

GC/MS Analysis of Organic Explosives: In-Injection Port Thermal Desorption.

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Introduction: A commonly used method for the analysis of organic explosives involves gas chromatographic separation coupled with one of several, somewhat selective, detection methods. Liquid injection is a common technique of introducing organic explosives into a GC; however, thermal desorption methods can also produce excellent chromatographic separation and low method limits of detection. A standard split/splitless injection port can be used with minimal modification to provide an affordable and effective thermal desorption method for organic explosives analysis.^{1,2}

Methods: A Hewlett Packard gas chromatograph was modified by attaching an on/off valve to one of the injection ports' split vent. With the on/off valve open, a carrier flow of 1 ml/min was established on the column and the split vent flow was adjusted to 30 ml/min. When the on/off valve was closed for thermal desorption, the column flow increased to ~13 ml/min. In-injection port thermal desorption was accomplished by cooling the injection port to near ambient temperature, the port was opened, the on/off valve closed and a sample placed in the port. Samples were introduced on Teflon supports and vapor sampling adsorbent tubes containing Tenax TA and sol-gel derived adsorbents, as shown in Figure 1.^{1,2} The injection port was then closed and thermal desorption was performed by heating the injection port to 170°C. The GC oven was held near 30°C during the thermal desorption to allow the organic explosives to condense and focus on the head of the column. After thermal desorption was complete, the on/off valve was opened, a flow of 1 ml/min was established on the column and a normal GC run was initiated. Separation of the explosives was accomplished on a HT-5 column (5% phenyl polycoarborane siloxane, 0.22 mm i.d., 12 mm ID, 12 m length with 0.1 mm film thickness).

The three most sensitive detection methods for the analysis of organic explosives are reported to be electron capture (EC), thermal energy analyzer (TEA) and negative ion chemical ionization (NICI) mass spectrometry. Employing a mass spectrometer for explosive detection provides moderate information content that assists in analyte identification. A Hewlett Packard 5989B quadrupole mass spectrometer operating in the NICI mode was utilized in this work. NICI is almost always a more sensitive ionization mode for the detection of organic explosives than either electron impact (EI) or positive ion chemical ionization (PICI) modes. A recently published study gives a direct comparison of the method detection limits for GC/MS analysis (mass scanning) of organic explosives by NICI, EI and PICI on a single instrument by standard liquid sample injection techniques.³

Results: The in-injection port thermal desorption GC/MS(NICI) data for a standard set of 15 organic explosives and related analytes is shown in Figure 2. The analyte set includes five

dinitrotoluene isomers, dinitrobenzene, trinitrotoluene, trinitrobenzene, two amino-dinitrotoluenes, three nitroesters and two nitramines. Method limits of detection are given in Table 1 for 2,4-dinitrotoluene and three common organic explosives, as determined by NICI for



Figure 1: Inserting a sample for in-injection port thermal desorption

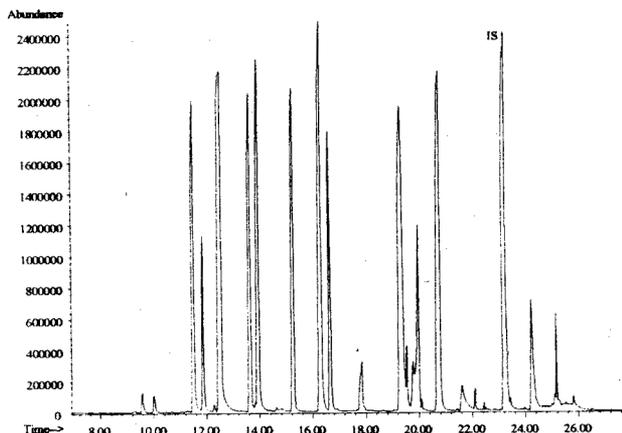


Figure 2: In-injection port GC/MS(NICI) chromatogram from the analysis of a standard set of explosives and related analytes.

Analyte	Liquid Injection (ng)	In-Injection Port Thermal Desorption (ng)	
		Desorption off Teflon	Desorption off Tenax TA
2,4-DNT	0.18	0.68	3.30
TNT	0.19	0.40	2.32
RDX	1.10	1.84	35.41
PETN	0.78	5.62	25.97

liquid injection and in-injection port thermal desorption from Teflon supports and Tenax TA adsorbent tubes. Detection limits for a wider range of explosives have been published elsewhere.¹⁻³

Conclusions: In-injection port thermal desorption can provide a useful tool for the analysis of organic explosives. The method is inexpensive to implement and therefore accessible to most forensic laboratories.

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Reference:

- (1) Sigman, M. E.; Ma, C. Y. *Anal. Chem.* **1999**, *71*(19), 4119.
- (2) Sigman, M. E.; Ma, C. Y.; Ilgner R. H. *Anal. Chem.* **2001**, *73*(4), 792.
- (3) Sigman, M. E.; Ma, C.Y. *J. Forensic Sci.* **2001**, *46*(1), 6.

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