Separate Explosives and Propellant Residues

Using Ultra C18 and Pinnacle™ II Biphenyl Columns

by Robert Freeman, Environmental Innovations Chemist

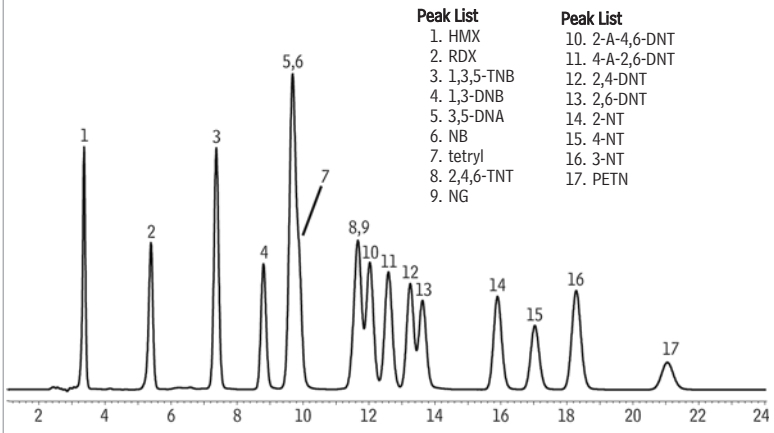
- Easily quantify and confirm new US EPA Method 8330B target analytes.
- Excellent resolution, improved accuracy.
- Simple, easy to use, isocratic method.

US EPA 8330, a test method for determining trace amounts of 14 nitramines and nitrate esters, was recently revised to include three new target analytes. The new method, EPA 8330B, includes nitroglycerin (NG), pentaerythritol tetranitrate, (PETN), and 3,5-dinitroaniline (3,5-DNA) and now covers 17 analytes that are commonly found in explosives and propellants residues. This method uses reversed phase HPLC and dual wavelength UV detection (210 & 254nm) in conjunction with a primary and a confirmation column.

We recently assessed the performance of our current column offerings relative to the elution order and retention times of the new analytes in the revised method. Separations on all columns were accomplished using a simple, isocratic water:methanol mobile phase (Table 1). The primary and confirmation columns that we recommend for the EPA 8330 analysis are the Ultra C18 and Pinnacle™ II Biphenyl columns, respectively. Based on this work, we conclude this combination will work well for the revised method, EPA 8330B, as shown by the chromatograms in Figures 1 and 2. Both columns provide excellent resolution of the EPA 8330B analytes and their differing selectivity provides a true confirmation analysis.

As an alternative to the Ultra C18/Pinnacle™ II Biphenyl combination, a Pinnacle™ II C18 column and a Pinnacle™ II Cyano column work well together as a primary-confirmation column set. Another column of interest is the Allure® Biphenyl column. A high organic mobile phase was required on this column but the analysis was completed in approximately six minutes (Table 1).

Figure 1 Excellent resolution of EPA 8330B target analytes on the Ultra C18 column.



Sample:	50µg/mL each compound diluted in acetonitrile 8330 Calibration Mix #1 (cat.# 31450) 8330 Calibration Mix #2 (cat.# 31451) PETN Standard(cat.# 31600) 3,5-dinitroaniline Reference Mix (cat.# 31661) Nitroglycerin Reference Mix (cat.# 31498)	**Conditions:** Mobile phase: Flow: Temp.: Det.:	 water:methanol (44:56 v/v) 1.0mL/min. 30°C UV detection @ 210nm
Inj.:	10µL		
Column:	**Ultra C18**		
Cat.#:	9174575		
Dimensions:	250mm x 4.6mm		
Particle size:	5µm		
Pore size:	100Å		

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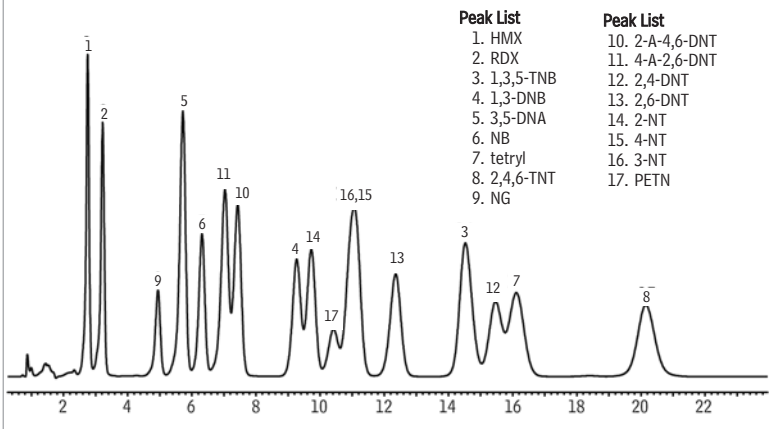
Figure 2 Alternate selectivity of EPA 8330B analytes on the Pinnacle™ II Biphenyl column confirms compound identity.



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Inj.:	10µL		
Column:	**Pinnacle™ II Biphenyl**		
Cat.#:	9209565-700		
Dimensions:	250mm x 4.6mm		
Particle size:	5µm		
Pore size:	110Å		

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Table 1 Retention times for EPA 8330B analytes on various Restek columns.

- new target analytes are shown in red
- highlighted cells indicate coelution.

	Primary Columns		Confirmation Columns		
H_2O:MeOH	**50:50**	**44:56**	**45:55**	**50:50**	**20:80**
Flow	1.5 mL/min	1.0 mL/min	1.2 mL/min	1.5 mL/min	1.5 mL/min
Analytes	**Pinnacle™ II C18**	**Ultra C18**	**Pinnacle™ II Biphenyl**	**Pinnacle™ II Cyano**	**Allure® Biphenyl**
HMX	2.29	3.38	2.76	18.65	1.61
RDX	3.63	5.41	3.22	9.38	1.75
1,3,5-TNB	4.89	7.39	14.54	4.78	5.69
1,3-DNB	5.94	8.82	9.26	4.59	3.92
3,5-DNA	6.63	9.71	5.73	6.34	2.30
tetryl	6.97	9.71	16.12	11.47	4.42
NB	6.97	9.88	6.31	3.80	2.79
2,4,6-TNT	8.23	11.69	20.17	5.94	6.22
NG	8.23	11.69	4.94	8.52	1.98
2-A-4,6-DNT	8.94	12.05	7.43	7.24	2.50
4-A-2,6-DNT	8.94	12.61	7.02	6.34	2.41
2,6-DNT	9.73	13.27	12.36	5.10	4.09
2,4-DNT	9.73	13.64	15.46	5.58	5.14
2-NT	11.92	15.92	9.73	4.38	3.40
4-NT	12.76	17.05	11.07	4.38	3.72
3-NT	13.74	18.32	11.07	4.38	3.73
PETN	16.13	21.08	10.43	17.24	2.67

Ultra C18 Column (USP L1)

5µm Column, 4.6mm	cat. #
250mm	9174575
250mm (with Trident™ Inlet Fitting)	9174575-700

Pinnacle™ II Biphenyl Column (USP L11)

5µm Column, 4.6mm	cat. #
150mm	9209565
150mm (with Trident™ Inlet Fitting)	9209565-700

ordering note

For guard cartridges for these columns, visit our website at **www.restek.com**.

8330 Calibration Mix #1 (7 components)

1,3-dinitrobenzene
2,4-dinitrotoluene
HMX
nitrobenzene
RDX
1,3,5-trinitrobenzene
2,4,6-trinitrotoluene

1,000µg/mL each in acetonitrile, 1mL/ampul

cat. # 31450 (ea.)

8330 Calibration Mix #2 (7 components)

2-amino-4,6-dinitrotoluene
4-amino-2,6-dinitrotoluene
2,6-dinitrotoluene
2-nitrotoluene
3-nitrotoluene
4-nitrotoluene
tetryl

1,000µg/mL each in acetonitrile, 1mL/ampul

cat. # 31451 (ea.)

Single-Component Explosives Reference Mixes

Volume is 1mL/ampul unless otherwise noted. Concentration is µg/mL unless otherwise noted.

3,5-dinitroaniline	ACN	1,000	31661
nitroglycerin	M	1,000	31498
PETN (pentaerythritol tetranitrate)	M	1,000	31600

ACN=acetonitrile
M = methanol