

Detection of Trace Organic Explosives by Solid Phase Extraction and Liquid Chromatography

References

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Goal

To identify and semi-quantitate any organic high explosives remaining at a post-blast crime scene.

Suggested Method of Analysis

Collection of the explosives from a surface by wiping with a wetted cotton swab; isolation of the explosives by solid phase extraction; analysis of the extract by reverse phase liquid chromatography using a diode array detector

Available Standards

HMX	1,3,5,7-tetranitro-1,3,5,7-tetraazacyclohexane
RDX	1,3,5-trinitro-1,3,5-triazacyclohexane
Tetryl	2,4,6-trinitrophenylmethylnitramine
EGDN	ethyleneglycoldinitrate
NG	glyceroltrinitrate
PETN	pentaerythritoltetranitrate
TNT	2,4,6-trinitrotoluene
DNT	2,4-dinitrotoluene
NT	4-nitrotoluene

Standard solutions (1000 µg/mL) of explosive compounds for analytical work are commercially available. For example, refer to a Cerilliant company catalog.

Sample Preparation

Simulate post-blast residue from one of the following common types of explosive materials.

<i>Material</i>	<i>Composition</i>	<i>Preparation from standard solutions</i> 1-mL mixture in acetonitrile
Cyclotol	RDX, TNT	2.0 µg/mL RDX, 0.5 µg/mL TNT
Semtex	RDX, HMX, PETN	1.0 µg/mL RDX, 0.25 µg/mL HMX, 1.0 µg/mL PETN
Tetrytol	Tetryl, TNT	2.0 µg/mL Tetryl, 0.5 µg/mL TNT
Triple-base propellant	NG, DNT	2.0 µg/mL NG, 0.5 µg/mL DNT

To create some *post-blast debris* place the 1 mL of the explosives mixture dropwise at random spots on one side of a 1-quart plastic milk jug. Let the acetonitrile solvent evaporate at room temperature and then place the jug in a plastic bag in the refrigerator until time for analysis.

Collection of the Explosives

Wet a clean cotton ball with 0.50 mL of 50:50 water:methanol. Swab the surface of the *post-blast debris* with a cotton ball to collect the explosives residue. Rotate the swab occasionally as you wipe in order to use all of its surface. Next, extract the captured explosives from the cotton ball into water. Place the cotton ball at the bottom of a 10-mL plastic syringe, draw 10 mL of water into the syringe, let the syringe stand for 15 minutes, and finally push the water from the syringe into a collection tube.

Solid Phase Extraction

Apply solid phase extraction to the 10-mL extract to isolate the organic explosives. A 3-mL, Waters Oasis sorbent, SPE tube is conditioned, loaded with extract, washed, and explosives eluted with methanol. Follow the procedure outlined in the table below. Flow rates are maintained by adjusting the vacuum applied to the SPE manifold.

STEP	REAGENT	VOLUME	FLOW RATE
Condition	methanol	10 mL	5 mL/min
	water	10 mL	5 mL/min
Load Sample	cotton ball extract	10 mL	2 mL/min
Wash	75:25 water:methanol	3 mL	2 mL/min
Elute	methanol	1 mL	<2 mL/min

Volume Reduction and Solvent Exchange

Add 0.1 mL of water to the 1-mL methanol eluate. The volume is reduced to less than 0.25 mL by heating the liquid to 60 °C while passing nitrogen over it. Next, 0.50 mL of water are added to the solution to lessen the strength of the sample solvent for subsequent injection into the liquid chromatograph.

Liquid Chromatography

Prepare a standard solution containing each of the eight explosive compounds at 2.0 $\mu\text{g/mL}$ in 50:50 water:methanol solvent. Separate standards and samples for 15 minutes under the following conditions:

Injection Volume	Mobile Phase	Flow Rate	Column	Detector
100 μL	50:45:5 mixture of water: methanol:acetonitrile	0.8 mL/min	C ₁₈ , 15 cm, 4.6 mm i.d., 5 μm particles	200 – 250 nm ; monitored @210 nm

Data Analysis

Qualitative analysis: perform a spectrum analysis of the standards and unknowns to confirm the identity of the unknown explosives. Visually compare the pairs of spectra.

From the results of the qualitative analysis and the table below determine the type of explosive material that may have been the cause of the explosion under investigation.

<i>Material</i>	<i>Composition</i>
Cyclotol	RDX, TNT
Semtex	RDX, HMX, PETN
Tetrytol	Tetryl, TNT
Triple-base propellant	NG, DNT

Semi-quantitative analysis: by comparing the peak areas of the components of the standard with the peak areas for each of the identified unknown compounds in the sample and by accounting for the volume changes, calculate the mass of each explosive found on the *post-blast debris*.