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**U.S. Army  
Environmental  
Center**

# Composting of Nitrocellulose Fines Regulatory and Logistical Feasibility RAAP Installation Position Paper

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13. ABSTRACT (Maximum 200 words) The production of nitrocellulose for munitions purposes results in the production of nitrocellulose fines (NC fines). RAAP currently produces approximately 1,250 lbs/day of NC fines (dry basis). Composting has been evaluated as a means of managing these fines and yielding a nonreactive beneficial soil amendment. This paper summarizes the logistical and regulatory feasibility of the following end-use options for the compost: (1) land application (with harvesting) by the installation; (2) providing local farmers with compost as a soil amendment; (3) land application of finished compost to reclaim land that mining operations have disturbed; and (4) disposal at a landfill. All options were found to be feasible with regard to regulatory constraints. Because finished NC compost is not specified in federal or Virginia State regulations for solid waste, the nonhazardous nature of the compost needs to be assured through demonstration of nonreactivity and/or chemical content determination. Based on predicted application rates, the anticipated 450 tons/year of compost would require approximately 320 acres/year of land. Based on preliminary site selection criteria, these land requirements appear to be achievable. Total costs per ton of compost (including disposal) range from \$1,005/ton to \$1,200/ton.				
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**CONTRACT NO. DACA31-1-D-0079**  
**TASK ORDER 0011**  
**DRAFT NC FINES COMPOSTING POSITION PAPER**  
**RADFORD ARMY AMMUNITION PLANT**

## **INTRODUCTION**

The production of nitrocellulose (NC) for munitions purposes also results in the formation of nitrocellulose fines (NC fines). The U.S. Army Environmental Center (USAEC) is evaluating methods to recover these NC fines or to recycle them into usable products. One technology, which the USAEC has considered for NC fines or residual NC fines in soil, is biological treatment via composting.

NC is a highly substituted cellulose fiber, which is synthesized from cellulosic materials, such as wood pulp or cotton, by nitration using nitric and sulfuric acids, followed by various additional processing steps.<sup>(1,2)</sup> NC is used by the Army as a propellant (alone or in combination with other constituents) in munitions and rocket motors.

The manufacture of NC results in the production of NC fines, which are difficult to recover during production due to their small size. These NC fines have historically been discharged with process water into lagoons. Fines that settled in the lagoons were periodically removed for recycle into product or storage.

While NC fines are not considered toxic by the U.S. Environmental Protection Agency (EPA)<sup>(3)</sup>, they may be reactive under certain conditions. The Army is investigating options to maximize both the recovery of the NC fines and the recycling of NC fines into usable products.<sup>(4)</sup> The USAEC is evaluating composting as a method for treating NC fines, which have not or cannot be effectively recovered or recycled into propellant products. Previous testing by the USAEC has shown that composting can treat NC fines in soil.<sup>(5)</sup>

Currently, NC fines are being generated from NC production operations at Radford Army Ammunition Plant (RAAP) in Radford, Virginia at an average rate of approximately 1,250 lb/day on a dry basis.<sup>(4)</sup> It is anticipated that these NC fines could be treated by composting to yield a nonreactive soil amendment suitable for beneficial use. Testing has shown that a compost matrix containing approximately 10 to 35% NC by weight, with a moisture content of 30%, may be handled safely.

Several methods of composting, derived from solid waste composting practice, may be applicable for NC Fines. Based upon USAEC's work to date, it is likely that NC fines would be composted on site, using the windrow composting method. NC fines would be mixed with organic amendments which serve to support the composting process. As with other types of explosives/propellant composting, the basic process would consist of the following principal steps:

1. NC fines receipt and storage.
2. Organic amendment receipt and preparation.
3. Windrow construction.
4. Windrow operation.
5. Windrow removal and disposition of finished compost.

The general windrow composting procedure to be used is depicted in Figure 1. Amendments would be layered in long, parallel rows ("windrows"). In this approach, NC fines would be transported to the composting pad from storage. The NC fines would be placed on the amendments and mixed with a windrow turner. The windrows would be periodically turned and monitored during the process. At the end of the required composting period, the finished compost would be removed from the pad for use as a beneficial soil amendment.

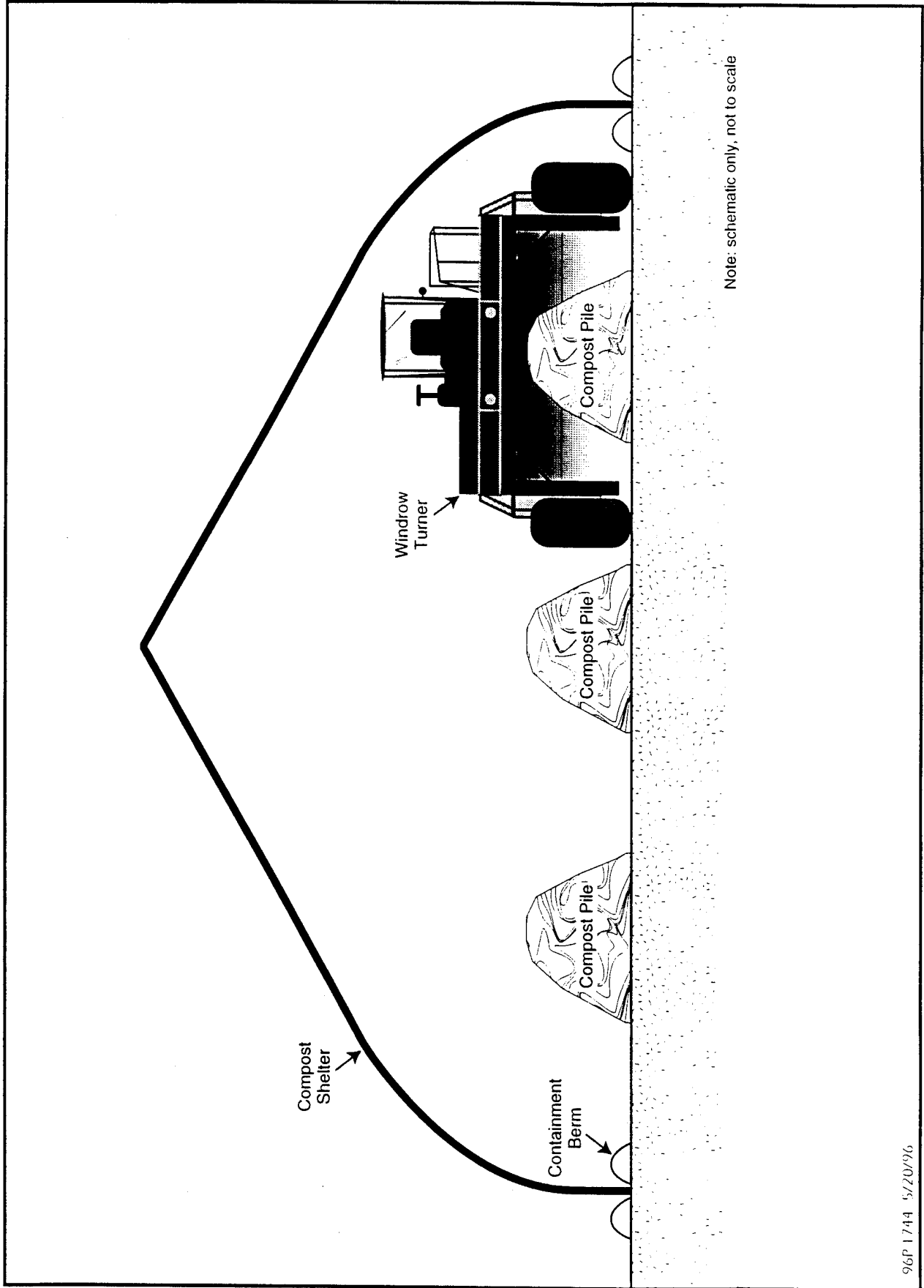
## **REGULATORY REQUIREMENTS FOR COMPOST USE**

### **Status of NC Fines**

A federal and state regulatory review was conducted to determine regulations and technical requirements that may affect management and disposal options for NC compost. Based on RAAP's location in southwestern Virginia, both Virginia and West Virginia regulations were considered. The use of NC fines compost as a soil amendment, or for similar uses, may require demonstration that NC fines are not RCRA Listed or Characteristic hazardous wastes.

The Army has successfully held that NC fines resulting from their production processes are not RCRA Listed wastes. NC fines do not exhibit the RCRA characteristics of ignitability, corrosivity, or toxicity. Although NC fines can be reactive under specific conditions, particularly when dry, the finished compost mixtures will not be reactive because NC fines will have been treated to levels determined to be nonreactive in tests conducted by Radford Army Ammunition Plant (RAAP) USAEC. In particular, final NC concentrations will be less than 10% NC. Results of the preliminary tests by RAAP for USAEC indicated that at concentrations less than 12% NC, NC fines compost was nonreactive at all moisture levels. Results of the RAAP Analysis were summarized in the "Composting of Nitrocellulose Fines-Hazards Analysis" report.<sup>(5)</sup>

It may be necessary to confirm that the finished NC compost is not hazardous and does not exhibit the RCRA Characteristic of reactivity prior to placement of the finished product on land. Reactivity tests such as the Bureau of Mines procedures used in the explosives industry may be used in demonstrating the presence or absence of reactivity. Federal regulations describing the properties of solid wastes that exhibit this characteristic are found in 40 CFR 261.23. Alternatively, it may be possible to use chemical analysis methods to confirm that the final NC levels are below the reactive level.



**FIGURE 1 CROSS-SECTIONAL SCHEMATIC OF WINDROW COMPOSTING**

Review and comparison of the state (VA, WV) and federal regulations indicates that their criteria for identifying hazardous wastes are equivalent. Therefore, a solid waste considered nonhazardous under the federal regulations would be considered the same under the Commonwealth of Virginia or West Virginia regulations. Since it is expected that the finished compost will be considered a nonhazardous solid waste under federal regulations, it is also expected to be considered the same under the state regulations.

#### **Evaluation of or Potential End-Use Options:**

Hazardous and solid waste regulations were examined with respect to specific applications of the finished compost. These applications may include:

- Land application (with crop harvesting) by the installation.
- Providing local farmers with compost as a soil amendment.
- Land application for reclaiming lands disturbed by mining.
- Landfilling the finished NC fines compost.

The following paragraphs contain a discussion of applicable or potentially applicable regulations for these end-use options.

#### **Land Application (by installation or by farmers):**

In the absence of other regulations concerning the land application of compost, the Virginia Sewerage regulations for land application of sludge were considered to be potentially applicable for land application of finished compost. Based on these regulations, it is anticipated that the finished compost materials must meet the following regulatory criteria to be acceptable for land application:

- The compost must be certified not to be a hazardous waste.
- The compost must meet allowable concentration limits for heavy metals.

These regulations specify that the operation of a land application facility cannot result in a hazard to public health, wildlife, water quality, or other environmental resource. In addition, the agricultural use of the finished NC compost can not result in harm to threatened or endangered species of plant, fish, or wildlife, nor result in the destruction or adverse modification of the critical habitat of a threatened or endangered species.

#### **Mining Reclamation:**

In addition to land disposal and land application, reclamation of surface mined land was also considered as a potential end use for the finished NC compost generated at RAAP. It was found that the neighboring state of West Virginia contained a number of surface mining sites that may require reclamation to restore vegetation to land disturbed by mining operations. Due to the number and proximity of potential reclamation sites to

RAAP in neighboring West Virginia, the state of West Virginia regulations were reviewed to determine the viability of this option.

According to the West Virginia Office of Surface Mining and Reclamation, before a solid waste is applied to surface mined land, it must be demonstrated that the waste is not hazardous. Also, it needs to be demonstrated that the use of the waste will be beneficial in the reclamation of the land. These requirements will apply to the finished NC compost. Finally, the existing Revegetation Plan for the site must be modified and approved, or a new plan submitted and approved prior to work.

#### Landfilling:

The Commonwealth of Virginia solid waste regulations state that sanitary landfills may receive compost so long as "the product can be handled, stored, and/or applied to the land without adversely affecting public health or the environment." Since it is expected that the finished NC compost can be considered in this category, placement in a sanitary landfill can be a viable disposal option in the Commonwealth of Virginia.

#### **TECHNICAL ISSUES TO BE ADDRESSED**

Based on this analysis of end-use options, it appears that composting is a technically feasible approach for management of NC fines once their potential reactivity has been reduced.

Prior to implementation of an NC fines composting process, there are four key technical issues that must be addressed. These issues are:

- Measurement of reactivity.
- Compostability of NC fines.
- Safety during treatment.
- Economics and reuse of NC fines compost.

These issues will be discussed individually.

#### **Measurement of Reactivity:**

As noted previously, NC fines do not pose an environmental concern due to toxicity, but only due to their potential reactivity. This suggests that treatment to destroy the reactive characteristics would render these materials environmentally safe. Therefore, the first key technical issue to be addressed involves measurement of NC fines reactivity. Due to the reactive nature of NC fines, particularly when dry, an accurate assessment of NC fines and NC fines compost is needed. As noted above, reactivity of NC fines compost could be evaluated either by direct reactivity testing or by NC analysis and correlation of residual NC levels to reactivity.

RAAP performed reactivity testing on NC fines compost mixtures for USAEC. <sup>(6)</sup> Testing was conducted using NC alone and in combination with three compost mixtures at various moisture levels. Reactivity to flame and shock stimuli were evaluated using standard test procedures to determine maximum NC levels and minimum moisture levels to control reactivity. Additional testing for sensitivity to impact, friction, and electrostatic discharge was conducted on selected formulations for use in evaluation of potential risks during actual composting operations.

The RAAP study concluded that NC compost mixtures exhibited similar reactivity under flame and shock stimuli at moisture levels up to 25%. Above this moisture level, NC compost required more moisture to avoid flame reactivity than shock reactivity. In other words, at a given moisture level, NC compost was more sensitive to flame stimulus than shock. NC alone (no compost) required 55% moisture to be nonreactive in the Deflagration to Detonation Transition (DDT) test.

The reactivity testing program results also indicated that dry NC compost mixtures did not react under the DDT test at less than 12% NC. Dry NC compost mixtures containing less than 10% NC did not propagate an induced explosive reaction in a <2.5-inch diameter, schedule 40 steel pipe in the Critical Diameter for Explosive Shock Propagation ( $C_d$ ) test. Based on these initial findings, it appears that NC fines composting can be accomplished under conditions such that the compost mixture is nonreactive.

Reactivity testing, such as the  $C_d$  test and the DDT test, would be a suitable way of confirming that final NC fines compost materials are nonreactive prior to their disposition as a soil amendment. Reactivity testing could also be used to confirm the safety of initial mixtures prior to composting. The precise tests to be used for these purposes have not been determined at this time. In addition, the cost for such testing if required on a frequent basis, may be of concern.

Another potential method of evaluating the reactivity of NC fines compost mixtures would be to correlate the measurable NC concentration in the compost to reactivity. After a correlation is established, only analytical results would be needed, thereby eliminating costly routine reactivity testing. The use of this approach requires an accurate, reliable, and cost effective method for analysis of NC in the compost matrix. As with most environmental analysis methods, analysis of NC in compost requires the quantitative extraction of NC from the matrix and accurate quantification of the NC in the extract, with minimal interferences from other components.

Previous NC composting studies utilized USATHAMA Method LY02, modified for the extraction and analysis of compost. Since this method analyzes for the nitrite ion, other extractable nitrogenous compounds naturally occurring in the amendment ingredients may affect the accuracy of this analysis.

The existing USATHAMA method uses an acetone extraction, followed by colorimetric measurement of a dye produced by reaction of the nitrite cleaved from the NC. While a

methanol washing step is used to remove other sources of nitrite and prevent interference, the possible presence of alcohol-insoluble nitrogenous compounds may still be of concern. NC mixed in soil or humic material may not be completely extracted with acetone. Therefore, the difficulties in acetone extraction in conjunction with the potential nitrogenous interferences make the analytical determination of NC content by this method uncertain.<sup>(25)</sup>

A preliminary literature search, identified several other classes of analytical methods which, with suitable development, may hold some promise for analysis of NC. Based on current information, the most promising techniques include Size Exclusion Chromatography/Gel Permeation Chromatography<sup>(7)</sup>, and Size Exclusion Chromatography followed by Electrochemical Detection<sup>(8)</sup>. Further development of such procedures may be warranted in order to provide a reliable method for analysis of NC in compost.

### **Compostability of NC Fines:**

The second key technical issue to be addressed is the compostability of NC fines. The evaluation of biological waste treatment must address the degree to which the target components can be either biodegraded to mineral products, or biotransformed to environmentally innocuous or acceptable products. In this regard, several sources have concluded that NC is resistant to direct microbial attack, even by cellulolytic organisms.<sup>(9,10)</sup> According to various reports<sup>(11,12,13,14,15,16)</sup> as cited in Wendt and Kaplan<sup>(9)</sup>, substituted celluloses are also generally resistant to microbial attack, with the degree of resistance increasing with the degree of substitution.

Duran et.al.<sup>(17)</sup> tested the anaerobic degradation of NC and found that, although some evidence of toxicity was observed, microorganisms derived from an anaerobic digester would degrade relatively high concentrations of NC (up to 54,000 mg/l) with appropriate acclimation. The presence of co-substrates, including cellulose, appeared to suppress the toxicity of NC. Other anaerobic degradation tests by Hsieh and Tai<sup>(18)</sup> used acclimated microorganisms. Various enzymatic inducing agents were evaluated for their ability to foster NC degradation. The authors concluded that the tested reagents were not effective in inducing NC degradation in the test system.<sup>(18)</sup>

However, microbial transformation of NC may be possible under certain conditions. Kaplan also reports that NC is not directly metabolized by microorganisms and suggests that other studies, in which growth of microorganisms on NC was reported, may represent cases where the observed growth was on other contaminants, on unsubstituted cellulose, or due to the effects of secondary metabolites on NC structure<sup>(16)</sup>. Wendt and Kaplan<sup>(9)</sup> also cite Urbanski's<sup>(19)</sup> conclusions that microorganisms growing on other substrates may produce metabolic products that adversely affect the stability of NC. Brodman and Devine<sup>(20)</sup> report that a fungus indirectly utilized nitrogen from NC (by a hydrolysis reaction) when supplied with a supplemental carbon source without attacking the cellulose (carbon) backbone. Recent studies conducted using three fungal strains concluded that

none of these organisms used NC as a source of carbon under the conditions tested.<sup>(21)</sup> This suggests that the carbon backbone was not attacked.

USAEC has previously conducted tests of composting of NC in soils, primarily as a potential remedial technology for sites with residual NC in soils from previous manufacturing activities.<sup>(22,24)</sup> The first USAEC study used both laboratory and pilot scale testing to evaluate whether NC in soils from Badger Army Ammunition Plant (BAAP) in Baraboo, Wisconsin, could be metabolized by composting.<sup>(24)</sup> In addition to residual NC soil trials, radiolabelled NC (uniformly labeled  $^{14}\text{C}$ -NC) was added to soil used in test composts (in addition to the NC contributed by the contaminated soil) in order to assess the fate of NC in the compost.<sup>(24)</sup> The evolution of  $^{14}\text{CO}_2$  from radiolabeled substrates is generally taken as evidence of mineralization; alternatively, the  $^{14}\text{C}$  tracer may allow analysis of the partitioning of the organic compound within the matrix. In contrast to previously cited sources, results of the BAAP testing indicated that, under simulated composting conditions, biodegradation of the cellulose backbone occurred. Subsequent pilot-scale testing at BAAP indicated that NC degradation well in excess of that attributable to thermal degradation was seen to occur.

Finally, a field-scale demonstration was conducted at BAAP to evaluate the potential utility of aerated static pile composting as a treatment and remediation technology for NC fines and residual NC fines in soil.<sup>(22)</sup> Results of this field demonstration indicated that composting is a feasible technology for reducing the extractable NC concentration in soils. In addition, this field demonstration provided evidence that NC at a high concentration can be degraded when incorporated into a compost mixture, using small amounts of compost spiked with concentrations of pure NC incubated in porous bags placed within some of the test piles.

Although mineralization (i.e., conversion of organic components to carbon dioxide and mineral products) is often the optimal treatment result, it may not be strictly necessary in all cases. Rather, treatment to render the material nonhazardous may be acceptable. As noted previously, NC is not toxic. Thus a process that transforms NC into nonreactive material may meet the primary treatment goal.

### **Safety During Treatment:**

The third technical issue that merits attention is safety during treatment. Due to the energetic nature of propellants, which can result in detonation under shock or thermal stimuli, safety criteria and procedures to avoid these stimuli are of critical importance in all materials handling aspects of NC treatment. Safety criteria must consider the levels of propellant that can safely be handled in the treatment process. NC is known to be a reactive material, particularly when dry. As previously stated, results of the RAAP Hazards Analysis indicated that dry NC compost mixtures did not exhibit reactivity at less than 12% NC. It was demonstrated that NC fines at loading rates between approximately 10 and 35% in a compost matrix, with a moisture content of 30%, may be handled safely. Therefore, it is anticipated that composting operations could be safely conducted if

starting compost mixtures were maintained within these parameters i.e., NC levels below 35%, with moisture maintained at or above 30%. Treatment would be continued until NC concentrations were less than 10%, at which point the material would be nonreactive regardless of moisture content.

In addition, an explosives hazards safety analysis would be conducted on the selected compost materials handling and operating equipment prior to their use. This analysis would consider the potential initiating forces which might be produced by the machinery, relative to the reactivity of the material. Only equipment deemed safe on the basis of such a review would be approved for use in the NC fines composting operation.

### **Economics and Reuse of NC Fines Compost:**

The last technical issue to be addressed involves the economics and reuse of NC fines compost. The economics of the composting process itself were discussed in "Composting of Nitrocellulose Fines- Hazards Analysis".<sup>(5)</sup> Potential end-use options for the compost were evaluated based on logistical and economic factors discussed in "Composting of Nitrocellulose Fines - Regulatory and Logistical Feasibility, RAAP Installation."<sup>(23)</sup>

Project costs for the NC fines composting facility were estimated using a NC fines throughput of 1,250 lb/day (on a dry basis) or equivalently 1,790 lb/day (on a wet basis), and a 35% NC fines loading at 30% moisture. The total 20-year project cost, was estimated to be \$6,532,000. This corresponds to a cost of \$1,000/ton of NC fines, or \$310/yd<sup>3</sup> of NC fines.<sup>(23)</sup>

It is anticipated that the nonreactive soil amendment product of the composting process would be suitable for beneficial reuse. The follow end-use options have been evaluated for RAAP:

- Land application (with crop harvesting by the installation).
- Providing local farmers finished with compost as a soil amendment.
- Land application of finished compost to reclaim land that mining operations have disturbed.
- Disposal of finished compost in a landfill.

All of the end-use options were found to be feasible with regard to regulatory constraints. To apply the anticipated 450 tons/year of finished compost, approximately 320 acres/year of land will be needed, based on predicted application rates. Table 1 shows approximate annual costs for each end-use option.<sup>(23)</sup>

**Table 1**

**Annual Cost Summary of End-Use Alternatives for NC Fines Compost**

<b>Alternative</b>	<b>Estimated Annual Cost (\$/year)</b>	<b>Estimated Cost per Ton of Finished Compost (\$/ton)<sup>a</sup></b>	<b>Estimated Cost per Ton of Original NC Fines (\$/ton)<sup>b</sup></b>
Land Application by the Installation	\$62,500	\$100	\$280
Supply to Local Farmers for Land Application	\$8,300	\$20	\$40
Compost to reclaim land disturbed by mining operations	\$33,100	\$60	\$150
Compost Landfilling	\$35,700	\$55	\$160

<sup>a</sup>Based on 640 tons/year of compost, wet basis.

<sup>b</sup>Based on 225 tons/year of NC fines, dry basis.

Based on these analyses, it is projected that composting could be implemented as a management method for NC fines at RAAP at a total cost ranging from \$1,005 to \$1,200/ton (dry basis), depending on the end use option. Total costs are presented in Table 2.

### **Recommendations:**

USAEC has previously tested composting of NC in soils, primarily as a potential remedial technology for sites with residual NC in soils from previous manufacturing activities. Results of these studies, in conjunction with findings from a preliminary literature review, indicate that NC fines may be treatable via composting. The composting process is anticipated to yield a nonreactive soil amendment suitable for beneficial use. For further development of composting as a treatment technology for NC fines, the following factors should be addressed:

- Determining the composting period and operating conditions necessary to achieve the desired NC destruction, and corresponding reactivity reduction.
- Evaluating the need for adding additional carbon sources (amendment supplementation) after temperatures have dropped in the windrow to prolong the composting period and achieve additional NC degradation, if needed.
- Maintaining environmental parameters (i.e., moisture, temperature, pH, and oxygen) such that transformation of NC is optimized and the potential for reactivity of NC fines is minimized.
- Determining safe materials handling procedures for initial NC fines/compost mixing and compost pile maintenance.
- Confirming the correlation between measurable NC levels and reactivity.
- Evaluating the current analytical method versus potential new analytical methods for NC fines compost.

Table 2

**Total Composting Cost  
(20 Year Project)**

<b>Alternative</b>	<b>Compost Process (\$/ton NC Fines)</b>	<b>Compost End Use (\$/ton NC Fines)</b>	<b>Total (\$/ton NC Fines)</b>
Land application by the installation	1,000	200	1,200
Supply to local farmers for land application	1,000	30	1,030
Compost to reclaim land disturbed by mining operations	1,000	105	1,005
Compost landfilling	1,000	115	1,115

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