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Application News

No.G281

Gas Chromatography

High-Sensitivity Analysis of Ammonia, Methylamine, and Trimethylamine in Environmental and Energy Fields

Nitrogencompounds such as ammonia and various amines are known as malodorous substances, and besides concern associated with the offensive odors, they have potential adverse affects on human life. On the other hand, ammonia's high energy density per unit volume and the ease with which it can be stored and transported has resulted in increased research in its use as a medium for the storage of hydrogen in fuel cells. The increased use of ammonia and the close proximity of associated amines to humans accentuate the necessity for methods of accurate and rapid detection and quantitation of these substances. The flame thermionic detector (FTD) is known as a high-sensitivity detector for nitrogen compounds, but because the FTD does not respond to ammonia, it cannot be used for its detection. Typically, ammonia analysis by gas chromatography (GC) is conducted using a thermal conductivity detector (TCD), but measurement is difficult using TCD unless the concentration is greater than about 100 ppm.
The dielectric barrier discharge ionization detector (BID) permits detection of nearly all compounds, except for helium and neon, at higher sensitivity than that possible with TCD and FID detectors. Here, we introduce examples of analysis at the ppm level of ammonia and methylamine in water, and of trimethylamine in water by GC-BID.

Analysis of Ammonia and Methylamine

Ammonia and methylamine were diluted with water to prepare solutions at 4.8 ppm, 24 ppm and 120 ppm, respectively, and the solutions were then measured by GC-BID.
The 4.8 ppm and 24 ppm chromatograms are shown in Fig. 1, the linearity is shown in Fig. 2, and the analytical conditions are shown in Table 1. Calculating the lower limit of detection (S/N = 3) from the 4.8 ppm S/N ratio, the results indicated 1.2 ppm for ammonia and 2.5 ppm for methylamine.
Linearity may be sacrificed at low concentrations of components that display adsorption. In this analysis, good linearity was obtained over the range including 4.8 ppm, 24 ppm, and 120 ppm. It should be noted that in this analysis, base-deactivated wool (RESTEK P/N: 20999) was used to pack the glass insert to prevent adhesion of ammonia and the amines at the injection port.

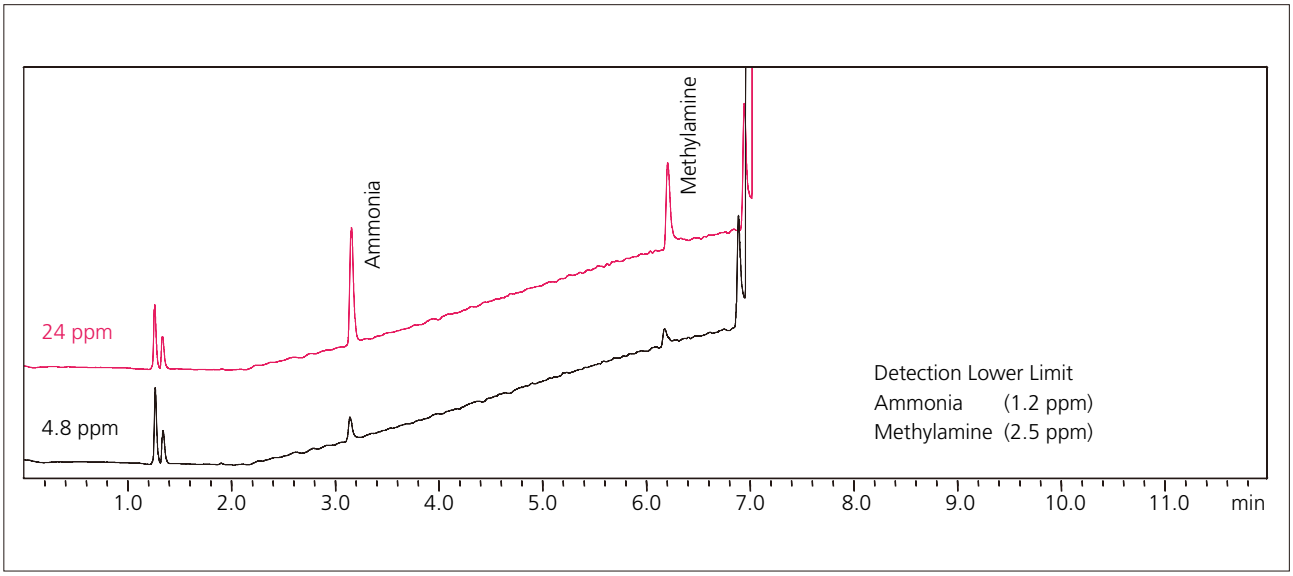

Fig. 1 Chromatograms of 4.8 ppm and 24 ppm Standard Solutions

Table 1 Analytical Conditions for Ammonia and Methylamine

Model	: Tracera (GC-2010 Plus + BID-2010 Plus)
Inj. Mode	: Split 1:5
Inj. Temp.	: 220 °C
Carrier Gas	: He 50 cm/sec. (Constant Linear Velocity Mode)
Column	: PoraPLOT Amines (25 m × 0.53 mm I.D., df = 20 µm)
Column Temp.	: 80 °C (2 min) - 10 °C/min - 130 °C - 20 °C/min - 200 °C (1.5 min)
Det. Temp.	: 220 °C
Discharge Gas	: 50 mL/min (He)
Glass Insert	: Split insert Restek Base Deacts FS wool
Inj. Volume	: 1.0 µL

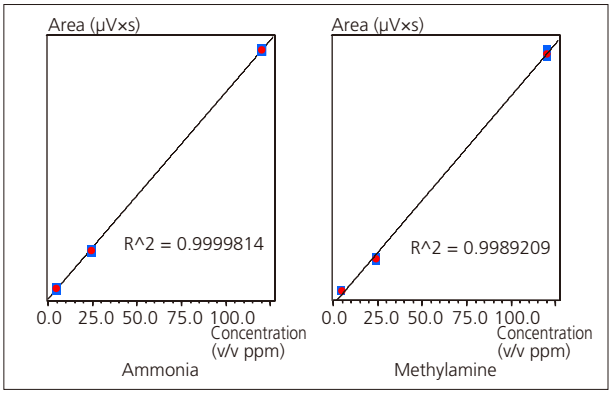

Fig. 2 Linearity of Ammonia and Methylamine (4.8, 24, 120 ppm)

Analysis of Trimethylamine

Since the above analytical conditions do not permit separation of the trimethylamine and water peaks, analysis was conducted using a different column. (Using these analytical conditions, ammonia and methylamine cannot be separated.)
The trimethylamine was diluted with water to obtain concentrations of 4.8 ppm, 24 ppm, and 120 ppm, respectively. These solutions were analyzed by GC-BID. The chromatograms obtained with the 4.8 ppm and 24 ppm solutions are shown in Fig. 3, the linearity is shown in Fig. 4, and the analytical conditions are shown in Table 2.
Calculating the lower limit of detection from the S/N using the 4.8 ppm solution, the result was 0.06 ppm. Further, excellent linearity was obtained over the range of concentrations including 4.8 ppm, 24 ppm and 120 ppm.

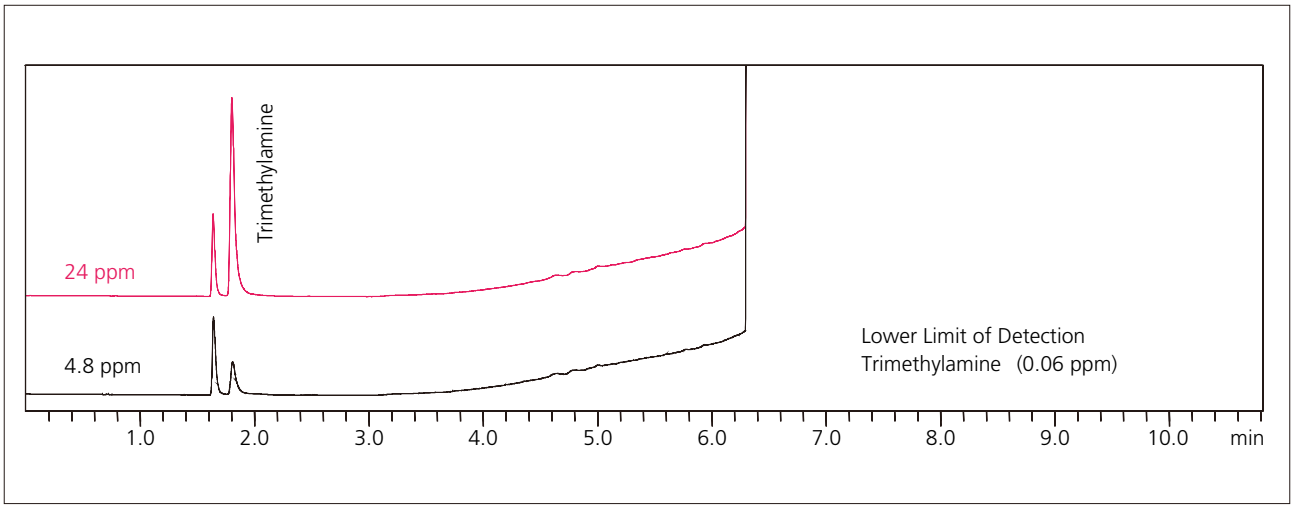

Fig. 3 Chromatograms of 4.8 ppm and 24 ppm Standard Solutions

Table 2 Analytical Conditions for Trimethylamine

Model	: Tracera (GC-2010 Plus + BID-2010 Plus)
Inj. Mode	: Split 1:5
Inj. Temp.	: 220 °C
Carrier Gas	: He 40 cm/sec. (Constant Linear Velocity Mode)
Column	: RESTEK Stabilwax-DB (30 m × 0.53 mm I.D., df = 1.0 µm)
Column Temp.	: 35 °C (3 min) - 30 °C/min - 180 °C (3.0 min)
Det. Temp.	: 220 °C
Discharge Gas	: 50 mL/min (He)
Glass Insert	: Split insert Restek Base Deacts FS wool
Inj. Volume	: 1.0 µL

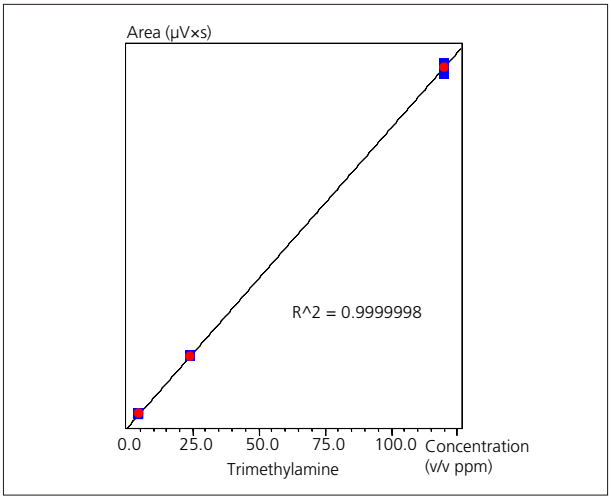

Fig. 4 Linearity of Trimethylamine

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