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Abstract

This application note describes methods for analyzing C1-C6 light hydrocarbons. An Agilent 6820 Gas Chromatograph configured with a 6-port gas-sampling valve interfaced to a split/splitless inlet and flame ionization detector was used. The Agilent HP-AL₂O₃ "M" deactivated 50 m PLOT column was used for separation.

Introduction

Measurement of light hydrocarbons is necessary in refinery gas streams, liquefied petroleum gas (LPG), and natural gas. Knowing the concentration and distribution of hydrocarbons in these gases is critical for controlling manufacturing processes and production quality. Light hydrocarbon measurements are also useful in the development of processes for new plant production. Those who produce, transport, or consume natural gas also require accurate analyses to monitor and to calculate ratios of different gas streams in order to optimize furnace performance. Detailed information about natural gas hydrocarbon content is also used to establish market pricing.

Both packed and capillary columns are used individually for analyzing light hydrocarbons. In packed columns, hydrocarbons heavier than C5 are usually backflushed ($C6^+$) by a switching valve [1]. Compared to packed columns, capillary PLOT columns have superior separation power with much lower bleed. Alumina columns can easily separate C4 and C5 isomers that are difficult or impossible to separate well with packed columns. They can also elute C6 hydrocarbons in most applications [2]. In this application, the HP-PLOT AL_2O_3 "M" deactivated column was used.

Experimental

The Agilent 6820 Gas Chromatograph was used for this work using helium as the carrier gas. The system is configured with a split/splitless capillary inlet and flame ionization detector (FID). An automatic 6-port gas-sampling valve heated to 80 °C injects gas samples. The gas sample valve is interfaced to the carrier line of the split inlet using a 1/16-inch stainless steel tubing. An aluminum tube is used to jacket the transfer line for improved thermal performance. A valving diagram for the system is shown in Figure 1.





Figure 1. System configuration.

PLOT Alumina 50 m \times 0.53 mm "M" deactivated column was used to separate hydrocarbons, including saturated, unsaturated, and aromatic compounds. An Agilent split liner (p/n 19251-60540) and bleed/temperature optimized Agilent advanced green septa (p/n 5183-4759) were used. Agilent Cerity NDS for chemical QA/QC was used for data acquisition and data analysis. Various temperature programs were used depending on sample complexity. Table 1 lists conditions representative for the hydrocarbon separations. Variation in the oven program, sample loop size, and split ratio are necessary to optimize the system for a given application. Care must be taken to optimize sample loop size to avoid overload of the alumina PLOT column.

Table 1. Gas Chromatographic Conditions

GC	Agilent 6820 Gas Chromatograph
Data system	Agilent Cerity NDS for chemical QA/QC, option 335
S/S Inlet	175 °C, 30:1 Split ratio typical
Valve	Gas sampling valve, 6-Port, option 701
Valve temperature	80 °C
Sample loop	0.1 mL typical
Column flow (He)	5 mL/min
Column	PLOT Alumina "M" 50 m \times 0.53 mm \times 0.25 μm (p/n: 19095P-M25)
Oven	40 °C (2 min) to 140 °C (5 min) at 4 °C/min
Detector	FID, 300 °C
H ₂	35 mL/min
Air	350 mL/min
Column and make up	26 mL/min

Results and Discussion

This system is suitable for analyzing light hydrocarbons, including C4 isomers, in a typical C1-C5 refinery gas stream. The FID is used for wide dynamic range hydrocarbon measurements, while for fixed gas determinations such as carbon dioxide, hydrogen, oxygen, and nitrogen, a second channel using a Thermal Conductivity Detector (TCD) is needed. The fully digital signal path employed in the 6820 allows measurement of ppm level impurities and very high percent level determinations in a single run. This is not possible for many gas chromatographs that use an analog

signal path. Split ratios and sample loop sizes were modified as needed, according to the concentrations present in the sample. This application demonstrates a simple but powerful 6820 configuration suitable for a wide variety of hydrocarbon samples. Figure 2 and Figure 3 show chromatograms for refinery gas standards. For some of the more complicated hydrocarbon analyses (compounds heavier than approximately C8), a 10-port valve with precolumn and backflush to vent can be employed to protect the PLOT column. As Figure 3 shows, 21 hydrocarbon components and isomers present in a standard sample achieve baseline separation.



Chromatogram of refinery gas test sample Split ratio: 30:1, Sample loop 0.1 mL. Figure 2.



12 c-2-Butene 13 Cyclopentane iso-Pentane 15 n-Pentane 1,3-Butadiene Methyl acetylene 18 t-2-Pentene 19 3-Methyl-1-butene 20 1,2-Pentadiene

- c-2-Pentene

Chromatogram of hydrocarbon standard sample Split ratio: 30:1, Sample loop 0.05 mL. Figure 3.

Conclusions

The Agilent 6820 gas chromatograph configured with a 6-port gas sampling valve, split/splitless inlet, and FID was used for detecting light hydrocarbons. The Agilent AL_2O_3 "M" PLOT column exhibited low bleed and baseline separation of C4 isomers. This system offers an easy-to-use configuration where the valve is interfaced directly to the split inlet for accurate sample transfer. Applications include refinery gas, LPG, natural gas, and C4 streams.

References

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