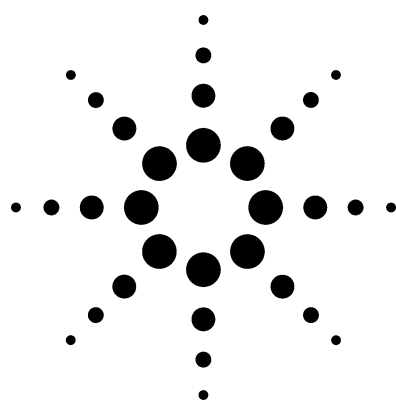# GC Analysis of Nitroaromatics and Explosives Using the Agilent 255 Dual Plasma NCD

Technical Overview

Introduction

Nitroaromatics are frequently used as solvents and as precursors to other high value organic compounds, such as pharmaceuticals. In addition, some nitroaromatics are either themselves explosive or are precursors to other explosive materials. Many high-energy (unstable) nitrogen containing species detonate by way of heat or shock and are useful as explosives, as is the case with several commonly known nitroaromatics. Their analysis is necessary for process, research and development, forensic, environmental, and other reasons.

The nature of these materials places some limitations on how they can be handled in their analysis. In gas chromatography, it is not advisable to use a conventional or untreated split-injector for sample introduction because the high temperature encountered in this injector can lead to the thermal decomposition of the analytes of interest. On the other hand, high temperatures may be required for adequate analyte vaporization. Analyte decomposition can also occur on the column, so the column for analysis should be chosen to elute the analyte at the lowest reasonable temperature, providing adequate separation. It should also be designed of a material that does not catalyze analyte decomposition.

The Agilent Dual Plasma Nitrogen Chemiluminescence Detector (NCD) offers a more cost effective, stable, sensitive, and selective solution for detection of nitrogen containing species. Owing to its high selectivity toward nitrogen species, greater than 10^8 over carbon, it is possible to measure these species in the presence of potentially interfering species. The fact that the detector operates under vacuum, similar to a mass spectrometer, allows for lower elution temperatures, which is a benefit for the analysis of thermally labile compounds. In addition, selectivity is often more important in determining method detection limits than sensitivity. In the case of the NCD, both selectivity and sensitivity are high, allowing large injections or the use of GC columns that would overload a mass spectrometer for example.

This overview briefly illustrates the potential for the analysis of nitrogen-containing species, such as nitroaromatics and related explosive compounds, by GC with NCD detection. A special Restek GC column developed specifically for this type of analysis was used. Restek has also developed a related column with slightly different selectivity that can be used for confirmatory analysis.

Detectors such as mass spectrometry (MS) and electron capture detectors (ECDs) are commonly used for this type of analysis. These detectors possess some advantages, such as identification capability by MS and sensitivity of ECD. The NCD with its inherent selectivity and sensitivity cannot only be used as a replacement for a mass spectrometer, electron capture detector, or nitrogen phosphorous detector, it can also be used as a complementary or confirmatory measurement technique. The experimental conditions used for this overview are given in Table 1.

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Table 1.   Experimental Conditions

Gas chromatograph	Agilent 6890 GC
Injector	Cool on-column set to oven track mode; 2 psi head pressure; 0.2 µL injection volume
Oven	60 ºC for 1 minute to 160 ºC at 10 ºC/min then to 300 ºC at 30 ºC/min (hold for 3 minutes)
Column	Rtx-TNT, 6 m, 0.53 mm id, 1.5 µm – Restek catalog number 12998
Detector	Agilent Model 255 NCD Dual Plasma Base temperature 300 ºC; Burner temperature 900 ºC Oxygen 8 SCCM; Hydrogen 4 SCCM

Test samples included Agilent NCD Test Mixture with nitroaromatics added and mixtures and dilutions of Restek Cal Mixtures 8095 A and B (Restek catalog number 31607 and 31608, respectively).

Figures 1, 2, and 3 show a nitrogen containing test mixture in toluene, a 1:50 dilution of 8095 Calibration Mixture A in toluene and a 1:100 dilution of equal parts of Calibration Mixtures A and B in toluene. Note that no solvent peak is observed in the chromatogram obtained from the nitrogen containing test mixture. This is because of the very high nitrogen to carbon selectivity. In the other mixtures, the solvent peak at the front of the chromatograms results from the presence of high levels of acetonitrile. Note the same numeric peak identifications are used in Figures 2 and 3. In all cases, excellent peak shape is observed for these compounds with the exception of HMX, which exhibits some fronting and tailing due to on-column decomposition. Nevertheless, this note demonstrates the capability for successful detection of all of these species at low ng levels. Sub-ng level detection is possible due to the high sensitivity of the NCD.

Restek Corporation (www.restekcorp.com; Bellefonte, PA, USA) is acknowledged for its generous donation of the GC column used for this work.

For More Information

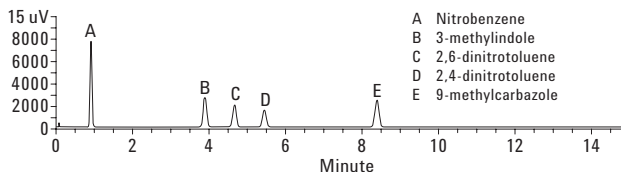For more information on our products and services, visit our Web site at www.agilent.com/chem.

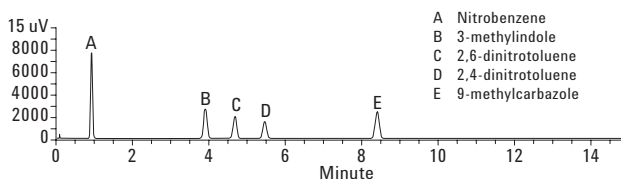**Figure 1.   Nitrogen containing test mixture, peaks represent ~ 2 ng as nitrogen on-column (0.2 µL of ~ 10 µg/mL).**

Figure 2.   1:50 dilution of Cal Mix 8095 A in toluene. Peaks represent ~ 4 ng as compounds on-column (0.2 µL of 20 µg/mL as compounds).
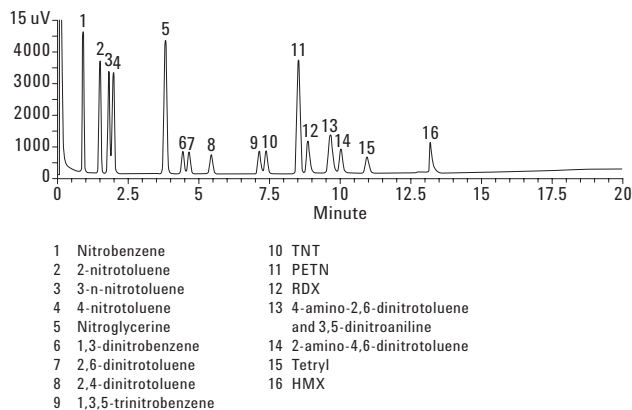

Figure 3.   1:100 dilution of Cal Mix 8095 A and B in toluene. Peaks represent ~ 2 ng as compounds on-column (3,5-dinitroaniline at ~ 10 ng as compound).

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