

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.

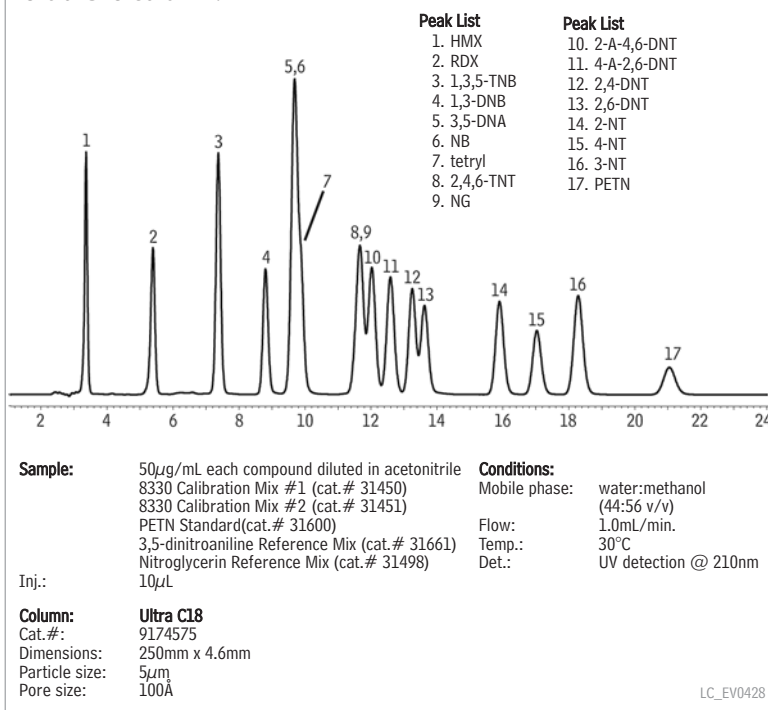


Figure 2 Alternate selectivity of EPA 8330B analytes on the Pinnacle™ II Biphenyl column confirms compound identity.

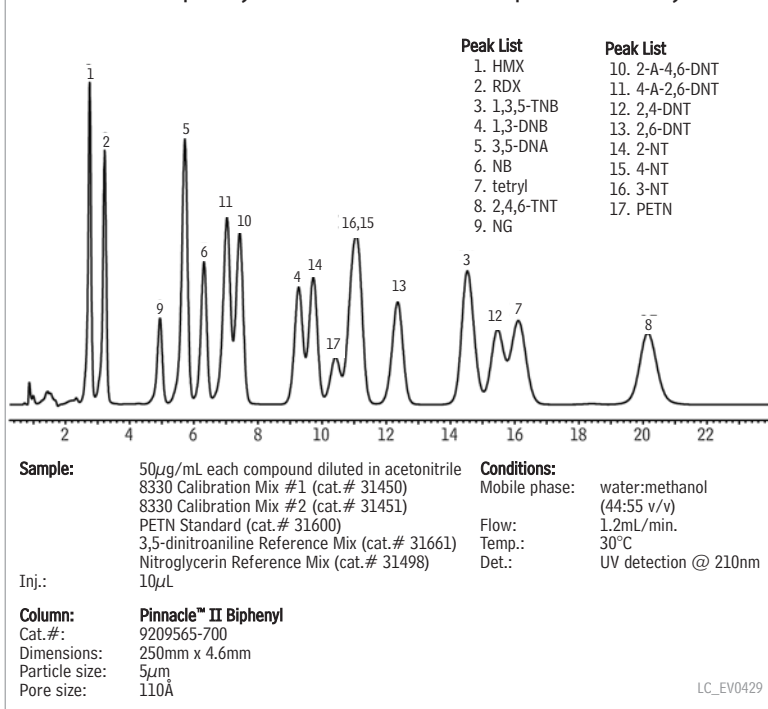


Table 1 Retention times for EPA 8330B analytes on various Restek columns.

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

H ₂ O:MeOH	Primary Columns		Confirmation Columns		
	50:50 1.5 mL/min	44:56 1.0 mL/min	45:55 1.2 mL/min	50:50 1.5 mL/min	20:80 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)

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

Pinnacle™ II Biphenyl Column (USP L11)

5 μ m 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	RDX
2,4-dinitrotoluene	1,3,5-trinitrobenzene
HMX	2,4,6-trinitrotoluene
nitrobenzene	
1,000 μ g/mL each in acetonitrile, 1mL/ampul	
	cat. # 31450 (ea.)

8330 Calibration Mix #2 (7 components)

2-amino-4,6-dinitrotoluene	3-nitrotoluene
4-amino-2,6-dinitrotoluene	4-nitrotoluene
2,6-dinitrotoluene	tetryl
2-nitrotoluene	
1,000 μ g/mL each in acetonitrile, 1mL/ampul	
	cat. # 31451 (ea.)

Single-Component Explosives Reference Mixes

Volume is 1mL/ampul unless otherwise noted. Concentration is μ g/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

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