APPLICATION NOTE

Analysis of a Gas Mixture Containing Ammonia and Methane by Using a Compact High-Resolution Multi-Turn Mass Spectrometer

AN-MS-001

Overview

Analysis of a chemical ionization reagent gas consisting of a 5% mixture of ammonia in methane is demonstrated by using the JEOL InfiTOF™ compact high-resolution mass spectrometer. the InfiTOF's high mass-resolving power clearly separates and identifies Isotope peaks and trace water contamination with excellent mass accuracy.

The InfiTOF Compact High-Resolution Mass Spectrometer

The InfiTOF™ is a compact time-of-flight mass spectrometer that is ideal for the high-resolution analysis of gases. The entire InfiTOF system (Figure 1), including the vacuum pumps, is approximately the size of a personal computer tower. Unique multi-turn ion optics (described in the following section) provide detection of ions from *m/z* 1 (H^+) to *m/z* 1000 with mass resolving power adjustable from unit resolution to greater than 30,000.

Figure 1. The InfiTOF™ Compact High-Resolution Mass Spectrometer System.

Multi-turn mass spectrometers

Although increasing the length of the flight tube can improve the resolving-power of a time-of-flight mass spectrometer, this can result in an impractically large system. Multi-turn ion mass spectrometers[1] with Perfect Focusing ion optics[2] solve this problem by using compact "figure-8" ion optics. The flight path can either be an "open-loop" series of figure-8 segments (Figure 2, left side) as in the JEOL *SpiralTOF* MALDI-TOF/TOF mass spectrometer or a closed-loop flight path with a single figure-8 segment as in the *InfiTOF* (Figure 2, right side).

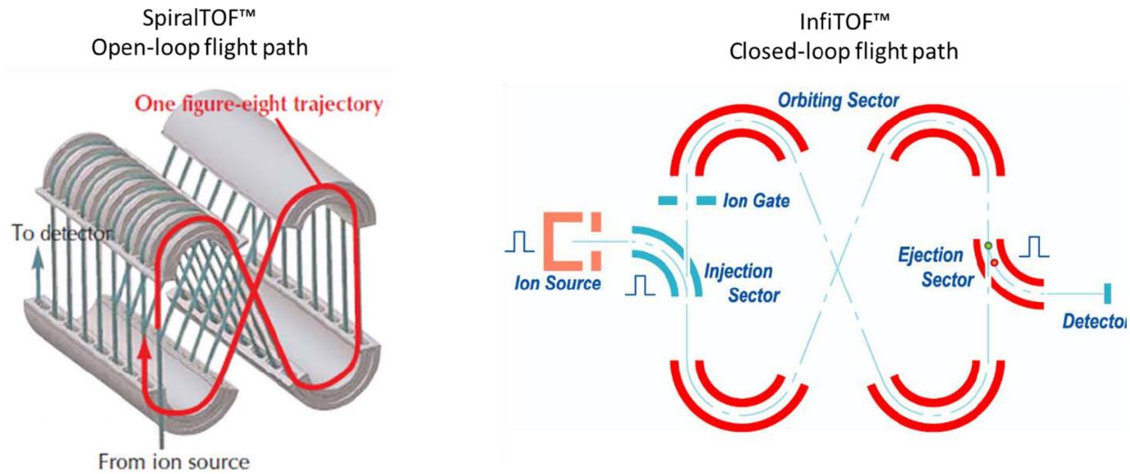

Figure 2. Two multi-turn mass spectrometer configurations.

A variable-resolution time-of-flight mass spectrometer

The InfiTOF's closed-loop configuration provides a variable-length flight path depending on how many turns ("laps") the ions make around the figure-8 "racetrack". The InfiTOF's control system provides timed ejection pulses eliminate the overtake problem (fast ions catching up with slow ions after several turns) by ejecting ions outside the programmed *m/z* ranges. Mass spectra can be measured in three modes: (1) low-resolution full-scan mass spectra, (2) multiple high-resolution segments, and (3) a continuous set of segments to provide full-range high-resolution mass spectra.

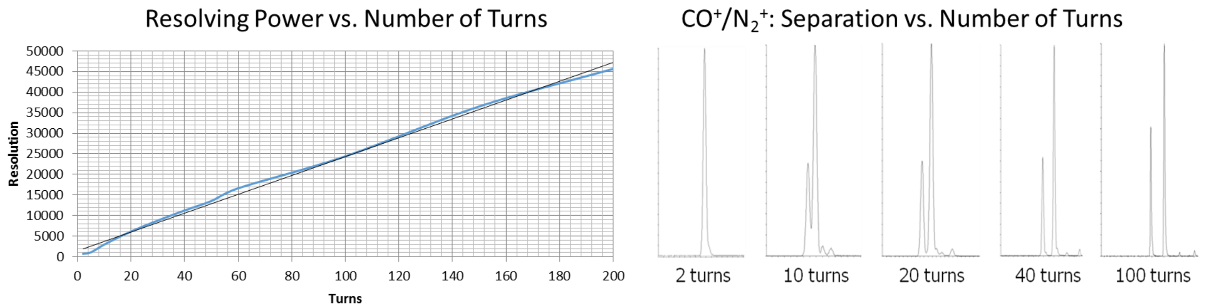

Figure 3. Mass resolving power increases with multiple turns around the InfiTOF ion optics.

Ammonia/methane gas mixture

Mixtures of ammonia in methane are easier to handle than pure ammonia and provide improved sensitivity for ammonia chemical ionization. [3-5] However, the electron ionization (EI) mass spectra of methane and ammonia contain a number of isobaric peaks that cannot be distinguished by low-resolution mass spectrometry alone.

In the absence of gas chromatography, characterization of low levels of ammonia in methane requires high resolution mass spectrometry. Figure 1 shows the NIST Mass Spectral Database electron ionization (EI) mass spectra of ammonia (Figure 1A) and methane (Figure 1B) and a summed mass spectrum for 5% ammonia in methane (Figure 1C). Except for slight increase in the relative abundance of the peak at m/z 17, the mass spectrum of 5% ammonia in methane is almost indistinguishable from the methane mass spectrum. Smaller ammonia concentrations would be indistinguishable from pure methane.

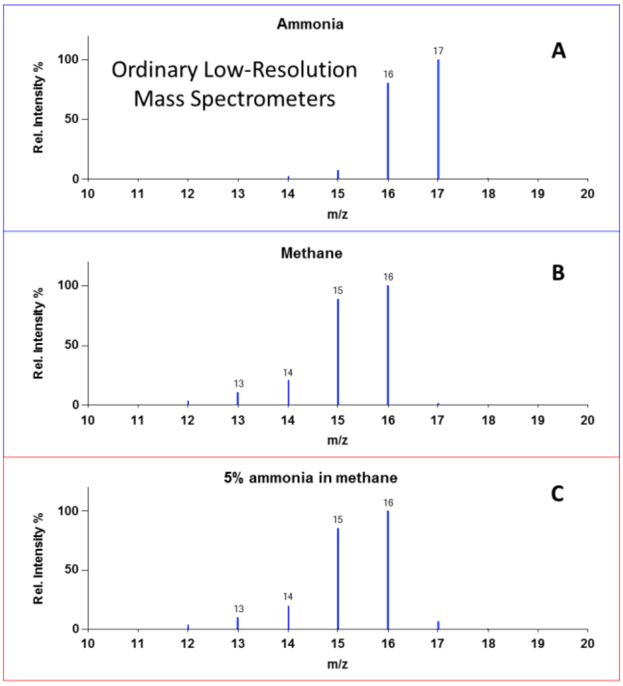

Figure 4. Low-resolution electron ionization (EI) mass spectra of ammonia (A) and methane (B) and a 5% mixture of ammonia in methane (C).

Here we demonstrate the use of a compact, high-resolution mass spectrometer with unique multi-turn ion optics to separate isobaric peaks in a sample of 5% ammonia in methane.

Experimental

Mass spectra were acquired by using the InfiTOF compact multi-turn high-resolution mass spectrometer. The ionizing electron voltage was set to 70 eV. High-resolution mass spectra were acquired in multi-segment mode (20 turns) to give a resolving power of approximately 6000 (FWHM) at *m/z* 16. The gas mixture was introduced directly into the mass spectrometer through a deactivated fused-silica capillary tube.

Results

Figure 2 shows that the InfiTOF has clearly resolved the isobaric ammonia and methane peaks, including the isotopic peaks and trace water peaks. Trace peaks corresponding to protonated methane (CH_5^+) and protonated ammonia (NH_4^+) from ion-molecule reactions are well separated from the $^{13}CH_4^{+\bullet}$ and $H_2O^{+\bullet}$ respectively. Accurate mass measurements were all within 0.001 u, providing unambiguous elemental composition assignments.

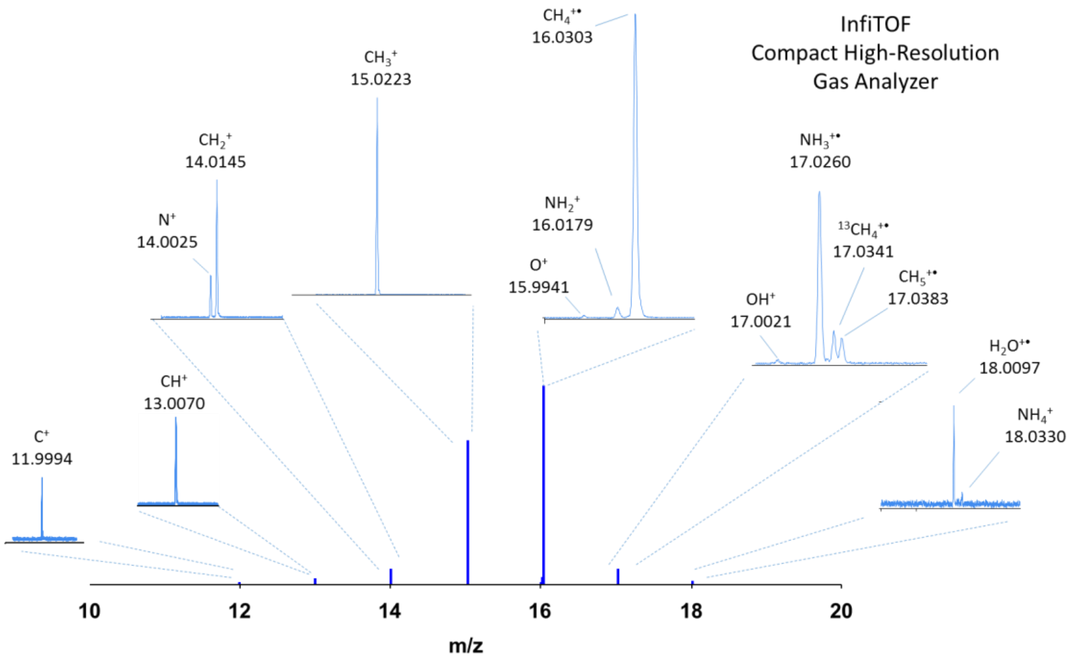

Figure 5. High-resolution InfiTOF mass spectrum of 5% ammonia in methane.

Conclusion

Trace peaks in gas mixtures are easily separated and identified without chromatography by the high mass-resolving power of the InfiTOF compact multi-turn mass spectrometer.

References

[1] M. Toyoda, D. Okumura, M. Ishihara, I. Katakuse. Multi-turn Time-of-Flight Mass Spectrometers with Electrostatic Sectors. *J. Mass Spectrom.*, **2003**, *38*, 1125.

[2] M. Ishihara, M. Toyoda, T. Matsuo. Perfect Spacial and Isochronous Focussing Ion Optics for Multi-turn Time of Flight Mass Spectrometer. *Int. J. Mass Spectrom.*, **2000**, *197*, 179.

[3] P. Rudewicz, B. Munson. Effect of ammonia partial pressure on the sensitivities for oxygenated compounds in ammonia chemical ionization mass spectrometry. *Analytical Chemistry*, **1986**, *58*, 2903.

[4] W. V. Ligon, H. Grade. Chemical ionization mass spectrometry utilizing an isotopically labeled reagent gas. *Journal of the American Society for Mass Spectrometry*, **1994**, *5*, 596.

[5] R. B. Cody. Separation of the reagent ions from the reagent gas in ammonia chemical ionization mass spectrometry. *Analytical Chemistry*, **1989**, *61*, 2511.