

## METHOD 8330

### NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

This method is intended for the analysis of explosives residues. This Method is limited to use by analysts experienced in handling and analyzing explosive residues.

#### 1.0 SCOPE AND APPLICATION

1.1 Method 8330 is used to determine the concentration of the following compounds in a water, soil or sediment matrix:

Compounds	Abbrev.	CAS No. <sup>a</sup>
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	1,3-DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
2-Amino-4,6-dinitrotoluene	2-Am-DNT	355-72-78-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2-Nitrotoluene	2-NT	88-72-2
4-Nitrotoluene	4-NT	99-99-0
3-Nitrotoluene	3-NT	99-08-1

a Chemical Abstracts Service Registry number

1.2 Method 8330 provides a salting-out extraction procedure for low concentration (parts per trillion or nanograms per liter) of explosives residues in surface or ground water. Direct injection of diluted and filtered water samples can be used for water samples of higher concentration (See Table 1).

1.3 All of these compounds are either used in the manufacture of explosives or are the degradation products of

compounds used for that purpose. When making stock solutions for calibration, treat each compound as if it were extremely explosive.

1.4 The practical quantitation limits (PQLs) of target analytes determined by Method 8330 in water and soil are presented in Table 1.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling explosive materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

2.1 Method 8330 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of certain explosives residues in water, soil and sediment matrix. Prior to use of this method, appropriate sample preparation techniques must be used.

2.2 Low-Level Salting-out Method: Aqueous samples of low concentration are concentrated by a salting-out extraction procedure with acetonitrile and sodium chloride. The acetonitrile extract is further concentrated to less than 1.0 mL using a Kuderna-Danish evaporator and brought to 1.0 mL using acetonitrile. The concentrated extract is diluted with 3.0 mL of reagent grade water, filtered, separated on a C-18 reverse phase column, determined at 254 nm, and confirmed on a CN reverse phase column.

2.3 High-Level Direct Injection Method: Aqueous samples of higher concentration can be diluted 1/1 (v/v) with methanol or acetonitrile, filtered, separated on a C-18 reverse phase column, determined at 254 nm, and confirmed on a CN reverse phase column. If HMX is an important target analyte, methanol is preferred.

2.4 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered and chromatographed as in Section 2.3.

## 3.0 INTERFERENCES

3.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from

interferences, under the conditions of the analysis, by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.1 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.

3.2 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

3.3 Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

#### 4.0 APPARATUS AND MATERIALS

##### 4.1 HPLC system

4.1.1 HPLC - equipped with a pump capable of achieving 4000 psi, a 100- $\mu$ L loop injector and a 254-nm UV detector (Perkin Elmer Series 3 or equivalent).

4.1.2 C-18 Reverse phase HPLC column, 25-cm x 4.6-mm (5  $\mu$ m), (Supelco LC-18 or equivalent).

4.1.3 CN Reverse phase HPLC column, 25-cm x 4.6-mm (5  $\mu$ m), (Supelco LC-CN or equivalent).

4.1.4 Strip chart recorder.

4.1.5 Digital integrator (optional).

4.1.6 Autosampler (optional).

##### 4.2 Other Equipment

4.2.1 Temperature controlled ultrasonic bath.

4.2.2 Vortex mixer.

4.2.3 Kuderna-Danish evaporator - 40 mL, micro Kuderna-Danish evaporator (Supelco #64718 or equivalent).



4.2.4 Water bath - Heated, with concentric ring cover, capable of temperature control ( $\pm 5^{\circ}\text{C}$ ). The bath should be used in a hood.

4.2.5 Balance -  $\pm 0.1$  mg.

#### 4.3 Materials

4.3.1 High pressure injection syringe - 500  $\mu\text{L}$ , (Hamilton liquid syringe or equivalent).

4.3.2 Disposable cartridge filters - 0.45  $\mu\text{m}$  Teflon filter.

4.3.3 Pipettes - 50 mL, 10 mL, 5 mL, 4 mL, 2 mL, 1 mL, volumetric, Class A, glass.

4.3.4 Pasteur pipettes.

4.3.5 Scintillation Vials - 20 mL, glass.

4.3.6 Vials - 15 mL, glass, Teflon-lined cap.

4.3.7 Vials - 40 mL, glass, Teflon-lined cap.

4.3.8 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.

4.3.9 Separatory funnel - 500 mL.

4.3.10 Volumetric flasks - 10 mL, 20 mL, 50 mL, 100 mL, 200 mL and 250 mL.

4.3.11 Vacuum desiccator - Glass.

4.3.12 Mortar and pestle - Steel.

4.3.13 Boiling chips - Solvent extracted, approximately 10/40 mesh (Teflon or equivalent).

4.3.14 Sieve - 30 mesh.

4.3.15 Oven - Forced air, without heating.

#### 4.4 Preparation

4.4.1 Prepare all materials to be used as described in Chapter 4 for semivolatile organics.



## 5.0 REAGENTS

5.1 HPLC grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

### 5.2 General

- 5.2.1 HMX - Standard Analytical Reference Material.
- 5.2.2 RDX - Standard Analytical Reference Material.
- 5.3.2 1,3-DNB - Standard Analytical Reference Material.
- 5.2.4 Tetryl - Standard Analytical Reference Material.
- 5.2.5 2,4,6-TNT - Standard Analytical Reference Material.
- 5.2.6 2-Am-DNT -
- 5.2.7 4-Am-DNT - Reagent grade (Aldrich Chemical or equivalent).
- 5.2.8 2,4-DNT - Standard Analytical Reference Material.
- 5.2.9 2,6-DNT - Standard Analytical Reference Material.
- 5.2.10 1,3,5-TNB - Standard Analytical Reference Material.
- 5.2.11 NB - Standard Analytical Reference Material.
- 5.2.12 2-NT - Reagent grade.
- 5.2.13 3-NT - Reagent grade.
- 5.2.14 4-NT - Reagent grade.
- 5.2.15 Reagent water - All references to water in this method refer to water in which an interference is not observed at the method detection limit of the compounds of interest. Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

5.2.16 Acetonitrile - HPLC grade.

5.2.17 Methanol - HPLC grade, distilled in glass.

5.2.18 Sodium Chloride, NaCl - Reagent grade. If possible use NaCl from glass bottles. High background levels have been observed from NaCl shipped in plastic containers.

5.2.19 Calcium Chloride,  $\text{CaCl}_2$  - Reagent grade. Prepare an aqueous solution of 5 g/L.

### 5.3 Stock Standard Solutions

5.3.1 Dry each analyte standard to constant weight in a vacuum desiccator in the dark. Place about 100 mg (weighed to the nearest 0.1 mg) of a single analyte into a 100-mL volumetric flask and dilute to volume with acetonitrile. Invert flask several times until dissolved. Store in refrigerator at 4°C in the dark. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1,000 mg/L). Stock solutions may be used for up to one year.

### 5.4 Intermediate Standards Solutions

5.4.1 If both 2,4-DNT and 2,6-DNT are to be determined, prepare two separate intermediate stock solutions containing (1) HMX, RDX, 1,3,5-TNB, 1,3-DNB, NB, 2,4,6-TNT, 2,4-DNT and 2-Am-DNT and (2) tetryl, 2,6-DNT, 4-Am-DNT, 2-NT and 4-NT. Dilute the intermediate stock standard solutions to prepare two solutions at 1,000 µg/L in acetonitrile.

5.4.2 Dilute the two intermediate stock concentrate solutions with acetonitrile to prepare intermediate standard solutions that cover the range of 2.5 - 1,000 µg/L. These solutions should be refrigerated on preparation and stored in the dark, and may be used for 30 days.

5.4.3 For the low-level method, the analyst must conduct a detection limit study and devise dilution series appropriate to the desired range. Standards for the low level method must be prepared immediately prior to use.

### 5.5 Working Standards

5.5.1 Calibration standards at a minimum of five concentration levels should be prepared through dilution of the intermediate standards solutions by 50% (v/v) with 5 g/L calcium chloride solution (Section 5.2.19). These solutions

must be refrigerated and stored in the dark, and prepared fresh on the day of calibration.

## 5.6 Surrogate Standards

5.6.1 The analyst should monitor the performance of the extraction and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with one or two surrogates (e.g., analytes not expected to be present in the sample).

## 5.7 Eluent

5.7.1 To prepare 1 liter of eluent, add 500 mL of methanol to 500 mL of reagent water.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

6.2 Samples must be kept below 4°C and in the dark from the time of collection through analysis, except during drying.

6.3 Soil and sediment samples should be air dried to constant weight at room temperature or colder after collection.

6.4 All water samples must be extracted within 7 days of collection and analyzed within 40 days after extraction. All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction.

## 7.0 PROCEDURE

### 7.1 Sample Preparation

7.1.1 Aqueous Samples: It is highly recommended that all samples of this type be screened with the high-level method (>50 µg/L) to determine if the low-level method (1-50 µg/L) is required.

#### 7.1.1.1 Low-Level Method (salting-out extraction)

7.1.1.1.1 Place a 400 mL aliquot of water sample in a 500 mL separatory funnel and add 130 g of NaCl. Vigorously shake the sample until all of the NaCl is completely dissolved. Be sure to



dissolve all salt before adding acetonitrile, or the dissolution process takes much longer.

7.1.1.1.2 Add a 100 mL volume of acetonitrile using a glass volumetric pipette. Shake the separatory funnel vigorously for 5 minutes. Allow the funnel to stand undisturbed for 30 minutes while the two phases separate. Discard the water (lower) layer and collect the acetonitrile (upper) layer (approximately 23 mL) in a 40 mL Teflon-capped vial. Rinse the separatory funnel with 5 mL of acetonitrile and add the rinsate to the extract.

7.1.1.1.3 If the collected sample was turbid, centrifuge the 40 mL vial at 4000 rpm's for 5 minutes. Remove the acetonitrile (upper) layer with a Pasteur pipette and transfer it to a clean vial.

7.1.1.1.4 Reduce the acetonitrile extract to less than 1.0 mL using a Kuderna-Danish evaporator and bring the total volume to 1.0 mL using acetonitrile. Dilute this concentrated extract with 3.0 mL of reagent water.

7.1.1.1.5 Filter the diluted extract through a 0.45- $\mu$ m Teflon filter. Discard the first 0.5 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4.

#### 7.1.1.2 High-Level Method

7.1.1.2.1 Sample filtration: Place a 5 mL aliquot of each water sample in a scintillation vial, add 5 mL of acetonitrile, shake thoroughly, and filter through a 0.45- $\mu$ m Teflon filter. Discard the first 3 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration.

#### 7.1.2 Soil and Sediment Samples

7.1.2.1 Sample homogenization: Dry soil samples in air at room temperature or colder, being careful not to expose the samples to direct sunlight. Grind and

homogenize the dried sample thoroughly in an acetonitrile rinsed mortar to pass a 30 mesh sieve.

#### 7.1.2.2 Sample extraction

7.1.2.2.1 Place a 2.0 g subsample of each soil sample in a 15 mL glass vial. Add 10.0 mL of acetonitrile, cap with Teflon-lined cap, vortex swirl for one minute, and place in an cooled ultrasonic bath for 18 hours.

7.1.2.2.2 After sonication, allow sample to settle for 30 minutes. Remove 5.0 mL of supernatant, and combine with 5.0 mL of calcium chloride solution (Section 5.2.19) in a 20 mL vial. Shake, and let stand for 15 minutes.

7.1.2.2.3 Place supernatant in a disposable syringe and filter through a 0.45- $\mu$ m Teflon filter. Discard first 3 mL and retain remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4.

### 7.2 Chromatographic Conditions

Primary Column:	C-18 reverse phase HPLC column, 25-cm x 4.6-mm, 5 $\mu$ m, (Supelco LC-18 or equivalent).
Secondary Column:	CN reverse phase HPLC column, 25-cm x 4.6-mm, 5 $\mu$ m, (Supelco LC-CN or equivalent).
Mobile Phase:	50/50 (v/v) methanol/organic-free reagent water.
Flow Rate:	1.5 mL/min
Injection volume:	100- $\mu$ L
UV Detector:	254 nm

### 7.3 Calibration of HPLC

7.3.1 All electronic equipment is allowed to warm up for 30 minutes. During this period, at least 15 void volumes of mobile phase are passed through the column (approximately 20 min at 1.5 mL/min) and continued until the baseline is level at the UV detector's greatest sensitivity.

7.3.2 Analyze working standards in triplicate, using the chromatographic conditions given in Section 7.2. Prepare calibration curve using peak heights or peak areas, as appropriate. The calibration curve should be linear with zero intercept.

7.3.3 Initial Calibration. Triplicate injections of each calibration standard over the concentration range of interest are sequentially injected into the HPLC in random order. Peak heights or peak areas are obtained for each analyte. Experience indicates that a linear calibration curve with zero intercept is appropriate for each analyte. Therefore, a response factor for each analyte can be taken as the slope of the best-fit regression line.

7.3.4 Daily Calibration. Analyze midpoint calibration standards, at a minimum, in triplicate at the beginning of the day, singly at the midpoint of the run and singly after the last sample of the day. Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within  $\pm 25\%$  of the response factor of the initial calibration for the first seven daily calibrations and within two standard deviations of the initial calibration for subsequent calibrations. If this criterion is not met, a new initial calibration must be obtained.

#### 7.4 HPLC Analysis

7.4.1 Analyze the samples using the chromatographic conditions given in Section 7.2. All positive measurements observed on the C-18 column must be confirmed by injection onto the CN column.

7.4.2 In limited applications (e.g., aqueous process wastes) direct injection of filtered and diluted sample into the HPLC system with a 100- $\mu$ L loop may be appropriate. The quantitation limits are high, therefore, it is only permitted where concentrations in excess of 50  $\mu$ g/L are expected.

7.4.3 Follow Section 7.6 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence. If column temperature control is not employed, special care must be taken to ensure that temperature shifts do not cause peak misidentification.



7.4.4 Table 2 summarizes the estimated retention times on both C-18 and CN columns for a number of analytes analyzable using this method. An example of the separation achieved by Column 1 is shown in Figure 1.

7.4.5 Record the resulting peak sizes in peak heights or area units. The use of peak heights is recommended to improve reproducibility of low level samples.

7.4.6 Calculation of concentration is covered in Section 7.8 of Method 8000.

7.4.7 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second HPLC column is required.

## 8.0 QUALITY CONTROL

8.1 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be run to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

8.2 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500.

8.3 Mandatory quality control to validate the HPLC system operation is found in Method 8000, Section 8.6.

8.4 The laboratory must, on an ongoing basis, analyze a method blank, a matrix spike, and a matrix spike duplicate/duplicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.5 A minimum of one duplicate sample shall be run with each analytical batch. If the samples are generally non-detect samples, a matrix spike duplicate must be run with the analytical batch.

### 8.6 Method Blanks

8.6.1 Method blanks for the analysis of aqueous samples should be reagent water carried through all sample storage, preparation and handling procedures.

8.6.2 Method blanks for the analysis of soil samples should be uncontaminated soil carried through all sample storage, extraction, and handling procedures.

## 9.0 METHOD PERFORMANCE

9.1 Method 8330 was tested by six laboratories. The results of this testing indicate that the results presented in Tables 3 through 5 are to be expected.

## 10.0 REFERENCES

1. Bauer, C.F., T.F. Jenkins, S.M. Koza, P.W. Schumacher, P.H. Miyares and M.E. Walsh (1989). Development of an analytical method for the determination of explosive residues in soil. Part 3. Collaborative test results and final performance evaluation. USA Cold Regions Research and Engineering Laboratory, CRREL Report 89-9.
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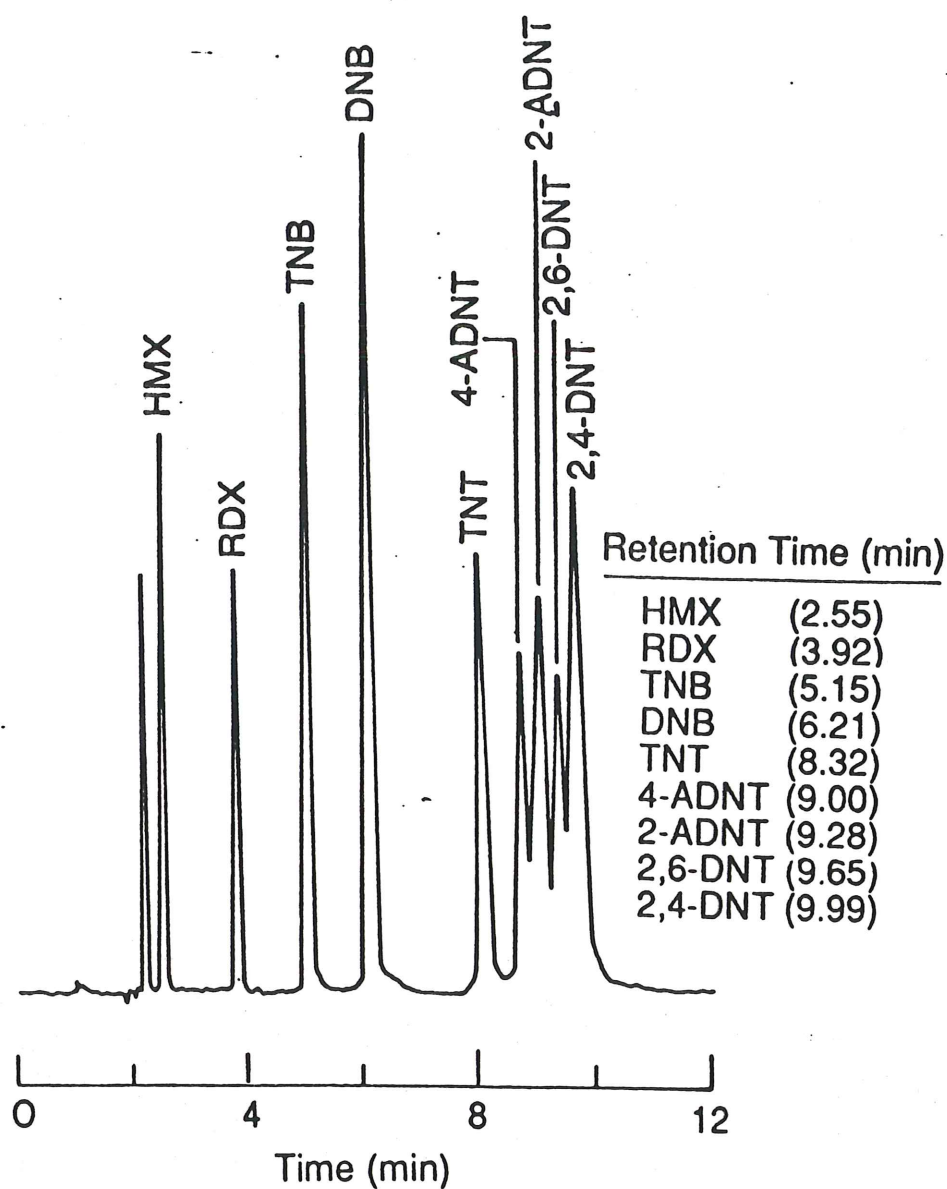
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#### 11.0 SAFETY

11.1 Standard precautionary measures used for handling other organic compounds should be sufficient for safe handling of the analytes targeted by Method 8330.





**Figure 3.** Separation for the direct injection RP-HPLC method (Jenkins et al. 1988a).

**Column:** LC-18 (25 cm  $\times$  4.6 mm, 5  $\mu$ m).

**Eluent:** 50/50 (v/v) MeOH/H<sub>2</sub>O.

**Flow:** 1.5 mL/min.

**$\lambda$ :** 254 nm.

Column: C-18 (25 cm x 4.6 mm, 5  $\mu$ m)  
Mobile Phase: 1/1 (v/v) Methanol/Water, 1.5 mL/min  
Detector: UV

Figure 1. Liquid Chromatogram of Explosives Residues.

TABLE 1  
PRACTICAL QUANTITATION LIMITS

Compounds	Water ( $\mu\text{g/L}$ )		Soil ( $\mu\text{g/g}$ )
	Low-Level	High-Level	
HMX	-	13.0	2.2
RDX	0.836	14.0	1.0
1,3,5-TNB	0.258	7.3	0.25
1,3-DNB	0.108	4.0	0.25
Tetryl	-	4.0	0.65
NB	-	6.4	0.26
2,4,6-TNT	0.113	6.9	0.25
4-Am-DNT	0.0598	-	-
2-Am-DNT	0.0349	-	-
2,6-DNT	0.314	9.4	0.26
2,4-DNT	0.0205	5.7	0.25
2-NT	-	12.0	0.25
4-NT	-	8.5	0.25
3-NT	-	7.9	0.25

TABLE 2  
RETENTION TIMES FOR ANALYTES ON C-18 AND CN COLUMNS

C-18		CN	
Compounds	Retention Time (min)	Compounds	Retention Time (min)
HMX	2.4	NB	3.8
RDX	3.7	1,3,5-TNB	4.1
1,3,5-TNB	5.1	1,3-DNB	4.2
1,3-DNB	6.2	2-NT	4.4
Tetryl	6.9	4-NT	4.4
NB	7.2	3-NT	4.5
2,4,6-TNT	8.4	2,6-DNT	4.6
2,6-DNT	9.8	2,4-DNT	4.9
2,4-DNT	10.1	2,4,6-TNT	5.0
2-NT	12.3	RDX	6.2
4-NT	13.3	Tetryl	7.4
3-NT	14.2	HMX	8.4



TABLE 3  
INTRALABORATORY PRECISION OF METHOD FOR SOIL SAMPLES

	Spiked Soils			Field-Contaminated Soils		
	Mean Conc. ( $\mu\text{g/g}$ )	SD	%rsd	Mean Conc. ( $\mu\text{g/g}$ )	SD	%rsd
HMX	46	1.7	3.7	14	1.8	12.8
				153	21.6	14.1
RDX	60	1.4	2.3	104	12	11.5
				877	29.6	3.4
1,3,5-TNB	8.6	0.4	4.6	2.8	0.2	7.1
	46	1.9	4.1	72	6.0	8.3
1,3-DNB	3.5	0.14	4.0	1.1	0.11	9.8
Tetryl	17	3.1	17.9	2.3	0.41	18.0
TNT	40	1.4	3.5	7.0	0.61	9.0
				669	55	8.2
2,4-DNT	5.0	0.17	3.4	1.0	0.44	42.3

TABLE 4  
INTRALABORATORY ERROR OF METHOD FOR SOIL SAMPLES

	Spiked Soils			Field-Contaminated Soils		
	Mean Conc. ( $\mu\text{g/g}$ )	SD	%rsd	Mean Conc. ( $\mu\text{g/g}$ )	SD	%rsd
HMX	46	2.6	5.7	14	3.7	26.0
				153	37.3	24.0
RDX	60	2.6	4.4	104	17.4	17.0
				877	67.3	7.7
1,3,5-TNB	8.6	0.61	7.1	2.8	0.23	8.2
	46	2.97	6.5	72	8.8	12.2
1,3-DNB	3.5	0.24	6.9	1.1	0.16	14.5
Tetryl	17	5.22	30.7	2.3	0.49	21.3
TNT	40	1.88	4.7	7.0	1.27	18.0
				669	63.4	9.5
2,4-DNT	5.0	0.22	4.4	1.0	0.74	74.0

TABLE 5  
INTERLABORATORY VARIANCE OF METHOD FOR WATER SAMPLES<sup>a</sup>

Compounds	Mean Conc. ( $\mu\text{g/L}$ )	SD	%rsd
HMX	203	14.8	7.3
RDX	274	20.8	7.6
2,4-DNT	107	7.7	7.2
2,4,6-TNT	107	11.1	10.4

a Nine laboratories.