesh ANAKROM Q
Column Oven Temperature:	190°C initial hold 2 minutes, then 5°C/minute to 210°C and hold

Injection Port Temperature: 230°C

Detector Temperature: 300°C

Carrier Gas Flow: 24 mL/min Nitrogen

Injection Volumn: 4μl manual

Appendix R

Gas Sample Analysis

Air samples from the compost piles were collected using Pressure-Lok push-button gas syringes. Two 100 µl aliquots were collected for each sample. Analysis was accomplished using a Varian 3700 Gas Chromatograph with thermal conductivity detection. These analyses were performed in two parts using two different columns to detect oxygen, nitrogen, carbon monoxide, carbon dioxide and ammonia.

O_2, N_2, and CO were separated and detected using a 6ft x 1/8in O.D. nickel column packed with 60-80 mesh, acid-washed molecular seives 5A. The GC was operated under the following conditions:

Column Oven Temperature = $60^{o}C$ Isothermal
Injection Port = $200^{o}C$
Thermal Conductivity Oven = $210^{o}C$
Filament Temperature = $310^{o}C$
Helium Carrier Gas Flow = 35 mL/min
Output Sensitivity = 2 x .05 mL full scale

Recording and integration were provided using Hewlett-Packard 5880 A GC computer.

CO_2 and NH_3 were separated and detected using a 5ft x 1/8in O.D. teflon-lined stainless steel column packed with Porpak N 80-100 mesh. The operating conditions were the same as for O_2, N_2, and CO analysis except the column oven was operated in a temperature programmed mode with the following parameters: Initial temperature was $85^{o}C$ for the first two minutes, then $5^{o}C$ per minute to $110^{o}C$ final temperature and hold for 3 minutes. Sensitivity, recording, and integration were the same as for O_2 and N_2 analysis.

Appendix S

Daily Composting Facility Inspection Sheets

COMPOSTING FACILITY INSPECTION

Inspector's Name

Inspection Date:____________________

	Integrity		Response Required		Remedial Action to be taken	Date Remedial Action Completed
Leachate Pump	1 good	leaking	Yes____	No____		
	2 good	leaking	Yes____	No____		
	3 good	leaking	Yes____	No____		
	4 good	leaking	Yes____	No____		
	5 good	leaking	Yes____	No____		
	6 good	leaking	Yes____	No____		
Plumbing	1 good	leaking	Yes____	No____		
	2 good	leaking	Yes____	No____		
	3 good	leaking	Yes____	No____		
	4 good	leaking	Yes____	No____		
	5 good	leaking	Yes____	No____		
	6 good	leaking	Yes____	No____		
Condensate Traps	1 good	leaking	Yes____	No____		
	2 good	leaking	Yes____	No____		
	3 good	leaking	Yes____	No____		
	4 good	leaking	Yes____	No____		
	5 good	leaking	Yes____	No____		
	6 good	leaking	Yes____	No____		

Inspector's Name ______________

Inspection Date: ______________

		Integrity	Response Required		Remedial Action to be taken	Date Remedial Action Completed
Composter Tanks	1 good ___	leaking ___	Yes ___	No ___	______	______
	2 good ___	leaking ___	Yes ___	No ___	______	______
	3 good ___	leaking ___	Yes ___	No ___	______	______
	4 good ___	leaking ___	Yes ___	No ___	______	______
	5 good ___	leaking ___	Yes ___	No ___	______	______
	6 good	leaking	Yes ___	No ___	______	______
Liner for leaks or damage	1 good	moisture or damage ___	Yes ___	No ___	______	______
	2 good	mositure or damage	Yes ___	No ___	______	______
Stored Waste Drums	1 good	leaking	Yes ___	No ___	______	______
	2 good	leaking	Yes ___	No ___	______	______
	3 good	leaking	Yes ___	No ___	______	______
	4 good	leaking	Yes ___	No ___	______	______
	5 good	leaking	Yes ___	No ___	______	______
	6 good	leaking	Yes ___	No ___	______	______
Fencing and gates	1 closed	open or damage ___	Yes	No ___	______	______
greenhouse / storage area	2 closed	open or damage	Yes	No ___	______	______
Fire Extinguishers	1 good	low or empty	Yes	No ___	______	______
	2 good	low or empty	Yes	No ___	______	______

COMPOSTING FACILITY INSPECTION

	Spills	Response Required	Quality & Dispositon of of Spilled Matter	Date Remedial Action Completed
B Transport Area	Yes ____ No ____			

C

Leachate Traps

Liquid Levels, ml.

1 volume ____ ml.
2 volume ____ ml.
3 volume ____ ml.
4 volume ____ ml.
5 volume ____ ml.
6 volume ____ ml.

Condensate Traps

1 volume ____ ml.
2 volume ____ ml.
3 volume ____ ml.
4 volume ____ ml.
5 volume ____ ml.
6 volume ____ ml.

Appendix T

Louisiana AAP Sediment Pilot-Scale Composting

T-1

Daily Temperatures (°C) - Louisiana AAP Pilot Scale Composts

	Hay-Horse Feed			Sewage Sludge-Wood Chips		
DATE	1	2	3	4	5	6
8/22/85	44	39	23	40	44	47
8/23/85	53	38	33	45	42	51
8/24/85	42	41	35	48	47	45
8/25/85	43	44	41	45	46	31
8/26/85	44	44	38	42	47	45
8/27/85	39	44	38	42	47	47
8/28/85	45	44	43	45	43	44
8/29/85	44	45	48	46	43	41
8/30/85	47	45	52	46	46	47
8/31/85	47	50	58	45	50	48
9/01/85	49	49	58	43	52	50
9/02/85	47	48	50	42	51	49
9/03/85	48	47	51	45	51	52
9/04/85	45	47	53	45	50	52
9/05/85	48	45	55	47	50	50
9/06/85	50	53	55	46	50	48
9/07/85	52	55	53	46	50	47
9/08/85	51	54	53	46	50	49
9/09/85	51	52	52	46	50	48
9/10/85	49	50	49	45	49	49
9/11/85	50	61	52	55	50	48
9/12/85	50	59	47	50	51	38
9/13/85	48	59	53	49	50	32
9/14/85	66	65	71	50	51	59
9/16/85	65	61	65	49	52	60
9/17/85	65	60	65	46	49	56
9/18/85	61	60	66	43	51	53
9/19/85	67	64	65	45	49	48
9/20/85	61	62	67	47	50	48
9/21/85	58	58	58	51	51	49
9/22/85	54	58	55	52	52	48
9/23/85	54	54	52	47	48	49
9/24/85	54	58	53	45	47	53
9/25/85	56	61	50	48	48	51
9/26/85	62	67	59	46	48	47
9/27/85	66	65	62	51	52	47
9/28/85	63	62	52	49	48	50
9/29/85	55	54	50	51	50	53
9/30/85	54	54	51	47	51	52
10/1/85	52	54	48	47	48	49
10/2/85	53	67	48	53	52	50
10/3/85	62	63	55	52	51	50
10/4/85	59	57	58	47	51	52
10/5/85	56	60	56	51	51	52
10/7/85	45	50	46	47	48	49

T-1A

Daily Temperatures (°C) - Louisiana AAP Pilot Scale Manure Composts

DATE	Tank 5	Tank 6
2/21/86	-	63
2/22/86	26	76
2/23/86	37	56
2/24/86	47	69
2/25/86	51	65
2/26/86	53	64
2/27/86	53	62
2/28/86	57	57
3/1/86	54	66
3/2/86	53	70
3/3/86	56	65
3/4/86	47	59
3/5/86	47	60
3/6/86	45	63
3/7/86	42	62
3/8/86	39	59
3/9/86	39	61
3/10/86	37	61
3/11/86	38	62
3/12/86	40	62
3/13/86	39	53
3/14/86	53	63
3/15/86	62	63
3/16/86	66	60
3/17/86	70	58
3/18/86	71	58
3/19/86	69	57
3/20/86	70	57
3/21/86	68	55
3/22/86	69	57
3/23/86	69	57
3/24/86	67	57
3/25/86	66	54
3/26/86	65	52
3/27/86	63	49
3/28/86	62	48
3/29/86	72	53
3/30/86	71	55
3/31/86	56	55
4/1/86	64	55
4/2/86	66	54
4/3/86	64	53
4/4/86	62	51
4/5/86	58	46
4/6/86	61	42
4/7/86	58	39
4/8/86	58	41
4/9/86	57	40
4/10/86	55	38
4/11/86	53	35
4/12/86	55	36
4/13/86	56	35
4/14/86	54	33
4/15/86	55	35
4/16/86	52	32
4/17/86	51	31
4/18/86	49	No reading taken

T-2

Metals Analysis of Louisiana AAP Compost Components

Component	Dry Weight lbs	Iron µg/g	Copper µg/g	Cadmium µg/g	Chromium µg/g	Lead µg/g	Zinc µg/g	Barium µg/g	Mercury µg/g
Tank #1									
Hay -	225	601.9	-	-	-	-	3.0	-	NA
Horse Feed	222	239.9	-	-	-	-	50.0	-	NA
LAAP Soil-B	54	13,479	38.16	-	423.7	2,430.9	-	65.4	0.75
Tank #2									
Hay-	214	2,755	-	-	-	-	23.9	-	NA
Horse Feed	211	248.0	-	-	-	-	60.1	-	NA
LAAP Soil-A	59	36,925	80.4	-	240.4	1,673.3	-	85.7	2.84
Tank #4									
SS-WC	410	56,366	284.4	3.272	171.77	151.3	285.38	430.5	0.62
LAAP Soil-B	155	13,479	38.16	-	423.7	2,430.9	-	65.4	0.75
Tank #5									
SS-WC	441	39,372	287.0	4.102	171.61	149.1	205.45	428.03	0.62
LAAP Soil-A	163	36,925	80.4	-	240.4	1,673.3	-	85.7	2.84

NA - Not Analyzed

- Not Detected

Detection Limits in Compost and Materials:

Fe	33	ppm
Cu	13	ppm
Cr	27	ppm
Cd	3.3	ppm
Zn	33	ppm
Hg	.2	µg

T-3

Leachate Metals*

Tank I - Hay-Horse Feed Compost with Louisiana AAP-B Soil

Leachate pH	Leachate Vol., L.	IRON ppm	IRON Amount Leached, g	COPPER ppm	COPPER Amount Leached, g	CHROMIUM ppm	CHROMIUM Amount Leached, g	LEAD ppm	LEAD Amount Leached, g	ZINC ppm	ZINC Amount Leached, g.
6.55	.503	10.096	.0051	-	-	-	-	2.972	.0015	612.7	.308
8.2	.716	2.85	.0020	.517	.00037	-	-	2.817	.0020	13.36	.0096
8.9	.780	.670	.0005	3.02	.0024	-	-	-	-	5.89	.0046
8.4	8.056	.679	.00547	1.83	.0147	-	-	-	-	8.98	.0723
8.7	14.85	-	-	2.22	.0330	-	-	-	-	8.77	.1302
8.9	9.30	.42	.00391	2.44	.0227	-	-	-	-	22.63	.2105
8.9	5.10	.61	.0031	1.984	.0101	-	-	-	-	9.403	.0480
			.0201		.0833		-		.0035		.7832

*No Cadmium or Mercury detected in leachate

- Not detected

Detection Limits: Fe .5 ppm Cd .05 ppm
Cu .2 ppm Pb 1 ppm
Cr .4 ppm Zn .5 ppm
Hg .2 μg

T-4

Leachate Metals*

Tank II - Hay-Horse Feed Compost with Louisiana AAP-A Soil

		IRON		COPPER		CHROMIUM		LEAD		ZINC	
Leachate pH	Leachate Vol, L.	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
5.65	.670	23.50	.016	-	-	-	-	1.971	.0013	1215.4	.8228
7.95	1.033	8.47	.0088	-	-	-	-	-	-	20.10	.0208
8.85	1.106	-	-	.55	.0006	-	-	-	-	9.29	.0103
8.5	8.588	.578	.0050	3.05	.0262	.51	.0044	-	-	31.63	.2716
8.7	12.935	.570	.0074	2.71	.0351	-	-	-	-	16.48	.2132
8.7	11.300	.825	.0093	1.66	.0188	-	-	-	-	17.85	.2017
8.7	7.25	1.319	.0096	1.963	.0142	-	-	-	-	28.41	.2060
			.0561		.0949		.0044		.0013		1.746

* No Cadmium, Barium or Mercury detected in Leachate

\- Not detected

Detection Limits:
Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 µg
Cd .05 ppm
Pb 1 ppm
Zn .05 ppm

T-5

Leachate Metals*

Tank III - Hay-Horse Feed Compost with Louisiana AAP Non-Contaminated Soil

Leachate pH	Leachate Vol, L.	IRON ppm	IRON Amount Leached, g	COPPER ppm	COPPER Amount Leached, g	LEAD ppm	LEAD Amount Leached, g	ZINC ppm	ZINC Amount Leached, g.
6.6	.583	9.151	.0053	-	-	-	-	607.67	.3543
8.3	1.162	-	-	-	-	5.775	.0067	10.54	.0123
8.75	1.599	.5	.0008	3.44	.0055	-	-	19.26	.0308
8.6	10.352	.724	.0075	3.52	.0364	-	-	49.06	.5079
8.7	4.229	1.52	.0064	2.24	.0095	-	-	38.35	.1622
8.9	2.314	1.10	.0026	2.27	.0053	-	-	31.63	.0732
8.9	2.080	2.195	.0045	2.139	.0045	-	-	14.224	.0296
			.0271		.0612		.0067		1.1703

* No Cadmium, Chromium, Barium or Mercury detected in leachate.

\- Not detected

Detection Limits:
Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 μg
Cd .05 ppm
Pb 1 ppm
Zn .05 ppm

T-6

Leachate Metals*

Tank IV - Sewage Sludge-Wood Chips with Louisiana AAP-B Soil

Leachate pH	Leachate Vol, L.	IRON		COPPER		LEAD		ZINC	
		ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
8.4	.592	4.009	.0024	-	-	-	-	13.7	.0081
8.4	.930	1.410	.0013	1.511	.0014	2.622	.0024	30.22	.0281
8.85	3.118	-	-	3.40	.0106	-	-	13.36	.0417
8.6	3.262	.713	.0023	3.21	.0105	-	-	20.66	.0674
8.9	1.143	.690	.0008	3.48	.0040	-	-	34.09	.0390
8.9	3.160	.940	.0030	3.36	.0106	-	-	30.78	.1004
8.9	2.995	.834	.0025	1.911	.0057	-	-	18.859	.0565
			.0123		.0428		.0024		.3412

* No Cadmium, Chromium, Barium or Mercury detected in leachate.

\- Not detected

Detection Limit: Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 μg
Cd .05 ppm
Pb 1 ppm
Zn .05 ppm

T-7

*Leachate Metals**

Tank V - Sewage Sludge-Wood Chips with Louisiana AAP-A Soil

		IRON		COPPER		LEAD		ZINC	
Leachate pH	Leachate Vol, L.	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
8.3	.724	9.818	.0071	-	-	-	-	8.850	.0064
8.3	1.054	2.69	.0028	-	-	2.077	.0022	8.644	.0091
8.75	.762	-	-	.35	.0003	-	-	?	?
8.8	1.646	9.46	.0156	1.08	.0018	-	-	33.60	.0553
8.8	.244	6.71	.0016	1.05	.0003	-	-	68.53	.0167
8.7	2.545	7.56	.0192	.103	.0003	-	-	25.72	.0655
8.7	4.00	9.056	.0362	.065	.0003	-	-	27.80	.0655
			.0825		.0030		.0022		.1092

* No Cadmium, Chromium, Barium or Mercury detected in leachate

\- Not detected

Detection Limits:

Fe .5 ppm	Cd .05 ppm
Cu .2 ppm	Pb 1 ppm
Cr .4 ppm	Zn .05 ppm
Hg .2 μg	

T-8

Leachate Metals*

Tank VI - Sewage Sludge-Wood Chips with Louisiana AAP Non-Contaminated Soil

		IRON		COPPER		LEAD		ZINC	
Leachate pH	Leachate Vol, L.	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
8.7	.591	2.822	.0017	-	-	-	-	18.50	.0109
8.55	.909	-	-	2.505	.0023	2.139	.0019	80.26	.0730
9.0	.924	-	-	1.62	.0015	-	-	92.06	.0851
8.5	10.105	1.216	.0123	2.51	.0254	-	-	9.263	.0936
8.8	2.783	1.66	.0046	1.93	.0054	-	-	29.21	.0813
8.9	5.06	2.59	.0130	.993	.0050	-	-	8.25	.0418
8.9	4.56	3.00	.0137	.366	.0017	-	-	1.523	.0069
			.0453		.0413		.0019		.3926

*No Cadmium, Chromium, Barium or Mercury detected in leachate

- Not detected

Detection Limits: Fe .5 ppm Cd .05 ppm
Cu .2 ppm Pb 1 ppm
Cr .4 ppm Zn .05 ppm
Hg .2 μg

T-9

Explosives in Leachate

Tank I - Hay-Horse Feed Compost with Louisiana AAP-B Soil

	TNT		RDX		HMX		2A-DNT		4A-DNT	
Leachate Vol., L.	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
.503	-	-	-	-	-	-	-	-	-	-
.716	-	-	-	-	-	-	-	-	-	-
.780	-	-	-	-	-	-	-	-	-	-
8.056	-	-	-	-	6.71	.0245	-	-	-	-
14.85	1.426	.0096	1.462	.0099	.16	.0011	-	-	-	-
9.30	.377	.0016	1.631	.0069	.57	.0024	-	-	-	-
5.10	-	-	.07	.0002	-	-	-	-	-	-
		.0112		.0017		.028		-		-

* Significant interference from compost materials

\- Not Detected

Detection Limits: TNT .285 ppm
RDX .303 ppm
HMX .248 ppm
2A-DNT .309 ppm
4A-DNT .263 ppm

T-10

Explosives in Leachate

Tank II - Hay-Horse Feed Compost with Louisiana AAP-A Soil

Leachate Vol., L	TNT ppm	TNT Amount Leached, g	RDX ppm	RDX Amount Leached, g	HMX ppm	HMX Amount Leached, g	2A-DNT ppm	2A-DNT Amount Leached, g	4A-DNT ppm	4A-DNT Amount Leached, g.
.670	1.86	.0006	-	-	.482	.0002	-	-	-	-
1.033	-	-	-	-	-	-	-	-	-	-
1.106	-	-	-	-	-	-	-	-	-	-
8.588	-	-	1.743	.0007	4.699	.0018	.423	.0017	.763	-
12.935	.889	.0052	1.795	.0105	.110	.0007	.308	.0018	-	-
11.300	.384	.0020	.404	.0021	-	-	.162	.0008	-	-
7.25	-	-	-	-	-	-	-	-	-	-
		.0078		.0133		.0027		.0043		-

* Significant interference from compost materials

- Not Detected

Detection Limits: TNT .285 ppm
RDX .303 ppm
HMX .348 ppm
2A-DNT .309 ppm
4A-DNT .263 ppm

T-11

Explosives in Leachate

Tank IV - Sewage Sludge-Wood Chips Compost with Louisiana AAP-B Soil

Leachate Vol., L.	TNT		RDX		HMX		2A-DNT		4A-DNT	
	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
.592	4.23	.0011	.672	.0002	-	-	.373	.0001	-	-
.930	1.694	.0007	.863	.0004	-	-	-	-	-	-
3.118	.440	.0006	9.300	.0132	3.973	.0056	-	-	.861	.0012
3.262	-	-	7.565	.0112	3.284	.0049	.779	.0012	.444	.0007
1.143	.056	.00003	3.946	.0021	.517	.0003	.24	.0001	-	-
3.160	-	-	4.348	.0062	.711	.0010	.815	.0012	.206	.0003
2.995	-	-	4.051	.0055	.317	.0004	1.969	.0027	1.296	.0018
		.00243		.0388		.0122		.0053		.0040

* Significant interference from compost materials

\- Not Detected

Detection Limits: TNT .285 ppm
RDX .303 ppm
HMX .248 ppm
2A-DNT .309 ppm
4A-DNT .263 ppm

T-12

Explosives in Leachate

Tank V - Sewage Sludge-Wood Chips with Louisiana AAP-A Soil

Leachate Vol., L.	TNT		RDX		HMX		2A-DNT		4A-DNT	
	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g.
.724	1.12	.0004	-	-	-	-	-	-	-	-
1.054	.676	.0003	-	-	.316	.0002	-	-	-	-
.762	-	-	-	-	-	-	-	-	-	-
1.646	-	-	-	-	-	-	-	-	-	-
.244	.103	.00001	1.708	.0002	.552	.00006	.161	.00002	-	-
2.545	-	-	6.258	.0072	1.754	.0020	.933	.0011	.567	.0007
4.000	-	-	4.541	.0083	.886	.0016	.119	.0002	-	-
		.00071		.0157		.00386		.00132		.0007

\- Not Detected

Detection Limits: TNT .285 ppm
RDX .303 ppm
HMX .248 ppm
2A-DNT .307 ppm
4A-DNT .263 ppm

T-13

Explosives in Leachate

Tank V - Manure-Hay-Saw Dust with Louisiana AAP-C Soil

Time Period Collected	Leachate Vol., L.	TNT ppm	TNT Amount Leached, g	RDX ppm	RDX Amount Leached, g	HMX ppm	HMX Amount Leached, g	2 & 4A-DNT ppm	2 & 4A-DNT Amount Leached, g	Tetryl ppm	Tetryl Amount Leached, g
3/1-3/7	.580	-	-	-	-	-	-	-	-	-	-
3/8 - 3/15	.200	.239	.00005	1.934	.00039	2.009	.00040	.347	.00007	-	-
3/16 - 3/21	1.845	-	-	*		*		-	-	-	-
3/22 - 3/28	10.950	-	-	-	-	**		-	-	-	-
3/29 - 4/4	19.650	-	-	-	-	-	-	-	-	-	-
4/5 - 4/18	4.100	-	-	**		-	-	-	-	-	-
Maximum Totals			.00005		.00039		.00040		.00007		0

* Trace (unreliable integration)

** Masked by compost organics

U. Badger AAP Sediment Pilot-Scale Composting

U-1

Daily Temperatures (^{o}C) - Badger AAP Pilot Scale Composts

	TANKS					
DATE	1	2	3	4	5	6
10/30/85	34	35	-	20	35	-
10/31/85	65	73	-	21	47	-
11/01/85	72	80	-	24	55	-
11/02/85	78	73	-	31	57	-
11/03/85	75	72	-	37	56	-
11/04/85	68	77	-	50	67	-
11/05/85	76	69	-	68	68	-
11/06/85	83	86	-	68	72	-
11/07/85	78	75	-	74	65	-
11/08/85	73	69	-	70	66	-
11/09/85	75	70	-	68	62	-
11/11/85	69	62	-	68	67	-
11/12/85	82	86	-	68	63	-
11/13/85	80	71	-	68	67	-
11/14/85	66	52	-	65	68	-
11/15/85	65	71	-	64	57	-
11/16/85	70	83	-	60	52	-
11/17/85	71	62	-	55	52	-
11/18/85	74	63	-	52	42	-
11/19/85	65	63	-	55	46	-
11/20/85	72	77	-	61	55	-
11/21/85	50	65	-	57	53	-
11/22/85	65	70	-	55	47	-
11/23/85	71	75	-	52	40	-
11/24/85	55	60	-	50	43	-

U-2

Metal Analysis of Badger Time Zero Compost

Tank #	Weight lbs	Iron µg/g	Copper µg/g	Cadmium µg/g	Chromium µg/g	Lead µg/g	Zinc µg/g	Barium µg/g	Mercury µg/g
1 Hay-Horse Feed with BAAP-B Sediment	508	3,837	25.2	-	-	-	740.0	-	.68
2 Hay-Horse Feed with BAAP-B Sediment	523	2,966	21.6	-	-	-	2,058.9	-	.675
4 Sewage Sludge-Wood Chips with BAAP-A Sediment	702	44,820	214.5	-	166.8	106.6	3,389.6	400.2	.776
5 Sewage Sludge-Wood Chips with BAAP-A Sediment	742	45,023	234.8	-	163.9	99.6	4,356.0	416.1	.715

- Not Detected

Detection Limits in Solids: Fe 33 ppm
Cu 13 ppm
Cr 27 ppm
Cd 3.3 ppm
Zn 33 ppm
Hg .2 µg

U-3

Leachate Metals *

Tank I - Hay-Horse Feed Compost with Badger AAP-B Soil

Time Period Collected	Leachate pH	Leachate Vol., L	IRON ppm	IRON Amount Leached, g	ZINC ppm	ZINC Amount Leached, g	COPPER ppm	COPPER Amount Leached, g
10/29 - 11/4		None						
11/5 - 11/11	8.1	.355	1.175	.00042	48.1	.01708	1.282	.00046
11/12 - 11/18	8.7	3.035	.711	.00216	12.03	.03651	1.495	.00454
11/19 - 11/24	8.8	1.725	2.843	.00490	8.34	.01439	1.920	.00331
				.00748		.06798		.00831

* No Cadmium, Chromium, Barium, Lead or Mercury detected in leachates

Detection Limits:
Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 μg
Cd .05 ppm
Pb 1 ppm
Zn .5 ppm

U-4

Leachate Metals*

Tank II - Hay-Horse Feed Compost with Badger AAP-B Soil

			IRON		ZINC		COPPER	
Time Period Collected	Leachate pH	Leachate Vol., L	ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g
10/29 - 11/4		None						
11/5 - 11/11	9.0	.845	.924	.00078	17.70	.01496	1.718	.00146
11/12 - 11/18	8.5	3.600	.284	.00102	10.21	.03676	1.473	.00531
11/19 - 11/24	8.8	4.825	2.592	.001251	4.87	.02350	2.334	.01126
				.00305		.07522		.01803

* No Cadmium, Chromium, Barium, Lead or Mercury detected in leachates

Detection Limits:

Fe .5 ppm	Cd .05 ppm
Cu .2 ppm	Pb 1 ppm
Cr .4 ppm	Zn .5 ppm
Hg .2 μg	

U-5

Leachate Metals*

Tank III - Sewage Sludge-Wood Chips Compost with Badger AAP-A Soil

Time Period Collected	Leachate pH	Leachate Vol., L	IRON ppm	IRON Amount Leached, g	ZINC ppm	ZINC Amount Leached, g	COPPER ppm	COPPER Amount Leached, g
10/29 - 11/4		None						
11/5 - 11/11	8.7	3.71	.460	.00171	44.15	.1638	.336	.00125
11/12 - 11/18	8.7	9.63	.535	.00515	42.40	.40831	1.930	.01859
11/19 - 11/24	8.6	3.075	1.764	.00542	40.50	.12454	2.982	.00917
				.01228		.69665		.02901

* No Cadmium, Chromium, Barium, Lead or Mercury detected in leachates

Detection Limits:
Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 μg
Cd .05 ppm
Pb 1 ppm
Zn .5 ppm

U-6

Leachate Metals*

Tank IV - Sewage Sludge-Wood Chips Compost with Badger AAP-A Soil

Time Period Collected	Leachate pH	Leachate Vol., L	IRON		ZINC		COPPER	
			ppm	Amount Leached, g	ppm	Amount Leached, g	ppm	Amount Leached, g
10/29 - 11/4		None						
11/5 - 11/11	9.1	.997	1.576	.00157	1290.0	1.2861	8.46	.00843
11/12 - 11/18	8.9	7.326	2.442	.01789	343.0	2.51282	1.526	.01118
11/19 - 11/24	8.6	2.965	1.037	.00307	15.5	.04611	1.792	.00531
				.02253		3.84503		.02492

* No Cadmium, Chromium, Barium, Lead or Mercury detected in leachates

Detection Limits:
Fe .5 ppm
Cu .2 ppm
Cr .4 ppm
Hg .2 µg
Cd .05 ppm
Pb 1 ppm
Zn .5 ppm

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