COMPOSTING OF SOILS/SEDIMENTS AND SLUDGES CONTAINING TOXIC ORGANICS INCLUDING HIGH ENERGY EXPLOSIVES

Final Report

Contract No. 02112412

By

Richard C. Doyle and Judith F. Kitchens

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PREFACE

Currently available technology is not adequate to assess environmental contamination at Department of Energy (DOE) sites, take permanent remedial action, and eliminate or minimize the environmental impact of future operations. Technical resources to address these shortcomings exist within the DOE community and the private sector, but the involvement of the private sector in attaining permanent and cost-effective solutions has been limited.

During 1990, on behalf of DOE's Office of Technology Development, Argonne National Laboratory (ANL) conducted a competitive procurement of research and development projects addressing soil remediation, groundwater remediation, site characterization, and contaminant containment. Fifteen contracts were negotiated in these areas.

This report documents work performed as part of the Private Sector Research and Development Program sponsored by the DOE's Office of Technology Development within the Environmental Restoration and Waste Management Program. The research and development work described herein was conducted under contract to ANL.

On behalf of DOE and ANL, I wish to thank the performing contractor and especially the report authors for their cooperation and their contribution to development of new processes for characterization and remediation of DOE's environmental problems. We anticipate that the R&D investment described here will be repaid many-fold in the application of better, faster, safer, and cheaper technologies.

Details of the procurement process and status reports for all 15 of the contractors performing under this program can be found in "Applied Research and Development Private Sector Accomplishments - Interim Report" (Report No. DOE/CH-9216) by Nicholas J. Beskid, Jas S. Devgun, Mitchell D. Erickson and Margaret M. Zielke.

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ABSTRACT

Laboratory and pilot-scale experimentation were conducted to evaluate composting as an on-site treatment technology to remediate soils contaminated with hazardous waste at DOE's PANTEX Plant. Suspected contaminated sites within the PANTEX Plant were sampled and analyzed for explosives, other organics, and inorganic wastes. Soils in drainage ditches and playas at PANTEX Plant were found to be contaminated with low levels of explosives (including RDX, HMX, PETN and TATB). Additional sites previously used for solvent disposal were heavily contaminated with solvents and transformation products of the solvents, as well as explosives and by-products of explosives. Laboratory studies were conducted using ^{14}C -labeled explosives and ^{14}C -labeled diacetone alcohol contaminated soil loaded into horse manure/hay composts at three rates: 20, 30, and 40%(W/W). The composts were incubated for six weeks at approximately 60°C with continuous aeration. All explosives degraded rapidly and were reduced to below detection limits within 3 weeks in the laboratory studies. ^{14}C -degradates from ^{14}C -RDX, ^{14}C -HMX and ^{14}C -TATB were largely limited to $^{14}\text{CO}_2$ and unextracted residue in the compost. Volatile and non-volatile ^{14}C -degradates were found to result from ^{14}C -PETN breakdown, but these compounds were not identified. ^{14}C -diacetone alcohol concentrations were significantly reduced during composting. However, most of the radioactivity was volatilized from the compost as non- $^{14}\text{CO}_2$ degradates or as ^{14}C -diacetone alcohol. Pilot scale composts loaded with explosives contaminated soil at 30% (W/W) with intermittent aeration were monitored over six weeks. Data from the pilot-scale study generally was in agreement with the laboratory studies. However, it was found that the ^{14}C -labeled TATB degraded much faster than the unlabeled TATB. It is suspected that some formulations of TATB may be more resistant to composting activity than others.

EXECUTIVE SUMMARY

Technology Description

Composting is a biological process that utilizes the unique ability of thermophilic microorganisms to degrade toxic organics such as high energy explosives in soils, sediments and sludges. To perform the remediation, soils containing the toxic organics are mixed with nutrient and bulking agents and laid in a pile. The nutrients and bulking agents used can be any high organic-containing waste that is available locally. Typically animal manure, animal bedding, corn stalks, waste hay or straw, etc. are acceptable for use as nutrients or bulking agents. For hazardous waste composting, hot (60-65°C), aerobic composts are used to facilitate the degradation of the toxic organics. In the field, this type of compost is achieved in forced-aerated windrows (long trapezoidal-shaped piles that are turned routinely and aerated).

In this project, IIT Research Institute evaluated composting on a laboratory and pilot-scale as a remediation technique for explosives found in the soils at DOE's Pantex Plant near Amarillo, Texas. In the initial task, a preliminary survey of Pantex soils was performed to determine the types and levels of explosives present in the soils. Sixty-nine soil samples and one water sample were analyzed for the explosives TATB, PETN, RDX, and HMX. Levels of these explosives in the soils were found to range from non-detectable to the following concentrations:

*	RDX	-	66 µg/g
*	HMX	-	109 µg/g
*	PETN	-	201 µg/g
*	TATB	-	1349 µg/g

Laboratory-scale composts were run to 1) determine the effectiveness of the composting process for degrading the combination of contaminants in the Pantex soil, 2) determine the soil loading levels tolerated by the compost microorganisms, and 3) identify the major degradates of the explosives and the mechanisms of degradation.

For the laboratory-scale composts, uncontaminated soil from the Barrow Pit at Pantex was spiked with the explosives at approximately twice the levels found in the Pantex soils during the preliminary survey. Four separate studies were run with all four explosives in the soil but with a ¹⁴C-label on a different explosive in each study. Three soil loading levels were also evaluated for each ¹⁴C-labeled explosive. To set up each study, 27 weighed soil samples (9 for each soil loading level) were spiked with the four explosives (one with the radiotracer). The soil samples were mixed with preweighed amounts of hay, horsefeed and horse manure in one-quart Mason jars. The jars (18 for each study) were placed in the test system shown in Figure 1. The prescrubbers and the Mason jars were then placed in an incubator at 60-65°C. The remaining 9 composts were taken as time zero samples. After three weeks (two weeks for ¹⁴C-PETN composts) and six weeks of composting, 9 additional samples at each time were taken for analysis. The compost samples were extracted with acetonitrile and analyzed by HPLC and liquid scintillation counting (LSA). All offgas traps were also analyzed for the presence of ¹⁴C degradates.

Pilot-scale composting was performed to determine realistic degradation rates in self-sustaining, large mass composts. For this study, 500 gal. stainless steel tanks were used as the composters as shown in Figure 2. For the pilot studies, a silt-loam soil was weighed, spiked with the four explosives and thoroughly mixed in a cement mixer. This soil was added (at a 30% soil loading level) to preweighed batches of hay, horsefeed and horse manure and mixed in the cement mixer. The resultant mixtures were dumped into the compost tanks. Three replicates of the treated compost and one control were run.

The pilot-scale composters were intermittently aerated and were turned at least twice weekly. The composters were sampled on days 0, 12, 18, and 42. The samples were analyzed by HPLC for the explosives.

Technology Performance

The laboratory-scale composts showed that the explosives found in Pantex soils were amenable to degradation by the composting process. The order of explosives degradation was PETN>RDX>HMX>TATB. RDX and HMX were mineralized in the composts as indicated by the large quantities of $^{14}\text{CO}_2$ given off by these composts. PETN was also mineralized to some extent, however, highly polar ^{14}C -compounds were found in the composts. These compounds slowly disappeared with production of $^{14}\text{CO}_2$ as the composting process continued. TATB was more slowly metabolized by the compost microorganisms with small amounts of $^{14}\text{CO}_2$ given off (15% of the total ^{14}C -activity after 6 weeks of composting).

Pilot-scale composting resulted in even more rapid degradation of RDX, HMX, and PETN. After 18 days of composting, these explosives were non-detectable in the compost extracts. Approximately 32-39% of the TATB was still present in the compost extracts after 42 days. The half-life of TATB was calculated to be 25.3 days. Although the pilot-scale composts were self-sustaining, excessive moisture in these composts resulted in anaerobic pockets and lower compost temperatures than desired. It is anticipated that the TATB degradation rate can be improved dramatically in large composts. Seeding the larger composts with acclimated microorganisms could also be used to improve the TATB degradation rate.

The compost volumes in the pilot-scale tests were reduced by about 70% or approximately twice the volume of the soil. When the compost residue was mixed with soil and utilized to grow rye and soybeans, healthy foliage was obtained indicating no phytotoxic effects from the compost residues.

Based on the data obtained in the pilot-scale studies, a demonstration plant was designed and costed. This demonstration plant would treat 55 cu. yds. of explosives contaminated soil using four windrows (92 ft. long by 14 ft. at the base). Capital and operating costs for this demonstration plant were estimated at \$187,874 and \$408,740, respectively.

Remediation Costs

A preliminary design and cost analysis for a full-scale composting plant for remediation of Pantex soils was performed. Composting to cleanup approximately two miles of Pantex ditches would cost approximately \$1,324,979 or \$226/ton and take about one year to complete. This cost is significantly less than incineration of explosives contaminated soils (\$272/ton in 1989 dollars). The trade-off between composting and incineration involves public opinion and the time-frame in which the remediation must be completed.

Figure 1. Laboratory-Scale Composting Test System

Figure 2. Schematic of Pilot-Scale Composter

1.0 INTRODUCTION

The main objective of this research program was to evaluate composting as a remediation technology for soils, sludges, and sediments contaminated with hazardous organics, including high energy explosives. This research program was specifically aimed at evaluating the composting process for cleanup of high energy explosives contamination in the soils at DOE's Pantex Plant.

The program was divided into four main tasks:

- * A preliminary survey of Pantex to determine the approximate levels and type of explosives present in the soil.
- * Laboratory-scale composting aimed at 1) determining the effectiveness of the composting process for degrading the combination of contaminants in the Pantex soil, 2) determining the soil loading levels tolerated by the compost microorganisms and 3) identifying the major degradates of the explosives and the mechanisms of degradation.
- * Pilot-scale composting to determine the degradation rates under self-sustaining composting conditions.
- * Design a demonstration plant for the decontamination of Pantex soils and perform a preliminary economic analysis for a full-scale cleanup of soils at Pantex.

1.1 Technology Scope

The specific DOE technology area addressed in this research effort was "Need for Remediating Soils Contaminated with Organics and Nitrates." Current methods for remediating soils contaminated with hazardous organics include:

- * excavation and landfill
- * excavation and on-site incineration followed by landfill of the nonhazardous soil
- * air or steam stripping
- * *in situ* biodegradation
- * radio frequency heating.

Air or steam stripping and radio frequency heating are applicable to the removal of volatile organics, *e.g.*, TCE and fuels, from soils. Bioremediation can be effectively applied to shallow soils containing low levels of degradable organics. Incineration and landfill have been the major technologies utilized for soils that are highly contaminated with recalcitrant organics. Composting, with its unique biodegradation capabilities, is an alternative to incineration and landfill. In

contrast with incineration, the resulting product is a fertile soil which can be returned to the original site or used elsewhere on the facility.

1.2 Technology Programmatic Requirements

This project is part of the Applied Research and Development phase of the overall DOE Technology Integration Program for environmental restoration and waste management operations. As part of the Applied Research and Development phase, the project was needs driven. A DOE site, namely the Pantex Plant near Amarillo, Texas, was known to have soils that are contaminated with high energy explosives, *i.e.*, TATB, PETN, HMX, and RDX. The end goal of this project was to obtain sufficient data on the composting of explosives contaminated soil that would allow the generation of engineering specifications for a demonstration plant.

2.0 METHODOLOGY AND APPROACH

2.1 Preliminary Survey of Pantex Plant

In December, 1990, IIT Research Institute (IITRI) personnel visited DOE's Pantex Plant in Amarillo, Texas. The purpose of this visit was to learn more about the past and present operations at Pantex and perform a preliminary survey to determine the types and amount of explosives and toxic organics that are present in the soils. Soils at 25 sites were sampled to a maximum depth of 24 in. with a soil probe and the samples were divided into six-in. segments. Water from a playa previously used for lagooning waste water from explosives operations was also sampled. A total of 69 soil samples and 1 water sample were sent to IITRI's Newington, Virginia laboratories for analysis. Each sample was analyzed for the explosives RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine), HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazocine), PETN (pentaerythritoltetranitrate) and TATB (triaminotrinitrobenzene) by High Performance Liquid Chromatography (HPLC). Pooled samples from the same sites were analyzed for phenol, di-n-butyl phthalate, total chromium, hexavalent chromium, and cyanide.

2.2 Laboratory Scale Composting

2.2.1 Experimental Design

Two types of contaminated soil from Pantex were evaluated for cleanup by composting:

- * soil from waste water ditches in zones 11 and 12 contaminated with a mixture of up to four explosives - RDX, HMX, PETN and TATB
- * soil from a solvent dumping pit contaminated with large quantities of diacetone alcohol, smaller quantities of mesityl oxide and polymers thereof, and by products of the synthesis of TATB.

The laboratory studies were conducted using uncontaminated soil spiked with an appropriate combination of contaminants to simulate the contaminated soils at Pantex. To simulate soil in the ditches, soil was spiked with RDX, HMX, PETN, and TATB at concentrations that were approximately twice the highest levels found at Pantex, simulating a worse case condition. Separate composting trials were run where one of each of the four explosives in the test soil was labeled with ^{14}C tracer. The ^{14}C -label was utilized to identify degradates from the breakdown of the contaminants and to determine the mechanisms of degradation, if possible.

A second soil was spiked with a mixture of ^{14}C -labeled diacetone alcohol, ^{14}C -labeled mesityl oxide, and ^{14}C -labeled acetone to simulate the solvent pit. ^{14}C -mesityl oxide and ^{14}C -acetone were impurities in the synthesis of ^{14}C -diacetone alcohol. Both the diacetone alcohol and mesityl oxide are contaminants in the solvent pit soil at Pantex.

The study design was as follows:

Soil Spike:

A	RDX	220 $\mu\text{g/g}$
	HMX	220 $\mu\text{g/g}$
	PETN	400 $\mu\text{g/g}$
	TATB	2700 $\mu\text{g/g}$

B	Diacetone alcohol	6020 $\mu\text{g/g}$
	Mesityl oxide	70 $\mu\text{g/g}$
	Acetone	1100 $\mu\text{g/g}$

Soil Loading in Compost: 20, 30, and 40% (W/W)

Sampling Intervals: 0, 3, and 6 weeks. Because of the rapid breakdown of PETN, samples were taken at 0, 2, and 6 weeks.

Replicates: Three

All composts were incubated at 60-65°C (60-70°C for diacetone alcohol) with continuous aeration. Volatiles were scrubbed through H_2SO_4 and then passed through charcoal and NaOH solution to capture all ^{14}C -organic volatiles and $^{14}\text{CO}_2$.

2.2.2 Materials

Each compost consisted of 10.0 g (dry weight basis) Purina Omolene 200® horsefeed, 20 g of chopped alfalfa hay, and 21.2 g of horse manure (All materials were preanalyzed for moisture content). Treated soil was added to the composts in 12.4, 21.3, and 33.2 g aliquots, corresponding to loading levels of 20, 30, and 40% by weight.

The soil used for all studies was an uncontaminated soil from Pantex. This soil was collected from the B horizon in the barrow pit at Pantex and was similar to the contaminated soils in the ditches and solvent pit. The soil was air dried, crushed, and sieved (2 mm) before use.

^{14}C -labeled explosives and ^{14}C -labeled diacetone alcohol were synthesized by IITRI. Unlabeled explosives were obtained from Pantex.

The test system was designed to continuously incubate and aerate each compost and to trap ^{14}C -volatiles from each compost (Figure 1). Composting was conducted in 1-quart glass Mason jars closed with a rubber stopper. A ring of 1/4 in. O.D. polyethylene tubing with holes drilled at 1/4 in. intervals was positioned underneath the compost in each jar. This aeration ring was connected via vinyl tubing, through the rubber stopper, to the aeration system as shown in Figure 1. A thermocouple was also inserted through the rubber stopper into the compost materials. Air was pulled through the system under slight negative pressure. Warm air entering the system was first scrubbed through NaOH solution and then deionized water to remove CO_2 and humidify the air. The air passed through the aeration ring, moving up through the compost. Off-gases from each compost moved through a series of traps. Water condensate was collected in a dead trap, then the offgases passed through concentrated H_2SO_4 and Orbo[®] 32 tubes (Supelco, charcoal solid sorbent) to trap ^{14}C -organics. The offgases were then scrubbed through 2.0 N NaOH to trap $^{14}\text{CO}_2$. A dead trap was placed between the NaOH trap and the Orbo tube to prevent the NaOH from flowing into the Orbo tube if the system lost vacuum. The prescrubbers and the composts were placed in an incubator held at 60-65°C.

2.2.3 Methods

Twenty-seven subsamples of soil were individually treated with RDX, HMX, PETN, And TATB. Weights of the soil subsamples were 12.5, 21.4, and 33.3 g (dry weight basis), nine samples each. RDX, HMX, and PETN were added in separate acetone solutions to obtain concentrations of 220 $\mu\text{g/g}$, 220 $\mu\text{g/g}$, and 400 $\mu\text{g/g}$, respectively. TATB was weighed into each soil sample to yield a concentration of approximately 2700 $\mu\text{g/g}$. The RDX spike included uniformly ring labeled ^{14}C -RDX. The solvent was evaporated from the treated samples by allowing them to set in a hood for a few hours.

Three additional sets of treated samples were prepared, as above, one each to include ^{14}C -labeled HMX, PETN, and TATB. Characteristics of the radiolabeled materials were as follows:

Compound	Label Position	Purity	Specific Activity*
RDX	Uniformly ring-labeled	100.0%	2542 dpm/ μg
HMX	Uniformly ring-labeled	94.6%	682 dpm/ μg
PETN	Uniformly labeled	>99.0%	322 dpm/ μg
TATB	Uniformly ring-labeled	100.0%	1362 dpm/ μg

*(Disintegrations per minute)/microgram

The ^{14}C -labeled materials used to simulate the primary contaminants in the solvent pit soil were volatile and could have partially volatilized from air dried soil before it was mixed into the compost. Therefore, the soil was mixed into the compost and an acetone solution of the test substances (^{14}C -diacetone alcohol, ^{14}C -mesityl oxide, and ^{14}C -acetone) was added directly to the

Figure 1. Laboratory-Scale Composting Setup

compost to give an approximate concentration of 7200 $\mu\text{g/g}$. The radiochemical composition of the spiking solution was:

- * 75.2% ^{14}C -diacetone alcohol
- * 14.4% ^{14}C -acetone
(labeled on number 2 carbon)
- * 10.3% ^{14}C -mesityl oxide.

The overall specific activity of the mixture was 161 dpm/ μg .

The treated soils were mixed with the appropriate amounts of compost materials and placed in the Mason jars. Eighteen of the Mason jars for each material were placed in the test system. The remaining 9 jars (3 at each soil loading level) were analyzed as time zero samples.

The composts were monitored daily. The temperatures were recorded and the NaOH traps changed when near saturation. After three (two for ^{14}C -PETN composts) and six weeks of composting, 9 composts (3 at each soil loading level) were removed from the test system. All composts were extracted with appropriate solvents using sonication. Total ^{14}C in the extracts was quantified by liquid scintillation analysis (LSA). Total explosive and ^{14}C -explosive in the extracts were determined by HPLC. The extracted composts were dried and then combusted to release residual ^{14}C as $^{14}\text{CO}_2$. The $^{14}\text{CO}_2$ released by combustion was trapped and then quantified by LSA. Radioactivity in the charcoal traps was also determined by combustion and LSA analysis. Total ^{14}C in the NaOH and sulfuric acid traps was determined by direct LSA analysis. NaOH traps with significant amounts of ^{14}C were pooled by replicates over short time intervals, and the $^{14}\text{CO}_2$ was precipitated using BaCl_2 to determine the percentage of ^{14}C -activity associated with $^{14}\text{CO}_2$.

2.3 Pilot-Scale Composting

2.3.1 Experimental Design

The experimental design for the pilot-scale compost was as follows:

Size of Composters:	500 gal. (control - 400 gal.)	
Number of Replicates:	3 plus an uncontaminated control	
Explosives Concentrations in Soil:	RDX	220 $\mu\text{g/g}$
	HMX	220 $\mu\text{g/g}$
	PETN	400 $\mu\text{g/g}$
	TATB	1500 $\mu\text{g/g}$
Aeration:	Intermittent	

Temperature:	Monitored daily
Turning:	2-3 times per week
Duration:	6 weeks
Sampling:	Days 0, 12, 18, and 42

2.3.2 Materials

The composting materials (horse manure, hay, and horsefeed) were the same as in the laboratory studies. Because of the relatively large mass of soil required, a locally obtained uncontaminated silt loam was used. The explosives used were obtained from Pantex.

Pilot-scale composting was conducted using three 500-gal. dished-bottom stainless steel tanks for the contaminated composts (soil spiked with explosives) and one 400-gal. dished-bottom stainless steel tank for the control compost (no explosives in the soil). A diagram of the pilot composters is shown in Figure 2. These tanks were placed in a fenced enclosure next to a greenhouse. The four composters were enclosed in two 5-ft. X 10-ft. X 6-ft. open top framed boxes with lids, sheaved on all exterior surfaces with CELOTEX TUFF-R insulated sheaving. A small space heater was placed inside each insulated box to provide warm air (70°F) for aerating the composts (Figure 3).

A perforated platform in the bottom (above the dish) of each tank served to hold the compost off the bottom of the tank to allow for drainage of leachate and for air to be pulled through the compost (Figure 4). Air was intermittently pulled down through the composts using a radial blower. Offgas from the composts was scrubbed with a water scrubber to remove ammonia and odors before release to the atmosphere. The scrubber and blower were housed in the heated greenhouse to prevent the water in the recirculation system from freezing during the winter. The operation of the aeration system fan was controlled by a programmable timer. A valve in each aeration line was used to individually regulate the rate of air flow through each compost (Figure 5).

2.3.3 Methods

For each contaminated compost, 230 lb of a silt-loam soil were spiked with 19.5 g HMX, 19.5 g RDX, 35.4 g PETN and 132.7 g TATB. The soil and explosives were thoroughly mixed in a 1/3 cu. yd. cement mixer. Alfalfa hay was coarsely chopped and then soaked in water for at least one hour before use. The hay, horsefeed, horse manure, and soil were weighed in the proper proportions and mixed using the 1/3 cu. yd. cement mixer. The mixed materials were dumped from the cement mixer into a 50 gal. horse trough. Random grab samples were taken as the mixture was dumped. These grab samples were placed in a separate 20 gal. bucket. The trough was then taken to the composter and dumped into the tank. After each composter was filled, the grab samples were thoroughly mixed and samples taken for moisture and explosives

Figure 2. Schematic of Pilot-Scale Composter

Figure 3. Composter Enclosures

Figure 4. Aeration Board in Bottom of Compost Tank

analyses. Approximately 20 lb of grab sample from each tank were placed in a double trash bag and immediately frozen for future reference. The remaining sample was added to the composter.

After each composter was filled, three thermocouples were placed within the compost and the insulated lid was placed over the composter. When all four composters were filled, the air flow system was started.

Twenty batches consisting of 45 lb hay, 27 lb horse manure, 3.4 lb horsefeed (Purina Omolene 100), and 11.5 lb spiked soil (all on wet weight basis) were required to fill each composter (to within 2 in. of the top of the tank). Average compost wet weight was 1738 lb (553-589 lb dry weight). Initial explosives concentrations in the composts were calculated to be:

*	RDX	76 $\mu\text{g/g}$
*	HMX	76 $\mu\text{g/g}$
*	PETN	138 $\mu\text{g/g}$
*	TATB	513 $\mu\text{g/g}$.

The control compost was mixed in the same manner except no explosives were added to the soil. Thirteen batches were required to fill the control composter yielding a total wet weight of 1130 lb (350 lb dry weight).

The pilot-scale composters were monitored daily for appearance and smell. At least once daily, temperature readings from the three thermocouples in each tank were taken and the leachate was collected and measured. Composts were dug out and turned at least twice a week to mix the materials and adjust moisture levels (Figure 6). Random samples from each compost were taken during the mixing operations on days 4, 12, 18, and 42. On day 42, the composts were dug out and weighed to determine weight loss.

Samples taken from the composts were split for analysis. A portion was taken for moisture analysis. Another portion was taken for analysis of the explosives. Triplicate 75g aliquots of compost were extracted with acetonitrile using sonication. Extracts were cleaned by passing through an activated florosil column and then were analyzed by HPLC (UV detection) for RDX, HMX, and PETN. The remaining compost was air dried, pulverized in a mill and extracted with dimethyl sulfoxide (DMSO). The DMSO extracts were analyzed by HPLC for TATB.

At the end of 42 days of composting, the residual compost was mixed with soil at loading rates of 15, 25, and 35%. Rye grain and soybeans were planted in the mixtures and control soil to determine if the compost had any residual phytotoxic effects on the growth of the plants.

2.4 Data Reduction and Interpretation

^{14}C -activity was quantitated using a Packard 2200CA Tri-Carb Liquid Scintillation Analyzer (LSA) and associated software. Samples were counted for 10 minutes or until the 2 Sigma error reached 0.5. The LSA was programmed to automatically correct for background and

Figure 5. Compost Aeration System

Figure 6. Turning the Compost

quench and to report dpm (disintegrations/minute). Quench was corrected using Packard's Transformed Spectral Index (an external standard method) as a quench indicator.

All explosives and organics were quantitated using a Beckman 338 Binary Gradient High Performance Liquid Chromatograph and utilizing Beckman's System Gold software for data collection and peak integration. A set of standards (made from SARMs provided by the U.S. Army Toxic and Hazardous Materials Agency [USATHAMA]) were run at the beginning and end of each set of samples. A standard curve was constructed via linear regression. This curve was used to determine the concentration of materials in the samples. These values were further corrected for matrix and extraction effects using USATHAMA's detection limit program.

2.5 Quality Assurance

This work was performed in accordance with the quality assurance plan provided to ANL. Audits of work in progress and data reviews were performed throughout the study. The audits of the work in progress were performed to assure that all activities were in compliance with written protocols, Virginia Technology Center SOPs, and Good Laboratory Practice (GLP) standards. Physical audits were performed for all key operations with adequate frequency to assure proper conduct of the study. The data reviews were conducted in compliance with the quality objectives given in the quality assurance plan.

Written reports of the audit findings were provided to management. All quality assurance audit reports shall be retained in the locked archives of the Virginia Technology Center for five (5) years from the date of the final report.

3.0 RESULTS AND DISCUSSION

3.1 Preliminary Survey of Pantex Plant

The soils from the ditches and playas at Pantex contained highly variable amounts of explosives with the following concentration ranges:

- * RDX: non-detectable to 66 $\mu\text{g/g}$
- * HMX: non-detectable to 109 $\mu\text{g/g}$
- * PETN: non-detectable to 201 $\mu\text{g/g}$
- * TATB: non-detectable to 1349 $\mu\text{g/g}$.

The highest concentrations were found in the ditches from building 11-44 (explosives filtration) and 11-36 (explosives synthesis) in zone 11; and 12-43 (filtering operations) and 12-19 and 12-17 (originally melt and cast operations) in zone 12. The ditches from the buildings in zones 11 and 12 flow to playa 1. Small amounts of PETN and TATB were also found in the sediment near the

inlet of playa 1 from zone 12. No cyanide, phenol or n-butyl phthalate were found in the soil samples. Total and hexavalent chromium levels were similar to those found in soils surrounding the plant; thus, indicating natural occurrence rather than contamination from plant operations.

Soil from the solvent pit was contaminated with large quantities of diacetone alcohol and smaller amounts of mesityl oxide. Other contaminants found in low concentration included reaction by-products of diacetone alcohol and mesityl oxide, impurities from TATB synthesis, and explosives (TATB, PETN, and HMX).

3.2 Laboratory-Scale Composting

3.2.1 ^{14}C -RDX

Breakdown of RDX was rapid in all composts. Within 3 weeks, less than 5% of the total radioactivity could be extracted from the composts (See Table I). Concentrations of the parent ^{14}C -RDX were too low in the extracts to be detected by HPLC. No ^{14}C -degradates in the extracts were detected by HPLC. After 6 weeks, less than 3% of the ^{14}C -activity was extracted from any individual composts. ^{14}C levels in the 6 week extracts were too low to detect ^{14}C -RDX or any potential ^{14}C -degradates.

The pattern of $^{14}\text{CO}_2$ evolution from the composts also indicated very rapid degradation of ^{14}C -RDX (Figures 7-9). Although the rates of $^{14}\text{CO}_2$ release varied substantially among replicate composts, it was evident that release of $^{14}\text{CO}_2$ began almost immediately, with the greatest rate of evolution occurring during the first two weeks of composting. The rates of $^{14}\text{CO}_2$ release decreased until reaching a relatively constant rate at 3 weeks. This pattern of $^{14}\text{CO}_2$ evolution suggests that the breakdown of RDX largely occurs during the first 10-15 days of composting. A portion of the carbon from the triazine ring was released as $^{14}\text{CO}_2$ during RDX metabolism, and the remaining ^{14}C could not be extracted from the compost. As the ^{14}C -RDX was depleted, the rates of $^{14}\text{CO}_2$ release decreased. After 3 weeks of composting, all or essentially all, of the ^{14}C -RDX had been degraded. The relatively slow release of $^{14}\text{CO}_2$ after 3 weeks was primarily the result of secondary metabolism.

Increased levels of soil loading in the compost did not slow the rate of RDX breakdown. In the 40% soil loading composts, an average of 49.3% of the added radioactivity was recovered as $^{14}\text{CO}_2$ with 38.4% of the ^{14}C remaining in the compost after extraction. $^{14}\text{CO}_2$ accounted for 32.0 to 35.8% of the ^{14}C -activity in the 20 and 30% soil loading composts at 6 weeks. Approximately 48.3% of the ^{14}C remained in these composts as bound residues.

^{14}C -volatiles other than $^{14}\text{CO}_2$ were negligible. The mass balance was generally good, averaging 92.3% over the entire study. Recovery of ^{14}C -activity in several of the 20 and 30% soil loading samples was below 90%. This low recovery was related to the problem of the NaOH traps saturating. The rates of total CO_2 evolution from the 20 and 30% soil loading composts were exceptionally high, resulting in frequent saturation of the NaOH traps. It is probable that some $^{14}\text{CO}_2$ was lost when the traps saturated. Thus, the total $^{14}\text{CO}_2$ recoveries for the 20 and 30% soil treatments were probably an underestimate.

Table I. Summary of the ^{14}C -Distribution from the Composting of ^{14}C -RDX¹

Sampling Time (Weeks)	Soil Loading Levels (%)	^{14}C -Activity (%) ²						Total
		Charcoal Trap	$^{14}\text{CO}_2$	H_2SO_4 Trap	Condensate	Extract	Unextracted Residue	
0	20					93.44 (5.97) ³	5.15 (1.48)	98.59 (7.07)
	30	No volatiles data for time = 0				90.20 (2.87)	7.45 (0.88)	97.65 (2.70)
	40					96.12 (2.53)	7.63 (0.82)	103.75 (1.83)
3	20	0.13 (0.12)	28.12 (9.76)	2.42 (1.58)	0.54 (0.56)	4.77 (0.00)	56.14 (3.06)	92.12 (6.48)
	30	0.07 (0.06)	35.76 ⁴ (4.68)	2.61 (0.79)	0.49 (0.22)	2.86 (2.85)	43.43 (11.40)	85.22 (13.14)
	40	0.05 (0.04)	35.12 (7.40)	1.52 (0.61)	0.24 (0.11)	2.86 (1.90)	53.14 (7.22)	88.97 (2.11)
6	20	0.32 (0.18)	31.95 ⁴ (6.58)	2.29 (1.67)	0.36 (0.61)	1.27 (1.45)	48.12 (8.50)	84.31 (12.51)
	30	0.12 (0.08)	35.79 ⁴ (10.20)	2.00 (1.36)	0.55 (0.60)	1.59 (0.55)	48.45 (8.42)	88.50 (5.62)
	40 ⁵	0.16 (0.05)	49.29 (4.68)	1.06 (0.15)	0.23 (0.27)	2.86 (1.34)	38.41 (1.41)	92.01 (1.56)

¹ All values are an average of 3 replicates unless otherwise noted.² Percent based on the average total ^{14}C -activity recovered from all time = 0 samples.³ Values in parenthesis indicate the standard deviation of the mean above it.⁴ Several NaOH traps were saturated; probably lost ^{14}C as $^{14}\text{CO}_2$.⁵ Values are an average of 2 replications. Figure 7. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -RDX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 8. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -RDX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 9. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -RDX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

3.2.2 ^{14}C -HMX

Degradation of ^{14}C -HMX was similar to that of RDX. After 3 weeks of composting, a very substantial drop in the extractable ^{14}C was observed (See Table II). The extractable ^{14}C (expressed as percent of applied ^{14}C) ranged from 50.8% to 0.6% in individual replicates. The results were variable, but extractable ^{14}C was less than 7% in 2 of every 3 replicate samples. By 6 weeks, extractable ^{14}C -activity was $\leq 4.1\%$ in all replicate samples, with an average of 2.2%.

The pattern of $^{14}\text{CO}_2$ evolution from ^{14}C -HMX composts was largely as described for the ^{14}C -RDX compost, *i.e.*, initial rapid release of $^{14}\text{CO}_2$ lasting approximately 1 to 3 weeks, followed by a slowing to a relatively low constant rate of $^{14}\text{CO}_2$ evolution for the duration of the study (Figures 10-12). This pattern of $^{14}\text{CO}_2$ evolution in conjunction with a significant decrease in solvent extractable ^{14}C indicates that ^{14}C -HMX was largely degraded in the compost during the first three weeks. ^{14}C from the ^{14}C -HMX ring was released partially as $^{14}\text{CO}_2$, and was partially recovered as bound residue. The overall average ^{14}C -activity found as $^{14}\text{CO}_2$ and unextracted residue was 35.8% and 43.4%, respectively, after 3 weeks of composting. During the 4 to 6 week period, smaller amounts of ^{14}C -HMX were degraded, and some of the ^{14}C in the bound residue was metabolized to release $^{14}\text{CO}_2$. At 6 weeks, the average cumulative $^{14}\text{CO}_2$ release was 42.2%, and the average unextracted ^{14}C -residue was 47.2%.

The rate of ^{14}C -HMX breakdown was not noticeably influenced by the amount of contaminated soil added to the compost, nor did variation in soil loading level change the ratio of the unextracted ^{14}C -residue to $^{14}\text{CO}_2$. Neither the soil nor the contaminants in the soil appeared to slow the composting process.

$^{14}\text{CO}_2$ was the only significant volatile recovered. Only trace amounts ($<0.03\%$) of ^{14}C were found in the charcoal traps. Very small amounts of ^{14}C were found in the H_2SO_4 traps ($\leq 1.93\%$).

The overall average mass balance for the entire study was 94.8%, with values ranging from 89.2% to 100.8% for individual treatments. One sample (30% soil loading, 3 week sampling, replicate 1) had a mass balance of 54.2% and was excluded from the data analysis. Evidently this sample was not accurately treated with ^{14}C -HMX. NaOH traps for 20% soil loading saturated a number of times because of the very high rates of CO_2 evolution at the initiation of the study. Some $^{14}\text{CO}_2$ was lost when the traps saturated, thus resulting in a somewhat low mass balance for these samples.

3.2.3 ^{14}C -PETN

^{14}C -PETN was approximately 76% extractable at time zero (See Table III). Extractable ^{14}C accounted for an average of 27.4% of the total ^{14}C -activity after two weeks of composting, and decreased to 10.4% of the total ^{14}C after 6 weeks. HPLC analysis of the extracts revealed that PETN levels were below detection limits by week 2. During the initial HPLC analyses, no ^{14}C -degradates were detected in the post time = 0 samples. However, it was found that the major portion of the ^{14}C in these extracts was retained on the florisol column during extract cleanup. Therefore, the 2 week and 6 week sample extracts which had the highest ^{14}C concentrations were

Table II. Summary of the ^{14}C -Distribution from the Composting of ^{14}C -HMX¹

Sampling Time (Weeks)	Soil Loading Levels (%)	¹⁴ C-Activity (%) ²						Unextracted Residue	Total
		Charcoal Trap	¹⁴ CO ₂	H ₂ SO ₄ Trap	Condensate	Extract			
0	20					96.88 (1.55) ³	3.95 (1.12)	100.83 (2.51)	
	30	No volatiles data for time = 0					96.37 (3.32)	3.71 (2.71)	100.08 (4.17)
	40					96.13 (4.94)	3.01 (1.10)	99.14 (3.94)	
3	20	0.01 (0.01)	26.16 ⁴ (13.32)	1.64 (0.73)	0.32 (0.06)	18.44 (28.07)	42.96 (6.72)	89.53 (17.97)	
	30 ⁵	0.02 (0.01)	42.23 (5.96)	0.91 (1.24)	0.10 (0.06)	2.37 (0.64)	43.57 (1.20)	89.25 (4.09)	
	40	0.01 (0.00)	39.00 (9.15)	1.16 (0.84)	0.14 (0.12)	11.49 (9.94)	43.52 (7.46)	95.32 (8.51)	
6	20	0.03 (0.04)	38.03 ⁴ (12.68)	0.98 (0.47)	0.16 (0.11)	2.24 (1.61)	48.88 (20.23)	90.32 (8.60)	
	30	0.06 (0.05)	49.26 (4.13)	0.83 (0.59)	0.14 (0.04)	1.75 (0.29)	43.44 (8.91)	95.48 (5.46)	
	40	0.00 (0.00)	39.21 (6.30)	1.93 (1.02)	0.17 (0.14)	2.53 (1.10)	49.36 (11.52)	93.20 (9.10)	

¹ All values are an average of 3 replicates unless otherwise noted.² Percent based on the average total ^{14}C -activity recovered from all time = 0 samples.³ Values in parenthesis indicate the standard deviation of the mean above it.⁴ Several NaOH traps were saturated; probably lost ^{14}C as $^{14}\text{CO}_2$.⁵ Values are an average of 2 replications. Figure 10. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -HMX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 11. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -HMX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 12. Cumulative $^{14}\text{CO}_2$ evolved from the degradation of ^{14}C -HMX in compost. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

reanalyzed by HPLC, without undergoing the florisil column cleanup. A large early eluting (elution time of 2.9 - 3.0 minutes) radioactive peak was found in all samples (PETN elution time was 10.3-10.4 minutes). Essentially all of the radioactivity extracted in the 3-week composts was associated with this peak. After 6 weeks of composting, the $^{14}\text{C}\%$ in this peak was reduced by 70% (approximately 8.5% of the total ^{14}C -activity remained associated with this compound at 6 weeks). A shoulder on this peak indicated the presence of a second ^{14}C -degradate (elution time of 2.5 minutes) accounting for 1.9% of the total ^{14}C -activity.

The elution time of the ^{14}C -degradates suggests that they are polar compounds. They co-eluted with a large number of organics extracted from the composting materials. Extracts were analyzed by GC-MS, but no conclusive results were obtained. The GC-MS analysis did confirm that PETN and pentaerythritol were not present in the extracts.

A substantial amount of ^{14}C -activity was volatilized from the ^{14}C -PETN composts. Most of the ^{14}C -volatiles were trapped in the NaOH traps. Small amounts were found in the H_2SO_4 traps (average of 1.5% of the total ^{14}C at 6 weeks) and in the water that condensed out of the compost offgases (average of 1.2% at 6 weeks). No radioactivity was detected in the charcoal traps. The cumulative ^{14}C -activity in the NaOH traps accounted for an average of 23.0% and 43.8% of the total radioactivity after 2 and 6 weeks of composting, respectively.

Neutralization of the NaOH traps only released a portion of the ^{14}C -activity, indicating that $^{14}\text{CO}_2$ was not the only ^{14}C -degradate retained in the traps. The unknown ^{14}C -degradate(s) was too dilute in the NaOH traps to permit isolation and analysis. It was theorized to be one or more low molecular weight volatile acids, which could pass through H_2SO_4 and charcoal without being retained.

The rates and patterns of ^{14}C collection in the NaOH traps are illustrated in Figures 13-15. The pattern shows an average release of approximately 1.5% ^{14}C /day for the first 25-30 days. All treatments, and essentially all replicate samples have the same pattern of ^{14}C volatilization. The initial volatile losses were primarily as the unknown ^{14}C -degradates (68-99% of the ^{14}C in the NaOH traps). However, after 2 weeks of composting, the ratio of unknown ^{14}C -degradates to $^{14}\text{CO}_2$ decrease, *i.e.*, $^{14}\text{CO}_2$ constituted an average of 55% of the ^{14}C found in the NaOH traps, and 45% was the unknown ^{14}C -degradates.

Unextracted residues increased with ^{14}C -PETN degradation. Average values for unextracted ^{14}C were 20.4% at time zero, 37.6% after 2 weeks, and 42.1% after 6 weeks of composting.

The rate of soil loading did not influence any of the parameters measured, and did not noticeably alter the composting processing. The mass balance was good, with an overall average of 94.9%.

3.2.4. ^{14}C -TATB

Loss of ^{14}C -TATB from all composts was rapid. By the third week of composting, an average of only 15.8% of the added ^{14}C -activity was found in the solvent extracts (See Table IV).

Table III. Summary of the ^{14}C -Distribution from the Composting of ^{14}C -PETN¹

Sampling Time (Weeks)	Soil Loading Levels (%)	¹⁴ C-Activity (%)						
		Charcoal Trap	NaOH ¹ Trap	H ₂ SO ₄ Trap	Condensate	Extract	Unextracted Residue	Total
0	20					75.47 (4.18) ¹	13.76 (2.02)	89.23 (4.10)
	30	No volatiles data for time = 0				76.37 (2.89)	25.03 (4.45)	101.40 (2.75)
	40					76.13 (8.36)	22.42 (7.42)	98.55 (1.75)
2	20	0.34 (0.17)	19.46 (2.40)	0.46 (0.13)	0.47 (0.08)	37.13 (5.57)	40.37 (3.65)	98.23 (7.03)
	30	0.51 (0.31)	25.68 (2.53)	0.84 (0.69)	0.64 (0.34)	20.83 (2.46)	36.50 (1.11)	85.00 (4.94)
	40	0.02 (0.03)	23.73 (3.53)	0.31 (0.14)	0.50 (0.07)	24.20 (2.78)	35.95 (3.48)	84.71 (4.10)
6	20	0.02 (0.03)	44.22 (5.44)	2.97 (1.62)	1.63 (0.40)	17.76 (6.04)	36.69 (10.50)	103.29 (5.43)
	30	0.01 (0.00)	43.34 (6.53)	1.09 (0.02)	1.23 (0.08)	4.44 (4.35)	49.98 (10.79)	100.09 (3.34)
	40	0.00 (0.00)	43.81 (5.17)	0.50 (0.19)	0.64 (0.31)	9.02 (7.58)	39.57 (5.69)	93.54 (2.32)

¹ All values are an average of 3 replicates unless otherwise noted.² NaOH traps contained a mixture of $^{14}\text{CO}_2$ and other ^{14}C -volatiles.³ Values in parenthesis indicate the standard deviation of the mean above it. Figure 13. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -PETN. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 14. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -PETN. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 15. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -PETN. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

HPLC analysis of the extracts did not detect any ^{14}C -TATB. However, a significant quantity of unlabeled TATB was found (HPLC analysis) in both the 3 week and 6 week composts (See Table V). An average of 39.5% of the unlabeled TATB remained after 6 weeks of composting. The discrepancy between the labeled and unlabeled TATB may be related to the formulation of the TATB. The ^{14}C -TATB, synthesized by IITRI, was not coated. The unlabeled TATB, synthesized at Pantex, was not fully characterized, and may have been coated. If coated, degradation of the TATB may have been delayed until chemical and/or microbial activity in the compost had largely removed the coating.

The mechanism of ^{14}C -TATB loss from the compost is not clear. Release of the $^{14}\text{CO}_2$ was observed from all composts. However, the rate of $^{14}\text{CO}_2$ release was low throughout the 6 weeks of composting. After a short lag period of 5-10 days the rate of $^{14}\text{CO}_2$ remained relatively constant, showing no significant increases when ^{14}C -TATB was degraded or any significant decrease following the degradation of ^{14}C -TATB. Total cumulative $^{14}\text{CO}_2$ at 6 weeks accounted for an average of 6.1% of the total ^{14}C -activity added. Other volatile losses were negligible. Most of the ^{14}C -activity at the 3 and 6 week samplings was recovered as unextracted ^{14}C -residue. It is believed that the TATB is polymerized and combines with the humus material in the soil to form an unextractable matrix similar to that found for TNT (trinitrotoluene) (Kaplan and Kaplan, 1982).

The mass balance was somewhat variable because of the difficulty associated with adding individually weighed 15 mg aliquots of ^{14}C -TATB to each compost. However, the overall mass balance of 93.7% indicated that the radioactivity was accounted for and that the analytical procedures were adequate.

3.2.5. ^{14}C -Diacetone Alcohol

^{14}C -diacetone alcohol dissipated from the compost via degradation and volatilization. Degradation of ^{14}C -diacetone alcohol was evident from the relatively high release of $^{14}\text{CO}_2$ from several samples. The cumulative ^{14}C found in the NaOH traps is plotted against time in Figures 16-18. There is an initial rapid release of $^{14}\text{CO}_2$ during the first 7-10 days of composting in a few samples indicating mineralization of ^{14}C -diacetone alcohol (and perhaps mineralization of ^{14}C -mesityl oxide and ^{14}C -acetone). As much as 37.7% of the total ^{14}C -activity was recovered from the NaOH traps within 3 weeks of composting. However, very little $^{14}\text{CO}_2$ was evolved from most of the composts. Accumulation of ^{14}C -activity from the NaOH traps did not exceed 1% for most composts at 3 weeks. After the first 10 days of composting, the small quantities of radioactivity trapped contained unidentified volatile ^{14}C -material(s) as well as $^{14}\text{CO}_2$. The relative amounts of $^{14}\text{CO}_2$ and unidentified $^{14}\text{CO}_2$ and unidentified ^{14}C -compound(s) was variable.

Significant amounts of ^{14}C -volatiles were found in the other offgas traps. After 6 weeks, average ^{14}C recoveries in the H_2SO_4 traps ranged from 21.5% to 27.8% of the total applied ^{14}C -activity (Table VI). Average radioactivity accumulated in the water condensate ranged from 12.3 to 31.3% at 6 weeks. As much as 56% of the total ^{14}C -activity was recovered from the condensate of one compost (Replicate 1, 30% soil loading). HPLC analysis identified the ^{14}C in the condensates as ^{14}C -diacetone alcohol. Only small amounts of ^{14}C were found in the charcoal traps. However, combustion results were erratic and it was suspected that ^{14}C -volatiles in the charcoal

Table IV. Summary of the ^{14}C -Distribution from the Composting of ^{14}C -TATB¹

Sampling Time (Weeks)	Soil Loading Levels (%)	^{14}C -Activity (%)						Total
		Charcoal Trap	$^{14}\text{CO}_2$	H_2SO_4 Trap	Condensate	Extract	Unextracted Residue	
0	20					82.97 (7.65) ²	4.99 (0.21)	87.96 (7.77)
	30	No volatiles data for time = 0				85.60 (1.57)	4.21 (0.64)	89.81 (1.95)
	40					75.63 (5.06)	4.60 (0.27)	80.23 (4.83)
3	20 ³	0.00 (0.00)	2.12 (0.74)	0.00 (0.00)	0.03 (0.03)	9.95 (3.04)	82.22 (10.59)	94.32 (8.60)
	30	0.00 (0.00)	2.02 (1.02)	0.00 (0.00)	0.02 (0.01)	21.10 (7.56)	78.80 (0.68)	101.94 (5.98)
	40 ⁴	0.00 (0.00)	2.13 (0.00)	0.00 (0.00)	0.03 (0.00)	16.30 (0.00)	95.30 (0.00)	113.76 (0.00)
6	20	0.00 (0.00)	3.86 (1.04)	0.00 (0.00)	0.04 (0.04)	16.40 (0.62)	74.40 (5.25)	94.70 (4.71)
	30 ³	0.00 (0.00)	4.88 (2.17)	0.00 (0.00)	0.07 (0.03)	18.60 (4.24)	65.40 (4.33)	88.95 (6.38)
	40	0.00 (0.00)	9.65 (0.63)	0.00 (0.00)	0.11 (0.01)	12.47 (3.61)	69.76 (8.82)	91.99 (6.88)

¹ All values are an average of 3 replicates unless otherwise noted.² Values in parenthesis indicate the standard deviation of the mean above it.³ Average of 2 replications.⁴ A single replication.

Table V. TATB Recovered in 0, 3, and 6 Week Composts

Sampling Time (Weeks)	Soil Loading Levels (%)	TATB in Extract ¹			
		Concentration (ug)	Standard Deviation	% of Added TATB	Standard Deviation
0	20	22,944	1748.6	121.4	9.4
	30	38,327	1970.5	118.9	6.5
	40	50,838	4137.7	101.6	8.5
3	20	3,587	728.5	19.1	4.0
	30	18,430	5907.5	57.3	18.3
	40	29,984	7035.3	60.0	14.1
6	20	6,615	1336.6	35.3	7.1
	30	14,576	2127.3	45.3	6.8
	40	19,022	1620.5	38.0	3.3

¹ All values are an average of 3 replicates.

were flushed through the oxidizer before complete oxidation could take place. Combustion analysis was adjusted to encourage more complete oxidation; however, results remained erratic. The data reported may underestimate the ^{14}C -volatiles collected in the charcoal traps.

The extraction procedure for ^{14}C -diacetone alcohol composts had a relatively low efficiency. Recovery of ^{14}C -activity in the time zero samples averaged 64.4%. However, the data clearly show a significant decrease of ^{14}C -diacetone alcohol and its impurities, *i.e.*, ^{14}C -mesityl oxide and ^{14}C -acetone, in the extracts. After 3 weeks of composting less than 12% of the total ^{14}C was extracted. After 6 weeks, extractable ^{14}C decreased to an average of 1.6%. Results of the HPLC analysis of the 3 week extracts were given in Table VII. ^{14}C -diacetone alcohol dropped from an average of 50.2% at time zero to $\leq 12.0\%$ at 3 weeks and could not be detected in three of the 3 week composts. ^{14}C -acetone was not detected in any compost extracts at 3 weeks, and ^{14}C -mesityl oxide was detected in only two of the nine compost extracts analyzed at 3 weeks. No ^{14}C -degradates were found in the extracts. Radioactivity in the extracts of the 6 week composts was too low to permit HPLC analysis.

The mass balance was poor, 66.7% for time zero analysis, 63.4% average over the entire study. Drying and grinding the extracted compost before combustion was a probable source of ^{14}C loss, which accounted for the poor mass balance. Low combustion efficiency with incomplete oxidation of volatile ^{14}C -compounds may also have contributed to the low mass balance. Combustion analysis did not detect any appreciable quantity of unextracted ^{14}C -residue in any sample (<4.6% all samples, 2.8% overall average).

Table VI. Summary of the ^{14}C -Distribution from the Composting of ^{14}C -Diacetone Alcohol¹

Sampling Time (Weeks)	Soil Loading Levels (%)	¹⁴ C-Activity (%)						
		Charcoal Trap	NaOH ² Trap	H ₂ SO ₄ Trap	Condensate	Extract	Unextracted Residuc	Total
0	20					56.60 (5.48) ¹	3.23 (1.09)	59.83 (6.57)
	30	No volatiles data for time = 0				69.87 (3.84)	2.02 (0.62)	71.89 (3.44)
	40					66.43 (3.37)	1.73 (0.02)	68.36 (3.39)
3	20	0.05 (0.02)	0.14 (0.01)	23.39 (3.26)	12.47 (1.99)	10.30 (2.86)	3.00 (0.76)	49.35 (8.01)
	30	0.14 (0.07)	0.33 (0.23)	23.29 (2.95)	16.30 (5.31)	5.89 (2.10)	3.48 (0.34)	49.43 (5.88)
	40	0.06 (0.05)	5.20 (8.09)	24.24 (10.18)	18.77 (18.73)	8.63 (5.49)	3.48 (1.31)	60.38 (16.78)
6	20	0.32 (0.09)	11.98 (18.67)	27.83 (5.57)	22.95 (8.38)	1.50 (0.72)	2.80 (0.61)	67.38 (5.85)
	30	11.94 (8.35)	4.15 (4.44)	21.48 (9.38)	31.34 (22.78)	2.53 (1.86)	2.58 (1.24)	74.02 (21.44)
	40	0.12 (0.13)	31.78 (8.08)	22.08 (5.64)	12.34 (6.03)	0.77 (0.15)	2.43 (0.20)	69.52 (3.47)

¹ All values are an average of 3 replicates unless otherwise noted.

² NaOH traps contained a mixture of $^{14}\text{CO}_2$ and other ^{14}C -volatiles.

³ Values in parenthesis indicate the standard deviation of the mean above it.

Table VII. Summary of ^{14}C -Diacetone Alcohol Compost Extracts:
At Time = 0 and After Composting for 3 Weeks

Sampling Time (Weeks)	Soil Loading Levels (%)	^{14}C -Activity (%)			
		Replicate	Mesityl Oxide	Diacetone Alcohol	Acetone
0	20	1	5.7	51.2	1.8
		2	4.0	41.9	4.5
		3	3.4	57.4	ND
	30	1	— ¹	— ¹	— ¹
		2	4.2	58.0	4.2
		3	9.4	57.6	2.1
	40	1	10.1	51.6	3.9
		2	6.3	53.7	3.9
		3	5.7	57.9	6.8
3	20	1	ND	12.0	ND
		2	ND	11.9	ND
		3	— ²	— ²	— ²
	30	1	2.4	5.6	ND
		2	— ²	— ²	— ²
		3	ND	5.9	ND
	40	1	ND	11.7	ND
		2	0.3	11.6	ND
		3	— ²	— ²	— ²

¹ Sample lost.

² Inadequate ^{14}C -residue in extract for HPLC analysis.

Figure 16. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -Diacetone Alcohol. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 17. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -Diacetone Alcohol. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

Figure 18. Cumulative ^{14}C in NaOH traps evolved from the degradation of ^{14}C -Diacetone Alcohol. Data expressed as percent of total ^{14}C added. Curves are drawn point to point with no smoothing.

3.2.6 Discussion

All of the explosives in the soil from the ditches in zones 11 and 12 were readily degraded in the laboratory composts. The rates of breakdown varied among explosives, but were not significantly influenced by the soil loading level in the compost. Thus, at the concentrations used in this study, RDX (88.4 ppm in the compost), HMX (88.4 ppm), PETN (160.8 ppm) and TATB (1080.0 ppm) were not toxic or otherwise inhibitory to the composting process. The rates of explosive breakdown were in the order of $PETN > RDX \geq HMX > TATB$.

In the discussion of degradation kinetics it should be noted that composting in a small container is much more difficult to control than in a large mass, self-sustaining compost. As a result, the composts in a laboratory study tend to be variable, and sub-optimal conditions may prevail in a number of individual replicates. Despite the variability, the small laboratory composts are accurate in predicting the relative susceptibility of a compound to be degraded via composting. Rates of waste destruction tend to be slower in the laboratory composts relative to full-scale composts.

3.3 Pilot-Scale Composting

All of the pilot-scale composts were well composted by day 42 as evidenced by the large mass reductions (See Table VIII). The analytical results for the explosives are presented in Tables IX and X. The concentration of each explosive (in mg/g) in the compost at each sampling time is given in Table IX. The actual amount of explosives remaining and the percent remaining are presented in Table X. The data in this table present a more accurate picture since the reductions in mass of the composts were taken into account in calculating these numbers.

Breakdown of RDX, HMX and PETN was very rapid. Substantial losses occurred within the first 4 days of composting. RDX could not be detected by day 12, and HMX and PETN were not detectable within 18 days of composting. Degradation of TATB was slower. The average TATB loss in the first 12 days of composting was 39.8%. After 42 days of composting, 32-39% of the TATB was recovered. The first-order half-life for TATB degradation was 25.3 days ($r^2=0.889$).

The presence of explosives did not appear to inhibit the composting process. There was no substantial difference in the compost mass reduction between the explosives treated and control composts. However, the treated composts tended to have high moisture levels and were somewhat difficult to manage. The high moisture levels resulted in partial anaerobic conditions and required that the composts be mixed frequently. The anaerobic conditions and frequent mixing reduced the compost temperatures and probably the degradation rates. Maintenance of fully aerobic conditions and higher composting temperatures may result in more efficient degradation of the explosives.

Phytotoxicity tests indicated that the explosives-treated 42 day compost did not inhibit plant growth. The addition of compost, with control compost or explosives treated compost, increased the fertility of the soil and thus increased the yield of plant tissues for both the rye grain table VIII, IX X

Table VIII. Mass Reduction of Composts

Tank #	Initial Weight Kg	Final Weight Kg	% Mass Reduction
1 (treated)	250.8	141.1	43.7
2 (control)	158.8	90.7	42.9
3 (treated)	257.6	136.5	47.0
4 (treated)	267.2	145.6	45.5

Table IX. Concentration of HMX, RDX, PETN, and TATB in Pilot Scale Composts as a Function of Composting Time

Sampling Interval	Tank #	HMX µg/g	RDX µg/g	PETN µg/g	TATB µg/g
0 days	1	40.8	54.4	67.7	350.6
	3	65.6	88.3	86.0	421.1
	4	56.4	72.8	86.0	383.6
4 days	1	10.8	ND	ND	-
	3	50.6	37.9	<24.6	-
	4	18.3	<19.1	ND	-
12 days	1	<10.3	ND	ND	217.4
	3	<10.3	ND	<24.0	329.6
	4	<10.3	ND	ND	251.1
18 days	1	ND	ND	ND	-
	3	ND	ND	ND	-
	4	ND	ND	ND	-
42 days	1	-	-	-	197.0
	3	-	-	-	215.5
	4	-	-	-	263.9

ND = No peak detected in chromatogram

< = Below certified detection limit

- = Sample not analyzed

Table X. Explosives Remaining in Composts as a Function of Composting Time

Sampling Interval	Tank #	HMX		RDX		PETN		TATB	
		g	%	g	%	g	%	g	%
0	1	10.2	72.5	13.6	73.1	17.0	82.0	87.9	88.2
	3	16.9	120.1	22.7	122.0	22.2	107.1	108.5	108.9
	4	15.1	107.3	19.5	104.8	23.0	110.9	102.5	102.9
4	1	2.6	18.5	ND		ND		-	
	3	12.5	88.9	9.3	50.0	<5.9	<28.5	-	
	4	4.7	33.4	<4.9	<26.3	ND		-	
12	1	<2.3	<16.4	ND		ND		48.9	49.1
	3	<2.3	<16.4	ND		<5.9	<26.0	73.5	73.2
	4	<2.4	<17.1	ND		ND		58.4	58.2
18	1	ND		ND		ND		-	
	3	ND		ND		ND		-	
	4	ND		ND		ND		-	
42	1	-		-		-		27.8	27.9
	3	-		-		-		29.4	29.5
	4	-		-		-		38.4	38.6

¹ = Each explosive expressed as the total mass (g) remaining in the compost and the % of average mass found in the time = 0 samples.

ND = No peaks detected

< = Less than the certified detection limit

- = Sample not analyzed

• = % Remaining

and soybeans. The rate of compost addition (15-35%) did not substantially alter the plant tissue yield.

3.4 Demonstration Plant Design and Economics

A demonstration composting plant was designed to remediate 55 cu. yd. of explosives contaminated soil from Pantex ditches. The demonstration plant consists of four windrows, each 92 ft. in length and 14 ft. at the base. Windrow was chosen over static piles to get better mixing and aeration, and higher pile temperatures. A forced air windrow is used in this design to assure aerobic conditions. A cross section of the windrow presented in Figure 19 and the layout of the windrows is shown in Figure 20. The capital and operating costs (including obtaining the permit) for this demonstration plant are \$187,874 and \$408,740, respectively (see Tables XI and XII).

4.0 TECHNOLOGY STATUS

4.1 Technology Development Evaluation

This project has investigated the feasibility of remediation of Pantex soils by composting. The significant achievements of the project were:

- * The amenability of TATB and PETN to compost bioremediation was demonstrated.
- * The ability of the compost microorganisms to effectively degrade all the explosives in the Pantex soil in combination with each other was demonstrated.
- * The presence of the explosives at approximately twice the levels in the Pantex soils did not hinder the composting process at the highest soils loading levels.
- * Sufficient rate data were obtained to design a demonstration plant.
- * The resulting compost residue was non-toxic to plants and is useful as a soil amendment.
- * Initial cost projections for cleanup of Pantex soils indicate that costs for composting of the soils will be about 20% less than those for incineration.

Cleanup of soil should be ideally 99.99% or better. In reality, the achievement of this goal for soil is often not observable because the available analytical methodologies do not have the required sensitivity. Therefore, the best that we can hope to prove is that the hazardous material remaining is below the detection limits of available analytical methods. The composting process was shown to reduce HMX, RDX, and PETN to below detection limits in 18 days. Some TATB remained in the compost materials after 6 weeks of composting. However, it is believed that with

Figure 19. Cross Sectional View of Demonstration Compost Windrow

Figure 20. Demonstration Plant Layout

Table XI. List of Demonstration Plant Equipment

Equipment	No.	Unit Price	Total Price
Polyethylene Liner, 100 mil	4 rolls	\$4,162.50	\$16,550.00
SCARAB Composter	4 rolls	\$130,000.00	\$130,000.00
Blowers	4 each	\$594.33	\$2,377.32
Timers	4 each	\$90.00	\$360.00
PVC Elbow, 6 in, sch 80	8 each	\$16.22	\$129.76
Flanged Gate Valves, 6 in	4 each	\$625.50	\$2,502.00
Flange Gasket & Hardware 6 in	8 each	\$19.07	\$152.56
Tanks, 300 gal, PE	4 each	\$326.08	\$1,304.32
Flanges, 6 in	8 each	\$12.37	\$98.96
Railroad Ties, 6X6	720 each	\$12.00	\$8,640.00
PVC Pipe, 6 in, sch 80	520 feet	\$11.93	\$6,203.60
Metal Grading, 4X8 sheets	70 each	\$89.00	\$6,230.00
Freight for Liner	4 rolls	\$300.00	\$1,200.00
Installation of liner	22500 sf	\$0.25	\$5,625.00
Excavation of Trenches	4 each	\$100.00	\$400.00
Grading of Site	1 day	\$400.00	\$400.00
Installation of Pipe	10 mandays	\$200.00	\$2,000.00
Installation of Grating	6 mandays	\$200.00	\$1,200.00
Installation of Trench Support	10 mandays	\$200.00	\$2,000.00
Electrical Work	2 mandays	\$200.00	\$400.00
TOTAL CAPITAL COSTS			187,873.52

Table XII. Operating Cost for Demonstration Plant

Description	Quantity	Cost, \$
Labor for Permit	0.5 man year	\$120,000
Travel for Permit	4 trips	\$4,000
Power for Blowers		
Laborers	2 @ \$25,000	\$50,000
Nutrients/Bulking Agent	20 truckloads @ \$50/truck	\$1,000
Chips	2 truckloads @ \$200/truck	\$400
Diesel Fuel	100 gallons @1.40/gallon	\$140
Analytical Labor		
Chemist	.5 manyear	\$60,000
Technician	.5 manyear	\$37,500
Site Supervisor	.5 manyear	\$120,000
Travel	10 trips @ \$70/trip	\$5,700
Subsistence	100 days @100/day	\$10,000
TOTAL OPERATING COSTS		\$408,740

acclimation of the microorganisms, the TATB should be reducible to below detection limits in a reasonable composting time.

The other technology most applicable to remediate of soils contaminated with high energy explosives is incineration. Costs data available from actual incineration of Louisiana Army Ammunition Plant sediments show incineration costs were \$272/cu. yd. (in 1989) of sediment. Initial projections for composting Pantex soils are \$226/cu.yd., however, there is a trade-off between the two processes. Incineration can process the soils rapidly but leaves an unfertile amorphous soil residue that will have to be landfilled. Incineration also produces a certain amount of air pollution. Because of the shoddy manner in which many incinerators have been operated in the past, incineration also suffers from a lack of public confidence in its ability to remediate hazardous materials without additional harm to humans and the environment. In contrast, composting takes a significant time period to complete, but the residue is a very fertile soil which can be returned to the area or used elsewhere on an installation. Essentially, the choice between the two technologies must be made based on the hazards posed to humans and the environment by the contamination. If the explosives are entering the groundwater and faster remediation is needed, incineration is the method of choice. However, if the explosives are in a stable environment and pose no immediate threat to humans or the surrounding environment, then composting may be the method of choice.

4.2 Technology Integration Evaluation

Although composting is an old technique, its applicability to waste problems in the United States and in particular, hazardous waste problems, has only been recently realized. As such, regulations regarding composting and other bioremediation processes are only now being considered. Further complicating the regulatory issues is the lack of sensitivity of the analytical methods for hazardous materials in soils and compost materials. It is expected that more definitive regulations for bioremediation in general will be forthcoming as public disfavor with incineration increases.

The acceptability of a hazardous waste destruction process to the public is based on fears and not scientific data. Incineration is feared to give off toxic pollutants because the public has observed smelly air emissions from incinerators. In contrast, the public has often used composting in their back yard to reduce the volume of leaves, manure, etc. and to yield a good soil amendment. In general, the public perceives composting as an innocuous technique.

DOE's selection criteria for proceeding into the Demonstration, Test and Evaluation (DT&E) phase of the Technology Integration Program is "faster, better, safer, and cheaper while achieving and maintaining compliance" (Hain *et al.*, 1990). Composting meets all of these criteria except "faster" as discussed in the previous section. The DT&E activity is the transition phase of technology development which links a laboratory curiosity with the full-scale plant. It is designed to provide semi-full-scale operations to determine the applicability and economics of the process for full-scale operations. Composting of explosives contaminated soils is ready for the demonstration phase. It has the potential to meet all the technical, legal and economic requirements for full-scale implementation.

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