



ENVIRO-CLEAN[®] DVB Cartridge

Part Number: **ECDVB156**

EPA Method 8330B

September 16, 2009

The UCT ECDVB156 styrene divinylbenzene cartridge is designed to provide a new level of performance in solid-phase extraction for the analysis of **nitroaromatics, nitramines and nitrate ester compounds--explosive and explosive residue compounds**. With its high capture efficiency, fast flow and excellent dry times, laboratory throughput can be significantly improved using this solid-phase product

Product Benefits

- SPE using styrene divinylbenzene polymeric gel
- SPE has been shown to provide equal or superior results as compared to liquid-liquid extraction LLE*
- No hydrolysis of the solid-phase DVB
- Fast flow rates for rapid analyte capture
- Teflon frits in the cartridge eliminate particle fines yielding clean extracts
- Works well at all levels of analyte loading
- No Lot to Lot variability
- Excellent analytical reproducibility
- Packaged in metalized, sealed pouches to maintain product purity

Product Features

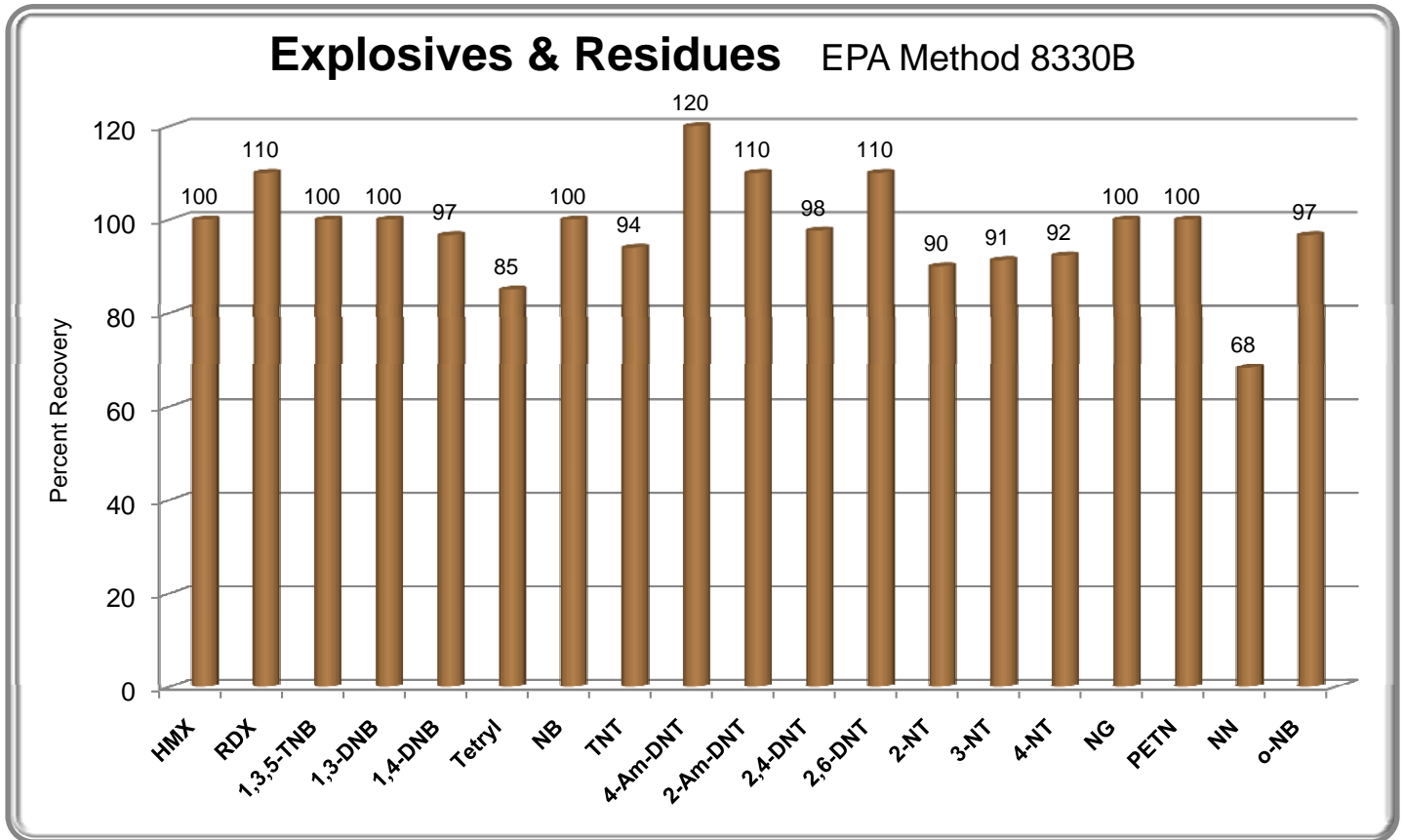
- 6 mL cartridge body manufactured from special polypropylene
- Each cartridge contains 500 mg of styrene divinylbenzene DVB sorbent
- Can be used on manual single or multi-station manifold systems
- Cartridges may be used with automated extraction systems

Nitroaromatics, Nitramines and Nitrate Ester Analytes and CAS Number

The following RCRA compounds have been determined by this method in water, soil & sediment matrices

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

Method 8330B Recovery Values



The UCT ECDVB156 styrene divinyl benzene cartridge for EPA Method 8330B shows excellent recovery of explosive and explosive residue analytes

Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)

Method 8330B

Method Summary

This method is used for the trace analysis (ppb) of explosive and propellant residues in water, soil and sediment matrices using high performance liquid chromatography (HPLC) and a dual wavelength UV or diode array detector. It is an updated method from 8330 promulgated September 1996. In this method aqueous samples are preconcentrated using the UCT styrene divinylbenzene SPE sorbent cartridge ECDVB156 as described in Method 3535 then eluted with acetonitrile or other appropriate solvent. The final extract is diluted with water as appropriate to bring the concentration into an analytical range suitable for HPLC analysis.

Interferences

- Solvents, reagents, glassware and other sample processing hardware may show interferences in sample analysis. All material must be demonstrated to be free from interferences under conditions of the analysis by analyzing method blanks
- 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° to 4° C prior to extraction and should be extracted within 14 days of collection

I Sample Preparation--Solid Matrices, (e.g. soil) (from Method 3535)

A soil sample is placed in a glass vial and dried with sodium sulfate. Acetonitrile (ACN) is added to the sample then mixed by vortex to suspend the soil in the solvent. The sample vial is then placed in a chilled ultrasonic bath for sonication. After 18 hours of sonication the sample is centrifuged for 15 to 20 minutes and the ACN solvent portion is removed from the vial. The volume of the removed aliquot is doubled by adding an equal volume of calcium chloride solution. The extract is then filtered through 1 μm Teflon filters.

1) Procedure (Ultrasonic Preparation)

- a) Weigh $2.0 \pm .04$ g of solid sample into a 25 mL glass vial
- b) Add 2 g of sodium sulfate and mix
- c) Add 0.1-mL explosives soil surrogate to all samples, blanks, and spikes
- d) Using a syringe or pipette, add 0.5 mL explosives spike to the LCS, LCSD if applicable, matrix spike, and matrix spike duplicate samples
- e) Using a graduated cylinder, add 10 mL of ACN and vortex swirl the sample for approximately 1 minute to suspend the soil in the ACN
 - a. Place the sample in a cooled ultrasonic bath ($<10^{\circ}\text{C}$). Make sure the water level in the sonicator is at least as deep as the level of solvent in the vial. Sonicate for 18 hours
 - b. After sonication, centrifuge the sample for 15 to 20 minutes to separate the solids from the solvent

2) Final Preparation

- a) Using a pipette, add 5 mL of a (5.0 gram/L) calcium chloride (CaCl_2) solution to a 10 mL volumetric flask
- b) Using a disposable pipette, bring to volume using the solvent layer of the centrifuged sample
- c) Mix thoroughly then allow the mixture to stand 15 minutes
- d) Filter the sample through 1 μm Teflon filters using a disposable syringe
- e) Discard the first 3 mL and retain the remainder in an appropriately labeled 12 mL vial
- f) Store in a refrigerator until analysis

II Sample Preparation--Aqueous matrices, (e.g. water) (from Method 3535)

A measured volume of the aqueous sample is adjusted to a specified pH then extracted using the UCT styrene divinylbenzene SPE sorbent cartridge **ECDVB156**. Two challenges are noted for aqueous sample preparation. First, any particulate matter in the original sample must be included in the sample aliquot that is extracted. Second, the sample container must be rinsed with solvent as the majority of organic analytes are hydrophobic and may adhere to the sample container surfaces.

Note

- 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 5.0 mL of methanol and surrogate standards to all samples and blanks
- Add matrix spikes standards to representative sample replicates

Note: Adjustment of sample pH may result in precipitation or flocculation reactions and potentially remove analytes from the aqueous portion. The analyst should note the formation of such precipitates or floc and transfer any such material with rinses to the SPE extraction cartridge. Do not let the cartridge dry out after cartridge conditioning with acetonitrile (ACN)

A. Glass Apparatus Washing:

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

Draw solvents through the cartridge under low vacuum

B. Cartridge Conditioning:

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		

*Soak time

Draw solvents through the cartridge under low vacuum

1) Initial Preparation

- a) Assemble a DVB extraction cartridge **UCT ECDVB156** in an all-glass manifold.
- b) Use of a manifold for multiple extractions or automated extraction equipment is acceptable

2) Cartridge Conditioning

- a) Follow the 4 steps in Table **Cartridge Conditioning for** solvent quantities

Do not let the cartridge dry out once the cartridge is conditioned as this may affect analyte recovery

b) 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 approximately 10 ml per minute. This will require about 1 hour for sample extraction
- 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

c) Cartridge Elution

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

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

Nitramines and Nitroaromatics

- b) Add 5 mL ACN, soak for 3 minutes
- c) Draw through using a gravity flow or very low vacuum into a collection tube
- d) Store extract in freezer until analysis

d) Extract Concentration

- a) Concentrate the extract to 0.7 mL under a gentle stream of nitrogen in a warm bath at 40^o C
- b) Transfer the extract to a 1 mL volumetric flask
- c) Add internal standard for a 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	<p>C-18 reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm</p> <p>C8 reversed-phase HPLC column, 15-cm x 3.9-mm, 4 µm</p>
Secondary Columns	<p>CN reversed-phase HPLC column, 25-cm x 4.6-mm, 5 µm</p> <p>Luna Phenyl-Hexyl reversed-phase HPLC column, 25-cm x 3.0-mm, 5 µm</p>

Injection volume: 100 µL

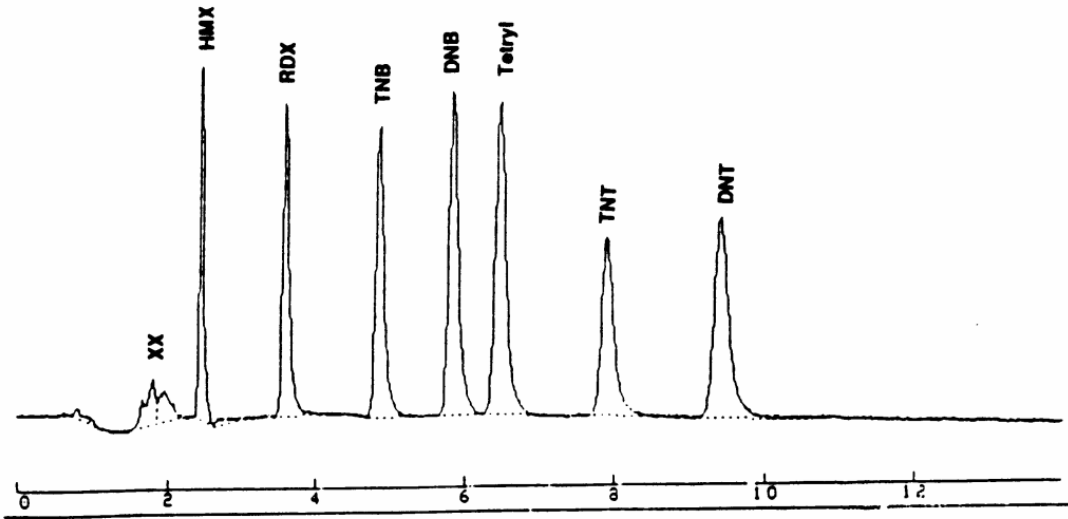
UV Detector: Dual 254nm & 210nm or Photodiode Array

Mobile phase: For C18 & CN column, 50:50 methanol:water

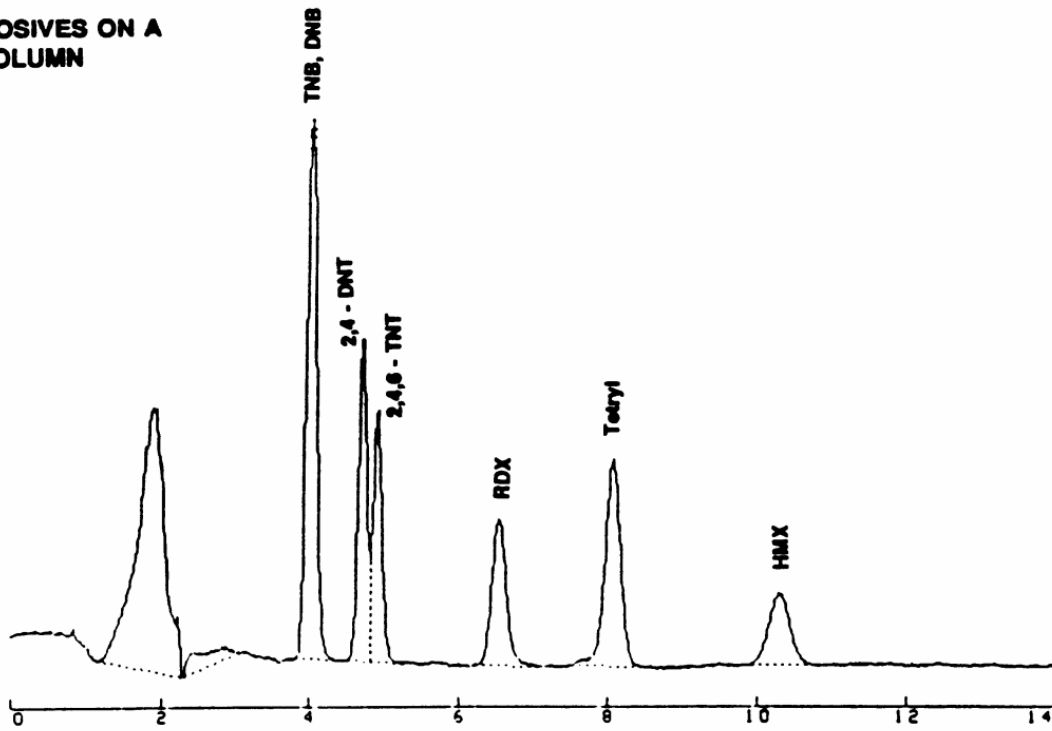
Energetic Compounds Currently Not Target Analytes of Method 8330

Compound	Symbol
picric acid (PA)/Ammonium picrate	AP
2,4-diamino-6-nitrotoluene	
2,6-diamino-4-nitrotoluene	
hexanitro-hexaazaisowurtzitane	CL-20
1,3,3-trinitroazetidine	TNAZ
hexahydro-1-nitroso-3,5-dinitro-1,3,5- triazine	MNX
hexahydro-1,3,5-trinitroso-1,3,5-triazine	TNX
nitrocellulose	NC
nitroguanidine	NQ
diphenylamine	DPA
n-nitroso-diphenylamine	NDPA
2-nitrodiphenylamine	
4-nitrodiphenylamine	
2,4-dinitrodiphenylamine	

**EXPLOSIVES ON A
C18 COLUMN**



**EXPLOSIVES ON A
CN COLUMN**



RETENTION TIMES AND CAPACITY FACTORS ON LC-18 AND LC-CN COLUMNS

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

*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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