Application Note

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Ammonia Analysis Using the Agilent 990 Micro GC

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Abstract

The Agilent 990 Micro GC with the Agilent J&W CP-Volamine column is introduced for the accurate and stable analysis of ammonia. The ammonia peak (100 ppm to 20%) has excellent retention time and area repeatability. The whole system is inert against the corrosion of ammonia with moisture.

Introduction

Ammonia is widely used in many industrial processes, such as a fertilizer, refrigerant, detergent, and pesticide. Ammonia analysis on the Agilent 990 Micro GC was unsatisfactory using the current channels. The ammonia peaks on the commonly used Agilent J&W CP-Sil 5 CB and Agilent J&W PoraPLOT U columns tail severely at all concentrations. Especially when the concentration is low (below 1,000 ppm), strong retention time shift and difficult integration due to peak tailing make quantitative analysis of ammonia impossible.

The Agilent J&W CP-Volamine column is now introduced to the 990 Micro GC system. This column is coated by a nonpolar stationary phase with MPD (multipurpose deactivation) technology, which is specially optimized for the analysis of ammonia and volatile amines. The column is inert and stable even when the sample contains high concentration of water vapor.

This application note demonstrates the analysis of ammonia standards (100 ppm and 20%) and real samples containing high concentration of water vapor using a 15 m J&W CP-Volamine straight channel. For all samples, the 990 Micro GC system provided excellent chemical performance, including peak shape and repeatability. The whole system inertness was verified by six months of consecutive injections.

Experimental

The 990 Micro GC was equipped with a 15 m J&W CP-Volamine straight channel. Table 1 shows the experimental conditions of this application. Tables 2 and 3 show the composition of the ammonia standard gases (low concentration and high concentration). Except for the standard gases, a real ammonia sample gas was also analyzed. This sample gas came from the headspace vapor of ammonia solution, containing approximately 10% ammonia and saturated water vapor.

Table 1. Experimental conditions of the ammonia analysis.

Carrier Gas	Helium
Pressure	150 kPa
Injector Temperature	50 °C
Column Temperature	50 °C
Injection Time	40 ms
Sampling Time	30 s
Run Time	60 s

Table 2. Composition of the low concentration ammonia gas standard.

Compound	Concentration
Ammonia	96.8 ppm
Nitrogen	Balance

Table 3. Composition of the high concentration ammonia gas standard.

Compound	Concentration
Ammonia	20.2%
Nitrogen	Balance

Results and discussion

The experimental results are listed in Table 4. Repeatability for retention time (RT) and peak area were measured as the percentage of the relative standard deviation (RSD) calculated from 10 runs of each standard and sample.

Table 4. Retention time and peak area repeatability of the ammonia gas samples.

Compound	RT (min)	RT RSD	Area (mV × s)	Area RSD
Ammonia (96.8 ppm)	0.617	0.0034%	0.0786	0.696%
Ammonia (20.2%)	0.600	0.0070%	279.3	0.018%
Ammonia (Real Sample)	0.605	0.0066%	143.4	0.262%

Figure 1 is the chromatogram of the low concentration ammonia (96.8 ppm), and Figure 2 is the chromatogram of the high concentration ammonia (20.2%). Ammonia shows good peak shape at both low and high concentrations. At low concentrations (approximately 100 ppm), eliminating ammonia adsorption in the sample flow path requires using short, inert tubing that is purged thoroughly before injection. For these experiments, an injection time of 40 ms was used. Injection time could be increased to 150 to 200 ms for a better peak response. At high concentration (approximately 20%), the ammonia peak was well resolved from the nitrogen peak (Rs >4), indicating good selectivity of the J&W CP-Volamine column for ammonia.

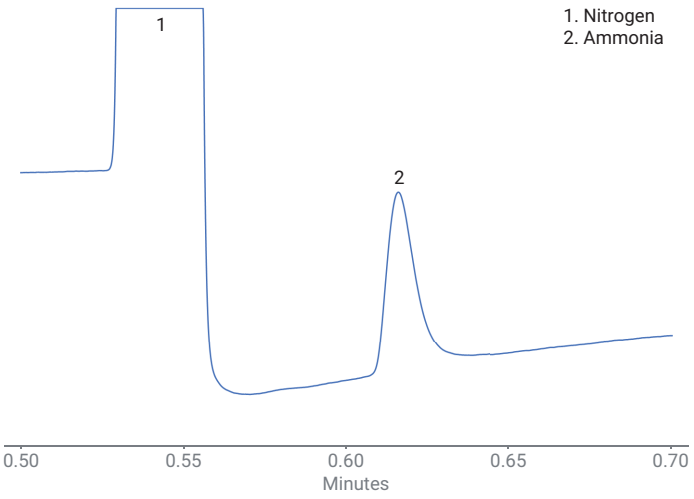

Figure 1. Chromatogram of 96.8 ppm ammonia on the 15 m Agilent J&W CP-Volamine straight channel.

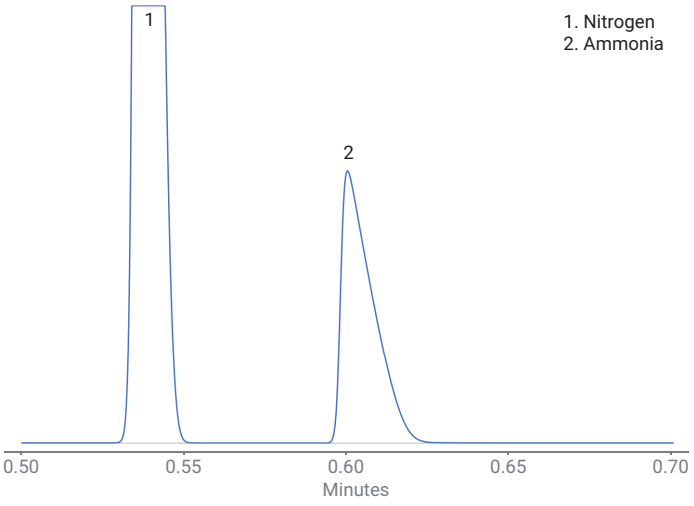

Figure 2. Chromatogram of 20.2% ammonia on the 15 m Agilent J&W CP-Volamine straight channel.

For the real ammonia sample, both the ammonia and water peaks elute within one minute. Figure 3 shows the chromatogram of the real ammonia sample. The peak area and retention time remained the same during the six months of the consecutive injections of samples containing high concentration of ammonia (approximately 10%) and saturated water vapor (3 to 5%). The whole sample introduction system of the 990 Micro GC was inert, and no corrosion was found. Table 5 shows the peak area and RT RSD of the 20.2% ammonia standard sample of the seven monthly quality check runs on three parallel channels, all of which are below 1%. Figure 4 shows the overlapped chromatograms of the previously mentioned seven quality check runs of one of the channels.

Table 5. Long-term (six months) chemical performance of the three channels.

Channel Number	Peak Area RSD	RT RSD
Channel #1	0.63%	0.41%
Channel #2	0.65%	0.23%
Channel #3	0.31%	0.37%

It should be noted that only gaseous samples can be injected into 990 Micro GC channels. Liquid water must be filtered out before injection. Although water elutes on this Volamine column with good peak shape, it is not recommended to quantify water amount using gas chromatography.

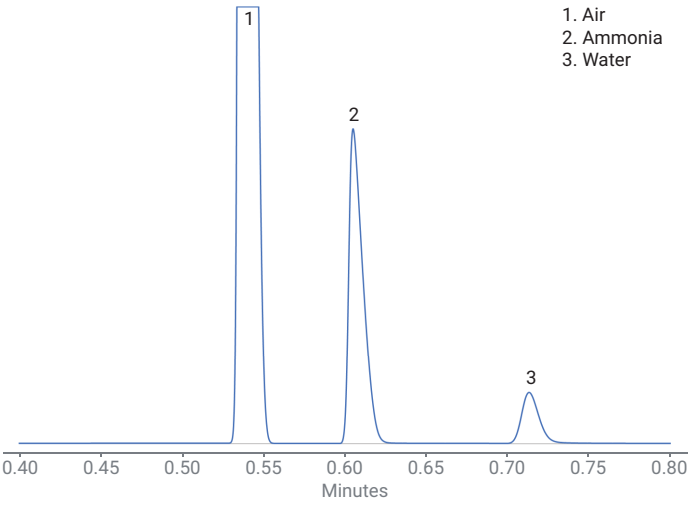

Figure 3. Chromatogram of headspace vapor of ammonia solution on the 15 m Agilent J&W CP-Volamine straight channel.

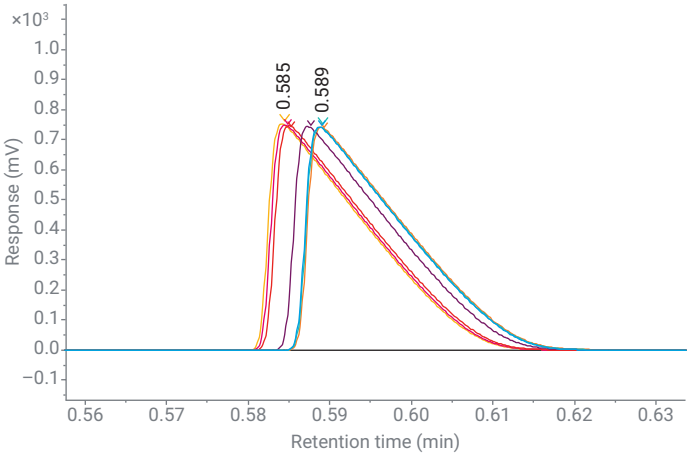

Figure 4. Chromatogram of 20.2% ammonia peaks of the seven monthly quality check runs during the long-term stability test.

Conclusion

This article shows the chemical performance of the Agilent 990 Micro GC system equipped with the Agilent J&W CP-Volamine column for the analysis of ammonia samples. The inert sample flow path provides long-term protection against corrosion of ammonia samples containing high moisture content. For both low and high concentration samples, the retention time and peak area repeatability of ammonia are excellent.

References

1. Luong, J. *et al.* Polar volatile compounds. *Agilent Technologies application note*, publication number A01915, **2011**.

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