

Permanent Gases on a COX Module Using an Agilent 490 Micro GC

Application Note

Environmental

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Abstract

This application note demonstrates the capabilities of the Agilent J&W CP-COX column with the Agilent 490 Micro GC, including separation of permanent gases and backflush possibilities to ensure extended column lifetimes.

Introduction

Separation of permanent gases is usually performed on a Molsieve column. This column offers the best separation for all permanent gases but also has some severe drawbacks. Water and carbon dioxide do not elute from a Molsieve column under regular GC conditions. A bake out at high temperatures (250 – 300 °C) is needed to fully regenerate the column. Regeneration is very time consuming in a Micro GC usually taking overnight or longer because the maximum temperature is 180 °C. In addition, it is likely that regeneration from moisture does not occur at this temperature.

If there is no need to separate oxygen and nitrogen, the CP-COX column is a better alternative. It delivers good separation of permanent gases, and carbon dioxide elutes from the column. CP-COX is an ideal alternative for a Molsieve column, offering prolonged lifetime and instrument uptime.



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Experimental

Instrumentation

An Agilent 490 Micro GC system with a CP-COX column module was used for these experiments. The CP-COX column module was equipped with a heated injector and an optimal precolumn with backflush.

Conditions

| | |
|-------------------------|----------------|
| Column temperature | 100 °C |
| Carrier gas | Argon, 100 kPa |
| Backflush to vent time | 13 s |
| Injection time | 80 ms |
| Injection temperature | 110 °C |
| Sample line temperature | 100 °C |
| Sampling time | 30 s |
| Stabilization time | 5 s |
| Run time | 200 s |

Sample Information

Standard gas samples were used. Concentrations were in % levels.

Table 1. Repeatability Figures Per Component on Peak Area

| Run | He | H ₂ | N ₂ | CO | CH ₄ | CO ₂ |
|---------|----------|----------------|----------------|---------|-----------------|-----------------|
| 1 | 943213 | 16024030 | 20593423 | 1439534 | 1535598 | 1064007 |
| 2 | 947355 | 16092042 | 20685887 | 1444814 | 1538714 | 1062243 |
| 3 | 949818 | 16142635 | 20749728 | 1446996 | 1544418 | 1070193 |
| 4 | 949808 | 16167426 | 20781405 | 1449939 | 1542239 | 1066091 |
| 5 | 952725 | 16194789 | 20815739 | 1453498 | 1539162 | 1066940 |
| 6 | 952107 | 16206479 | 20826967 | 1456289 | 1543749 | 1063772 |
| 7 | 954648 | 16228802 | 20856620 | 1455219 | 1548126 | 1074325 |
| 8 | 954635 | 16249294 | 20879589 | 1456795 | 1547760 | 1079645 |
| 9 | 955454 | 16251565 | 20883920 | 1456611 | 1552320 | 1064839 |
| 10 | 955872 | 16250493 | 20901246 | 1473831 | 1547242 | 1065483 |
| Average | 951563.5 | 16180756 | 20797452 | 1453353 | 1543933 | 1067754 |
| St. Dev | 4053 | 75870 | 97930 | 9249 | 5122 | 5456 |
| RSD% | 0.43 | 0.47 | 0.47 | 0.64 | 0.33 | 0.51 |

Results and Discussion

The above settings produce the chromatogram shown in Figure 1, with repeatability data in Table 1.

The chromatogram shows a baseline separation of helium and hydrogen. Oxygen and nitrogen eluted as a single peak but separate from carbon monoxide and methane. Carbon dioxide eluted perfectly.

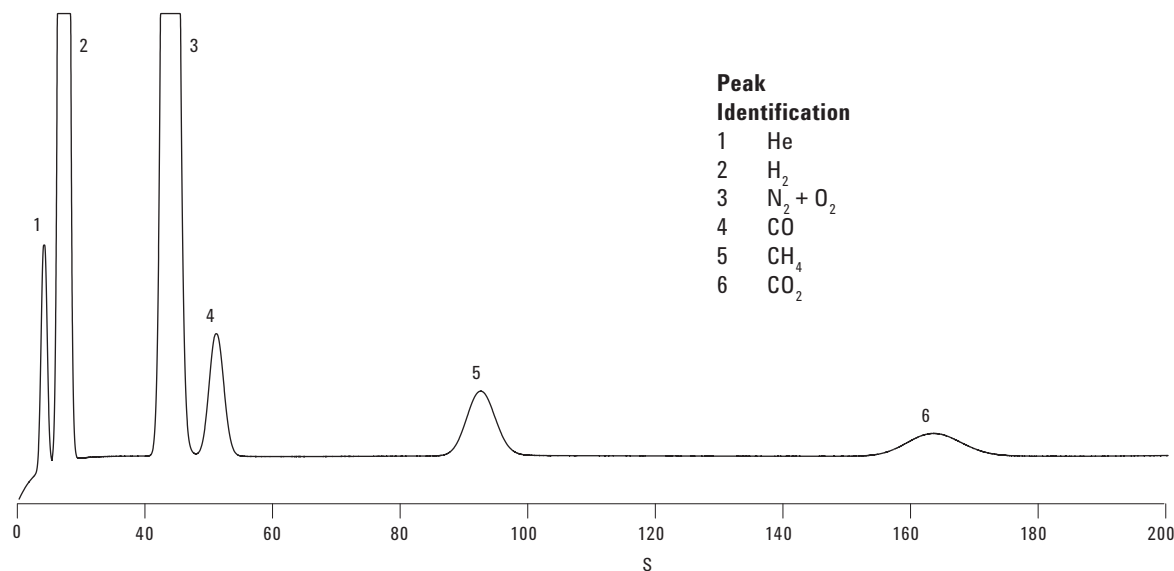


Figure 1. Excellent baseline separation of a gas sample on an Agilent CP- COX column.

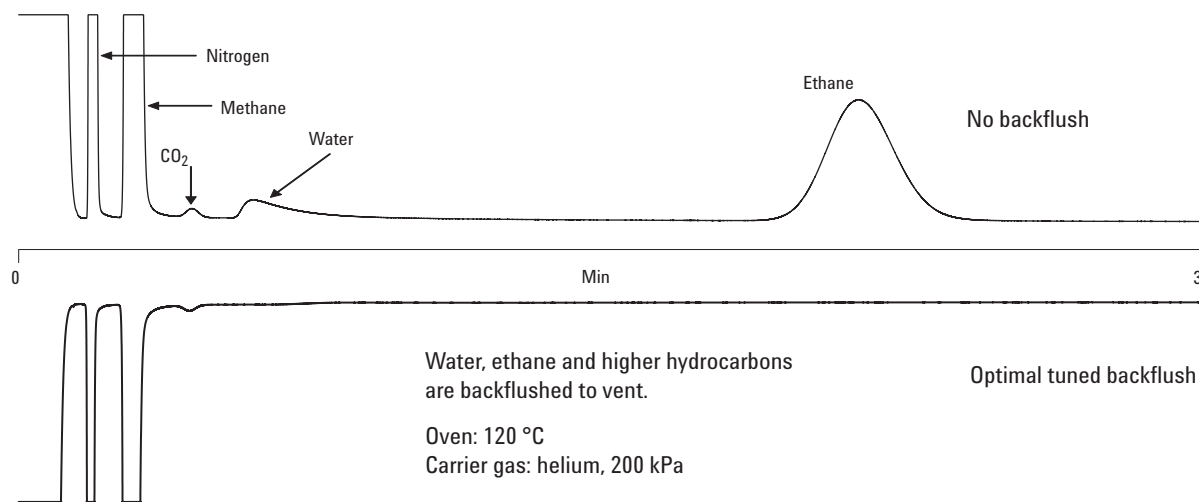


Figure 2. Backflush of water and ethane.

Other components such as water and higher hydrocarbons were backflushed to vent.

If the backflush time is set at a high value then virtually all the sample components enter the analytical column and eventually elute. However, if higher hydrocarbons are present the CP-COX column is polluted because these components elute late and can influence the succeeding analysis.

Figure 2 shows the elution of water and ethane if no backflush is applied. If the backflush time is optimally tuned, water, ethane and higher hydrocarbons are backflushed to vent and does not enter the analytical column.

Conclusion

For the analysis of permanent gases the Agilent J&W CP-COX column is a good alternative to the commonly used Molsieve column.

Although the CP-COX column does not separate oxygen and nitrogen, it does separate hydrogen and helium. In addition, carbon dioxide is analyzed and water elutes from the CP-COX column. Repeatability figures are good, ensuring reliable analysis results.

The COX module can be equipped with a precolumn. This allows backflush of higher components and prolongs column lifetime.

The Agilent 490 Micro GC is a rugged, compact and portable “lab-quality” gas analysis platform. When the composition of gas mixtures is critical, this fifth generation Micro Gas Chromatograph generates more data in less time for faster and better performance.

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