

HPLC Analysis of Explosives Using EPA Method 8330

Michael J. Lang
Varian Chromatography Systems

Key Words: Explosives, EPA, HPLC

Introduction

Since the end of the Cold War, various governments have begun to dismantle military installations and munitions plants in accordance with non-proliferation agreements and disarmament treaties. As a result of these efforts, major environmental problems are being discovered at many locations. Surrounding lands are found to be laden with explosive residues such as 2,4,6-trinitrotoluene (TNT) and associated nitroamine impurities. The highly toxic nature of many of these substances, coupled with their persistence in the environment, requires thorough characterization of contaminated areas.

Gas chromatography is often used to determine these substances. However, for the separation of thermally unstable and non-volatile compounds, high performance liquid chromatography (HPLC) with ultraviolet detection is ideally suited and offers adequate detection limits for nitroaromatics. HPLC methodology was first documented in the early 1980s by Bratin et. al.

The EPA Method 8330, first introduced in November 1990, is the most common way that explosives are analyzed. Analytes are able to be detected down to 2.5 ppb in water, soil, or sediment. The data generated in this report followed the EPA 8330 method. The EPA method documents co-elution of some of the compounds of interest. In order to identify and quantitate all explosive compounds, it was necessary to rerun the samples under different chromatographic conditions. This co-elution was observed by other investigators as well.

Complete separation of all compounds of interest in a single chromatographic run would improve sample throughput and decrease the cost of analysis.

Experimental

The HPLC Measurements were carried out on a Varian Star HPLC system which included: 9012 Gradient Solvent Delivery System, 9050 UV-VIS Detector, 9300 Refrigerated AutoSampler (fitted with a 20 uL loop), and a Star Chromatography Workstation.

NOTICE: This document contains references to Varian. Please note that Varian, Inc. is now part of Agilent Technologies. For more information, go to www.agilent.com/chem.



LC

Varian Application Note Number 24

The instrument conditions followed a modified version of EPA Method 8330 and are listed in Table 1. Standards were obtained from Accustandard and were diluted with water. A typical chromatogram showing the complete separation of these explosive compounds is shown in Figure 1. A typical calibration curve is shown in Figure 2.

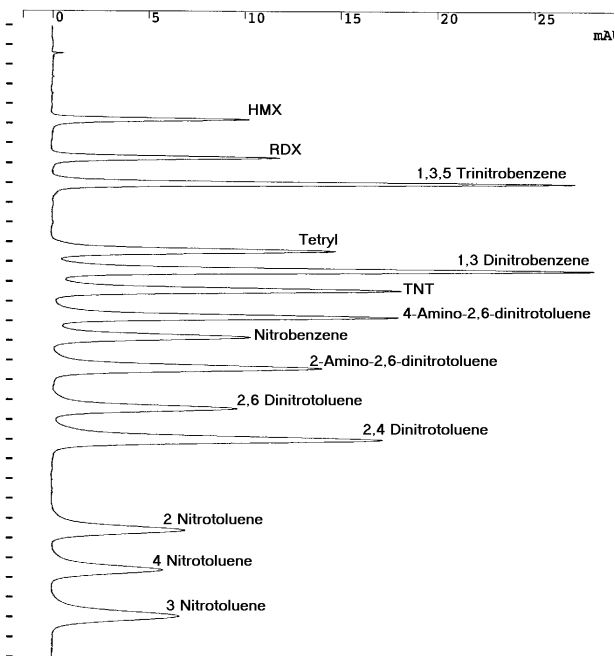


Figure 1. EPA Explosives at 10 ppb

Table 1

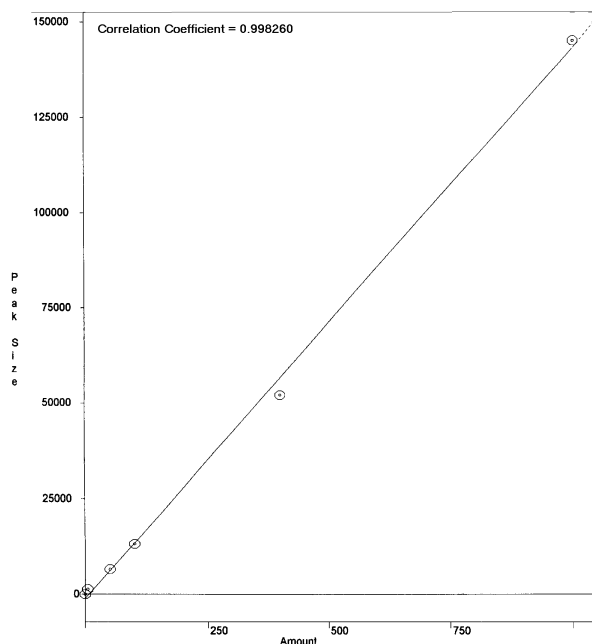
Columns	30 mm CN in series with a 250 mm Bondesil C - 18
Mobile Phase	50% Methanol / 50% Water
Flow Rate	1.3 ml/min
Detection	254nm
Sample	4 ^o C in water

Table 2**Conclusions**

The use of the unique combination of 30 mm Res Elut CN column in series with a 250 mm Bondesil C-18 column, produces complete separation of all explosives in a single run. This improves sample throughput, decreases analysis time, and eliminates the need for repeating injections on a second column. Reducing the sample injection volume from the typical 100 uL to 20 uL gave better peak shape and improved retention time reproducibility for early eluting compounds. The flow rate was also reduced from 2.0 mL/min to 1.3 mL/min without increasing the analysis time so solvent consumption was reduced. As illustrated in Figure 1, a complete separation of the 14 components in EPA Method 8330 is achieved. Table 2 shows the typical retention times under the chromatographic conditions outlined in this note. A second confirmation run, because of co-elution, is not required due to complete separation of the explosives in this modified method.

Excellent linearity of response is observed over the concentration range of 2.5 ppb to 1 ppm. Typical results of multi-level calibrations are shown in Figure 2 where the correlation coefficient is 0.998260.

<u>Component</u>	<u>Time</u>
HMX	4.585
RDX	6.450
1,3,5 Trinitrobenzene	7.757
Tetryl	10.937
1,3 Dinitrobenzene	11.931
TNT	12.905
4-Amino-2,6-dinitrotoluene	14.179
Nitrobenzene	14.974
2-Amino-2,6-dinitrotoluene	16.457
2,6 Dinitrotoluene	18.516
2,4 Dinitrotoluene	20.017
2 Nitrotoluene	24.394
4 Nitrotoluene	26.284
3 Nitrotoluene	28.466

**Figure 2. Calibration of TNT (1 ppm-2.5 ppb)****References:**

1. Kleiboher, et. Al., Journal of Chromatography, Vol. 638 (1993), pp 349-356.
2. Bratin, et al., Analytica Chima Acta, Vol. 130 (1981), pp. 295-311.
3. EPA Method 8330. U.S government. (1990).
4. Emmrich, et. Al., Journal of Chromatography, Vol. 645 (1993), pp. 89-94
5. Millennium App Brief, Waters Corp. (Aug. 1993).
6. Walsh and Thomas, Analytica Chima Acta, Vol.231, (1990), pp. 313-315
7. Kolla, Journal of Chromatography, Vol. 674, (1994) pp. 308-318.