CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Profiling Common Explosives

Purpose

Determination of explosives by reductive LCEC.

The trace determination of explosives (F1) has become important in several fields. Security agency personnel and forensic chemists have traditionally been interested in pre- and post-blast analysis of debris material in order to ascertain the origin, manufacturer, and type of explosive device used to perform a crime. Also important is the accurate determination of the purity, stability, and composition of explosives in order to evaluate their long-term effectiveness.

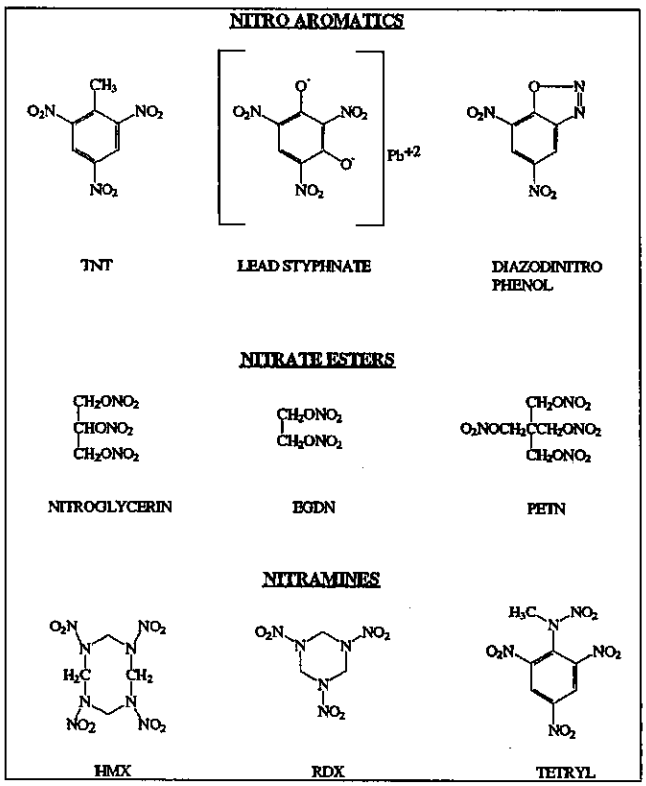

Figure 1. Representative compounds, found in military and commercial explosives and propellants, that are suitable for reductive LCEC.

Since most explosive compounds contain reducible functional groups, they are ready candidates for analysis via liquid chromatography with electrochemical detection (LCEC).

Existing Methods

Liquid chromatography is ideally suited for the determination of thermally unstable and non-volatile explosive compounds. Liquid chromatography with UV (1), thermal energy analysis (TEA) (2) and off-line chemical ionization mass spectrometry (CIMS) (3) have been used. LCUV methods usually offer adequate detection limits for nitro aromatic compounds; however, routine determinations below several nanograms of nitrate esters and nitramines is difficult.

Conditions

System: BAS LC-304, LC-44 or BAS-200A. Oxygen removal is built in on the BAS-200A. For other systems, use of a suitable oxygen-removal apparatus (4) is mandatory.
Detector: BAS LC-4B, LC-4C or BAS-200A
Electrode: BAS Mercury/Gold (Hg/Au) amalgam
Potential: -1.0 V *vs.* Ag/AgCl
Column: 5 µm Biophase C_{18}, 250 x 4.6 mm (MF-6017)
Mobile Phase: 78% (v:v) 20 mM monochloroacetic acid, 14.7 mM sodium acetate, 1 mM EDTA, pH 3.5; 5% ethanol; 17% 1-propanol. Flow rate was 1.7 mL/min.
Injection Volume: 20 µL
Detection Limits: See T1.

Compound	k'	Detection Limits (picomole) at S/N = 3	
		UV	EC at -1.0 V
HMX	0.21	5.1	0.98
Picric acid	0.58	2.1	0.28
RDX	0.88	4.0	0.76
Tetryl	2.6	2.7	0.73
TNT	3.0	2.9	0.62
Nitroglycerin	4.0	700	1.7
2,4-DNT	5.0	3.2	0.88
2,6-DNT	5.0	6.6	0.93
3,4-DNT	5.5	7.1	0.82
PETN	7.9	n.m.	1.3
n.m. - not measured			

Table 1. Typical detection limits (picomoles, at S/N = 3) of explosive compounds using EC and UV detection.

Notes

Chromatograms of various explosives are presented in F2 and F3.

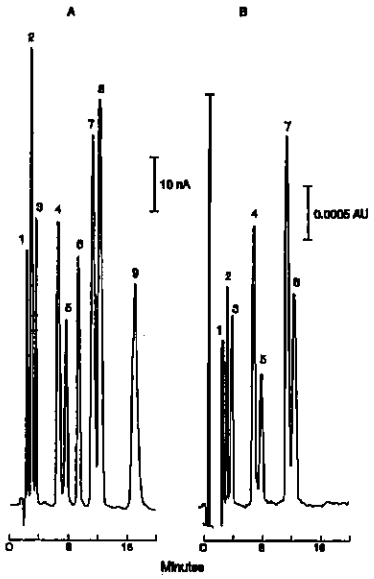

Figure 2. Comparison of EC and UV detection for a standard mixture of explosives. A = EC, B = UV at 254 nm. The mixture contained 34.5 ng HMX (1), 14.3 ng picric acid (2), 23.2 ng RDX (3), 30 ng tetryl (4), 12.1 ng TNT (5), 46.5 ng nitroglycerin (6), 29.6 ng 2,4-DNT (7), 34.0 ng 2,6-DNT (8), and 81.7 ng PETN (9).

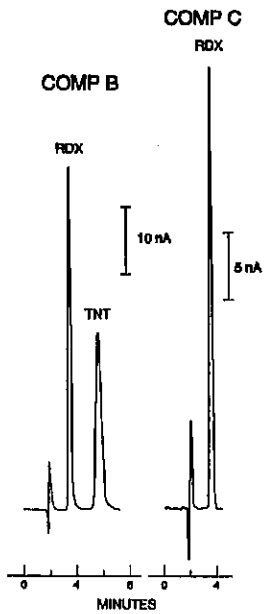

Figure 3. Chromatograms of 97 ng COMP B and 25 ng COMP C military explosives. Column: Biophase C_8, 5 µm, 250 x 4.6 mm (MF-6032). Mobile phase: 50 mM monochloroacetic acid, 37 mM sodium acetate, 1 mM EDTA (pH 3.5); 18% (v:v) 1-propanol; 5% ethanol. Flowrate: 2 µL/min.

Detection limits were 2–500 times lower for EC detection compared to UV detection (T1).

Cyclic voltammetry data for the various explosives are summarized in F4.

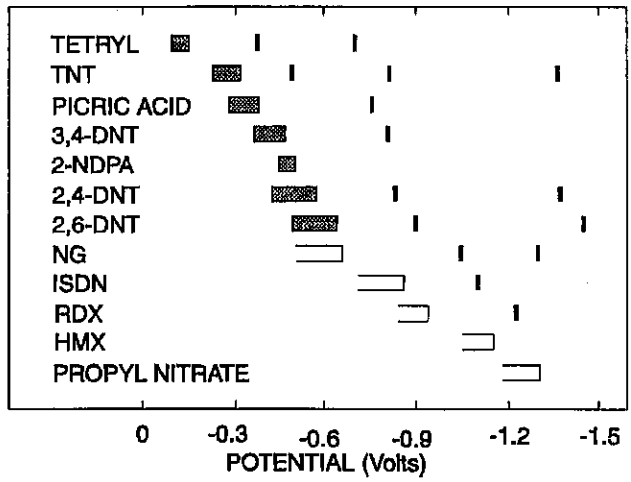

Figure 4. Cyclic voltammetry data for selected explosive compounds obtained with an Hg/Au electrode. A shaded bar indicates that there is one reduction peak, and that an oxidation peak is observed when the scan is reversed. A solid bar indicates the presence of additional forward peaks. An open rectangle indicates a poorly defined forward peak.

References

1. P. Vouros, B.A. Petersen, L. Colwell, B.L. Karger, and H. Harris, Anal. Chem. 49 (1977) 1039.

2. G.C. Whitnack, J.M. Nielsen, and E.S.C. Gantz, J. Am. Chem. Soc. 76 (1954) 4711.

3. D.T. Lewis, Analyst 79 (1954) 644.

4. W. Jacobs, Current Separations 4 (1982) 45.