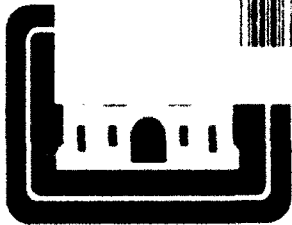


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US Army Corps of Engineers

Toxic and Hazardous
Materials Agency

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THE PREPARATION AND ANALYSIS OF SOIL COMPOST MATERIAL FOR INORGANIC AND EXPLOSIVE CONSTITUENTS

Prepared For:

U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, MD, 21010-5401

Prepared By:

U.S. Geological Survey
P.O. Box 25046
Denver, Colorado 80225



OCTOBER 1992

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Material for Inorganic and Explosive Constituents

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LIST OF ACRONYMS

Es	error in bulk sampling
Ep	error in physical preparation
Ed	error in analytical determination
Et	total error
g	gram
HMX	octahydro-1,3,5,7-tetranitro 1,3,5,7-tetrazocine
HPLC	high performance liquid chromatography
ICP-AES	inductively coupled argon plasma atomic emission spectroscopy
kg	kilogram
LAAP	Louisiana Army Ammunition Plant
mg	milligram
mm	millimeter
mL	milliliter
μm	micron
μL	microliter
NC	nitrocellulose
ppm	parts per million, mg/kg
RDX	hexahydro-1,3,5-trinitro-1,3,5 triazine
TNT	2,4,6, trinitrotoluene
UMDA	Umatilla Army Depot
USGS	United States Geological Survey
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
UV/VIS	ultra violet/visible
%	percent
%RSD	percent relative standard deviation
<	less than
>	greater than

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EXECUTIVE SUMMARY

A procedure is described for the physical preparation of explosives-contaminated soil compost for chemical analysis. The method involves macerating the air-dried compost mixture using a No. 4 Wiley mill followed by sample splitting using a traditional Jones-type riffle splitter. This procedure allows reliable homogenization and subsampling of compost material, thus improving overall analytical precision for replicate analyses. Comparison of previous results with the proposed method show a decrease in the percent relative standard deviation (%RSD) for replicate analyses from more than 200% to 3% for TNT analyses. Results using the proposed method are reported for selected major and trace elements, in addition to TNT.

INTRODUCTION

The treatment and rehabilitation of soils and sediments contaminated with a variety of organic and inorganic compounds is a priority at several military facilities within the United States. One group of compounds that is of highest concern is explosives left over from the production of munitions. The principle compounds include 2,4,6-trinitrotoluene (TNT), hexahydro-1,3,5-trinitro-1,3,5 triazine (RDX), octahydro-1,3,5,7-tetranitro

1,3,5,7-tetrazocine (HMX), and nitrocellulose (NC). The currently accepted treatment method is incineration of contaminated soil, but due to the high cost, more efficient methods are currently being examined.

A treatment method that has shown promise in early studies is the use of composting (Klausmeier et al., 1974; Kaplan and Kaplan, 1982; Williams et al., 1988, 1989, 1992). In these studies soil and or sediments contaminated with TNT, RDX, HMX, and NC were mixed with organic material such as straw, manure, alfalfa and wood products and allowed to react for several weeks under low-temperature (mesophilic) or high-temperature (thermophilic) conditions. The "average" concentrations of the explosives in the compost pile decreased dramatically by the end of the composting period. The thermophilic conditions proved the most effective, decreasing the concentrations of TNT, RDX, and HMX more than 90% (Williams et al.; 1988, 1992; Greist et al., 1990). A problem encountered during initial studies was the poor analytical reproducibility in explosive concentrations for samples collected during the initial stages of composting. An example of this variability is illustrated in figure 1, which traces the decomposition of TNT in thermophilic composting experiments conducted at the Louisiana Army Ammunition Plant (LAAP) near Shreveport, Louisiana. The wide variation in TNT concentrations is due to errors associated with compost pile sampling, sample

preparation, and laboratory analysis. The total variation as measured by the percent relative standard deviation (%RSD) for the first three data points increases from 7% to 68%. In a separate set of experiments, 10 replicate samples were prepared and analyzed for TNT, HMX, and RDX to investigate sample preparation of errors. The variation expressed as the %RSD ranged from 15% to 240% for the three compounds. This large variation in replicate data suggests that sample preparation was a key contributor to the total variation in the data and must be reduced if reliable evaluation of the composting process is to be made.

In any study involved with sampling of solid material, different sources of error exist. Several investigators have studied parameters involved with sampling and identified three general sources of error (Shaw, 1961; Wilson, 1964; Grant and Pelton, 1974; Ingamells, 1974; Kratochvil and Taylor, 1981). The first deals with sampling of the bulk material (E_s) and is governed by the number of samples obtained, the degree of sample inhomogeneity, and the size (mass or volume) of the samples collected. The second source of error is attributed to the physical preparation (E_p) of samples and is governed by the techniques used to reduce sample particle size and prepare subsamples for analysis. The third source of error is attributed to the variation in the property being determined (E_d) and is usually a combination of chemical sample preparation (for example, decomposition, extraction) and instrumental error. The three

sources of error combine as squares of their standard deviations (variance) to produce the total study error (E_t). Another measure of these errors, which will be used in this report, is percent relative standard deviation (%RSD).

In any laboratory, there are limitations on how much time and effort can be expended to obtain representative subsamples of material prior to chemical analysis. Because of these limitations, a number of methods can be used that attempt to fit subsampling requirements to the study's needs. The more common methods include 1) cone and quartering (Allman and Lawrence, 1972), 2) bottle shaking (Reeves and Brooks, 1978), 3) "spooning" (Carver, 1981), and 4) riffle splitting (Schumacher et. al., 1990). In the laboratory a key parameter in obtaining a representative split(s) is the ability to reduce sample particle size to an acceptable level before subsampling. In soil studies sample particle size is reduced usually by some form of grinding and, in the case of plant material, by some form of disaggregation such as maceration or blender grinding (Jackson et al. 1985).

In USGS geochemical studies subsampling designed to evaluate laboratory performance is generally preceded by a multistep grinding process, that ultimately reduces the particle size to less than 150 μm . Subsampling of this ground material followed by chemical analysis provides reliable information on method precision.

EQUIPMENT AND METHODS

Sample Collection

Testing of the proposed preparation process for inorganic constituents was performed by the USGS using an uncontaminated (no explosives) sample of fresh compost material. The sample contained 24% soil, 10% alfalfa, 25% horse manure/bedding, and 41% horse feed by weight. Testing the procedure on contaminated compost was performed by Roy F. Weston Inc., using soil samples obtained from the Umatilla Army Depot Activity (UMDA) site in north central Oregon. The contaminated soil mixture contained the same amendments as the uncontaminated soil mixture.

Sample Preparation

The experimental design used for the preparation of compost samples containing explosives is presented in figure 2. After drying, two samples of contaminated (CS1 and CS2) and uncontaminated compost material (US1 and US2) were obtained and alternately processed using the proposed procedure. Initially the samples were macerated using a model 4 Wiley mill equipped with a 2-mm screen. The Wiley mill, shown in figure 3 (adapted from Peacock, T., 1992) was equipped with four stainless steel knives bolted to a central hub that rotates in a counterclockwise

direction cutting the material against six stationary stainless steel knives bolted to the chamber's frame. The sample was continually processed (cut) until it passed the 2-mm screen into the receiving container. Sample processing times were on the order of 10-15 minutes for 250 g of material.

After processing, the sample was transferred to an 8-ounce container and tumbled mixed by hand for 5 minutes. The sample was then passed over a Jones-type riffle splitter similar to the one presented in figure 4 (reprinted with permission, Schumacker, B.A. et al. 1990). The splitting process continued until replicate samples of proper volume were obtained. The Jones-type riffle splitter consists of two sets of metal chutes that are offset from one another and designed to deliver processed material to receiving containers situated on either side of the unit. Part of the test was designed to verify that consecutive samples could be processed without significant cross-contamination using only a methanol rinse between samples followed by air drying.

Analysis of Explosives Constituents

TNT concentrations in compost samples were determined using the following procedure. A 10-g aliquot of prepared sample (<2 mm) was transferred to a 100-mL-wide-mouth glass jar equipped with a teflon-lined cap. Acetonitrile (40 mL) was added, the

container sealed and shaken for 1 minute. After shaking, the contents of the jar were allowed to settle for 15 minutes. At that time 2 mL of extractant were removed and filtered through a 0.2- μ m (micron) teflon filter into a 4-mL autosample vial and the vial sealed. At the time of analysis, 200 μ L of the filtered extract was diluted with 600 μ L of a 33% methanol/67% water solution and injected into a Perkin-Elmer Series 4, high performance liquid chromatograph (HPLC) equipped with a Perkin-Elmer ISS100 Auto-Injector and Micrometrics model 786 UV/VIS variable wavelength detector. Analyte separation was accomplished using a DuPont, Zorbax® HPLC column and a water/methanol/acetonitrile mobile phase. Analytical results were corrected to sample dry weight.

Analysis of Inorganic Constituents

Samples used for inorganic analysis were processed through the Wiley mill as described above and then split into three aliquots using the Jones splitter. The samples were identified as USGS-1, USGS-2, and USGS-3. Sample USGS-3 was further split, producing USGS-3a and USGS-3b. Samples were then ashed at 300°C overnight in a Mellen programmable ashing furnace. The samples were analyzed using U.S. Geological Survey standard methods of chemical analysis for geologic samples (Crock et al., 1983; Baedeker, 1987). In this procedure a sample of ashed material (0.100 g) was transferred to a 60-mL teflon container, spiked with 100 μ L of a 500- μ g/mL

(ppm) lutetium internal standard solution and decomposed at 110°C using a multi-acid digestion. The solution was taken to dryness and the residue redissolved with 1 mL of aqua regia. The final solution was brought to a mass of 10.0 g with 1% nitric acid. Samples were analyzed for 40 trace and major elements simultaneously using a Jarrell-Ash model 1160 ICAP-AES system. All inorganic results were corrected to dry weight using percent ash values.

RESULTS AND DISCUSSION

In two separate experiments compost material was processed using the proposed procedure and replicate samples prepared for TNT and inorganic analysis. The samples were analyzed for TNT content by the Roy F. Weston Inc. and inorganic constituents by the U.S. Geological Survey.

Organic Analysis

In these experiments two parameters were of interest. The first was to determine if the proposed processing scheme was capable of preparing replicate compost samples with an Ed+Ep value <10% RSD. The second was to examine the possibility of cross-contamination between samples during maceration (Wiley mill) or subsampling (Jones splitter) steps. The uncontaminated compost material (samples US1 and US2) did not contain detectable levels of

TNT, whereas the contaminated material (CS1 and CS2) contained approximately 3,000 ppm of TNT. Contaminated and uncontaminated samples were alternately processed through the Wiley mill (figure 2). Concentrations of TNT corrected to dry weight for each sample are presented in figure 2.

The information in figure 2 provides a measure of variation attributed to sampling (Es) and a combination of sample preparation and determination (Ep+Ed). Calculations using TNT concentrations in samples CS2a, CS2b, and CS2c yield an average value of $3,200 \pm 100$ ppm. This result corresponds to a combined Ep and Ed value of 3.1% RSD, which is a significant improvement over previous studies (Griest et. al., 1990) in which the %RSD for replicate TNT analyses was >200%. If one extrapolates the %RSD for sample CS2 to data for sample CS1 a standard deviation of ± 81 ppm is obtained. Using this approximation, it appears that the TNT concentrations in CS1 and CS2 are statistically different at the 95% confidence interval. A more rigorous evaluation of these assumptions awaits further analytical data. Results in figure 2 also indicate that cross-contamination using this procedure is not detectable in either the Wiley mill operation or the sample splitting stage.

Following this method validation study, the procedure was used to prepared 240 samples from the composting optimization field study conducted at UMDA by Roy F. Weston, Inc. (Williams and Marks, 1991). The method substantially reduced sample variation from that

observed in previous field demonstrations. Improved precision in the determination of explosive concentrations will allow scientists to more accurately evaluate changes in the composting process and their effects on the decomposition of soil explosives.

Inorganic Analysis

In these studies a sample of uncontaminated compost material was processed through the Wiley mill and split into three samples (USGS-1, USGS-2, USGS-3) using the Jones splitter. Sample three was further split (USGS-3a and USGS-3b) to examine the effect of additional sample splitting on analytical variation. Statistical results for total element concentrations in samples are presented in table 1, for selected elements.

Results in table 1 show that between sample and within sample variation (%RSD) for all the elements was less than 10%. The %RSD values for the within sample study (USGS-3a and USGS-3b) were lower for most elements except lead. The variation observed for between and within sample data reflects the sum of E_p and E_d . Inorganic analyses mimic those reported earlier for TNT; this suggests that the proposed sample preparation procedures would meet the goal of reducing sample preparation errors to 10% or less. Smaller $E_p + E_d$ value will allow investigators to more accurately monitor the decomposition of explosives during composting process, even in the initial treatment stage.

Table 1 Comparison of between and within sample variation for inorganic constituents in uncontaminated compost material

Element, unit of conc.	Between <u>sample variation</u>		Within <u>sample variation</u>	
	Avg. conc.	%RSD	Avg. conc.	%RSD
Al, %	2.02	5.4	1.92	1.2
Ba, ppm	216	4.2	207	0.9
Ca, %	1.28	4.0	1.23	0.8
Ce, ppm	15	8.0	14	2.6
Cr, ppm	15	8.5	14	0.3
Cu, ppm	12	4.1	12	1.4
Mn, ppm	312	2.9	303	0.7
Na, %	0.82	3.8	0.79	1.2
Pb, ppm	5	4.4	4	5.7
Sr, ppm	116	5.4	109	1.3
Zn, ppm	54	1.8	53	0.5

%RSD, percent relative standard deviation;
ppm, parts per million; (mg/kg) dry weight
%, percent dry weight

SUMMARY

Use of composting to decompose explosives such as TNT, HMX, and RDX in contaminated soils and sediments is an effective remediation procedure. In previous studies, accurate evaluation of the decomposition rate for explosives was hampered due to inhomogeneity of the compost material and large variations attributed to the physical methods of sample preparation. In an attempt to improve sample preparation precision, a procedure was developed that utilizes a Thomas Wiley mill and Jones-type riffle splitter to prepare replicate samples for analysis. Analysis of explosive-contaminated compost yielded an average TNT concentration of $3,200 \pm 100$ ppm. The 3 %RSD observed in this study represents a significant improvement over previous studies in which the %RSD value for replicate TNT analyses was 240%. Using the proposed procedure no detectable cross-contamination of TNT was observed between contaminated and uncontaminated samples. Analysis of inorganic constituents in replicate compost samples reveals a between sample variation of 3% to 6% RSD depending on element.

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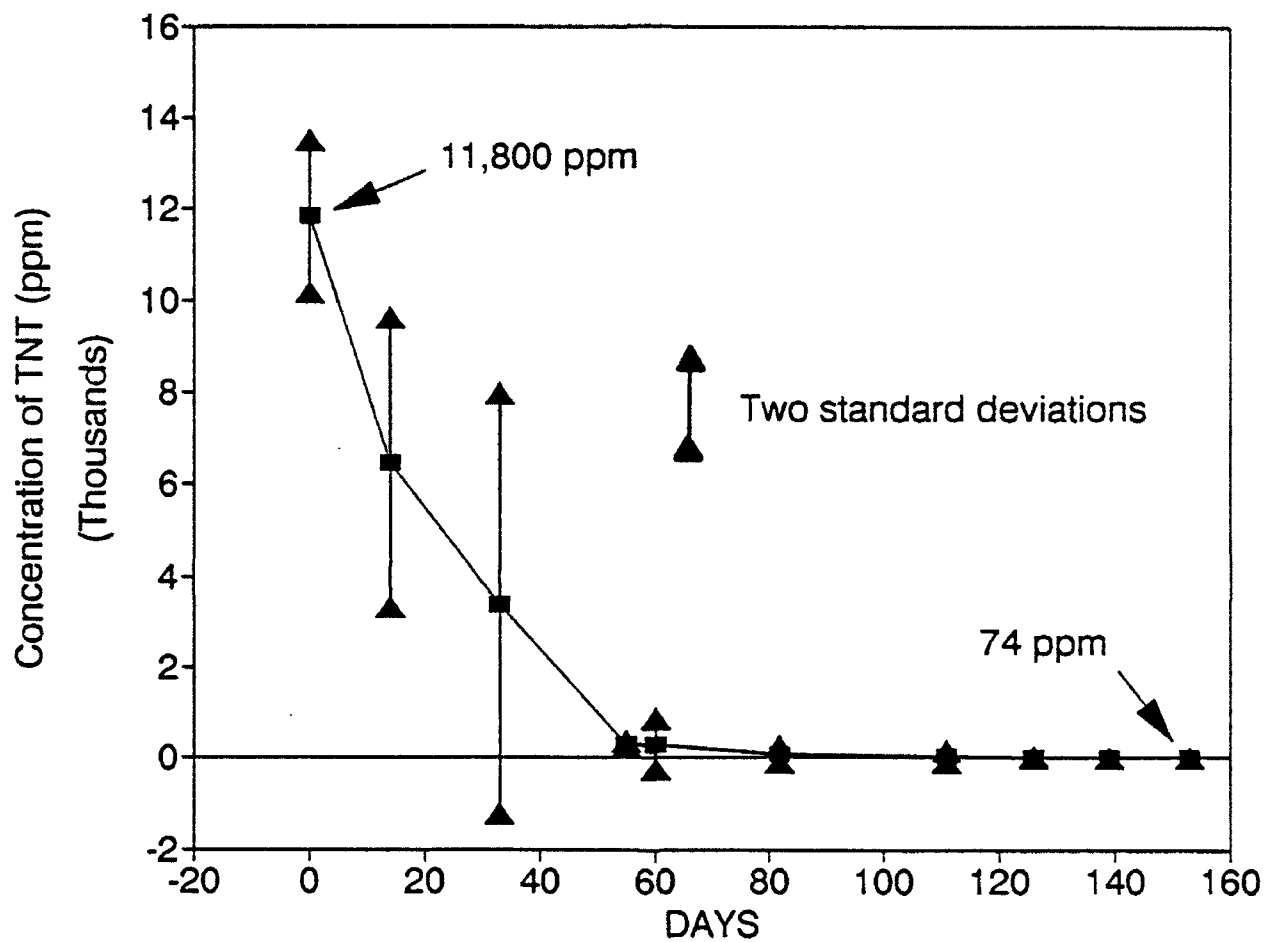
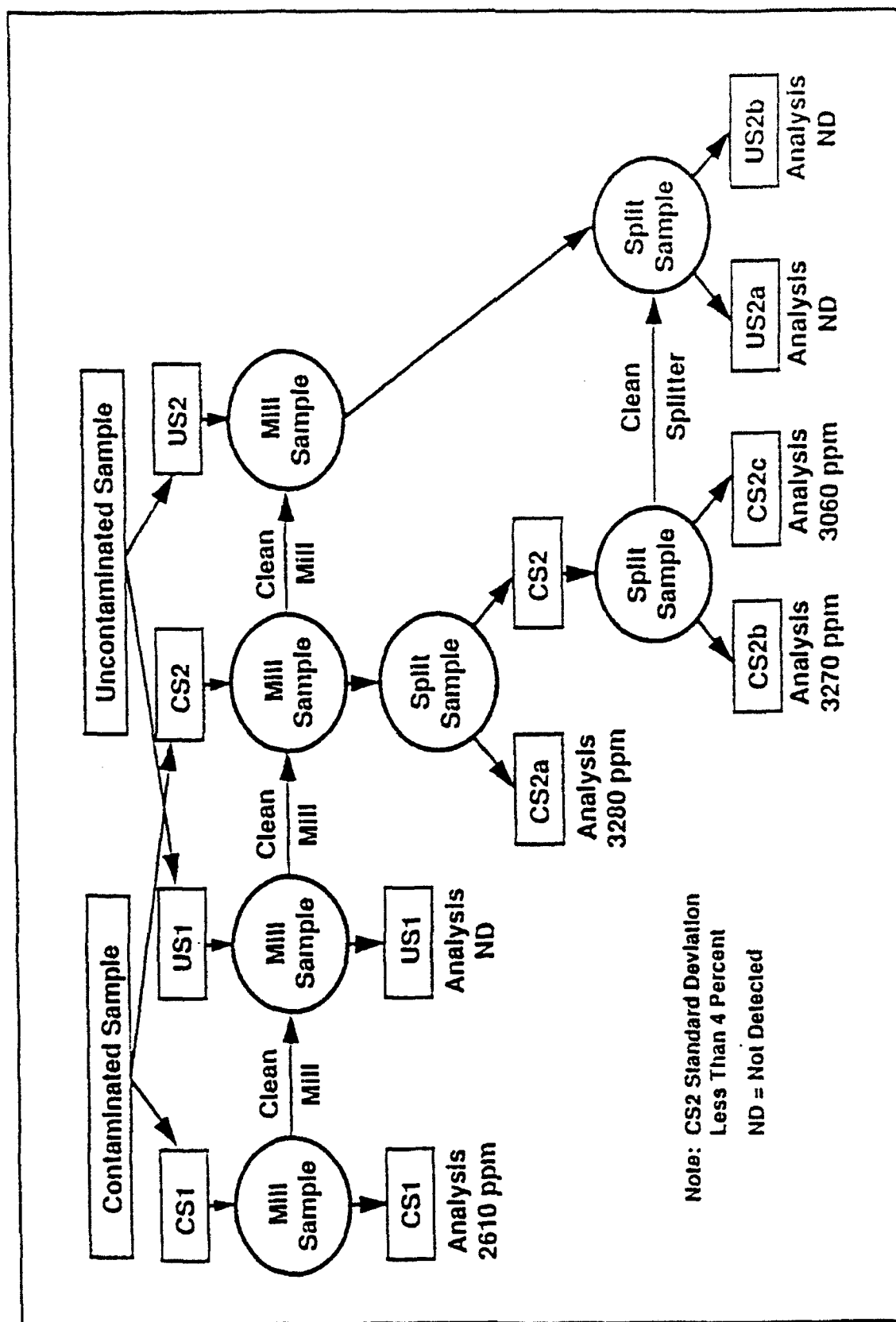


Figure 1 Decomposition of TNT during thermophilic composting



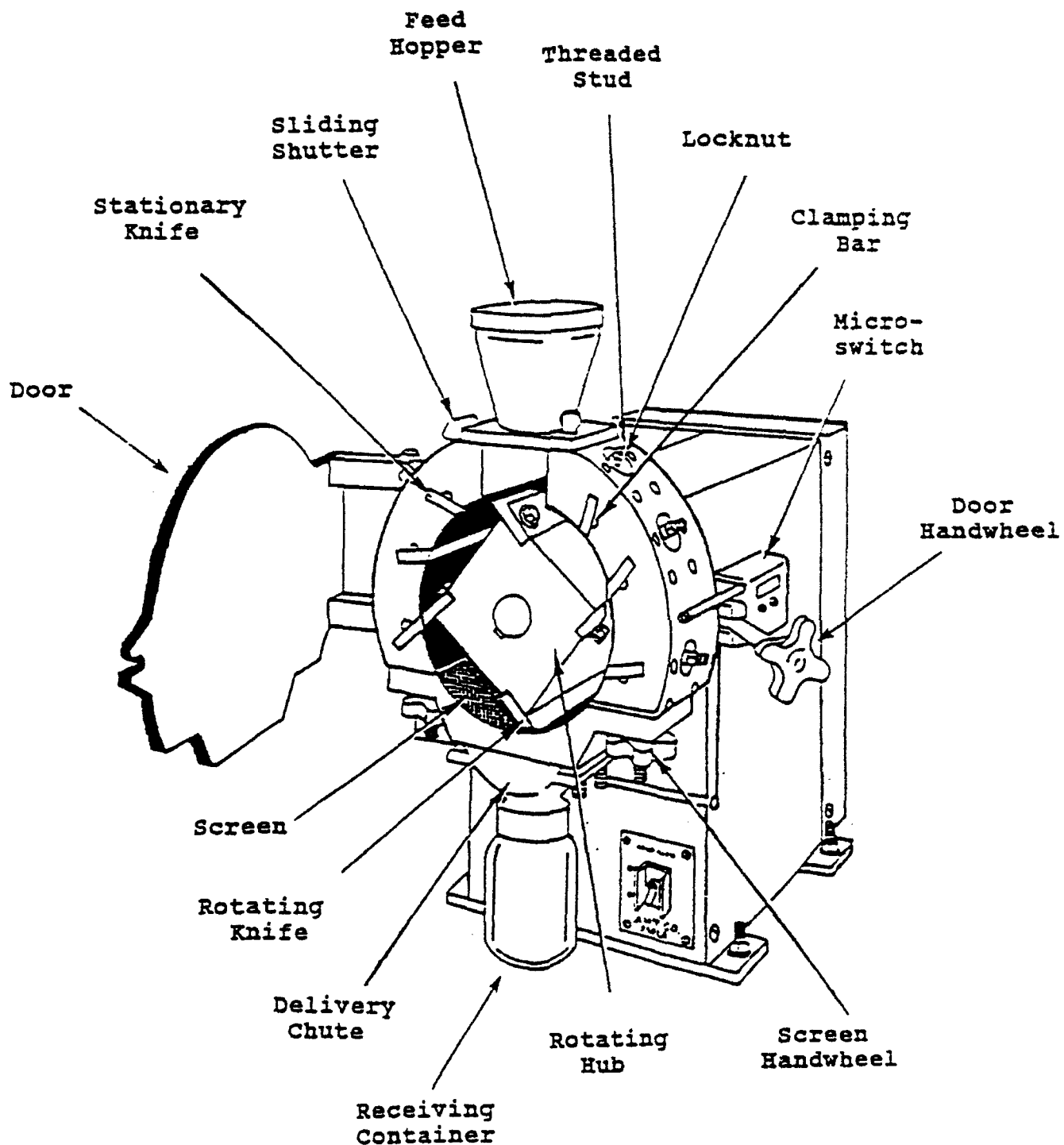


Figure 3 A Model 4 Wiley mill

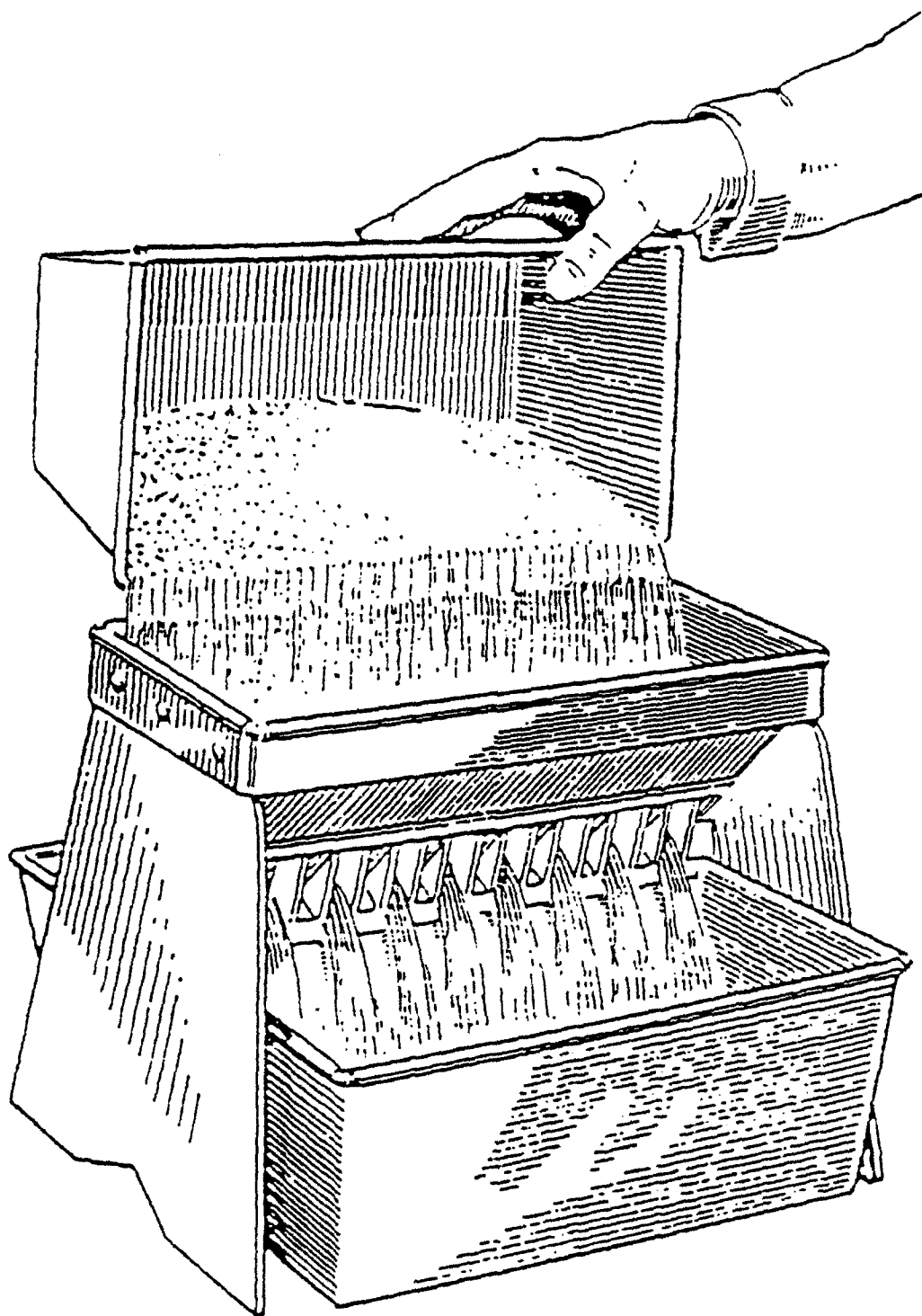


Figure 4 A Jones-type riffle splitter

HERCULES INCORPORATED
MISSILES, ORDNANCE & SPACE GROUP
ALLEGANY BALLISTICS LABORATORY
ROCKET CENTER, WEST VIRGINIA

HAZARD REVIEW OF WILEY MILL

MAY 1990

T. A. GROCE

PREPARED FOR
U. S. ARMY TOXIC and HAZARDOUS MATERIAL AGENCY
ABERDEEN PROVING GROUND, MARYLAND

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CAUTION

CONCLUSIONS PRESENTED IN THIS HAZARD ANALYSIS REPORT ARE BASED UPON THE HARDWARE (OR DESIGN), MATERIAL OF CONSTRUCTION, OPERATING CONDITIONS, PROCESS MATERIALS AND PROCEDURES AS THEY EXISTED AT THE TIME OF THE ANALYSIS (OR AS THEY WERE PRESENTED TO HERCULES FOR ANALYSIS). IF CHANGES IN ANY OF THESE PARAMETERS OCCUR IN THE FUTURE, THE CONCLUSIONS OF THE CURRENT HAZARD ANALYSIS MAY BE INVALIDATED.

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SUMMARY

OBJECTIVE

The objective of this work is to review the use of a Wiley Mill for use in the homogenization of a explosive contaminated soil sample. This review will assess the potential fire and explosion hazards associated with the operation of the Wiley Mill for this service.

CONCLUSIONS AND RECOMMENDATIONS

It is concluded that the operation of a Wiley Mill in grinding explosive contaminated soil is not an explosive operation if the sample contains less than 4% explosive. Although the sample containing less than 4% explosive is non-explosive, it is not necessarily non-flammable and precautions must be taken to protect the operator and lessen the effects should a fire occur. Recommendations are made in this section and discussed in this report that should allow this operation to be conducted safely and reduce the risk and potential effects of an incident from the use of a Wiley Mill with explosive contaminated soil.

The following recommendations are made:

1. The most important recommendation is to assure that sample contains the "less than 4% explosive" that USATHAMA expects. The use of a sample with a greater percentage explosive or the inclusion of primary explosives may invalidate this hazard review.
2. Protect the operator from possible fire by conducting the grinding within a ventilated laboratory hood with the shield lowered during operation and by using safety goggles or glasses with side shields and fire retardant gloves and arm protection. If the mill must be operated with the shield raised, the operator should have the added protection of a face shield and fire retardant suit/coveralls/lab coat.
3. Clean up sample spills as soon as possible.
4. Use dust rated electrical equipment if available. If not available, prevent build-up of sample on electrical equipment, because a fire could result from heat or spark.
5. Avoid generating a dust cloud with a dusty sample.

6. Do not allow the mill to continue to run with material within the mill and no output. Establish a reasonable time to accomplish the grind and shut down if this time is exceeded so as to minimize overheating in the mill.
7. If the mill feed clogs, use a non-metallic tool to assist feed. Do not allow the tool to enter the grinding chamber.
8. Operators should be trained in the importance in following proper operating procedures and the importance of using the proper personnel protective equipment provided.
9. Electrically ground the mill, auxiliary equipment, and sample preparation surfaces.

INTRODUCTION

BACKGROUND

The Army is currently investigating the use of composting for the remediation of explosive contaminated soils as an alternative to incineration. Hercules Incorporated recently completed a hazard review of a pilot composting reactor which bacteriologically treats explosive contaminated soil. As part of the overall study, samples are tested for explosives and byproducts and it is necessary that these samples be homogenized prior to being tested. A process for homogenization has been developed by the U. S. Geological Survey Geochemistry Section which utilizes a Wiley Mill for size reduction of the sample material.

SCOPE and LIMITATIONS OF THE REVIEW

The U. S. Army Corps of Engineers has contracted with Hercules/Allegany Ballistics Laboratory (ABL) through Purchase Order No. DACA31-90-P-1114 to conduct a hazard review of Wiley Mill for use in homogenizing samples from a study at Umatilla Depot Activity (UMDA). These samples are explosives contaminated soil compost samples. The scope of the review is limited to the Wiley Mill equipment that is planned to be used in homogenization process and excludes other areas of the process, such as subsequent handling of the sample material, and other equipment that might be used in the process, such as a Jones Splitter for separation of the sample. No specific safety/hazard analysis regulations, such as MIL STD 882 or ARROOM R 385-4, have been invoked by the purchase order.

DISCUSSION

DESCRIPTION OF WILEY MILL

The Wiley Mill is a grinding device utilizing rotating cutting blades against removable cutting bars (not in contact) through a screen to achieve particle size reduction. The mill is widely used and is available in a number of sizes. The mill at U.S.G.S. is a "Model 4" and is composed of the following components¹:

- a. Loading funnel - 3" diameter by 5" deep²
- b. Grind chamber - 8" diameter by 3" deep
- c. Cutting blades - 3" long
- d. Cutting bars - 3" long
- e. Screen - 2 mm openings
- f. Collection hopper - 4" diameter by 6" deep

The overall dimensions of the mill are 15" wide by 23" deep by 23" high and the components are steel. The cutting blades are attached to a wheel that turns at 800 revolutions per minute. An 1/2 HP motor (not explosion proof) integral to the mill drives the cutter wheel.

ABL has several Wiley Mills in use in various laboratories to reduce particle size of propellant and explosive samples. The mills are each smaller than the U.S.G.S. mill, roughly 1/4 to 1/2 size. Since 100 % explosive samples are processed at ABL, special precautions are taken such as grinding the sample with dry ice and grinding remotely or with shielding and operator protection.

DESCRIPTION OF SAMPLE

The following description¹ is of the sample that has been processed by U.S.G.S. and it is anticipated that future samples would be similar.

The sample (~1 gallon) contained 24% soil, 10% alfalfa, 41% horse feed, and 25% horse stable waste. The sample was moist when received and the first step was to dry the material in plastic trays (approximately 4" x 6" x 3") in a ventilated oven using room temperature air. The sample was dried overnight until visually dry and dry by feel. The material was hand processed to break-up clods then the sample was poured into the loading funnel of the mill (approximately 3/4 pint). The mill is located within a ventilated lab hood and the mill is operated with the operator in attendance. Occasionally the material would clog and the operator would have to

¹ Information from Steve Wilson, U.S.G.S. Geochemistry Section

² All dimensions are approximate to give a rough idea of the size

push the material using a wooden dowel. After the material passes through the screen and is collected, it is removed and the sample is split using a Jones Splitter.

The operator wore rubber gloves while manually sorting the material; however after sorting the only personnel protection was safety glasses. After the grinding operation, the mill was cleaned by disassembly, blowing with air, then washing with acetone. No unusual occurrences were observed during the milling operation.

HAZARD REVIEW

The use of a Wiley Mill in the homogenization of composted explosive soil has been reviewed for possible hazards. Only the unique hazards that are associated with the use of explosive contaminated soils in the Wiley Mill are addressed in this review; thus routine safety problems involving operation of this type of equipment (e.g. operator injury by the mechanical action of the mill, electric shock, improper operation) or the use of flammable solvents (i.e. acetone for cleaning) will not be discussed. It is felt that a laboratory, such as U.S.G.S. has the experience to deal with these kinds of problems. Table III identifies several scenarios by which the explosive sample may be initiated and represent potential hazards with the Wiley Mill.

By the nature of the design and construction of the Wiley Mill, there are initiation sources readily available during operation. Table I lists the sensitivity of typical explosives and explosive contaminated soil. The cutting blades normally rotate against the bars with only a small clearance, which could be negated by wear, misalignment, or improper assembly. The mill operates at 800 rpm so the cutter blades move at a speed greater than 10 ft/sec, which is higher than the speed for which we have sensitivity data. This means that there can be sufficient stimuli to initiate the explosive in the soil. Table III identifies other possible initiation sources.

The safe operation of the Wiley Mill in this service does not depend so much on the avoidance of potential initiation stimuli as controlling the extent of possible reactions. Table II presents the reactivity of various explosive/sand samples. It is concluded from these data plus other corroborative results⁽⁶⁾, that there should be no explosive reaction from any sample with less than 10 % explosive. [NOTE: These studies did not consider primary explosives so the results may not be valid if a sample contains primary explosives. The current effort, as well as the review of the Composter, is applicable to soils containing explosives such as TNT, RDX, HMX, etc.] Since the homogenization sample contains at most 4 % explosive, there should be no explosive reaction even if there is initiation within the mill. If there is sufficient initiation stimuli present, it is expected that there could be localized initiation of the explosive within the sample. This, in turn, might be immediately extinguished for lack of fuel or it may lead to a fire in flammable organic matter or other explosive in the sample. The worst case expected if initiation occurs in the mill is a mild fire.

Table III lists other less likely sources of ignition for the sample.

- If the mill continues to run with material in the grinding chamber, heat may build up eventually leading to thermal initiation. It is recommended that the mill be stopped when there is no output. A reasonable time for grinding the sample should be established and an excessive time should trigger a shutdown.
- If contaminated soil contacts electrical equipment, there could be initiation through thermal means or from exposure from an electric spark. Any sample spill or contamination should be cleaned promptly and the equipment should be grounded.
- If the sample is excessively dusty and a dust cloud is generated, ignition could be caused by a spark from electrical equipment or an operator. This combination of events is possible but not considered very likely.
- Should the mill clog or stop feeding, a common occurrence, the operator should use a non-metallic tool to restart the feed. Use of a metal tool increased the chance for localized initiation of explosive in the sample.

If the homogenization process were designed from scratch, different or modified equipment (e.g. dust rated electrical equipment) or process (e.g. remotely operated, conductive flooring) might be selected. However; the process should be able to be conducted safely in a non-explosive rated area if the above suggestions are followed. The most important single precaution is assuring that the sample contains a maximum of 4% explosive. In all steps from selecting the sample, through preparing for shipment, receiving, drying, and preparation for grinding operators should be on the look-out for excessive explosive such as pockets of pure explosive that could cause a more severe reaction. A good opportunity for this inspection is during the breaking up of clods prior to loading the mill.

Operators performing the grinding should be trained in the importance of proper operation of the mill, keeping the area clean, visually inspecting the sample, and not deviating from approved procedures. It is suggested that the grinding continue to be done in a ventilated hood with the shield lowered when the mill is running. Eye protection goggles or glasses with side shields should be required as well as fire retardant gloves and arm protection (e.g. long Nomex^R gloves). If the front shield must be raised during operation, the operator should have additional fire protection such as flame retardant coveralls and a face shield. The level of protection should be consistent with a fire, not an explosion, since the sample should be non-explosive but not necessarily non-flammable.

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TABLE I
TYPICAL SENSITIVITY DATA FOR
EXPLOSIVE CONTAMINATED SOIL AND SELECTED EXPLOSIVES

MATERIAL	TEST ³	THRESHOLD INITIATION LEVEL	REMARKS
Explosive Contaminated Soil	Friction, MPa	Range from 234 to 306 @ 3m/s & 380 to 520 @ 0.9 m/s	Dried Lagoon Sludge ⁴
	Impact, J/sq m	Range from 0.8 E4 to 13.9 E4	Dried Lagoon Sludge ¹
	Impact, J/sq m	4.75 E4	Composted Sample ⁵
	ESD, J	Range from 0.024 to 0.5	Dried Lagoon Sludge ¹
TNT	Friction, MPa	585 @ 2.4 m/s	Hercules Data
	Impact, J/sq m	5.25 E4	Hercules Data
	ESD, J	0.075	Hercules Data
	Impingement, m/s	193 [5/8x5/8" dia sample] 46 [1-5/8x5/8" dia sample]	Hercules Data Hercules Data
	Initiation Temp., deg C	570 @ 0.1 sec 465 @ 10 sec 328 @ 50 sec 275 @ 12 min 240 @ 30 min	AMCP 706-177 ⁶
	Crit Ht-to-Exp @ 2 in dia	12 in	Hercules Data
	Crit. Dia.	< 0.27 in	Hercules Data

³ FRICTION & IMPACT TESTS CONDUCTED WITH STEEL-ON-STEEL COMPONENTS

⁴ REFERENCE 2

⁵ REFERENCE 3

⁶ REFERENCE 4

TABLE I (Con't)

TYPICAL SENSITIVITY DATA FOR
EXPLOSIVE CONTAMINATED SOIL AND SELECTED EXPLOSIVES

MATERIAL	TEST	THRESHOLD INITIATION LEVEL	REMARKS
HMX	Friction, MPa	220 @ 2.4 m/s	Hercules Data
	Impact, J/sq m	0.27 E4	Hercules Data
	ESD, J	0.075	Hercules Data
	DSC Exotherm Temp., deg C	250 Onset	Hercules Data
	Initiation Temp., deg C	380 @ 0.1 sec 306 @ 10 sec	AMCP 706-177
	Crit. Ht-to-Exp	3 in	Hercules Data
	Crit. Dia.	< 0.27 in	Hercules Data
RDX [Cyclotrimethylene trinitramine]	Friction, MPa	313 @ 2.4 m/s	Hercules Data
	Impact, J/sq m	2.7 E4	Hercules Data
	ESD, J	0.075	Hercules Data
	Impingement, m/s	207	Hercules Data
	DSC Exotherm Temp., deg C	216 Onset 242 Peak	Hercules Data
	Initiation Temp., deg C	405 @ 0.1 sec 235 @ 15 sec	AMCP 706-177
	Crit Ht-to-Exp	2 in	Hercules Data
	Crit Dia.	< 0.27 in	Hercules Data

TABLE II
REACTIVITY OF EXPLOSIVE/SAND/WATER MIXTURES⁷

SAMPLE	TEST	RESULT	REMARKS
100 % RDX 0 % WATER	CRITICAL DIAMETER	< 0.25 INCH	1.05 g/cc Density RADFORD TEST
20 % RDX 0 % WATER 80 % SAND	CRITICAL DIAMETER	1.5 INCH	1.26 g/cc Density RADFORD TEST
15 % RDX 0 % WATER 85 % SAND	CRITICAL DIAMETER	2.0 INCH	1.25 g/cc Density RADFORD TEST
	DEFLAGRATION-TO- DETONATION (DDT)	1 of 10 POSITIVE REACTIONS	1.33 g/cc Density RADFORD TEST
	ZERO GAP	0 OF 20 POSITIVE REACTIONS	1.34 g/cc Density RADFORD TEST
13 % RDX 0 % WATER 87 % SAND	DDT	0 OF 20 POSITIVE REACTIONS	1.43 g/cc Density RADFORD TEST
10 % TNT 0 % WATER 90 % SOIL	# 8 CAP	5 TRIALS - NO FIRES; NO EXPL ABL TEST	Sample Spiked w/ 10 % TNT ⁸
	SPARK	5 TRIALS - NO FIRES; NO EXPL ABL TEST	

⁷ EXCERPTED FROM REFERENCE 5

⁸ REFERENCE 6

TABLE II (Con't)
 REACTIVITY OF EXPLOSIVE/SAND/WATER MIXTURES

SAMPLE	TEST	RESULT	REMARKS
10 % TNT 0 % WATER 90 % SOIL	THERMAL STABILITY [48 HR @72 DEG C]	1 TRIAL - NO IGN; NO DECOMPOSITION ABL TEST	THIS SAMPLE SPIKED WITH 10 % TNT ⁶
	BOE IMPACT	0 OF 10 POSITIVE REACTIONS	ABL TEST
	FRICITION	0 OF 5 TRIALS @ 330 LBF AND 8FPS ABL TESTS	
	ELECTROSTATIC DISCHARGE	10 TRIALS - NO REACTIONS @ 0.024 JOULE	ABL TEST
	DDT	NO REACTIONS - w/10 or 20 GRAM IGNITERS	ABL TEST
	U S GAP	3 TRIALS; NO DAMAGE TO WITNESS PLATE	ABL TEST
	FIRE TEST	1 TRIAL; NO REACTION	ABL TEST

TABLE III

POTENTIAL INITIATION SCENARIOS FOR
OPERATION OF THE WILEY MILL

HAZARD	INITIATION SOURCE	RESULT	REMARKS
1. Cutter blade strikes/rubs bar because of misalignment, improper installation, etc.	Impact and/or Friction Steel/Steel	Localized initiation of explosive	Should be no explosion
2. Blade strikes foreign material (Metallic) in sample being processed	Spark or Friction Steel/Metal	Localized initiation of explosive	
3. Cutter blade strikes non-metallic foreign material (Stone, etc.) in the sample being processed	Friction Steel/stone	Possible localized initiation of explosive; damage to blades	
4. Mill output slows or stops allowing heat build-up	Thermal	Possible localized initiation of explosive	
5. Sample excessively dusty; spark available from electrical equipment or from operator	Electric spark or Electrostatic Discharge	Initiation of dust cloud	Not very likely; requires dust cloud formation
6. Sample contaminates electrical equipment	Electrical spark	Initiation of dust layer	Remove sample spills immediately
7. Sample contamination allowed to remain on electrical equipment	Thermal	Initiation of dust layer	Remove sample spills immediately
8. Operator clears clogged mill with improper tool	Impact or Friction Steel/Metal	Localized initiation of explosive	Use only proper, non-metallic tools

TABLE III (Con't)

POTENTIAL INITIATION SCENARIOS FOR
OPERATION OF THE WILEY MILL

HAZARD	INITIATION SOURCE	RESULT	REMARKS
9. Operator steps on spilled sample	Friction Leather/concrete	Possible localized initiation of explosive	Remove sample spills immediately
10. Sample contains excessive explosive	Impact or Friction Steel/Steel	Initiation of sample; fire and strong reaction	SEE TEXT