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# Extraction of TNT from aggregate soil fractions

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**Abstract**

Past explosives manufacture, disposal, and training activities have contaminated soil at many military facilities, posing health and environmental risks through contact, potential detonation, and leaching into ground water. While methods have been confirmed for extraction and measuring explosives concentration in soil, no work has addressed aggregate size material (the $>2$ mm gravel and cobbles) that often occurs with the smaller soil fractions. This paper describes methods and results for extraction and measurement of TNT (2,4,6-trinitrotoluene) in aggregate material from 1/2 to 2-1/1 from a WWII era ammunition plant. TNT was extracted into acetonitrile by both Soxhlet and ultrasonic extraction methods. High pressure liquid chromatography analyses of extracts showed expected variation among samples. Also effective extraction and determination of TNT concentration for each aggregate size fraction was achieved. 

*Keywords:* Explosives; Soil; Aggregate; Remediation; Extraction; Analysis; TNT

## 1. Introduction

### *1.1. Problem statement*

Nitroaromatic explosives contaminate soil at many military installations and pose a unique environmental problem. Current remediation methods involving incineration or secure landfilling are expensive and consume scarce landfill space. These compounds have a solubility in water on the order of 100 ppm and do not rapidly dissolve from the soil. Instead, they slowly leach into ground or surface waters and present a chronic environmental threat over many decades.

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Current analytical methods use extraction and high pressure liquid chromatography or spectrophotometry to analyze soils smaller than 2 mm in size. Field experience has shown, however, that soil contamination can occur in aggregate-size (gravel and cobbles) soil fractions.

### *1.2. Scope and objectives*

US Army Corps of Engineers research efforts seek to clarify the association of explosives with soil and develop physical separations methods for their removal and methods for their destruction. The primary concern here was to develop and assess measurement of TNT in aggregate soil fractions from a WWII ammunition plant. The objective of this project was to provide practical options for finding the explosives content of aggregate or debris, in this case 1/2–2-1/2 in. in size.

## 2. Background

### *2.1. History*

Until the late 1960s, TNT was manufactured by a batch process yielding excessive amounts of waste effluent. Much of the discharge was released to surface waters with limited treatment, generally involving settling [1]. Contamination occurs around process facilities, streams, settling lagoons, and deactivation areas. The primary contaminants are TNT, RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine), HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine), and Tetryl (methyl-2,4,6-trinitrophenylnitramine) along with decomposition products [1].

Since 1975, research and development for the Installation and Restoration Program (IRP) has emphasized the development of cost-effective techniques. Information developed under this program has assisted with the cleanup of IR, FUDS (formerly used defense sites), and BRAC (base realignment and closure) sites.

### *2.2. Explosion hazards*

Explosion hazards result from potential propagation of detonation through soil with $>$ 10% contamination or from concentrated 'chunks' of explosive. These chunks contain essentially pure TNT crystallized from concentrated liquids after steam cleaning and burning of TNT-contaminated structures. These chunks can be 100 $cm^3$ or more in size, although chunks on the order of 1 $cm^3$ are more common. These may be hazardous to unprotected workers, particularly those using power equipment.

### *2.3. Toxicity*

Hazards occur in contaminated soil and groundwater. Discharge from an explosives processing or handling facility is regulated, and is referred to as 'pink' or 'red' waters due to its distinctive color. Tests have shown toxicity to rats and mice [2–4], and fish [5]. Also, plant growth is slowed [6], and mutagenic effects on microorganisms have been documented [7]. Few factors affecting the environmental fate of TNT are well defined [1]. Worker exposure has generally taken place by absorption through the skin

and inhalation [8]. Symptoms from overexposure include mild irritation of the digestive tract and paleness or purpling of the skin. More severe symptoms are methemoglobinemia, severe jaundice due to liver damage, and aplastic anemia caused by dysfunction of the bone marrow [9].

### *2.4. Recent research*

Current soil remediation methods include excavation and hauling to hazardous waste landfills (if reactivity is low) or to incinerators. Problems include cost and public concern over incineration. In response to these limited options, a number of projects are underway to develop improved soil remediation methods. These focus on developing or improving methods for stabilization, oxidation, biodegradation, and separation based on physical property differences. All these efforts require accurate analytical methods for determining explosives concentration in soils. Currently, the most widely accepted method is based on work by a team at the US Army Corps of Engineers Cold Regions Research and Engineering Laboratory at Hanover, NH [10–12]. This method, based on HPLC (high pressure liquid chromatography) analysis of acetonitrile extracts has been incorporated into EPA Method 8330 for nitroaromatics. Extraction procedures included Soxhlet and ultrasonic bath methods. It should be stressed that these studies, as others, used soil particles smaller than 2 mm, and not aggregate.

### *2.5. Difficulties presented by aggregate samples*

Aggregate material (cobbles and gravel) poses unique problems due to the larger size of individual particles and required sample size. Most medium-size Soxhlet extractors do not readily hold particle sizes $> 3/4$ in. (1.90 cm). Furthermore, statistical design requires at least 20 pieces of each aggregate size, and preferably 2–3 times this minimum. Twenty pieces of $1/2$–$3/4$ in. (1.25–1.90 cm) aggregate weigh about 80 g; 20 pieces of $> 1$ in. ($> 2.54$ cm) aggregate, 1800 g. The soil used in this study contained almost 35 wt.% of material greater than $1/2$ in. in size. Meaningful investigation required analysis of this aggregate fraction. Before proceeding, a number of questions were raised about the effectiveness of the Soxhlet and ultrasonic methods: (1) what ratios of solvent to aggregate would be effective; (2) how large a sample could be extracted at a time with existing ultrasonic and Soxhlet equipment; (3) would the established extraction times prove effective; (4) would one round of ultrasonic extraction be adequate; and (5) how can solvent volumes be minimized with the significantly larger sample sizes required?

## 3. Materials and methods

### *3.1. Experimental approach and rationale*

We based our extraction approach on the work of Jenkins and Leggett [13] who had confirmed the effectiveness of the Soxhlet and ultrasonic extraction methods for soil.

We decided to adapt these methods to the available equipment. Extractions of subsamples were done in parallel to allow extraction of a statistically valid sample size. The variation among smaller subsamples could also be measured. Multiple extractions were done to quantify residual TNT and the efficiency of each extraction stage. Aggregate and fines were separated after extraction to measure TNT content on the basis of fines as well as combined fines and aggregate weight.

### *3.2. Soil sample collection*

The soil used for the study was a TNT-contaminated soil-aggregate mixture from the Weldon Springs Training Area, a WWII-era ammunition plant, near St. Louis, MO. Contaminated soil and aggregate remain around building foundations and along pipelines. Steam cleaning and burning left recrystallized TNT 'chunks' in the soil. Most measure less than a 1 $cm^3$ in size, but can reach extremes of 100 $cm^3$ or more. These highly concentrated nuggets cause an explosion hazard, the main acute safety concern for sampling and processing. The sample was taken using hand shovels from the top 7–8 in. of soil. The soil had a characteristic chocolate color, indicating a high TNT concentration on the order of 1000 s of parts per million. Significant aggregate was present, probably placed around process buildings to stabilize the surface.

### *3.3. Soil preparation*

For the initial physical separation, a SWECO Vibro-Energy Separator loaded with a nest of 30-in. diameter sieves with openings of 1 in., 3/4 in., and 1/2 in. was used to separate the soil into four size fractions: $<1/2$, 1/2–3/4, 3/4–1, and $>1$ in. in diameter. (The $>1$ in. aggregate ranged up to approximately 2-1/2 in. in size). The soil and aggregate were then 'homogenized', mixed thoroughly to insure uniformity. Homogenized samples were sealed in 20-liter (5-gal) plastic buckets and stored at 4°C.

### *3.4. Summary of extraction procedures*

To investigate the extraction of aggregate, we used both ultrasonic and Soxhlet methods. Table 1 summarizes the types of extractions done for each size fraction. Soxhlet extraction could not be done on the $>1$ in. size due to the limited size of extraction glassware on hand. However, visual observation indicated that the aggregate was impermeable gravel/cobbles with fines of $<2$ mm size adhering to the exterior surface. The bulk of the TNT contamination probably occurs at and near the aggregate

Table 1
Extraction methods applied to size fractions of Weldon Springs aggregate

| Aggregate fraction | Extraction method |
|---|---|
| 1/2–3/4 | Repeated Soxhlet and ultrasonic extraction |
| 3/4–1 in. | Single Soxhlet and repeated ultrasonic extraction |
| $>1$ in. | Repeated ultrasonic extraction |

surface (caked in fines). Calculation of TNT on a 'fines basis', as reported in Section 4.6 of this paper, supports this view. Successful extraction of 1/2–3/4 in. and 3/4–1 in. material would thus validate extraction of TNT from these surface fines regardless of the size of the aggregate.

### 3.5. Equipment

Soxhlet extraction used medium-sized (45 × 140 mm) Soxhlet extractors with fritted glass thimbles. The 250 ml boiling flasks were heated using a 6-position extraction heater (300 watts each) with individual controls. The ultrasonic extractions used a NEY 300 ultrasonic bath and an International Equipment PR-7000 centrifuge for solids separation. Extracts were analyzed according to the EPA 8330 Method using a Perkin-Elmer Model LC 4000 High Pressure Liquid Chromatograph with a UV detector at a wavelength of 254 nm.

### 3.6. Soxhlet extraction procedures

In two trials with 1/2–3/4 in. aggregate, 10 to 12 pieces (approximately 40 g) were placed into each thimble and extracted for 5 h with 175 ml of acetonitrile. The acetonitrile was removed and replaced with fresh acetonitrile for two subsequent stages of extraction of 4 h. HPLC analysis of the extracts from these three sequential extractions showed that all the TNT was extracted in the first stage. Thus, for the remainder of this study, only one stage of extraction was performed for 6 h on each sample. Extraction was repeated for three more 1/2–3/4 in. samples and for five 3/4–1 in. samples.

### 3.7. Ultrasonic extraction procedures

For the 1/2–3/4 in. aggregate, a three stage ultrasonic extraction was first performed with 50 g of aggregate and 250 ml of acetonitrile in each of two, 1 l centrifuge bottles (a solvent/aggregate ratio of about 5 ml/1 g). The bottles were sonicated for 18 h, centrifuged, and decanted of solvent. Two subsequent extractions were performed for 2–4 h periods, and the extracts for all three stages were analyzed separately. HPLC results indicated that the TNT was effectively removed in the first two stages, and the third stage extraction was eliminated for subsequent samples. Two-stage extraction was repeated on three more 1/2–3/4 in. aggregate samples and five 3/4–1 in. samples. The extractions of the 3/4–1 in. material used approximately the same solvent, but aggregate weights of about 120–130 g, a the solvent/aggregate ratio of 2 ml/1 g.

The > 1 in. samples involved a modified procedure to limit solvent use. We placed about 200–240 g of > 1 in. aggregate into each of six 500 ml beakers. Acetonitrile was added to bring the liquid level to 400 ml. These were sonicated for 18 h, the solvent decanted and solids centrifuged. Acetonitrile was added to the beakers and sonication repeated for 3.5 h. The aggregate was removed and washed with acetonitrile into the

beakers. The beakers were then emptied into centrifuge bottles and centrifuged at the above conditions to separate solids. The suspended solids that were entrained and removed during the first decanting were sonicated again for 5.75 h. The three extract liquids were analyzed separately.

For all three aggregate sizes the dried solids were slurried with water and poured into a pan. The aggregate were picked out, washed off into the pans, and dried at 60°C overnight. The fines remained in the water in each pan. These were dried for two days at 60°C. This approach provided data on the weight fraction of aggregate vs. the $< 2$ mm size fines caked onto the surface of the aggregate.

## 4. Results and discussion

The following observations were made based on the Soxhlet and ultrasonic extraction of three size fractions of the Weldon Springs aggregate. Results show the overall aggregate size distribution followed by a Soxhlet and ultrasonic extraction analysis of TNT concentration in subsamples. Results reveal the impact of aggregate size range, extraction method, and solvent to solid ratio on the TNT concentration measured and the extraction efficiency.

### *4.1. Overall aggregate size distribution*

The original soil was screened into four fractions with a 30-in. sieve as noted in Section 3.3. The weight distribution appears in Fig. 1. Weight percent and subsequently reported TNT concentrations are expressed on an oven-dried basis (overnight, 60°C). Two-thirds of the material was $< 1/2$ in. in size, leaving approximately 35 wt.% of the material consisting of $> 1/2$ in. aggregate caked with contaminated fines.

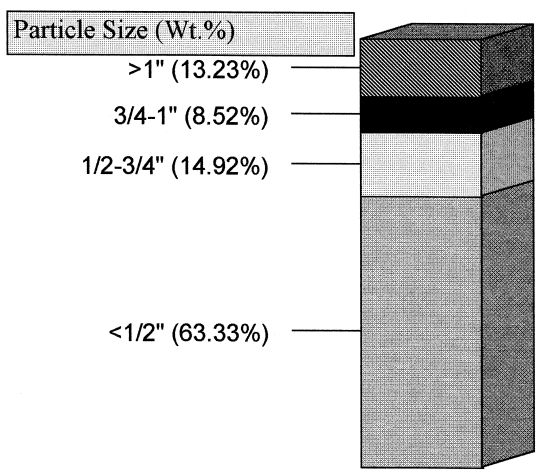


Fig. 1. Weight percent distribution vs. size, Weldon Springs soil/aggregate.

Table 2
Soxhlet extraction of 1/2–3/4 in. Weldon Springs aggregate

| Sample | Aggregate TNT (mg/kg) |
|---|---|
| S1 | 14,415 |
| S2 | 12,892 |
| S3 | 12,558 |
| S4 | 10,678 |
| S5 | 6655 |
| Weighted average | 11,423 |

### *4.2. TNT in 1/2–3/4 in. aggregate based on Soxhlet and ultrasonic extraction*

Soxhlet extraction of the 1/2–3/4 in. aggregate involved five samples ranging from 39 to 41 g in weight. Table 2 shows that after 6 h, the recovered TNT measured from 6655 to 14,415 mg/kg with a weighted average of 11,423 mg/kg. Statistical analysis (confidence interval for the mean, assuming a normal distribution function) gave a 95% confidence limit of $\pm 2618$ mg/kg for the mean TNT content of the five samples (Table 3).

Ultrasonic extraction of the 1/2–3/4 in. aggregate involved five samples ranging from 52–53 g in weight. Table 3 shows the TNT extraction (mg/kg) for each stage. Summing the extracted TNT for each stage gave a total recovered TNT ranging from 4832–19,882 mg/kg with a weighted average of 10,334 mg/kg. Table 4 summarizes the descriptive statistics for TNT concentration in 1/2–3/4 in. and 3/4–1 in. aggregate. The 95% confidence limit is $\pm 5150$ mg/kg for the mean TNT content of the five 1/2 to 3/4 in. samples tested. It should be noted that the descriptive statistics do not correct for weight variations among sample sizes. Thus the 'weighted' averages in Table 5 and the means in Table 4 from the statistical analysis are close, but different in value. Cumulative extraction of 90 + % of the TNT present occurred in the first stage and 98 + %, including the second, as illustrated by Fig. 2. The average percent recovered in the first stage extractions was 94.5%.

Table 3
Repeated ultrasonic extraction of 1/2–3/4 in. Weldon Springs aggregate—TNT concentration and percent recovery

| Sample | TNT (mg/kg) recovery for each extraction | | | Percent (%) TNT recovered for each extraction | | |
|---|---|---|---|---|---|---|
| | 1st | 2nd | 3rd | 1st | 2nd | 3rd |
| S1 | 4382 | 394 | 56 | 90.68 | 8.15 | 1.17 |
| S2 | 8560 | 102 | 86 | 97.85 | 1.17 | 0.99 |
| S3 | 5057 | 223 | – | 95.77 | 4.23 | – |
| S4 | 18,775 | 1107 | – | 94.43 | 5.57 | – |
| S4 | 11,841 | 811 | – | 93.59 | 6.41 | – |
| Average | 9733 | 529 | 71 | 94.57 | 5.1 | 1.1 |

Table 4
Descriptive statistics for Soxhlet and ultrasonic extraction of TNT from Weldon Springs aggregate

| Statistical parameters | Soxhlet extraction | | Ultrasonic extraction | |
|---|---|---|---|---|
| | 1/2–3/4 in. | 3/4–1 in. | 1/2–3/4 in. | 3/4–1 in. |
| Mean[a] | 11 440 | 4224 | 9723 | 6658 |
| Median | 12 558 | 4189 | 8560 | 6282 |
| Standard deviation | 2987 | 668 | 5875 | 2242 |
| Range | 7760 | 1719 | 14,393 | 5581 |
| Minimum | 6655 | 3446 | 4382 | 4604 |
| Maximum | 14 415 | 5165 | 18,775 | 10,185 |
| 95% confidence level[b] | 2618 | 586 | 5150 | 1966 |

[a]Mean TNT content for the sample set, not weighted for varying sample weights.
[b]Confidence limit for mean of sample set extracted.

### *4.3. TNT in 3/4–1 in. aggregate based on Soxhlet and ultrasonic Extraction*

Soxhlet extraction of the 3/4–1 in. aggregate involved five samples ranging from 76 to 97 g in weight. Fig. 3 shows that after 6 h extraction, the recovered TNT measured from 3446 to 5165 mg/kg with a weighted average of 4186 mg/kg. Statistical analysis gave a 95% confidence limit of 586 mg/kg for the mean TNT content of the five samples tested (Table 4).

Ultrasonic extraction of the 3/4–1 in. aggregate involved five samples ranging from 105 to 131 g in weight. Fig. 4 shows that after an 18 h primary sonication and a 3 h secondary sonication, the recovered TNT measured 4604 to 10,185 mg/kg. The weighted average was 6525 mg/kg. Statistical analysis gave a 95% confidence limit of 1966 mg/kg for the mean TNT content of the five samples tested (Table 4). At least 67 + % of the TNT was extracted in the first stage. However, 4 of 5 primary extractions recovered an average of 84.1%.

### *4.4. TNT in > 1 in. aggregate based on ultrasonic extraction*

Since no Soxhlet extraction glassware at hand was large enough for the > 1 in. aggregate (maximum of 2-1/2 in.), only ultrasonic extraction was performed. Results

Table 5
Weighted average TNT concentrations and mass and TNT distributions vs. size fraction, Weldon Springs aggregate

| Size fraction (in.) | TNT (mg/kg) ultrasonic | TNT (mg/kg) (Soxhlet) | Soil fraction (wt.%) | TNT (%) of total in whole soil |
|---|---|---|---|---|
| < 1/2 in. | 12 103 | – | 63.33 | 74.70 |
| 1/2–3/4 in. | 10 334 | 11,423 | 14.92 | 14.78 |
| 3/4–1 in. | 6525 | 4186 | 8.52 | 5.09 |
| > 1 in. | 4506 | – | 13.23 | 5.43 |
| Average | 10 261 | – | – | – |

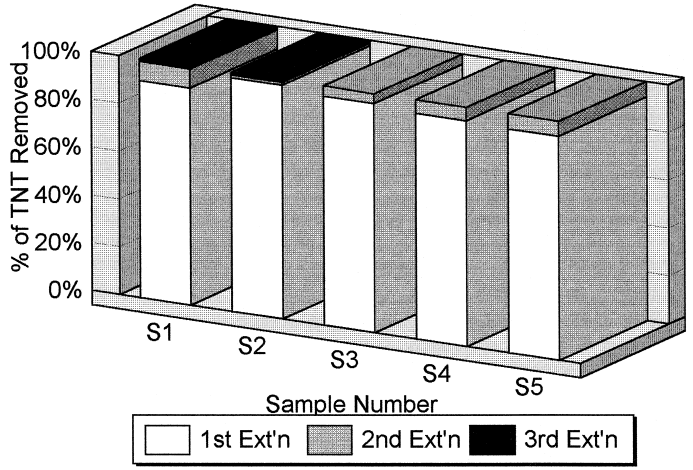


Fig. 2. Percent TNT recovery by repeated ultrasonic extraction, Weldon Springs 1/2–3/4 aggregate.

reflect the lowered extraction efficiency from use of a solvent to aggregate ratio of 1 ml/g vs. 5 ml/g for the 1/2–3/4 in. material. After the primary sonication, these were combined for centrifugation and solvent/TNT recovery. On a weight adjusted basis, the first stage extraction removed a total of 2370 mg/kg TNT. The second stage extraction (3.5 h) recovered 2066 mg/kg TNT. The sediment recovered from the liquid decanted from the first stage extraction was extracted again for 5.75 h. Only 69 mg/kg TNT was recovered from this last fraction. Fig. 5 illustrates the lessened extraction efficiency (percent of total) for each extraction stage of the $>1$ in. aggregate. No statistical analysis was done on the extraction of the $>1$ in. material.

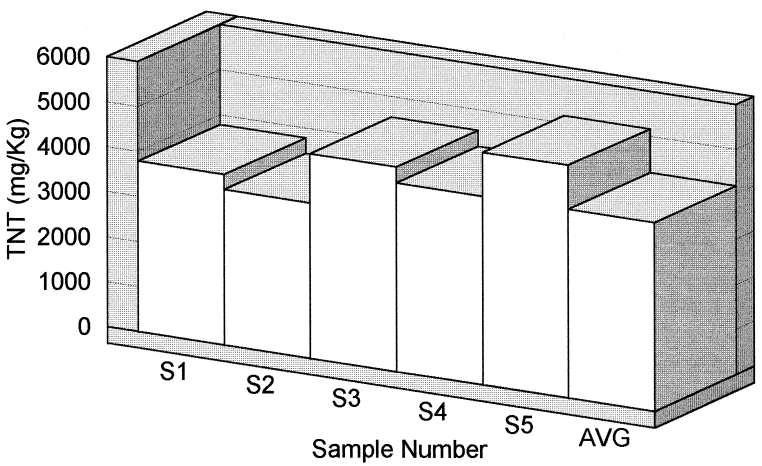


Fig. 3. Soxhlet extraction of TNT, Weldon Springs 3/4–1 in. aggregate.

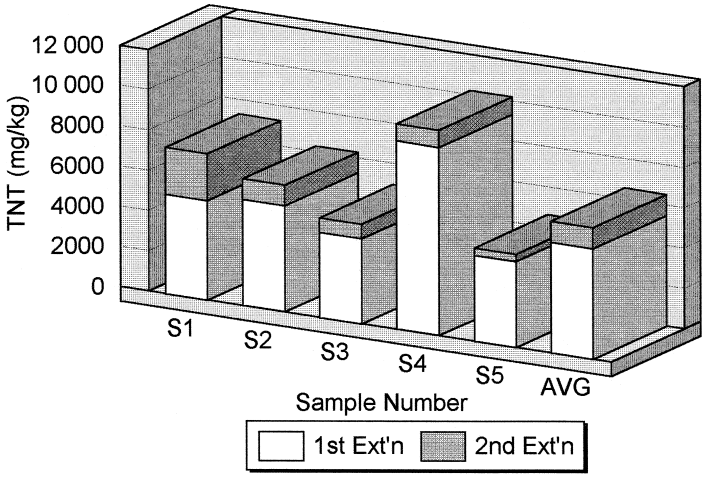


Fig. 4. Repeated ultrasonic extraction of TNT, Weldon Springs 3/4–1 in. aggregate.

### *4.5. Summary of TNT concentration in aggregate size fractions*

The TNT content for all the size fractions of aggregate and the $< 1/2$ in. soil is presented in Fig. 6. The TNT content declines with increasing aggregate size to a low of 4506 for the $> 1$ in. fraction. The weighted average for all size fractions is 10,261 mg/kg. Note that the TNT content of the 1/2–3/4 in. fraction measured by Soxhlet extraction is about 10% higher than that by ultrasonic extraction. For the 3/4–1 in. fraction, the TNT measured by the Soxhlet extraction was about 35% lower.

Comparing the weight and TNT distributions shown in Fig. 7 indicates that the $< 1/2$ in. fraction is somewhat enriched in TNT content. The $> 3/4$ in. fractions are

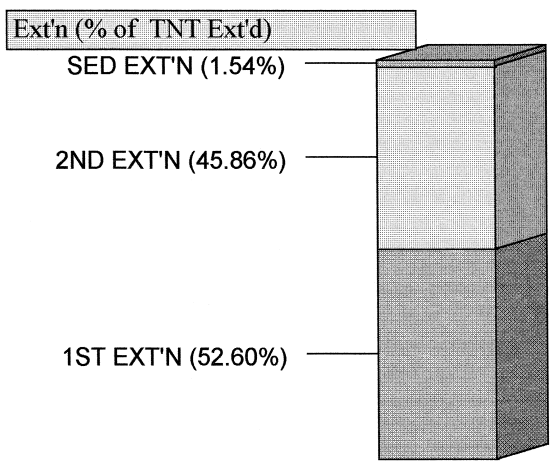


Fig. 5. Percent TNT recovery by repeated ultrasonic extraction, Weldon Springs $> 1$ in. aggregate.

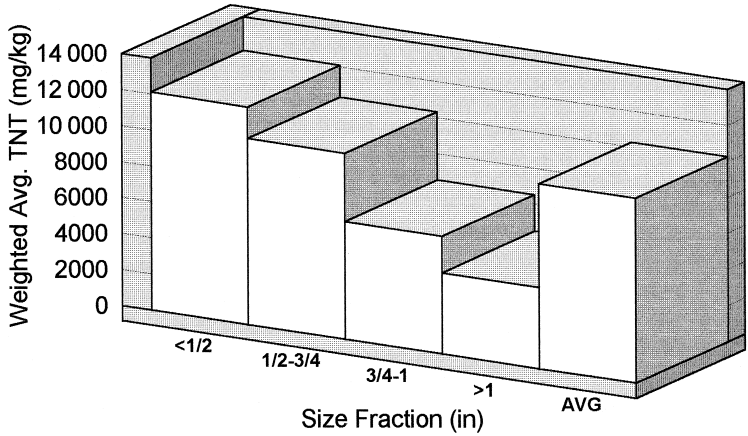


Fig. 6. TNT content vs. size fraction, Weldon Springs aggregate.

somewhat depleted. The distribution is made less extreme because the larger fractions carry contaminated fines caked on them.

### *4.6. TNT in fines caked on aggregate*

Visual inspection revealed that the aggregate was solid and relatively impermeable gravel and cobbles. Thus the TNT was predominantly in fines, caked onto the surface of the aggregate. The presence of impermeable and inert aggregate would thus 'dilute' the finer, contaminated material. The TNT content for each fraction based on fines (mg TNT/kg fines) appears in Table 6. The TNT content based on fines for ultrasonic extraction varied from 25,070 mg/kg for the 1/2–3/4 in., 33,522 mg/kg for the 3/4–1 in., and 35 012 mg/kg for the $>$ 1 in. fractions. The Soxhlet and ultrasonic TNT

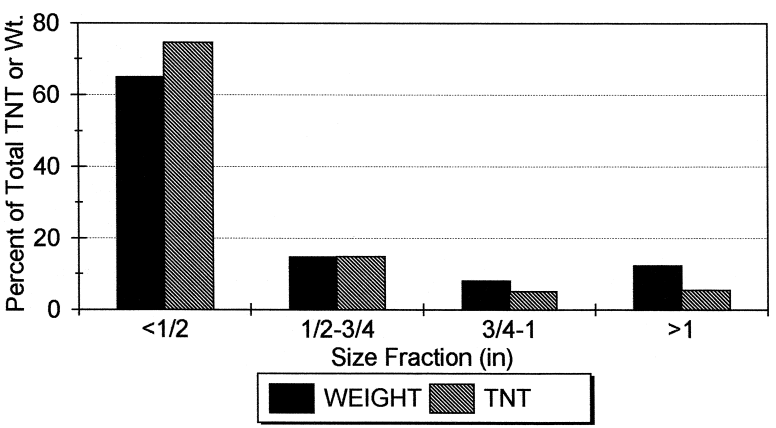


Fig. 7. Weight and TNT percent distribution vs. size, Weldon Springs aggregate.

Table 6
TNT content in fines caked on aggregate—by size and extraction method, Weldon Springs aggregate

| | TNT (mg/kg) Soxhlet extraction | TNT (mg/kg) ultrasonic extraction |
|---|---|---|
| 1/2–3/4 in. | 25 601 | 25 070 |
| 3/4–1 in. | 12 364 | 33 522 |
| > 1 in. | – | 35 012 |

recoveries, based on fines for 1/2–3/4 in. aggregate, agree within about 2%. However, the Soxhlet and ultrasonic results for the 3/4–1 in. aggregate differed greatly at 12 364 and 33 522 mg/kg TNT. Finally, the ultrasonic results for > 1 in. aggregate agreed within about 4% for the 3/4–1 in. aggregate.

## 5. Conclusions

Based on the test results, the following conclusions were made.

(1) Soxhlet and ultrasonic extraction efficiently recover TNT from aggregate up to 1 in. in size.

(2) Use of a solvent/aggregate ratio of 4–5 ml/g insures efficient one-stage extraction of over 90% for either Soxhlet or ultrasonic methods.

(3) Variations in TNT content reinforces the importance of using statistically meaningful sample sizes, at a minimum, 40–50 pieces of randomly selected aggregate.

(4) TNT content based on total aggregate weight includes the diluting effect that varies with the size of the aggregate. TNT content based on fines removes this effect to show the concentration in the adhering fines.

(5) A Soxhlet extraction period of 6 h is effective for aggregate up to 1 in. in diameter.

(6) An ultrasonic extraction period of 18 h is effective for aggregate up to 2 in. in size.

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