Waters

HIGH SPEED EXPLOSIVES MONITORING USING UPLC™

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INTRODUCTION

Rapid identification of explosive residues whether for pollution or terrorist concerns has become increasingly important in today's world. In particular, the decommissioning of military bases, both national and global has resulted in a multitude of sites requiring remediation.

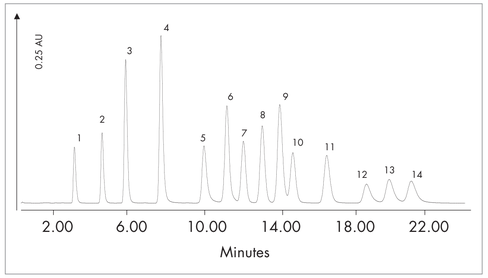
In addition to the obvious infernal effects, these compounds can damage the nervous system and are teratogenically active.

EPA Method 83301 describes the separation of 14 nitroaromatic and nitramine compounds using HPLC and UV detection. For peak confirmation, the same separation is run on a cyano column with a different selectivity.

In 1995, Bouvier and Oehrle[2] described a separation of these analytes using a C_8 and Cyano column in series with photodiode array confirmation. In 2002, Jenkins, Grumbach and Young[3] baseline baseline resolved these 14 analytes using an XTerra® Phenyl column.

An example of this separation follows.

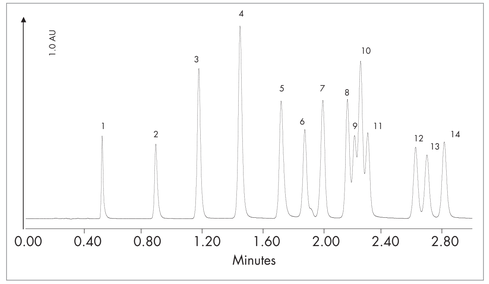
System:	Waters Alliance® HPLC System
Column:	Waters XTerra Phenyl 2.1 x 150 mm, 3.5 µ
Col Temp:	40 °C
Mobile Phase:	80% 10 mM Ammonium Formate / 20% IPA pH to 3.8 / Formic Acid (Isocratic)
Flow Rate:	0.25 mL/min
Inj Vol:	10 µL
Detection:	UV @ 254 nm
	10 ppm each analyte



1. HMX
2. RDX
3. 1,3,5- Trinitrobenzene
4. 1,3 Dinitrobenzene
5. Nitrobenzene
6. TNT
7. Tetryl
8. 2- Amino - 4,6 Dinitrotoluene
9. 2,4 Dinitrotoluene
10. 4- Amino - 2,6 Dinitrotoluene
11. 2,6 Dinitrotoluene
12. 4- Nitrotoluene
13. 2- Nitrotoluene
14. 3- Nitrotoluene

Recently, a new generation of column technology has been developed. These consist of a 1.7 micron bridged ethyl-siloxane hybrid (BEH) particle. These particles can operate at pressures as high as 15000 PSI using eluents with a pH range of 1–12. This results in a low dispersion system which improves peak shape and enhances sensitivity. Run times are significantly shortened. An example of the previous separation run under these conditions is shown for comparison.

System:	Waters ACQUITY UPLC™ System
Column:	Waters ACQUITY UPLC™ 2.1 x 100 mm, 1.7 µ
Col Temp:	40 °C
Mobile Phase:	Water (A) - Methanol (B) (Gradient) 30% B to 54% B in 3 minutes, back to initial conditions in 0.2 minutes
Flow Rate:	0.50 mL/min
Inj Vol:	3 µL
Detection:	UV @ 254 nm
	90 ppm each analyte



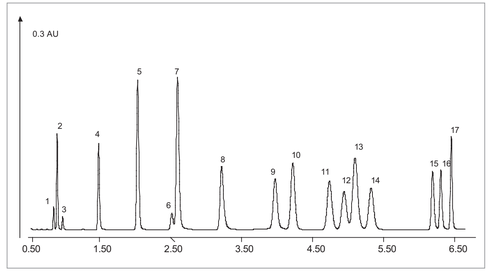
1. HMX
2. RDX
3. 1,3,5- Trinitrobenzene
4. 1,3 Dinitrobenzene
5. Nitrobenzene
6. Tetryl
7. TNT
8. 2- Amino - 4,6 dinitrotoluene
9. 2- Amino - 2,6 dinitrotoluene
10. 2,4 Dinitrotoluene
11. 2,6 Dinitrotoluene
12. 2- Nitrotoluene
13. 4- Nitrotoluene
14. 3- Nitrotoluene

Note that the run time has been reduced by a factor of almost ten. Also while the column load is 2.7X the previous example, peak response has quadrupled.

Another interesting feature is the differing analyte selectivity (high-lighted in blue) due to the change in column chemistry. However the rapid analysis time makes peak identification either through use of a spectral library, or spiking with known standards, quite easy when transferring methods.

By modifying the water methanol gradient to slightly lengthen the separation time we can achieve almost baseline separation of the analytes. In addition, we can include such surrogate compounds as 2,6- Diamino-4-nitrotoluene, 2,4- Diamino-6-nitrotoluene and 1,2- Dinitrobenzene.

System:	Waters ACQUITY UPLC System
Column:	Waters ACQUITY 2.1 X 100 mm, 1.7 µ
Col Temp:	40 °C
Mobile Phase:	Water (A) - Methanol (B) (Gradient) 31% B for 4 minutes, then to 60% B in 2 minutes using curve 8, hold 1 minute then immediate return to initial conditions (31 % B).
Flow Rate:	0.50 mL / min
Inj Vol:	5 mL
Detection:	UV @ 254 nm
	10 ppm each Analyte



1. 2,6 Diamino-4-nitrotoluene*
2. HMX
3. 2,4 Diamino-4-nitrotoluene*
4. RDX
5. 1,3,5- Trinitrotoluene
6. 1,2- Dinitrobenzene*
7. 1,3- Dinitrobenzene
8. Nitrobenzene
9. Tetryl
10. Trinitrotoluene
11. 2- Amino-4,6-dinitrotoluene
12. 2- Amino-2,6-dinitrotoluene
13. 2,4- Dinitrotoluene
14. 2,6- Dinitrotoluene
15. 2- Nitrotoluene
16. 4- Nitrotoluene
17. 3- Nitrotoluene

*Surrogate Compounds

SUMMARY

The ACQUITY UPLC™ is well suited for the analysis of explosives and their degradation products. The rapid analysis time along with the enhanced sensitivity and peak resolution makes a multi analyte analysis such as this easy and economical to perform.

REFERENCES

1. US EPA Method 8330, 1994.
2. Bouvier and Oehrle, LC.GC Vol. 13 No. 2, Feb. 1995 pp 120–130.
3. Jenkins et al. Poster Presentation ACS 2002 Boston, August 2002.

ACKNOWLEDGEMENT

The authors would like to thank Dr. Michael Swartz, Waters Corporation, for his assistance during this study.