Application Brief

Agilent
Trusted Answers

Argon and Oxygen Analysis Using the Agilent 990 Micro GC

Authors

Tingting Bu and Fei Jiang
Agilent Technologies, Inc.

Introduction

Argon (Ar) and oxygen (O_2) detection is always a challenge for the permanent gas manufacturers in their gas quality analysis. Separating Ar and O_2 is a challenging task, especially when the concentrations of both columns vary. The Agilent 990 Micro GC is equipped with a MEMS technology-based chip injector, ultrafast narrow bore columns with a Molesieve 5Å stationary phase, an industry-leading sensitive micro thermal conductivity detector (μ-TCD), and is plumbed by near zero dead volume connectors. The 990 Micro GC is ideally suited to face this detection challenge.

Experimental

In this test, two channels in the 990 Micro GC were used. One channel was a 20 m Molesieve 5Å using helium as carrier gas to detect similar concentrations of argon and oxygen or low-level argon in oxygen. The other channel was a 10 m Molsieve 5Å with Ar as carrier to detect low-level oxygen in argon.

As shown in Figure 1, the 20 m Molsieve 5Å channel has excellent argon/oxygen baseline separation using standard gas 1 with resolution >2.0. To verify detection of ppm level argon in oxygen, the standard gas 2 was used.

Figure 2 is a full picture of gas 2 with approximately 95% oxygen and approximately 5% nitrogen. Enlarging the RT from 2.5 to 3.5 minutes, shows that the 22.8 ppm argon is well separated from oxygen by the 20 m Molesieve 5Å channel. The peak tailing of O_2 is because of O_2 concentration higher than 90% and the column is overloaded.

Table 1. Analytical methods for sample analysis.

Channel Type	20 m CP-Molesieve 5Å, Straight	10 m CP-Molesieve 5Å, Straight
Carrier Gas	Helium	Argon
Column Pressure	150 kpa	250 kpa
Injector Temperature	50 °C	50 °C
Column Temperature	40 °C	80 °C
Injection Time	10 ms	80 ms

Table 2. Composition of standards gas 1.

Component	Concentration
Neon	18.0 ppm
Hydrogen	1.01%
Argon	0.201%
Oxygen	0.201%
Nitrogen	0.201%
Helium	Balance

Table 3. Composition of standards gas 2.

Component	Concentration
Argon	22.8 ppm
Nitrogen	5.04%
Oxygen	Balance

Table 4. Composition of standards gas 3.

Component	Concentration
Oxygen	23.5 ppm
Nitrogen	5.03%
Argon	Balance

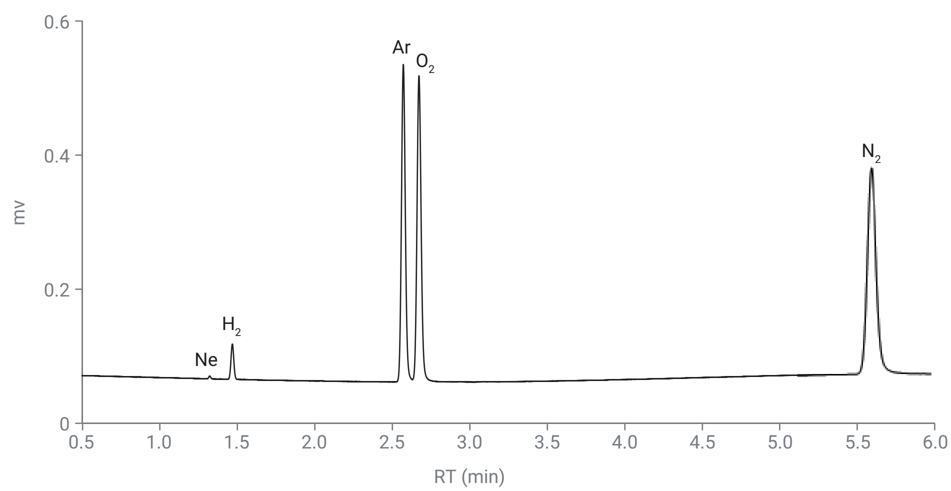

Figure 1. Chromatogram of standards gas 1 on a 20 m Agilent Molsieve 5Å channel.

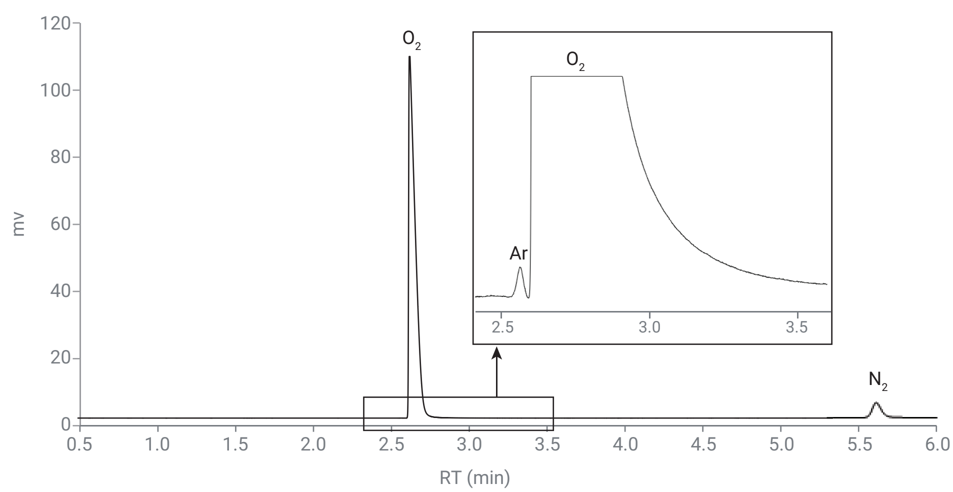

Figure 2. Chromatogram of standards gas 2 on a 20 m Agilent CP-Molsieve 5Å channel.

For low ppm O_2 in Ar, Ar was used as the carrier gas on another 10 m Molesieve 5Å. Figure 3A is full picture of gas 2 with approximately 95% Ar and approximately 5% nitrogen. Figure 3B is an enlarged picture of gas 3 and 23.5 ppm oxygen is well detected.

Table 5 shows the gas 2 and gas 3 repeatability results in 2 channels. The RSD of RT% is less than 0.04% for all components. The RSD of Area % is less than 1% except 23.5 ppm O_2 in 10 m Molesieve 5Å channel with RSD of 3.51%. The higher RSD is due to Ar as a carrier gas. The low detection limit value is larger with Ar than in He as carrier, and 20 ppm O_2 is close to the lowest detection limit. Repeatability is a little worse, as the RSD% of O_2 in the carrier Ar will be reduced to 1% when O_2 concentration is higher than 100 ppm.

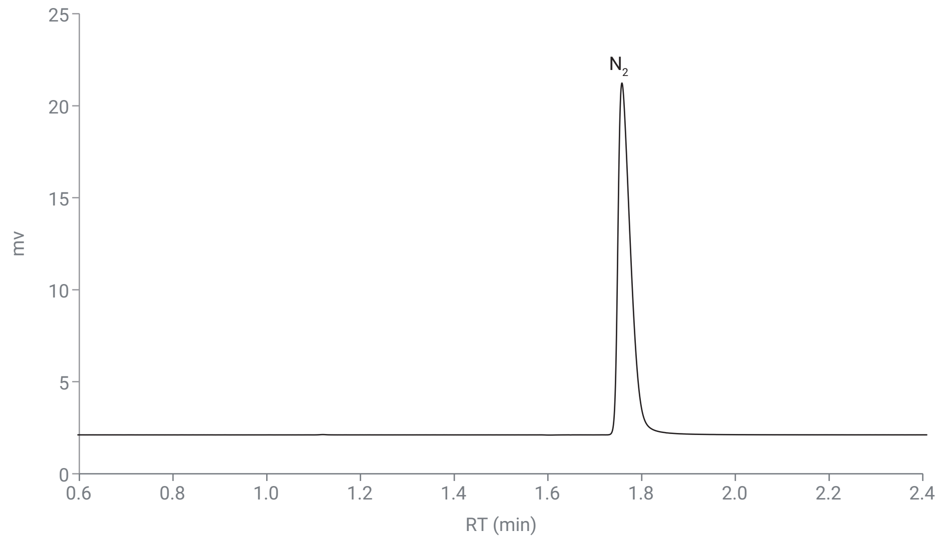

Figure 3A. Chromatogram of standards gas 3 on a 10 m Agilent CP-Molsieve 5Å channel using Ar as the carrier gas.

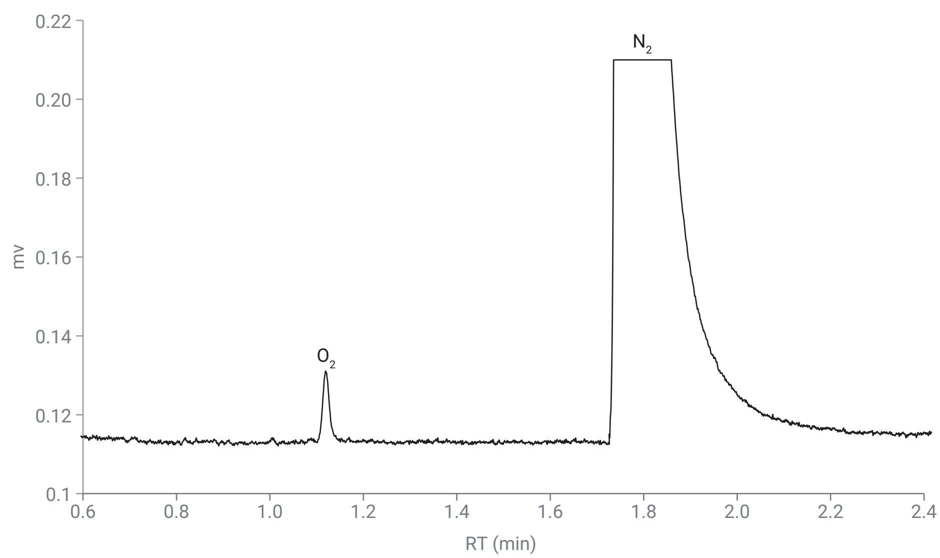

Figure 3B. Chromatogram of standards gas 3 on a 10 m Agilent CP-Molsieve 5 Å channel using Ar as the carrier gas.

Table 5. RT and area repeatability of 10 runs of standards gas 2 and gas 3.

Channel	Compound	RT (min)	RT RSD%	Area (mV × s)	Area RSD%
20 m Molsieve 5Å, He Carrier	22.8 ppm Ar	2.562	0.011%	0.01167	0.39%
	5.04% N_2	2.616	0.005%	313.84	0.19%
	Balance O_2	5.612	0.004%	18.39	0.33%
10 m Molsieve 5Å, Ar Carrier	23.5 ppm O_2	1.119	0.031%	0.02035	3.51%
	5.03% N_2	1.757	0.012%	36.07	0.09%

Conclusion

This application brief shows the applicability of the Agilent 990 Micro GC in permanent gas analysis. Fast and accurate separation of oxygen and nitrogen was achieved using two Molesieve 5Å channels. By choosing the right column length and the clever use of carrier gas, this instrument is a valuable tool for permanent gas manufacturers to qualify their gases. With the 990 Micro GC, ultra-fast results are obtained without any compromise to the quality of those results. This was clearly shown by the repeatability data on retention time and peak area.

References

1. Van Loon, R. Permanent Gas Analysis-Separation of Helium, Neon and Hydrogen a MolSieve 5Å column using the Agilent 490 Micro GC, *Agilent Technologies application note*, publication number 5990-8527EN, **2011**.
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 number5990-8700EN, **2011**.

www.agilent.com/chem

DE.4532407407

Printed in the USA, June 30, 2020
5994-2139EN