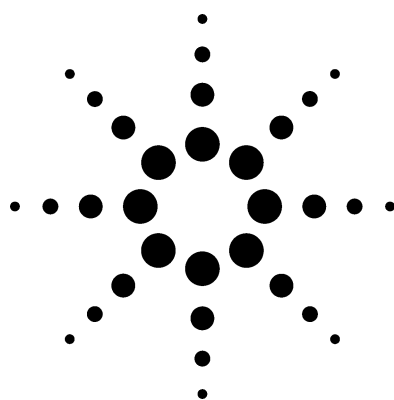


Analysis of Trace Nitrogen Species in Benzene



Technical Overview

Introduction

Specifications for trace nitrogen species in benzene are being driven lower by the use of more selective processing catalysts. Analytical laboratories must keep up with these trends and improve method sensitivity to measure these trace species. In many cases, interference from other trace species precludes the use of a universal detector, such as a flame ionization detector (FID). Instead, a nitrogen chemiluminescence detector is needed to deliver not only the required sensitivity, but specificity as well.

This technical overview describes the measurement of trace nitrogen species that can be found in high purity benzene, including acetonitrile, morpholine, 1-methyl-2-pyrrolidinone and 4-formyl-morpholine.

Discussion

In many cases, it is possible to measure nitrogen species in aromatic solvents like benzene using a universal detector such as an FID. However, when trying to measure nitrogen or sulfur species at trace concentration levels, other trace species can interfere because it is often not possible to resolve these species chromatographically. In these circumstances, chemiluminescence detectors provide a good analytical solution.

The Agilent 255 Nitrogen Chemiluminescence Detector (255 NCD) delivers the sensitivity required for this analysis without suffering from any hydrocarbon interference.

Figure 1 illustrates the selectivity of the detector, in that no response from benzene is observed even though it comprises over 99 percent of the sample. Figure 2 illustrates a linear response to nitrogen (morpholine) over the concentration range of interest.

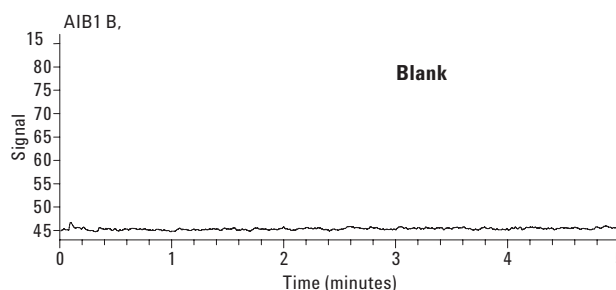


Figure 1. Agilent 255 NCD analysis of a benzene blank. This chromatogram demonstrates the absence of hydrocarbon interference. Note: A small amount of acetonitrile is observed at approximately 20 seconds.

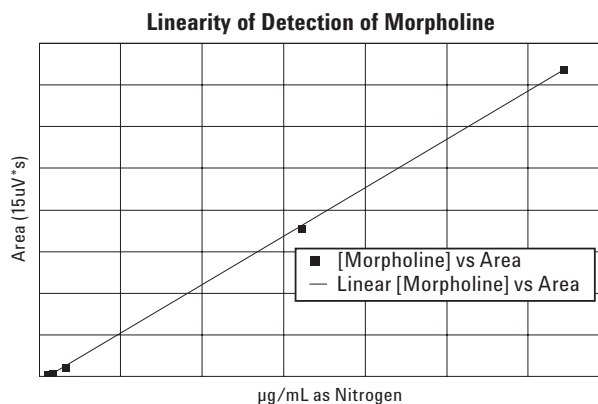


Figure 2. The linearity of response demonstrated in this chart for morpholine illustrates this beneficial feature of the 255 NCD.



The 255 NCD can be installed on nearly all commercially available GCs (gas chromatographs). In many cases, it is desirable to install the 255 NCD on an existing GC that is already being used for product quality or process measurements. In most cases, it is possible to simply add the detector to an existing instrument as a second channel. On the other hand, some laboratories prefer to keep separate testing stations for different measurement parameters.

In general, nitrogen compounds are difficult to chromatograph at trace levels and will exhibit tailing on most columns due to adsorption. This is a chromatographic issue; therefore, tailing will be observed with all detectors depending on the nitrogen species, its concentration level, and the column used for separation. Nevertheless, the following chromatograms illustrate the ability of the 255 NCD to sensitively detect ppm and sub-ppm levels of these trace nitrogen species in benzene.

Figure 3 shows good signal-to-noise for these species at the 0.5-ppm level as nitrogen. In fact, it was possible to detect levels as low as 0.2 ppm nitrogen with the operating conditions described in this technical overview. Even lower detection levels could be achieved using special techniques such as large volume injection. This technique would only work for those components that have a higher boiling point than benzene.

Figure 4 shows even better signal-to-noise for these species at about the 5-ppm level as nitrogen. Note that the peak shape is slightly improved at this concentration level. Even better peak shape is observed at higher concentrations. It was found

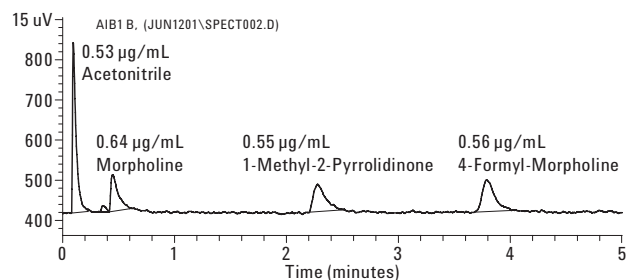


Figure 3. Chromatogram of trace nitrogen species in benzene at ca. the 0.5-ppm level as nitrogen.

that peak shape was generally better using columns with thicker films and/or base deactivation treatments. Tailing certainly limits the accuracy and precision of integration, but still good repeatability was observed for all species, even at these low concentrations (approximately 10% RSD at the 0.5- to 1-ppm level).

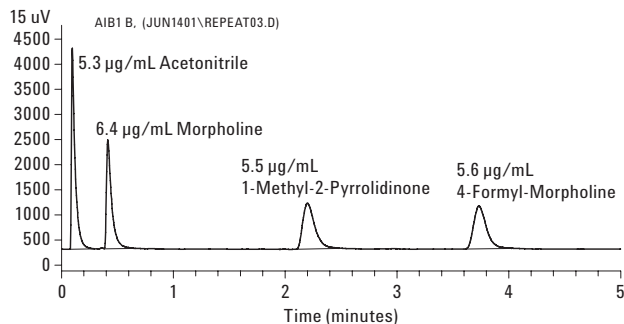


Figure 4. Chromatogram of trace nitrogen species in benzene at the 5- to 6-ppm level as nitrogen.

Operating Conditions

GC

| | |
|-----------------------|--|
| Temperature: | 50° C ISO |
| Helium carrier: | 7.4 mL/min |
| Column: | 10 m, HP-1 0.53 mm id 2.65 mm film thickness |
| Split injection: | 1:4 |
| Injector temperature: | 220° C |
| Injection size: | 2 mL |

Detector

| | |
|---------------------|-----------|
| Burner temperature: | 800° C |
| Hydrogen: | 20 mL/min |
| Oxygen: | 14 mL/min |

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