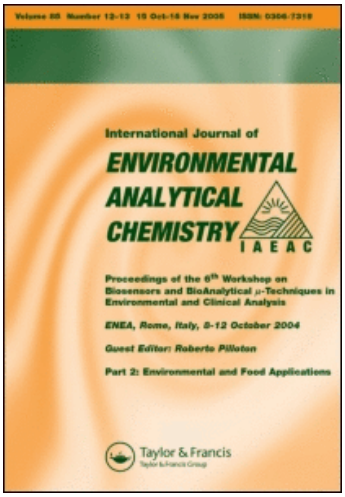

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METHOD FOR SCREENING AND ANALYSIS OF RESIDUES COMMON TO MUNITION OPEN BURNING/OPEN DETONATION (OB/OD) SITES

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Procedures are presented for qualitative screening and subsequent quantitation of residues of explosives and related compounds found at sites contaminated by open burning/open detonation (OB/OD) disposal of munitions. Methods are reported for recovery of explosives and polycyclic aromatic hydrocarbons (PAHs) from soil via sonic extraction into acetonitrile; and explosives from water via trapping onto C18 disposable cartridges. Complimentary HPLC procedures were used for identification and quantification of these compounds. A qualitative HPLC gradient method was developed, and used to screen samples for a wide range of explosives and PAHs. Quantitation of explosives and their environmental reduction products was accomplished using HPLC isocratic methods. Recoveries of explosives and degradation products doped into sandy loam soil were greater than 90%. Corresponding recoveries from aqueous samples were comparable for the nitroaromatics, but were poorer for the nitramines. Criteria of detection for individual munition residues ranged from 0.067 mg l^{-1} to 0.37 mg l^{-1}.

KEY WORDS: Munition residues, explosives, TNT, HMX, RDX, open burning/open detonation, OB/OD.

INTRODUCTION

Out-of-date and out-of-specification munitions have commonly been disposed of by burning, or by detonation, on unprotected ground.[1] The practice of open burning/open detonation (OB/OD) of munitions historically involved quantities of explosives up to thirty tons per disposal event, and generated a mixture of contaminants into the immediate area at high concentration.[2] At many military installations OB/OD sites consist of multiple disposal areas. These OB/OD sites number in the hundreds,

and have been developed and used by both the military and their civilian contractors during much of this century. Many of these sites have records inadequate to predict the nature and extent of the contamination. Residue from OB/OD consists primarily of unburned explosives, but environmental weathering and microbial action are known to produce modifications of these compounds.[3]

Estimation of the environmental impact of OB/OD contamination at an individual site requires detailed knowledge of the type and amount of the chemical contaminants present and an understanding of their migration behavior within the soil. Our approach to these investigations was based on a two step process.

The first step was qualitative analysis of highly contaminated surface samples to screen for compounds present in environmentally significant concentrations. Due to the variety of military explosives and their environmentally modified forms, a new method was required to chromatographically isolate the majority of the compounds likely to be encountered.

The second step was quantitation of the OB/OD contaminants in soil at various depths, and in water that leached through this soil. Quantitation required greater analytical sensitivity than the above screening method could provide. Moreover, the large number of analytical determinations involved in leaching experiments necessitates use of more rapid procedures. There are a number of simple isocratic HPLC separation methods which have been used to quantitate explosives. However, each of these methods has proven effective for only a limited number of compounds.

Appropriate quantitative HPLC methods were selected for each OB/OD site on the basis of the suite of compounds present. Herein, we review the performance of one isocratic system because of its utility, and to illustrate the problems common to analysis of OB/OD residues by isocratic HPLC and UV absorbance.

The compounds used to demonstrate the qualitative and quantitative methods were selected because they have been reported in association with the burning, incineration, or detonation of explosives. These compounds are representative of the mixtures likely to be encountered, but should not be construed as a complete list of OB/OD contaminants. Discussion of the origin and nature of selected OB/OD compounds follows.

Brueggemann's analysis[4] of the ash from munitions burned in incinerators revealed substantial concentrations of polycyclic aromatic hydrocarbons (PAHs). However, these compounds are not mentioned in Jenkin's report of a method for quantitation of explosive residues in soil.[5] PAHs are an important class of compounds and will be included in our screening procedures until the question of their existence at OB/OD sites is answered with some certainty.

Nitroglycerin (NG) is a component of several military munitions[6] but is not generally reported as a contaminant at OB/OD sites. Failure to detect NG may be attributed either to an actual absence of this compound, or to an inability of current methods to detect it. Failure to detect this compound could be due to its very weak absorbance at UV wavelengths greater than 220 nm, where most munition analyses are performed.

The military use of nitroguanidine (NQ) is primarily in M30 propellant, which is a mixture of NQ, NG and nitrocellulose. Since NQ is more polar than other

munitions, it is predicted to leach more rapidly and may therefore have a short residence time at the soil surface. It is also readily degraded by UV light.[7] In addition, because of its high polarity, methods which utilize reversed phase HPLC often have difficulty in separating NQ from extractable natural soil components.[8]

The nitramine munitions cyclotrimethylenetrinitramine (RDX) and cyclotetramethylenetetranitramine (HMX) are chemically similar and are widely used as explosives and as propellants. Industrial synthesis methods for these explosives do not produce pure compounds. All military grades of HMX contain RDX as an impurity and most RDX contains HMX.[6] They are normally found together as environmental contaminants. Nitramines are easily extracted from soil samples and readily lend themselves to quantitation by reverse phase HPLC and UV absorbance.

Trinitrotoluene (TNT) and its environmental breakdown products are the most common contaminants at many OB/OD sites. These compounds are less polar than the preceding compounds discussed, and have excellent UV absorbance. They are readily quantitated by reverse phase HPLC and UV detection methods. Nitroaromatics undergo a variety of modifications in the environment but generally tend to remain identifiable as related forms because frequently their ring structure is not degraded.[3]

Numerous HPLC methods have been reported for determination of explosive-residues. But adoption of a standard screening method among laboratories has not been pursued. This paper describes a new HPLC method that is useful for screening of any explosive-contaminated site, and an established quantitation method for use when only certain nitramines and nitroaromatics are found to be present. Sites such as these, contaminated primarily with TNT and RDX, are often encountered because these explosives are the most common in the U.S. military arsenal.

These methods were developed to support research into the environmental fate of residues from OB/OD operations, but are applicable to sites contaminated by the manufacture of explosives or by munition load/assemble/pack operations. The chromatographic methods presented herein are useful for analysis of (1) acetonitrile extracts of explosive-contaminated soils, (2) aqueous leachates, and (3) methanol concentrates of aqueous leachates.

METHODS

Sample preparation and extraction procedures were adapted from a method developed and extensively tested by Jenkins.[9–11] These modified procedures entailed grinding air-dried soil samples, and extracting 1 g of the sample into 10 ml acetonitrile with 18 hours of sonication in a bath at 20°C. Extracts were then centrifuged at 3900 × G for 15 min, passed under piston pressure through a Gelman 0.45 μm Acrodisc-CR disposable filter, and analyzed by HPLC. The latter portion of the sequence differs from Jenkin's method in that a step requiring mixing the acetonitrile extract with an aqueous flocculation solution was eliminated, and that the internal standard 1,3-dinitrobenzene (DNB) was incorporated.

An estimation of the efficiency of extraction of each compound was obtained by

doping subsamples of uncontaminated surface soil (A horizon, Wheeling sandy loam [Fine-loamy, mixed, mesic Ultic Hapludalfs]) with acetonitrile containing a mixture of selected OB/OD compounds plus DNB. The soil was air-dried and extracted as above, and the efficiency of extraction was calculated from the amount of each compound recovered. Because the efficiency of extraction of the OB/OD components at our test sites was similar to that of DNB, a simplified recovery correction system was possible. All soil samples were extracted with acetonitrile containing 2.5 ml l^{-1} of DNB as an internal standard.

Observed concentrations of OB/OD components in the extraction mixture were corrected for losses of internal standard that occurred during the extraction process. Corrections were also made for any increases in concentration due to evaporation of the extraction solvent, acetonitrile.

Aqueous leachates were collected, and subsamples adjusted to pH 6.00 $\pm$ 0.05 then made to contain 300 g l^{-1}NaCl. Two hundred mL of the resultant solution was put through a J.T. Baker 40 μm Sep-Pak Octadecyl (C18) disposable cartridge at a rate of 1.8 ml min^{-1}. Cartridges were prepared for use by wetting with 2 ml methanol, followed by 2 ml water. Munition residues were eluted from the cartridges with 2 × 1 ml additions of methanol, and eluates were analyzed by HPLC. Efficiencies of recovery were determined for this procedure using aqueous standards.

HPLC analyses were preformed with a Hewlett-Packard (HP) 1050 HPLC system that consisted of an autoinjector, pumping module, and UV detector. Signal integration was performed with an HP 3396A integrator. All analyses except screening tests for the presence of NG were done by UV absorbance at 244 nm. NG was determined at 220 nm.

Extracts of uncontaminated soils (background) and highly contaminated surface soils were screened by the gradient method developed for this investigation. A 15 μl sample was injected onto a 4.6 × 250 mm Rainin Microsorb C18 column with a 5 μm particle size, in series with a 4.6 × 250 mm Supelcosil LC-PAH column. Elution was accomplished with a methanol:water gradient (Table 1).

Simpler isocratic methods were used to substantiate the identification of contaminants, and for quantitation. The method of Miyares and Jenkins[12] entailed isocratic pumping of a mobile phase of 70.7% water, 27.8% methanol and 1.5% tetra-

Table 1 HPLC time/gradient (methanol: water mixture) for initial screening of samples for a broad range of munition-related analytes and PAHs

Time, min	*Percent methanol (% MeOH)*
0	30
1.5	33.5
6.0	47.5
24.0	51.0
35.0	54.5
60.0	100.0
80.0	100.0

hydrofuran at a flow rate of 2 ml min^{-1} through a 4.6 × 75 mm Supelco LC8 column of 3 μm particle size. This mobile phase and column combination were also used to screen for the presence of NG.

RESULTS

The above procedures have proven effective in recovering and quantitating OB/OD residues in sandy loam soil (Table 2); they have the additional advantage of being simple and reproducible. However, several shortcomings were encountered. Efforts to identify some minor components of the OB/OD soil contaminant mixture were not successful due to interferences from natural soil components. Although the majority of UV-absorbing soil components elute from reverse phase chromatography before most explosives, some elute at later retention times causing a rough baseline at high sensitivities thereby making quantitation of extremely small peaks unreliable.

The gradient procedure presented here effectively separated components of a mixture that included most compounds likely to be encountered during analysis of soils from OB/OD contaminated sites (Figure 1). It was able to detect many compounds that would otherwise be missed by previous methods, and produced sharp symmetrical elution peaks for all compounds tested. However, this chromatography required 90 min to complete, and could not be run as a routine procedure at a high sensitivity (for compounds < 1 mg l^{-1}) because of problems with baseline drift.

The isocratic HPLC method of Miyares and Jenkins proved effective in quantitating intact RDX, TNT, and DNTs (2,4-, and 2,6-dinitrotoluene) in water, acetonitrile, and methanol but performed less well with the aminodinitrotoluenes because they were later eluting and exhibited significant peak broadening (Figure 2). Peak

Table 2 Efficiencies of recovery of munition residues from soil and water, using an isocratic quantitation method[12]

Compound	*Percent recovered (%), ±s*		
	From soil extracted with acetonitrile		*From aqueous leachate concentrates in MeOH*
	doped uncontam.	*doped contam.*	
HMX	99 ± 6	112 ± 4	29 ± 10
TNB	102 ± 2	114 ± 3	123 ± 4
RDX	95 ± 1	91 ± 2	38 ± 1
TNT	107 ± 1	94 ± 9	90 ± 4
2,4-DNT	103 ± 1	110 ± 5	108 ± 7
2,6-DNT	103 ± 1	103 ± 2	104 ± 20
2-Amino-DNT	100 ± <1	103 ± 1	112 ± 15
4-Amino-DNT	98 ± 3	102 ± 4	137 ± 40

1 Nitroguanidine (NQ)
2 2,4,6-Trinitrophenol (Picric acid)
3 1-Acetyloctahydro-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX)
4 Cyclotetramethylenetetranitramine (HMX)
5 1-Acetylhexahydro-3,5-dinitro-1,3,5-triazine (TAX)
6 Cyclotrimethylenetrinitramine (RDX)
7 1,3,5-Trinitrobenzene (TNB)
8 1,3-Dinitrobenzene (DNB)
9 2,4,6-Trinitrophenylmethylnitramine (Tetryl)
10 2,4,6-Trinitrotoluene (TNT)
11 4-Amino-2,6-dinitrotoluene (4-Amino-DNT)
12 2-Amino-4,6-dinitrotoluene (2-Amino-DNT)
13 2,6-Dinitrotoluene (2,6-DNT)
14 2,4-Dinitrotoluene (2,4-DNT)
15 Naphthalene
16 Acenaphthylene
17 Fluorene
18 Phenanthrene
19 Anthracene
20 Fluoranthrene
21 Pyrene
22 Benz(a)anthracene
23 Chrysene
24 Benzo(a)pyrene

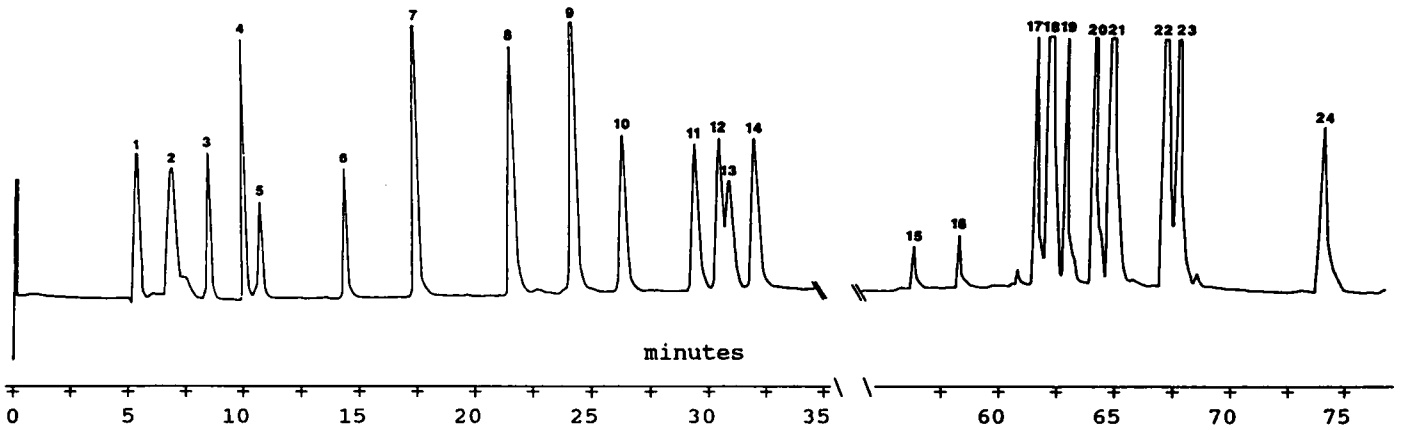

Figure 1 HPLC chromatogram showing the separation of a series of munition residues, environmental degradation products of explosives, and PAHs, using the gradient chromatographic (screening) method.

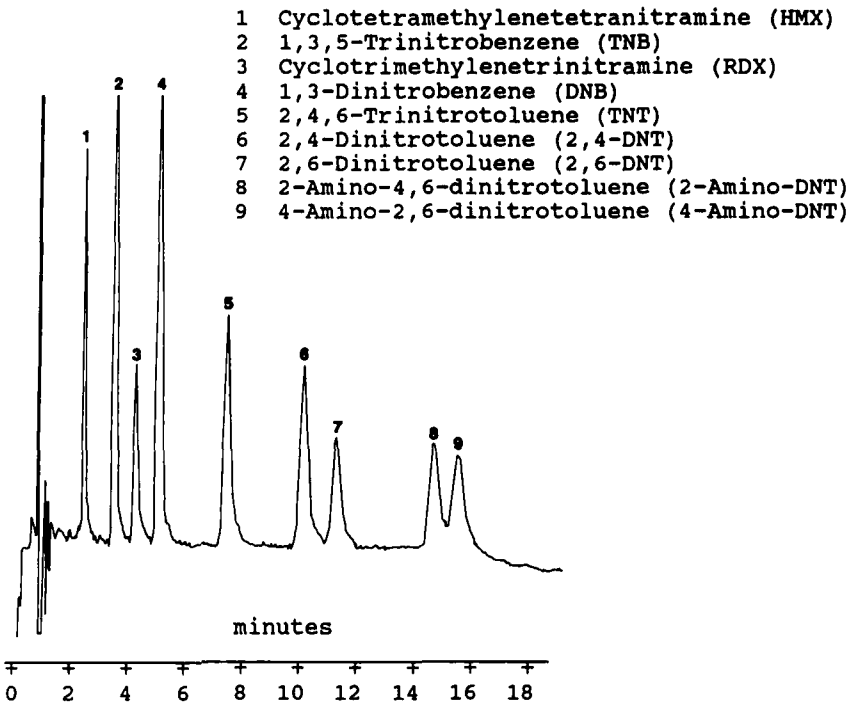

Figure 2 An example of the separation of a series of munition residues and associated co-contaminants, by the isocratic HPLC method.[12]

broadening caused problems with quantitation because it caused erratic start times during electronic integration of peak areas. We also observed that this solvent and column combination was unusually sensitive to temperature. At room temperatures the large negative absorbance peak from acetonitrile interfered with the quantitation of HMX. At temperatures above 23°C retention times were shortened, and at 30°C the system no longer resolved the two aminodinitrotoluenes.

Recovery of explosives doped into uncontaminated soil were nearly quantitative (Table 2); adjustments of recoveries due to gain or loss of the DNB internal standard were insignificant. Conversely, recoveries from the soil and water after leaching experiments ranged from 20–25% for TNT, 2–5% for 2,4-DNT, and even less for 2,6-DNT. Due to these low recoveries from the leached soils, the concentrations of explosives in soil extracts, and in aqueous leachates, were often diminished to levels below our criteria of detection (Table 3). The criterion of detection is defined as the lowest certifiable limit for quantitation. The analytical detection values reported in Table 3 represent the power of the HPLC method alone. These criteria of detection do not include multipliers used to calculate concentrations in soil or water, nor do they include factors to account for preconcentration of the samples prior to analyses. The criteria of detection were calculated using the computerized Quality Assurance

Table 3 Criteria of detection[a] for quantitation of standards in acetonitrile, determined by HPLC using an isocratic quantitation method[12]

Compound	*mg* l^{-1}
HMX	0.15
TNB	0.15
RDX	0.067
DNB	0.15
TNT	0.093
2,4-DNT	0.17
2,6-DNT	0.37
2-Amino-DNT	0.14
4-Amino-DNT	0.12

[a] The x value on the standard regression line that has the same y value as y at $x = 0$ on the upper confidence limit (95% conf. level) curve[13].

Program of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA), based on the methods of Hubaux and Vos.[13–14] When a compound was identified but present at levels below the criteria of detection, it was termed to be a "trace" quantity.

Concentration of the OB/OD residues in soil extracts was attempted by evaporating the acetonitrile into a stream of dry nitrogen at 60°C. This procedure was not used because of significant losses of TNT, the DNTs, and some PAH compounds. Concentration by simple evaporation of the acetonitrile at ambient laboratory temperatures (20–25°C) was also unsuccessful due to unacceptable losses of these analytes.

Because of the very low concentration of explosives in most of the soil leachates, a procedure[15] developed by Brueggemann was used to concentrate munition residues from these solutions. This method entailed trapping residues with disposable C18 cartridges, followed by elution with methanol. It was found to be useful for the nitroaromatics, however recoveries of the nitramine compounds were substantially lower (Table 2). Passage of volumes of leachate greater than 30 mL had the effect of rinsing a portion of the nitramines from the cartridge. In addition, this method concentrated the naturally occurring water-soluble soil components that interfered with determination of TNB and HMX. An alternative procedure for concentrating nitramines from aqueous samples is that of Richard and Junk,[16] who reported that nitramines can be trapped efficiently using vinyl-divinyl benzene resins. Quantitation of TNB in acetonitrile extracts of soil was occasionally limited by background absorption due to unidentified material co-extracted from contaminated soils but methanol concentrates of the aqueous leachates consistently suffered from this problem.

DISCUSSION

PAH compounds have been detected in the waste products from munition incinerators, but detectable quantities of these compounds were not found in the ash or soils from the three OB/OD sites we investigated thus far. A possible explanation for this difference is that the high energy intermediates that are responsible for ring fusion may accumulate at higher concentrations in contained combustion. PAHs may arise from the explosives themselves or from residual petroleum products associated with shell casings and bursting devices. Thus, the possibility of PAH production in conjunction with OB/OD activities is suspect but cannot be ruled out at this time.

TNT remaining exposed on the surface at OB/OD sites is converted to TNB, with the latter's concentration often exceeding that of the parent compound.[17] In many environments TNT is microbially degraded by reduction to aminodinitrotoluenes, and may also be transformed into phenolic compounds, and diazo forms.[3] Although the aromatic ring structure of these compounds is resistant to degradation, evidence exists of other environmental processes in which these compounds may become strongly bound to soil.[3] The internal standard selected for this investigation was 1,3-dinitrobenzene, chosen primarily for its similarity to the analytes under study. However, caution is recommended in selecting an internal standard. The internal standard should be selected only after a thorough screening of the site has been completed to ensure that the preferred compound is not already present as a pollutant.

Unlike the nitroaromatics, nitramine munitions and NQ undergo reactions which may leave little trace of the original compound.[3,17] Therefore it is not surprising that recovery of munitions from soil is generally poor, both in the on-site environment and in soils under simulated field conditions. Green *et al.*[18] were able to recover only a small fraction of added TNT after soil columns were leached. Banwart and Hassett[19] found that TNT extracted from soil declined from 2000 mg kg^{-1} to < 20 mg kg^{-1} when the soil was amended with straw and used to grow plants for ninety days.

The time dependent disappearance of explosive-residues in the environment may very well be due to covalent or other non-equilibrium bonding to natural soil components. This bonding should be considered separately from the equilibrium partitioning of explosives between soil and water, and between soil and organic solvent. Therefore, experiments in which explosives are amended to soil, air-dried, then immediately extracted test the "potential" efficiency of the extraction process, rather than indicate the performance of the system with weathered samples.

Several factors affected our choice to use a screening procedure for OB/OD residues in field samples, prior to quantitation. Characterizing the specific compounds that contaminate an OB/OD site, and measuring their movement within the soil, requires numerous accurate analyses. OB/OD residues found in high concentration at the soil surface typically decrease in concentration with depth, thus the concentration of explosives in extracts of subsurface soil samples, and aqueous leachates, may be quite low. Furthermore OB/OD sites differ, both in the explosives that are present, and in

their soil types which contain diverse natural compounds that interfere with analyses. For these reasons, the use of separate HPLC procedures for screening and for quantitation is essential.

When an accurate characterization of OB/OD residues for a given site is completed using the gradient screening procedure, an isocratic method with sufficient sensitivity and resolution is selected from the literature or developed, and optimized for the local conditions. The column and mobile phase selected should provide a quick isocratic separation while avoiding co-elution of OB/OD residues with UV-absorbing soil components, and also produce sufficiently sharp and symmetrical chromatographic peaks for successful electronic quantitation.

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Disclaimer

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