Application Note

Energy & Chemicals

Agilent
Trusted Answers

Noble Gases Analysis Using the Agilent 990 Micro GC

Authors

Fei Jiang and Tingting Bu
Agilent Technologies, Inc.

Abstract

This application note describes the use of the Agilent 990 Micro GC for noble gases analysis, which can be applied for fast and accurate detection of the noble gases. Two channels were used: the Agilent CP-Molsieve 5Å, and the Agilent CP-PoraPLOT U, to analyze noble gases such as neon, argon, krypton, and xenon.

Introduction

Noble gases that are commonly used in industry refer to argon, neon, helium, krypton, and xenon. These noble gases are widely used in the smelting and welding of rare metals, aerospace technology, superconducting technology, deep-water operation, electron tubes, laser and electric light sources, medical treatment, and so on. These noble gases are important materials for the development of modern science and technology.

Extraction of noble gases, such as argon by air separation, and helium from natural gas, have been widely used in industrial production. The output of neon, krypton, and xenon is also increasing, and the purity is improving.

With the wide application of noble gases and the development of technology, there is more demand for the analysis of noble gases during the noble gas extraction process. In this application note, the 990 Micro GC was applied for fast and accurate detection of noble gases, which can help improve the analysis efficiency during the extraction process.

Experimental

Channel 1: An Agilent CP-Molesieve 5Å, 20 m, with traditional backflush (BF) and retention time stability (RTS) options for neon, hydrogen, argon, and oxygen analysis. The BF and RTS options are used to protect the Molesieve 5Å column from moisture and carbon dioxide, and to reduce the retention time (RT) shift. This is beneficial to the long-term RT repeatability and column performance of the Molesieve 5Å column.

Channel 2: An Agilent CP-PoraPLOT U backflush channel, 20 m, for krypton, methane, and xenon analysis.

Analytical methods for sample analysis are outlined in Table 1, and the composition of the standards gas is outlined in Table 2.

Table 1. Analytical methods for sample analysis.

Conditions	Channel Type	
	Agilent CP-Molesieve 5Å, 20 m, RTS, Backflush	Agilent CP-PoraPLOT U, 20 m, Backflush
Carrier Gas	Helium	Helium
Injector Temperature	50 °C	50 °C
Column Temperature	50 °C	40 °C
Column Pressure	300 kpa	200 kpa
Injection Time	300 ms	40 ms
Backflush Time	10 s	15 s

Table 2. Composition of standards gas.

Components	Concentration (µmol/mol)
Neon	20.0
Hydrogen	27.4
Argon	34.0
Oxygen	23.8
Krypton	20.0
Xenon	20.0
Methane	20.6
Nitrogen	Balance

Results and discussion

The Molesieve 5Å, 20 m channel exhibited excellent neon/hydrogen and argon/oxygen baseline separation using helium as the carrier gas, as shown in Figure 1. Enlarging the RT from 0.8 to 1.2 minutes showed that 20.0 parts per million (ppm) neon and 27.4 ppm hydrogen in standard gas were well detected. Although the carrier gas was helium, the hydrogen still showed a response, and the signal-to-noise ratio (S/N) for hydrogen was 2.9.

The 20.0 ppm krypton, 20.6 ppm methane, and 20.0 ppm xenon were well-separated from nitrogen within 2.4 minutes on the CP-PoraPLOT U, 20 m, backflush channel, as shown in Figure 2. The resolution of krypton and methane was greater than 1.0 (R >1.0).

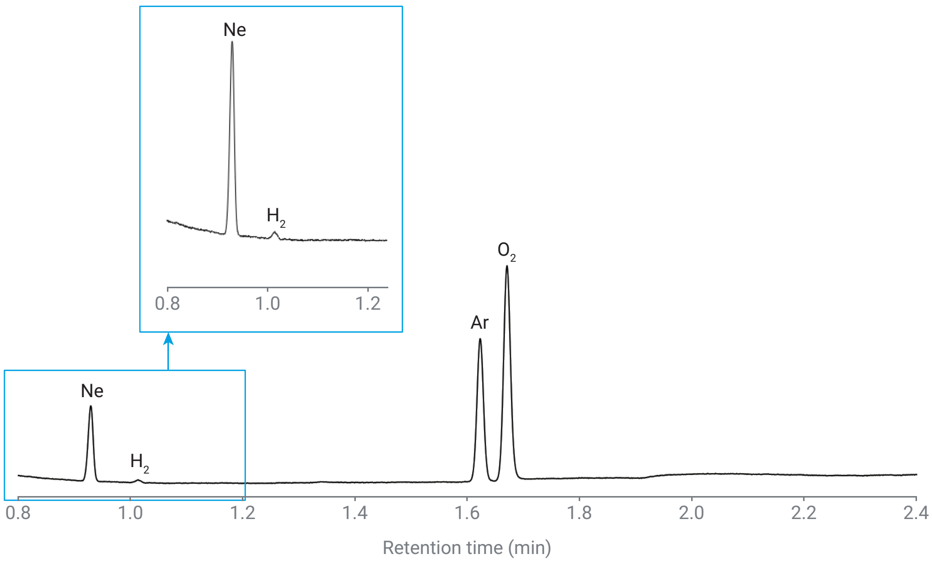

Figure 1. Chromatogram showing neon (Ne), hydrogen (H_2), argon (Ar) and oxygen (O_2) on the Agilent CP-Molesieve 5Å, 20 m channel.

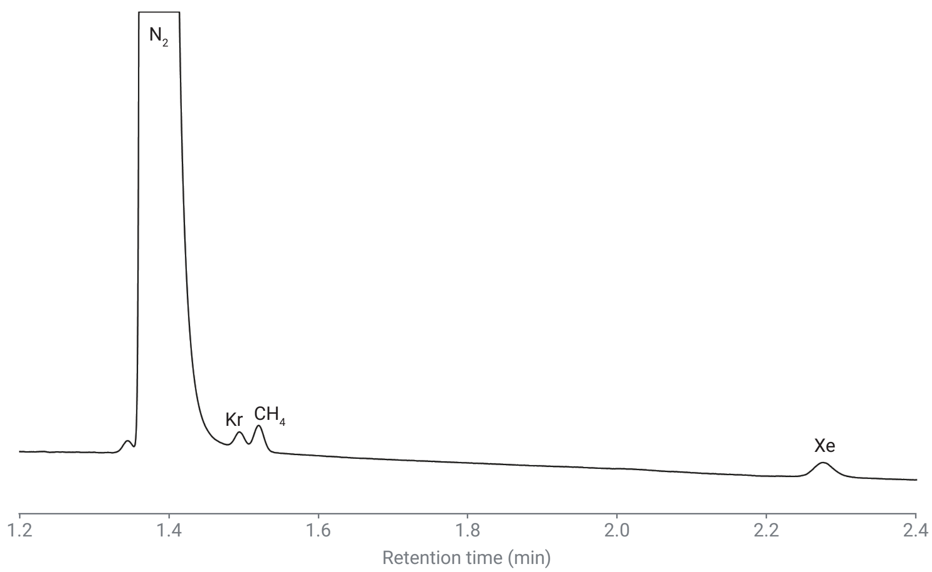

Figure 2. Chromatogram showing krypton (Kr), methane (CH_4) and xenon (Xe) on the Agilent CP-PoraPLOT U, 20 m channel.

The repeatability results and limits of detection (LODs) calculated from the 10 sample runs are shown in Table 3. The LOD was calculated as the signal of three times the noise. All components showed excellent RT relative standard deviation (%RSD), with values below 0.1%. For peak area %RSD, the values were smaller than 5%, except for hydrogen and oxygen, which had a %RSD of 6.69% and 6.94%, respectively. The calculated LODs were less than 3 ppm, except for hydrogen, which had an LOD of 28.3 ppm.

Table 3. RT, area repeatability, and LOD of 10 runs of the standards gas.

Compounds	Concentration (ppm)	RT/min	RT RSD%	Area (mV·s)	Area %RSD	LOD (ppm)
Neon	20.0	0.093	0.00	0.051	1.33	0.74
Hydrogen	27.4	1.014	0.06	0.003	6.69	28.3
Argon	34.0	1.624	0.00	0.124	4.16	0.65
Oxygen	23.8	1.672	0.00	0.206	6.94	0.27
Krypton	20.0	1.495	0.03	0.010	1.01	2.80
Methane	20.6	1.520	0.03	0.019	2.48	1.65
Xenon	20.0	2.275	0.04	0.026	4.05	2.50

Conclusion

This study demonstrates the applicability of the Agilent 990 Micro GC for the analysis of noble gases. Fast and accurate separation of noble gases in nitrogen/air was achieved using the Agilent CP-Molesieve 5Å, 20 m channel and the Agilent CP-PoraPLOT U, 20 m, backflush channel.

References

1. Remko van Loon. Permanent Gas Analysis – Separation of Helium, Neon and Hydrogen a MolSieve 5A column using the Agilent 490 Micro GC, *Agilent Technologies Application note*, 5990-8527EN, **2011**.
2. Bajja, M. Permanent Gas Analysis – Separation of Argon and Oxygen on a MolSieve 5A column using the Agilent 490 Micro GC, *Agilent Technologies Application note*, publish number: 5990-8700EN, **2011**.

www.agilent.com/chem

DE71770973

Printed in the USA, January 18, 2022
5994-4552EN