Application Brief

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Analysis of Impurities in Helium Using the Agilent 990 Micro GC

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Introduction

Helium is a noble gas that is extracted from natural gas. Because of its chemical inertness, low density, and low boiling point, helium is widely used as the protective atmosphere in arc welding and in the semiconductor industry. It is also used as the filler of balloons and airships, and as the cooling medium in NMR and other instruments.

The common impurities in helium are neon, oxygen, nitrogen, methane, carbon monoxide, and carbon dioxide. For example, helium must meet the requirement of 99.999% purity for use as the carrier gas in an Agilent GC system. This means that the concentration of impurities should not exceed 10 ppm. Therefore, it is critical to detect ppm level impurities in helium.

This application brief demonstrates the analysis of impurities in helium, on the Agilent 990 Micro GC system. The system was equipped with a 10 m CP-Molsieve 5Å Straight channel and a 10 m CP-PoraPLOT U Straight channel. The analysis was done in 1.5 minutes with excellent low detection limits (LODs) and repeatability. The typical concentrations of the detected impurities were 2 to 1,000 ppm.

Experimental

The 990 Micro GC system is equipped with a 10 m CP-Molsieve 5Å Straight channel with RTS filters, and a 10 m CP-PoraPLOT U Straight channel.

The sample gas components are listed in Table 2. The 10 m CP-Molsieve 5Å Straight channel was used to separate 5 ppm neon, oxygen, nitrogen, methane, and carbon monoxide. The 10 m CP-PoraPLOT U Straight channel was used to separate 2 ppm carbon dioxide, ethylene, ethane, and acetylene.

Table 3 shows the chemical performance of the 20 sample runs. For all components, the RT RSDs% were less than 1%, the area RSDs% were less than 3%, and the calculated LODs were less than 1 ppm. The LOD was calculated as the signal of 2 times the noise (4-sigma), and all LODs met the published specification, which is 2 ppm. These results show excellent repeatability and detection limitation of the 990 Micro GC.

Table 1. Test conditions for the impurities in helium.

Channel Type	10 m CP-Molsieve 5Å, RTS, Straight	10 m CP-PoraPLOT U, Straight
Carrier Gas	Helium	Helium
Column Pressure	300 kPa	150 kPa
Injector Temperature	50 °C	50 °C
Column Temperature	150 °C	60 °C
Injection Time	200 ms	200 ms
Sampling Time	30 s	

Table 2. Composition of the standard gas.

Component	Concentration (ppm)
Neon	5.00
Oxygen	5.17
Nitrogen	5.07
Methane	5.13
Carbon Monoxide	5.12
Carbon Dioxide	2.39
Ethylene	1.94
Ethane	1.98
Acetylene	1.93
Helium	Balance

Table 3. Retention time, area repeatability, and LOD of 20 runs of the standard gas.

Compound	RT (min)	RT RSD%	Area (mV × s)	Area RSD%	LOD (ppm)
Neon	0.287	0.227	0.00766	1.26	0.30
Oxygen	0.397	0.058	0.0128	1.19	0.19
Nitrogen	0.480	0.031	0.0142	3.04	0.19
Methane	0.644	0.075	0.0111	2.86	0.30
Carbon Monoxide	0.745	0.081	0.0122	1.16	0.31
Carbon Dioxide	0.493	0.157	0.0121	1.05	0.11
Ethylene	0.540	0.094	0.0129	0.93	0.086
Ethane	0.583	0.059	0.0132	1.51	0.089
Acetylene	0.706	0.041	0.0106	1.44	0.11

Figure 1 shows that on the 10 m CP-Molsieve 5Å channel, 5 ppm neon, oxygen, nitrogen, methane, and carbon monoxide were eluted and well separated within 1 minute. Figure 2 shows that on the 10 m CP-PoraPLOT U channel, 2 ppm carbon dioxide, ethylene, ethane, and acetylene were eluted and well separated within 1 minute.

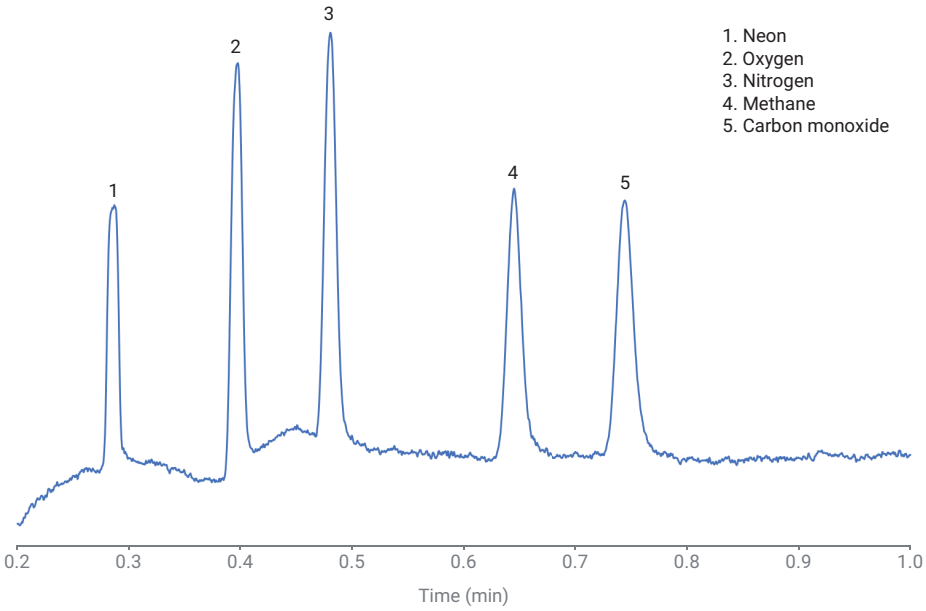

Figure 1. Chromatogram of neon, oxygen, nitrogen, methane, and carbon monoxide on an Agilent 10 m CP-Molsieve 5Å channel.

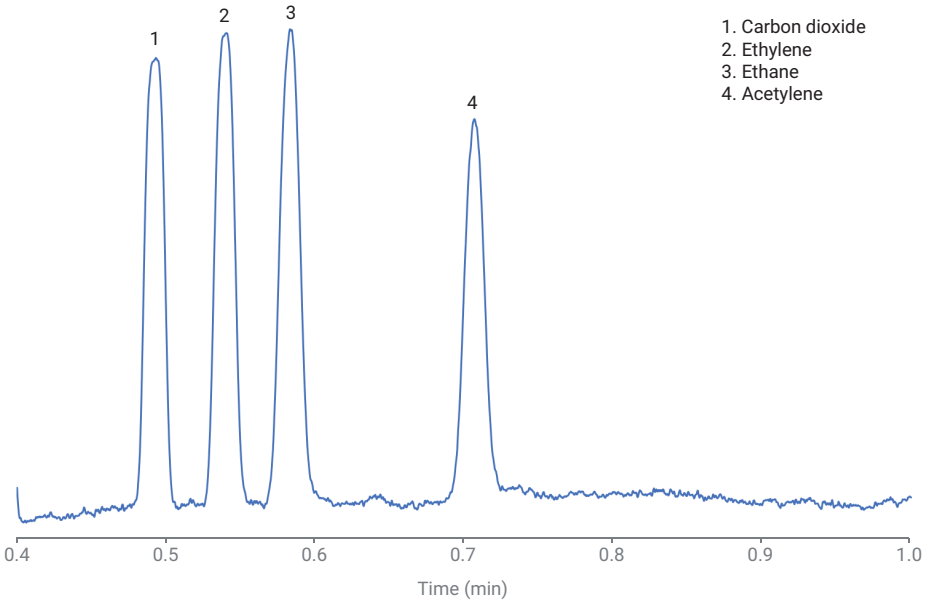

Figure 2. Chromatogram of carbon dioxide, ethylene, ethane, and acetylene on an Agilent 10 m CP-PoraPLOT U channel.

Conclusion

This application brief demonstrates the analysis of ppm level impurities in helium with the Agilent 990 Micro GC. The LODs of the target components are <2 ppm. The RT RSD% and the peak area RSD% are <0.3% and <3%, respectively. The analysis time is approximately 1.5 minutes. These results show excellent chemical performance of the Agilent 990 Micro GC as a platform for the analysis of the impurities in helium.

References

1. Remko van Loon. Permanent Gas Analysis – Separation of Helium, Neon and Hydrogen a MolSieve 5 Å column using the Agilent 490 Micro GC, *Agilent Technologies application note*, publication number 5990-8527EN, 2011.
2. Remko van Loon. C1 – C3 Hydrocarbon Analysis Using the Agilent 490 Micro GC – Separation Characteristics for PoraPLOT U and PoraPLOT Q Column Channels, *Agilent Technologies application note*, publication number 5990-9165EN, 2011.

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