Application Brief

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Hydrogen Impurity Analysis Using the Agilent 990 Micro GC

Author

Tingting Bu
Agilent Technologies, Inc.

Introduction

As a clean energy source, the application of hydrogen (H_2) in fuel cell cars is very promising. In Japan, many H_2 energy vehicles have been developed and are on the market. In China, the government has an ambitious plan to develop H_2 energy, build H_2 stations, and encourage local companies to develop H_2 vehicles.

There are various methods to produce H_2 such as electrolysis of water, coal gasification, natural gas steam reforming, etc. The different methods of H_2 production may introduce impurities into the H_2 product. When H_2 is used in a fuel cell, those impurities may damage the cell. The impurities He, Ne, N_2, Ar, CH_4 will dilute H_2 concentration, and CO/CO_2 will decrease the lifetime of the fuel cell. It is important to detect ppm level impurities before H_2 is used in a fuel cell.

The Agilent 990 Micro GC is a portable GC integrated with a low-dead-volume micro thermal conductivity detector (µ-TCD). The 990 Micro GC can provide fast and sensitive analysis for 2 to 10,000 ppm level H_2 impurities analysis.

Experimental

Table 1. Analytical methods for sample analysis.

Channel Type	20 m CP-Molesieve 5Å, RTS, Straight	10 m CP-PoraPLOT U, Straight
Carrier Gas	Hydrogen	Hydrogen
Column Pressure	230 kPa	120 kPa
Injector Temperature	50 °C	50 °C
Column Temperature	75 °C	50 °C
Injection Time	40 ms	50 ms

Table 2. Composition of standards gas 1.

Component	Concentration (ppm)
Helium	301
Neon	18.0
N_2	Balance

Table 3. Composition of standards gas 2.

Component	Concentration (ppm)
Argon	5.8
Oxygen	5.0
Nitrogen	8.5
Carbon Monoxide	5.8
Methane	5.3
Carbon Dioxide	6.2
Hydrogen	Balance

Table 4. RT and area repeatability of 10 runs of standards gas.

Compound	RT (min)	RT RSD%	Area (mV × s)	Area RSD%
Helium	0.367	0.045	0.02622	0.71
Neon	0.378	0.045	0.00552	1.15
Argon	0.569	0.011	0.00428	2.11
Oxygen	0.582	0.013	0.00371	5.28
Nitrogen	0.875	0.011	0.00741	1.78
Carbon Monoxide	2.235	0.070	0.00286	3.46
Methane	0.364	0.029	0.00738	2.84
Carbon Dioxide	0.505	0.012	0.00991	2.19

High purity H_2 with >99.9995% was used as the carrier gas in this test. H_2, with the highest thermal conductivity, is a good fit as a carrier and provides excellent sensitivity for components. Besides sensitivity, H_2 as a carrier eliminates matrix effects in this application.

Two standard gases were used from Table 2 and Table 3 and most of the component concentrations were in the 5 to 10 ppm range. Two straight channels were used, CP-Molesieve 5Å and CP-PoraPLOT U, to separate and detect components from He to CO_2. The analytical methods are in Table1.

Figure 1 shows that helium and neon are well resolved on a 20 m CP-Molesieve 5Å channel. Figure 2 shows the detection from Ar to CO, and in 5 ppm level concentration, the baseline and peak can be well identified. The separation of Ar and O_2 is also well finished with a resolution value 1.13. The response of methane in CP-PoraPLOT U is much higher than in 20 m CP-Molsieve 5Å, and the quantitative analysis is suggested in CP-PoraPLOT U together with CO_2. A repeatability test was run based on 10 injections, and the results are in Table 4. All components show excellent retention time RSD%. The value is below 0.1% for peak area RSD% and the area values are smaller than 5%, except for oxygen with an RSD of 5.28%.

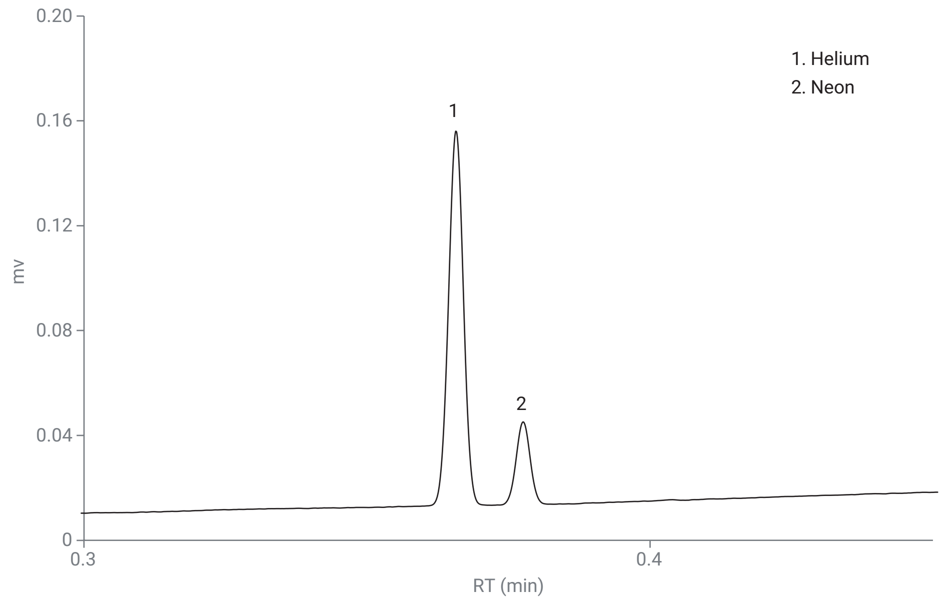

Figure 1. Chromatogram of standards gas 1 on a 20 m Agilent CP-Molsieve 5Å channel.

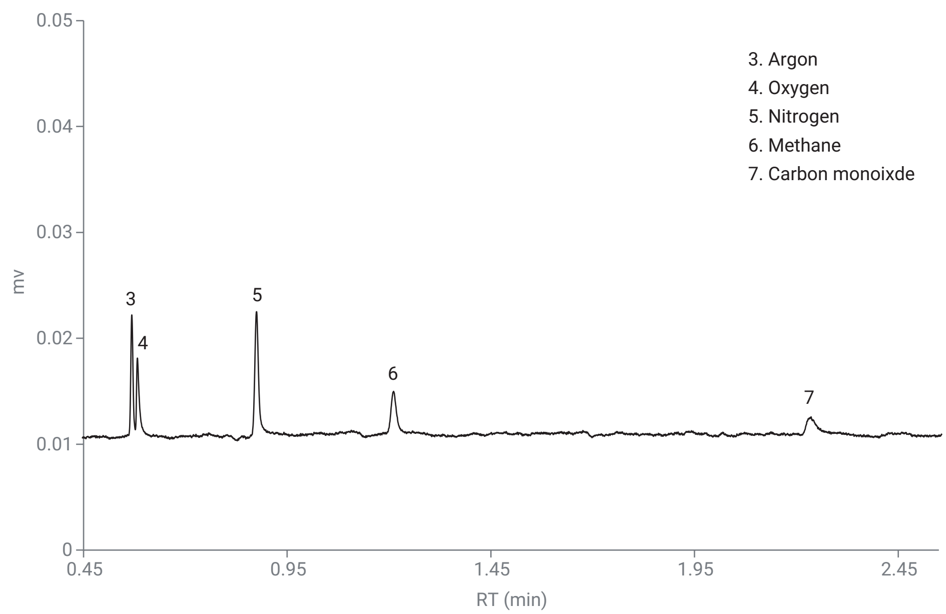

Figure 2. Chromatogram of standards gas 2 on a 20 m Agilent CP-Molesieve 5Å channel.

Conclusion

This application brief demonstrates the applicability of the 990 Micro GC for a fast analysis of impurities in hydrogen. The results show excellent retention time and peak area repeatability. Furthermore, the total run time was less than 150 seconds. This is extremely helpful to make quick decisions about hydrogen quality.

References

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2. Bajja, M. Permanent Gas Analysis – Separation of Argon and Oxygen on a MolSieve 5Å column using the Agilent 490 Micro GC, *Agilent Technologies application note*, publication number 5990-8700EN, **2011**.
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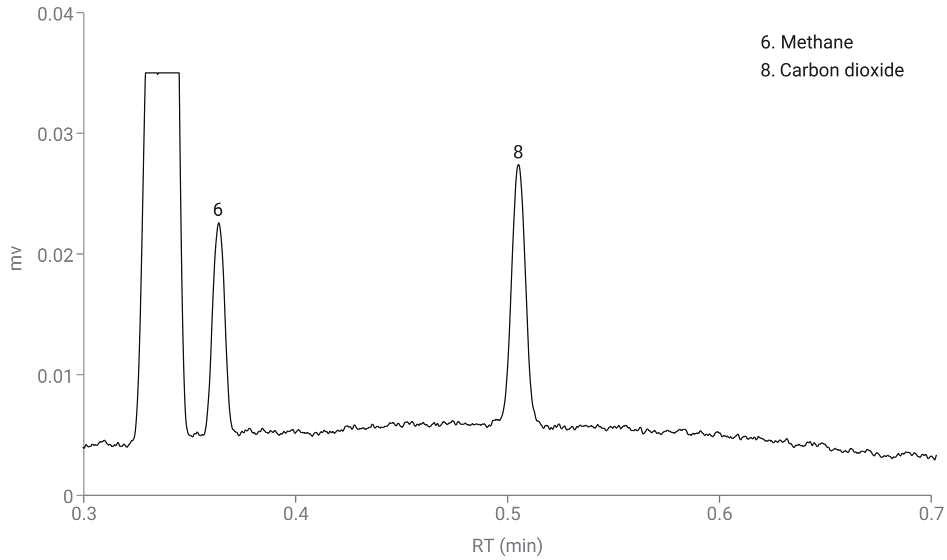

Figure 3. Chromatogram of standards gas 2 on a 10 m Agilent CP-PoraPLOT U channel.

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Printed in the USA, June 30, 2020
5994-2138EN

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