Fast and Accurate Analysis of Refinery Gas using Multi Channel Micro Gas Chromatography

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Introduction

The source and composition of each refinery gases varies considerably, therefore confident and precise analysis of refinery gases could be challenging. To succeed, your refinery gas analyzer must be able to quickly separate and accurately measure complex mixtures. Micro Gas Chromatography performs isothermal analysis, no column cool-down and stabilization is required, this results in very fast analysis times. The 490 Micro GC analyzes refinery gases in approximately 150 seconds.

Instrument Setup

The Agilent 490 Micro GC can be equipped with 1 to 4 independently controlled column channel. Each GC channel includes electronic carrier gas control, micro-machined injector, narrow-bore analytical column and micro thermal conductivity detector (µTCD).

For the results obtained and highlighted in this poster the 490 Micro GC with 4 column channel was used. Stationary phase and column length were optimized for refinery gas analytes.

Instrument control, data aqcuisition and report generation was done with Agilent OpenLAB CDS EZChrom Edition.

Figure 1. Agilent 490 Micro GC.

Results and Discussion

Molecular Sieve for Permanent Gases

The first column channel was equipped with a MolSieve 5A analytical column for the separation of permanent gases, except carbon dioxide (*Figure 2*).

Back flush to protect analytical column

Moisture and carbon dioxide tend to adsorb quickly to the molecular sieve stationary phase and change its chromatographic properties. This would results, over time, in retention shifts and loss of separation. To overcome this problem and to maintain the separation efficiency of this column, these compounds were back flushed.

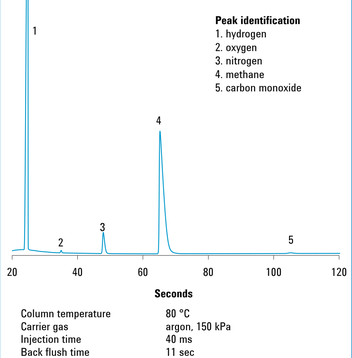

Figure 2. Chromatogram for refinery gas on MolSieve 5A column.

Results and Discussion

C2 hydrocarbons, CO_2 and H_2S on Plot U

Ethane, ethylene, acetylene as well as carbon dioxide and hydrogen sulfide were analyzed on a second column channel equipped with a PoraPLOT U column *(Figure 3)*.

Back flush to speed up analysis time

Hydrocarbons propane and above, present in the refinery gas, were back flushed to improve analysis time. A back flush Micro GC channel consists of a pre-column and an analytical column; the two columns are coupled at a pressure point. This setup enables the possibility to invert the carrier gas flow direction through the pre-column at any time. As a result the undesired compounds will not enter the analytical column and send to vent.

Saturated and unsaturated hydrocarbons analyzed on an alumina oxide column

On the third Micro GC channel, with an alumina oxide column, the C3 to C5 saturated and unsaturated hydrocarbons were separated. This channel was also equipped with the back flush option to prevent later eluting compounds intefering with the next analysis.

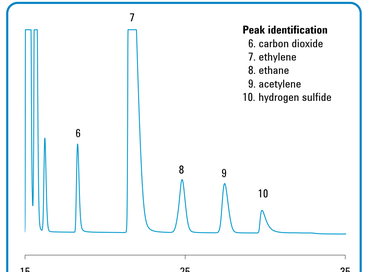

Figure 3. Chromatogram for refinery gas on PLOT U column.

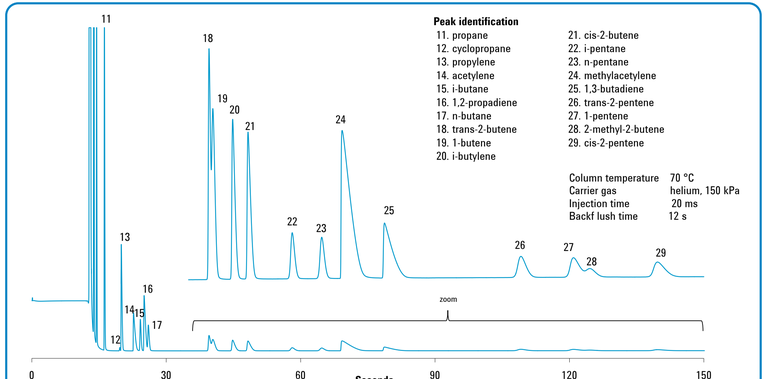

Figure 4. Chromatogram for refinery gas on 10 meter Al_2O_3 column (20 cm pre-column).

Results and Discussion

Non-polar column for C6+ hydrocarbons

A fourth channel, equipped with a non-polar phase column (CP-Sil 5) was used for the analysis of the hydrocarbons C6 and above. *Figure 5* shows the chromatogram for this channel with a total analysis time until n-decane of less than 2 minutes.

Reporting tools for physical properties

Reporting tools, integrated with the chromatography data system were used to calculate normalized Mole% and physical properties, such as BTU value. These calculation are conform official methods from American Society of Testing and Materials (ASTM), Gas Processors Association (GPA) and International Standards Organization (ISO).

Other Micro GC applications

Apart from refinery gases, the Agilent 490 Micro GC is used for a wide range of gas analysis, like natural gas, biogas, oil and gas exploration, catalyst reaction monitoring, fuel cell gases, air monitoring and mine safety analysis.

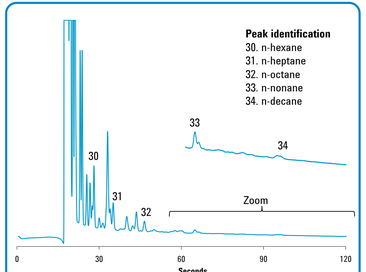

Figure 5. C6+ hydrocarbons on CP-Sil 5 CB.

Conclusions

- The 490 Micro GC was successfully used for fast analysis of refinery gases with a total analysis time of 150 seconds.
- Complex refinery gas mixture was simultaneously analyzed and quantified on 4 independently controlled Micro GC channels:
  - Molecular sieve column for permanent gases (except carbon dioxide),
  - Plot U column for C2 hydrocarbons, hydrogen sulfide and carbon dioxide,
  - Alumina oxide column for C3, C4 and C5 saturated and unsaturated hydrocarbons,
  - Non-polar phase (Sil 5) for C6+ hydrocarbons.
- Benefit of back flush functionality is protecting your analytical column as well as preventing later eluting compounds interfering with the next analysis.

References

To learn more about the Agilent 490 Micro GC Refinery Gas Analyzer visit www.agilent.com/chem/microgc

[1] SI-02233; Application Note – Fast Refinery Gas Analysis Using the 490 Micro GC QUAD; 2010.

[2] 5990-6664EN; Brochure – Agilent 490 Micro GC; 2010.

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