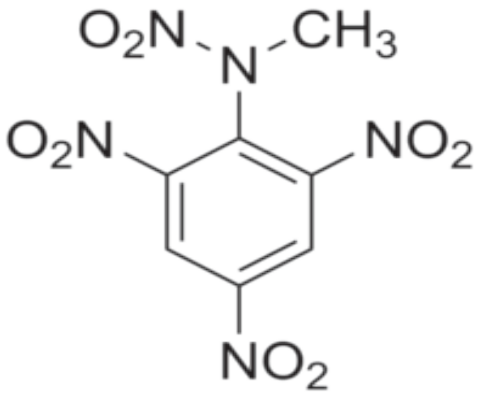
EPA Method 8330B: Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) Aqueous Matrices



UCT Part Numbers

ECDVB156P
Enviro-Clean® DVB 500 mg, 6 mL cartridge, PE Frit

ECHLD156-P
Enviro-Clean® HL DVB 500 mg, 6 mL cartridge, PE Frit

RCRA Compounds Using This Method

Table 1

Analyte	CAS	Abbreviation	% Recovery n=3
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	2691-41-0	HMX	100
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	RDX	110
1,3,5-Trinitrobenzene	99-35-4	1,3,5-TNB	100
1,3-Dinitrobenzene	99-65-0	1,3-DNB	100
1,4-Dinitrobenzene	10025-4	1,4-DNB	97
Methy-2,4,6-trinitrophenylnitramine	47945-8	Tetryl	85
Nitrobenzene	98-95-3	NB	100
2,4,6-Trinitrotoluene	118-96-7	TNT	94
4-Amino-2,6-dinitrotoluene	19406-51-0	4-Am-DNT	120
2-Amino-2,6-dinitrotoluene	35572-78-2	2-Am-DNT	110
2,4-Dinitrotoluene	121-14-2	2,4-DNT	98
2,6-Dinitrotoluene	606-20-2	2,6-DNT	110
2-Nitrotoluene	88-72-2	2-NT	90
3-Nitrotoluene	99-08-1	3-NT	91
4-Nitrotoluene	99-99-0	4-NT	92
Nitroglycerin	55-63-0	NG	100
Pentaerythritol tetranitrate	78-11-5	PETN	100
3,5-Dinitroaniline	618-87-1	3,5-DNA	68
1-Nitronaphthalene	86-57-7	NN	97
o-Dinitrobenzene	528-29-0	o-NB	100

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Safety:

- Extra caution that should be taken when handling the analytical standard neat material to prevent detonation.

Interferences:

- 2, 4-DNT and 2, 6-DNT elute at similar retention times on C18 columns using method separation conditions. If it is not apparent that both isomers are present or are not detected an isomeric mixture should be reported.

- Tetryl is thermally labile (decomposed with heat at temperature above room temperature) and decomposes in methanol/water solutions. All aqueous samples expected to contain tetryl should be diluted with acetonitrile and acidified with sodium bisulfate to pH <3 prior to filtration.

- Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak when using C18 columns.

Note: All samples should be stored at 2 °C to 4 °C prior to extraction and should be extracted within 14 days of collection

Sample Preparation--Aqueous matrices, (e.g. water) (from Method 3535)
Important Notes:

- Any particulate matter in the original sample must be included in the sample aliquot that is extracted.

- The sample container must be rinsed with solvent as the majority of organic analytes are hydrophobic and may adhere to the sample container surfaces.

- Do not concentrate explosives residue to dryness as they may **DETONATE.**

- For explosives and nitramines or nitroaromatics the extraction pH should be as received in the sample.

- Using a graduated cylinder, measure 1 liter of sample water. A smaller sample size may be used when analytical sensitivity is not a concern.

- Add methanol (ACN if tetryl is being analyzed) so that the sample is 0.5% v/v. Add surrogate standards to all samples and blanks.

- Add matrix spikes standards to representative sample replicates.

- Adjustment of sample pH may result in precipitation or flocculation reactions and potentially remove analytes from the aqueous portion. Transfer the precipitate with rinses to the SPE extraction cartridge.

- Do not let the cartridge dry out after cartridge conditioning with acetonitrile (ACN).

Glass Apparatus and SPE Cartridge Washing

Analyte	1st Solvent Wash	2nd Solvent Wash	3rd Solvent Wash
Explosives	5 mL acetone	15 mL isopropanol	15 mL methanol
Nitramines, Nitroaromatics	5 mL ACN	15 mL ACN	

1. Cartridge Conditioning

a) Follow the 4 steps in the table below for solvent quantities and soak times.

Analyte	Condition Step 1	Step 2	Step 3	Step 4
Explosives	20 mL ACN, 3 min*	20 mL ACN	50 mL DI water	50 mL DI water
Nitramines, Nitroaromatics	15 mL ACN, 3 min*	30 mL DI water	----	----

b) Draw solvents through the cartridge under low vacuum.

Do not let the cartridge dry out once cartridge is conditioned. This will affect analyte recovery.

2. Sample Extraction

a) Add the contents of the sample bottle to the cartridge.

b) Adjust vacuum to about 10-15 mm Hg to obtain a uniform flow rate of about 10 ml per minute.

c) After all the sample is drawn through, draw air through the cartridge for 15 minutes to dry it.

d) Do not dry for longer than 20 minutes as lower recovery may result.

3. Cartridge Elution

a) Insert a collection tube in the base of the vacuum manifold.

For Explosives

b) Add 4 mL of ACN and soak for 3 minutes.

c) Draw through using gravity flow or very low vacuum into a collection tube.

d) Store extract in freezer until analysis.

4. Extract Concentration (if necessary)

a) Concentrate the extract to 0.7 mL under a gentle stream of nitrogen in a warm bath at 40 °C.

b) Transfer the extract to a 1 mL volumetric flask.

c) Add internal standard for an extract concentration of 5 µg/mL.

d) Extract is now ready for analysis by HPLC.

RP-HPLC Columns for the Analysis of Explosive Residues	
Primary Columns	C-18 reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm C8 reversed-phase HPLC column, 15-cm x 3.9-mm, 4 µm
Secondary Columns	CN reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm Luna Phenyl-Hexyl reversed-phase HPLC column, 25-cm x 3.0-mm, 5 µm

Injection volume:	100 µL
UV Detector:	Dual 254 & 210 nm or Photodiode Array
Mobile phase:	For C18 & CN column, 50:50 methanol:water
Flow Rate:	1.5 mL/minute

Retention Times and Capacity Factors

Analyte	LC-18 RT minutes	LC-CN RT minutes
HMX	2.44	8.35
RDX	3.78	6.15
1,3,5-TNB	5.11	4.05
1,3-DNB	6.16	4.18
3,5-DNA	6.90	NA
Tetryl	6.93	7.36
NB	7.23	3.81
NG	7.74	6.00
2,4,6-TNT	8.42	5.00
4-Am-DNT	8.88	5.10
2-Am-DNT	9.12	5.65
2,6-DNT	9.82	4.61
2,4-DNT	10.05	4.87
2-NT	12.26	4.37
4-NT	13.26	4.41
PETN	14.10	10.10
3-NT	14.23	4.45

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References:

[1] *For complete details on Method 8330 "Nitroaromatics and Nitramines by High performance Liquid Chromatography" Revision 2 October 2006, the analyst is referred to: National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268 and Method 3550 Revision 0, December 1996

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