Application Note

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Light Hydrocarbons on the Agilent Intuvo 9000 GC with a Gas Sampling Valve

A reproducibility study with a gas sampling valve and GC detector splitter

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Abstract

This Application Note describes a method for analysis of a standard hydrocarbon gas mixture with an Agilent Intuvo 9000 GC. The system featured a gas sampling valve, a postcolumn D1/D2 detector splitter chip, and detection by flame ionization (FID) and thermal conductivity (TCD). Results were comparable, in terms of retention time and peak response, for both detectors in the flowpath.

Introduction

Samples can be introduced to a GC using various techniques, including a gas sampling valve. The Intuvo 9000 GC is amenable to this type of sampling, and is available with a single six-port gas sampling valve. This feature allows a smaller footprint in labs analyzing process streams, compressed gases, or other sample types already in the gas phase. Splitting the postcolumn sample to a second detector can add a level of efficiency to the analysis by extending the calibration range, or by adding more selectivity to the analysis. The Intuvo 9000 GC with gas sampling valve and D1/D2 detector splitter chip, combined with Agilent J&W HP-PLOT columns, provides a smaller, more productive option to separate and quantify gas samples with confidence.

This Application Note presents the reproducibility of results when using the gas sampling valve with a compressed gas standard, as configured on the Intuvo 9000 GC mainframe.

Sample preparation

This reproducibility study did not require sample preparation. The Agilent refinery gas test sample (p/n 5080-8755) was used to test the system. The cylinder containing the gas mix was connected through a stream selector for automation. Table 1 lists the compounds and approximate concentrations.

Experimental

Instrumentation

Intuvo Configuration	
Gas Sampling Valve	6-port, 0.1 mL loop
Inlet	Split/splitless
Inlet Liner	Ultra Inert with glass wool (p/n 5190-2295)
Intuvo Flowpath	Guard Chip (p/n G4587-60565) Inlet Flow Chip (p/n G4581-60031) D1/D2 GC detector splitter (p/n G4588-60402)
Analytical Column	Agilent J&W HP-PLOT AL2O3 M, 50 m × 0.32 mm × 8.0 µm
Detector 1	Flame ionization detector (FID)
Detector 2	Thermal conductivity detector (TCD)

Intuvo Operating Conditions	
Gas Sampling Valve (Thermal Aux #1)	150 °C
Loop Load Time Loop Inject Time	0.5 minutes 0.5 minutes
Inlet	150 °C, 200:1 split, 3 mL/min septum purge
Guard Chip	Track oven mode
Bus	225 °C
Analytical Column	Helium at 37.3 psig (5 mL/min)
Column Oven	75 °C, no hold Ramp at 15 °C/min to 175 °C, hold 1 minute Total run time = 7.67 minutes
FID Settings	250 °C Hydrogen = 30 mL/min Air = 400 mL/min Make up (N_2) = 25 mL/min
TCD Settings	200 °C Reference gas = 10 mL/min Make up gas = 5 mL/min

Table 1. Components of Agilent refinery gas test sample.

Compound	Concentration (%)
Methane	5
Ethane	10
Ethylene	1
Propane	5
Propylene	1
Isobutane	10
n-Butane	5
trans-2-Butene	5
1-Butene	10
cis-2-Butene	5
Isopentane	2
n-Pentane	1
Hydrogen	15
Nitrogen	15
Carbon dioxide	5
Carbon monoxide	5

Results and discussion

For this evaluation, 30 replicate injections were made using the gas sampling valve and an integrated stream selector for automation. The J&W HP-PLOT AL2O3 M column is not expected to separate the permanent gases in the standard, nor will the FID detect them. For these reasons, hydrogen, nitrogen, carbon dioxide, and carbon monoxide are excluded from the statistics. Figure 1 shows chromatograms for the FID and TCD, and Table 2 includes reproducibility statistics. The permanent gases coelute with methane on the TCD, and some additional variability is noted in the statistics for this peak.

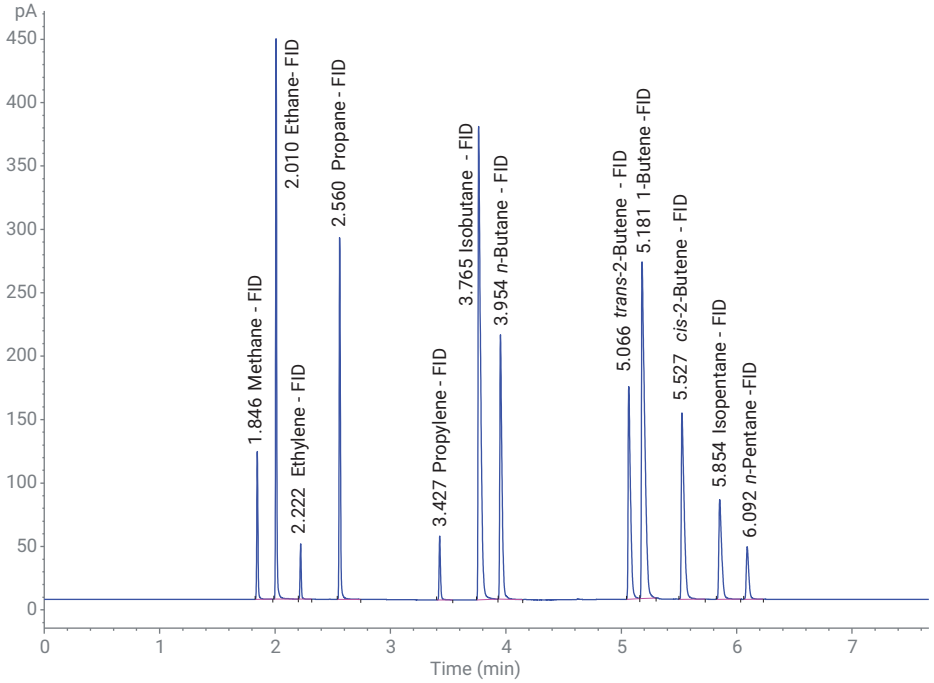

Figure 1A. FID chromatogram with retention time and compound labels.

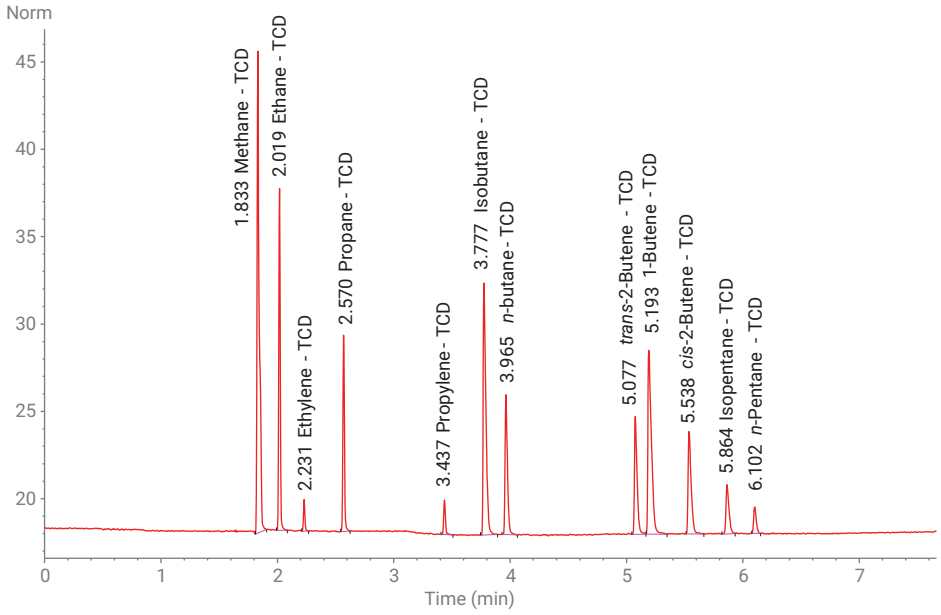

Figure 1B. TCD chromatogram with retention time and compound labels.

Conclusion

The Agilent Intuvo 9000 GC configured with a single gas sampling valve and Agilent J&W HP-PLOT columns delivers consistent results for gas streams. The evaluation of a postcolumn D1/D2 detector splitter for synchronous detection yields comparable results in retention time and peak response for both detectors in the flowpath. The Intuvo 9000 GC with smart key technology eliminates the need for flowpath configuration, complex calculations, and multiple column installations, adding another level of efficiency to a workflow.

Table 2. Reproducibility results for hydrocarbon compounds on TCD and FID detectors.

Compound	TCD - RT	TCD - Area	TCD - Height	FID - RT	FID - Area	FID - Height
Methane	0.00%	2.89%	3.99%	0.01%	1.05%	1.01%
Ethane	0.01%	0.93%	0.92%	0.02%	1.08%	1.02%
Ethylene	0.02%	3.07%	1.25%	0.02%	1.27%	1.03%
Propane	0.02%	1.20%	1.02%	0.02%	1.06%	1.02%
Propylene	0.04%	3.34%	1.29%	0.04%	1.06%	1.07%
Isobutane	0.03%	1.26%	1.07%	0.03%	1.10%	0.88%
n-Butane	0.03%	1.38%	1.05%	0.03%	1.24%	1.03%
trans-2-Butene	0.05%	1.48%	1.12%	0.05%	1.20%	1.09%
1-Butene	0.05%	1.32%	1.06%	0.05%	1.11%	1.14%
cis-2-Butene	0.05%	1.78%	1.27%	0.05%	1.21%	1.26%
Isopentane	0.04%	2.20%	1.38%	0.04%	1.26%	1.21%
n-Pentane	NA	NA	NA	0.04%	1.54%	1.49%

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