



C₅ and Lower Boiling Dienes, Olefins, and Paraffins in Naphthas by GC

UOP Method 980-17

Scope

This method is for determining C₅ and lighter diolefins, mono-olefins, and paraffins in olefinic naphthas. Resolved acetylenes and resolved C₆ hydrocarbons, including resolved dienes, are also identified. The method is applicable to selective hydrogenation process feeds, products, and other process streams with similar matrices having a final boiling point of 260 °C or lower. Components that do not elute from the chromatographic system under the specified conditions, if present in the sample, are not determined. The lower limit of detection for any compound is 0.01 mass-%. The *Appendix* describes an extension of the method to analyze LPG samples.

References

ASTM Method D 4307, "Preparation of Liquid Blends for Use as Analytical Standards,"

www.astm.org

ASTM Method D 5854, "Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products," www.astm.org

Scanlon, J. T. and Willis, D. E., *Journal of Chromatographic Science*, **23**, 333-340 (1985)

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The sample to be analyzed is injected into a gas chromatograph that is equipped with a fused silica capillary column, internally coated with (bonded) poly-dimethyl siloxane, and a flame ionization detector (FID). The mass-% composition of the sample is obtained by the internal normalization technique, wherein the peak areas are first corrected for differences in response and then normalized to 100%.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, readable to 0.1 mg

Chromatographic column, 100 m of 0.25-mm ID fused silica capillary, internally coated to a film thickness of 0.5 micron (bonded) with poly-dimethyl siloxane, Petrocol DH, Sigma-Aldrich, Cat. No. 24160-U

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Data system, electronic, for obtaining peak areas. This device must integrate areas at a sufficiently fast rate so that narrow peaks, typically obtained from a capillary column, can be accurately measured. The integrator must have programmable parameters for controlling baseline events, and have graphics capabilities. ChemStation, Agilent Technologies.

Electronic leak detector, Agilent gas leak detector, Agilent, Cat. No. G3388B

Freezer, flammable-materials storage, Fisher Scientific, Cat. No. 05-FFE-ETSA

Gas chromatograph, temperature programmable with cryogenic cooling, built for capillary column chromatography, utilizing a split injection system, equipped with a glass injection port insert, and a flame ionization detector that will give a minimum peak height response of five times the background noise for 0.01 mass-% *n*-heptane when operated at the recommended conditions, Agilent Technologies, Model 7890. The inclusion of an autosampler (or auto injector) is recommended but not required.

Gas purifier, hydrogen carrier gas purifier, VICI Metronics, Cat. No. P200-1

Regulator, air, two-stage, high purity, Matheson Tri-Gas, Model 3120A-590

Regulator, hydrogen, two-stage, high purity, Matheson Tri-Gas, Model 3120A-350

Regulator, nitrogen or helium, two-stage, high purity, Matheson Tri-Gas, Model 3120A-580

Sample injector, syringe or injector capable of introducing a 0.5 μ L volume of sample. An autosampler (or auto injector) is recommended, Agilent Technologies, Model 7693A.

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Air, zero gas, total hydrocarbons less than 2.0 ppm as methane

Autosampler vials, glass, 12- x 32- mm, for Agilent autosampler, Fisher Scientific, Cat. No. 03-391-8

Benzene, 99.9% purity, Aldrich, Cat. No. 270709

Caps, for autosampler vials, open hole with septum, screw, 9- mm, with Tef/Sil/Tef liner, Fisher Scientific, Cat. No. 03-377-2A

Carbon Dioxide, commercial liquid, 99.5% minimum purity, with full-length eductor tube for liquid withdrawal, for cryogenic cooling

Liquid Nitrogen, commercial liquid, obtained locally.

n-Heptane, 99.9+% purity, Sigma-Aldrich, Cat. No. 270512

Hydrogen, zero gas, 99.95% minimum purity, total hydrocarbons less than 0.5 ppm as methane

Pipet bulb, 1 mL, Fisher Scientific, Cat. No. 03-448-25

Pipets, disposable, Pasteur, Fisher Scientific, Cat. No. 13-678-20A

Nitrogen or helium, zero gas, total hydrocarbons less than 0.5 ppm as methane

Syringe, for sample injector, 5- μ L, Agilent Technologies, Cat. No. 51811273

Toluene, 99.8% purity, Sigma-Aldrich, Cat. No. 270377

Vials, 15- mL, with screw cap, Fisher Scientific, Cat. No. 03-339-21J

p-Xylene, 99+% purity, Sigma-Aldrich, Cat. No. 317195

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of gas chromatography, and the equipment being used. Dispose of used supplies and samples in an environmentally safe manner according to applicable regulations.

Sampling

Obtain the sample by following the procedures described in ASTM Practice D 4307, "Preparation of Liquid Blends for Use as Analytical Standards," ASTM Practice D 5854, "Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products," or other reliable technique.

Chromatographic Technique

The analyst is expected to be familiar with the technique of gas chromatography and the equipment being used.

1. Install the gas purifier in the supply line between the carrier gas source and the carrier gas inlets on the gas chromatograph.
 - Column life is significantly reduced if the gas purifier is not used.
2. Install the fused silica capillary column in the gas chromatograph, according to the column and gas chromatograph manufacturer's instructions.
 - CAUTION: Hydrogen leakage into the confined volume of the column oven can cause a violent explosion. It is mandatory to check for leaks each time a connection is made and periodically thereafter.

Table 1
Recommended Operating Conditions

Carrier gas	hydrogen
Mode	constant flow
Head pressure	207 kPa gauge (30 psig)
Linear velocity @ 15 °C	38 cm/sec
Equivalent flow @ 15 °C	2.6 mL/min
Split flow	200 mL/min
Injection port temperature	215 °C
Column temperature program	
Initial temperature	15 °C
Initial hold time	15 min
Programming rate A	8 °C/min
Intermediate temperature	75 °C
Intermediate hold time	14 min
Programming rate B	20 °C/min
Final hold temperature	250 °C
Final hold time	27.5 min
Detector	flame ionization
Detector temperature	250 °C
Hydrogen flow rate*	30 mL/min
Air flow rate*	400 mL/min
Makeup gas	nitrogen or helium
Makeup gas flow rate*	30 mL/min
Sample size	0.5 µL

*Consult the manufacturer's instrument manual for suggested flow rates.

3. Establish the recommended operating conditions as given in Table 1.
 - Different conditions may be used provided they produce the required sensitivity and chromatographic separations equivalent to those shown in the Typical Chromatograms (Figures 1, 2, and 3).
4. Program the column oven to 250 °C. Maintain this temperature until a stable baseline has been obtained at the required sensitivity.
5. Cool the column oven to a stabilized 15 °C.
7. In order to retain the light components, all samples should be placed in a freezer for about 30 minutes prior to sampling into vials for injections. Inject 0.5 µL of the sample to be analyzed into the gas chromatograph and start the integrator and column temperature programming sequence.
 - Better precision is obtained when an autosampler is used.
 - If using an autosampler, multiple samples may be transferred into autosampler vials and preloaded into the tray.
 - If samples are not to be run immediately, they should be placed in a freezer until they are analyzed. Samples should be removed from freezer and well mixed prior to injection.
8. Identify the components by comparing the resultant chromatogram with the Typical Chromatograms (see Figures 1, 2, and 3) and Table 2. The Peak No. in Table 2 refers to the identifications in the Typical Chromatograms, Figures 1, 2, and 3. If necessary, confirm the sites of the aromatic compounds by comparison with the calibration blend (see *Calibration*). Group all unidentified components as Unknowns.
 - Any individual peak or peaks can be identified and reported separately.

Table 2
Identified Components

Peak No.	<u>Component Identification</u>
	Ethylene*
	Ethane*
1	Propylene
2	Propane
3	Isobutane
4	1-Butene & Isobutylene
5	1,3-Butadiene
6	<i>n</i>-Butane
7	Vinylacetylene
8	<i>trans</i>-2-Butene
9	Ethylacetylene & 2,2-Dimethylpropane
10	<i>cis</i>-2-Butene
11	1,2-Butadiene
12	3-Methyl-1-butene
13	Isopentane
14	1,4-Pentadiene
15	1-Pentene
16	2-Methyl-1-butene
17	<i>n</i>-Pentane
18	2-Methyl-1,3-butadiene
19	<i>trans</i>-2-Pentene
20	3,3-Dimethyl-1-butene
21	<i>cis</i>-2-Pentene
22	2-Methyl-2-butene
23	1-<i>trans</i>-3-Pentadiene

Peak No.	Component Identification
24	3-Methyl-1,2-butadiene & Cyclopentadiene
25	1- <i>cis</i> -3-Pentadiene
26	2,2-Dimethylbutane
27	3-Methyl-1,4-pentadiene
28	Cyclopentene
29	4-Methyl-1-pentene
30	3-Methyl-1-pentene
31	Cyclopentane
32	2,3-Dimethylbutane
33	2,3-Dimethylbutene
34	<i>cis</i> -4-Methyl-2-pentene
35	2-Methylpentane
36	<i>trans</i> -4-Methyl-2-pentene
37	2-Methyl-1,4-pentadiene
38	1,5-Hexadiene
39	3-Methylpentane
40	2-Methyl-1-pentene
41	1-Hexene
	1,4-Hexadiene*
42	2-Ethyl-1-butene
43	<i>n</i> -Hexane
44	<i>cis</i> -3-Hexene
45	<i>trans</i> -3-Hexene
46	<i>trans</i> -2-Hexene
47	2-Methyl-2-pentene & 3-Methylcyclopentene
48	<i>cis</i> -3-Methyl-2-pentene
49	<i>cis</i> -2-Hexene
50	2,3-Dimethyl-1,3-butadiene
51	3-Methyl- <i>trans</i> -2-pentene
52	<i>trans</i> -1,3-Hexadiene
53	<i>cis</i> -1,3-Hexadiene
54	Methylcyclopentane
55	2,3-Dimethyl-2-butene
56	Methylenecyclopentane
57	Benzene & 1-Methylcyclopentene
58	Toluene
59	Ethylbenzene
60	<i>m</i> -Xylene
61	<i>p</i> -Xylene
62	<i>o</i> -Xylene

*Unnumbered components are not shown in Figures 1, 2, and 3.

Calibration

This analysis uses internal normalization of the total sample to be quantitative, and requires that all components in the sample which elute from the column under the specified conditions are measured. Any components which do not elute from the column under the specified conditions are not measured.

In order to accurately measure the total sample, certain components beyond the C₅ range must be identified and calibrated. This is necessary because C₆ through C₈ aromatic components that may be present in the sample do not have the same detector response as the non-aromatic components. All non-aromatic components have essentially the same detector response.

1. Prepare a blend as described in ASTM Method D4307 to contain approximately equal quantities of *n*-heptane, benzene, toluene, and *p*-xylene.
2. Analyze the blend in triplicate as described under *Chromatographic Technique*.
 - The peak areas from each of the three runs should not deviate from the average by more than 3%. If greater deviations occur, make certain that there are no problems with the equipment or technique, and then make additional runs until the required repeatability is obtained on three consecutive runs.
 - A calibration blend is run when the method is initially set up and thereafter when changes have been made to the equipment or periodically to verify proper calibration.
3. Based upon three replicate runs of the blend, determine the average relative response factors to four decimal places for each component using Equation 1 and *n*-heptane as reference:

$$F = \frac{AB}{CD} \quad (1)$$

where:

- A** = component of interest, mass-%
B = area of *n*-heptane peak
C = *n*-heptane, mass-%
D = area of component of interest
F = relative response factor

Calculated relative response factors should be similar to the theoretical relative response factors shown in Table 3. The theoretical relative response factors were calculated using the effective carbon number (ECN) concept as described by Scanlon and Willis. If the determined relative response factors differ by more than 10% from those shown in Table 3, check the apparatus, operating conditions, and blend preparation procedures.

Table 3
Theoretical Relative Response Factors

<i>n</i>-Heptane	1.000
Benzene	0.909
Toluene	0.919
<i>p</i>-Xylene	0.927

All C₈ aromatics will use the same factor as *p*-xylene. Due to the volatility of C₆ and lighter material, and the similarity of response factors, the response factor for *n*-heptane can represent all paraffins, olefins, diolefins, and unknowns.

Calculations

Obtain peak areas for each individual component or group of components and calculate the concentration of each identified peak in the sample to the nearest 0.01 mass-% using Equation 2:

$$\text{Component, mass-\%} = 100 \frac{PF}{T} \quad (2)$$

where:

- F** = relative response factor, from Equation 1
P = area of individual component or group of components
T = the sum of the products, **PF**, of all the recorded peaks

Precision

Precision statements were determined using UOP Method 999 from precision data obtained using an autosampler.

Repeatability, Site Precision and Reproducibility

A nested design was carried out for determining components in one olefinic hydrocarbon sample by two analysts on two separate days, performing two analyses each day in three separate laboratories for a total of 24 analyses. Using a stepwise analysis of variance procedure, the within-day estimated standard deviations (esd) were calculated at the concentration means listed in Table 4. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 4 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 4 with 95% confidence. Two tests performed by different analysts in different laboratories on different days should not differ by more than the reproducibility allowable difference shown in Table 4 with 95% confidence.

Table 4
Repeatability, Site Precision and Reproducibility, mass-%

<u>Component</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>		<u>Reproducibility</u>	
		<u>Within Day esd</u>	<u>Allowable Difference</u>	<u>Within Lab esd</u>	<u>Allowable Difference</u>	<u>Between Lab esd</u>	<u>Allowable Difference</u>
Cyclopentane	0.10	0.019	0.06	0.020	0.06	0.020	0.06
Isopentane	1.08	0.010	0.10	0.006	0.10	0.020	0.12
Pentane	13.02	0.065	0.20	0.066	0.20	0.250	1.52

The data in Table 4 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Time for Analysis

The elapsed time for one analysis is 1.5 hours. The labor requirement is 0.5 hour.

Suggested Suppliers

Agilent Technologies, 2850 Centerville Rd., Wilmington, DE 19808-1610, USA, 1-302-633-8000, www.agilent.com

Fisher Scientific, 585 Alpha Dr., Pittsburgh, PA 15238 (412-490-8300) www.fishersci.com

Matheson Tri-Gas, 150 Allen Rd. #302, Basking Ridge, NJ 07920, USA, 1-908-991-9200, www.mathesontrigas.com

Sigma-Aldrich, 3050 Spruce St., St. Louis, MO 63103, USA, 1-314-771-5765, www.sigma-aldrich.com

VICI Mat/Sen, 7806 Bobbitt, Houston, TX 77055, USA (713-688-9345) www.vicimatsen.com

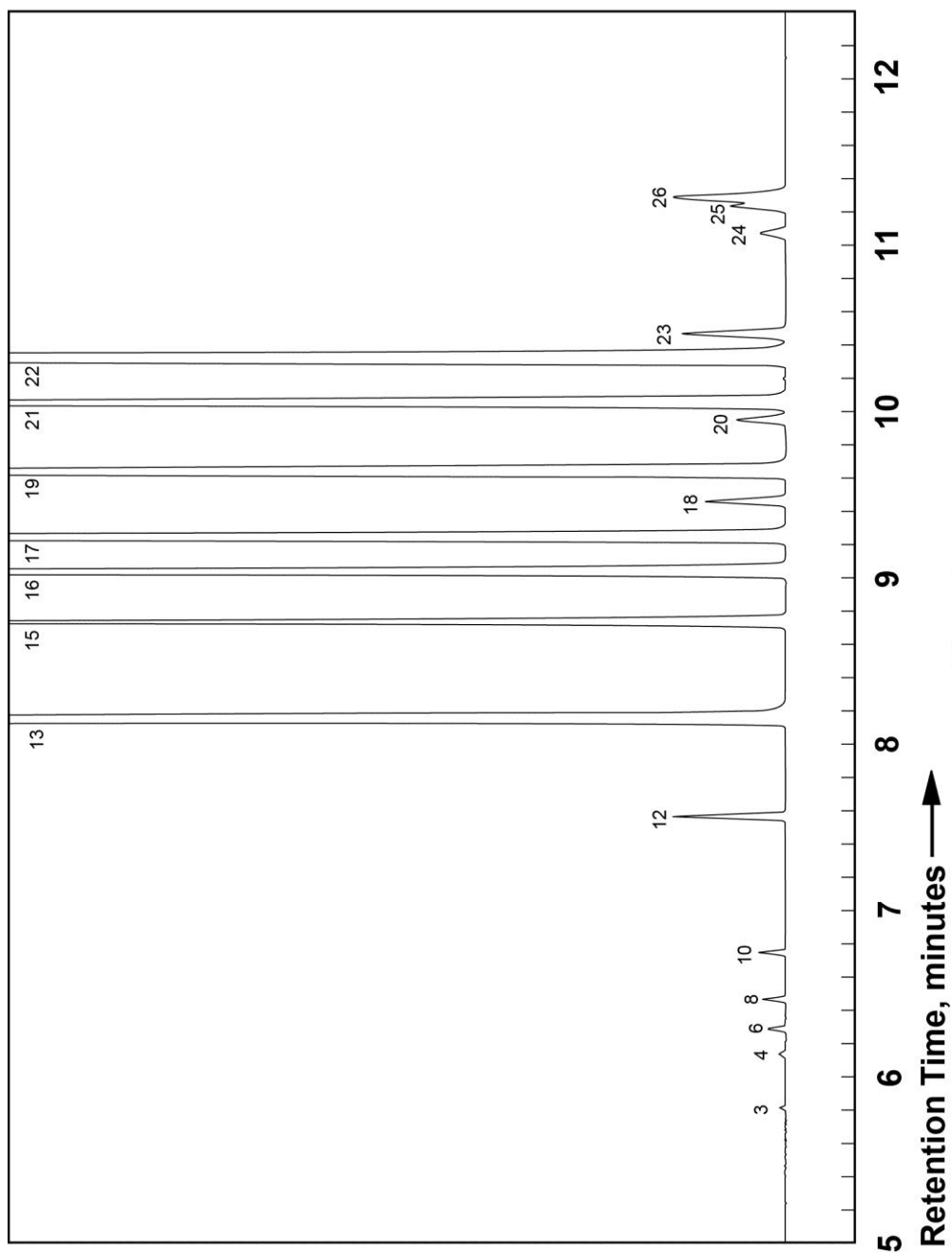


Figure 1

Typical Chromatogram (5-12.5 minutes)

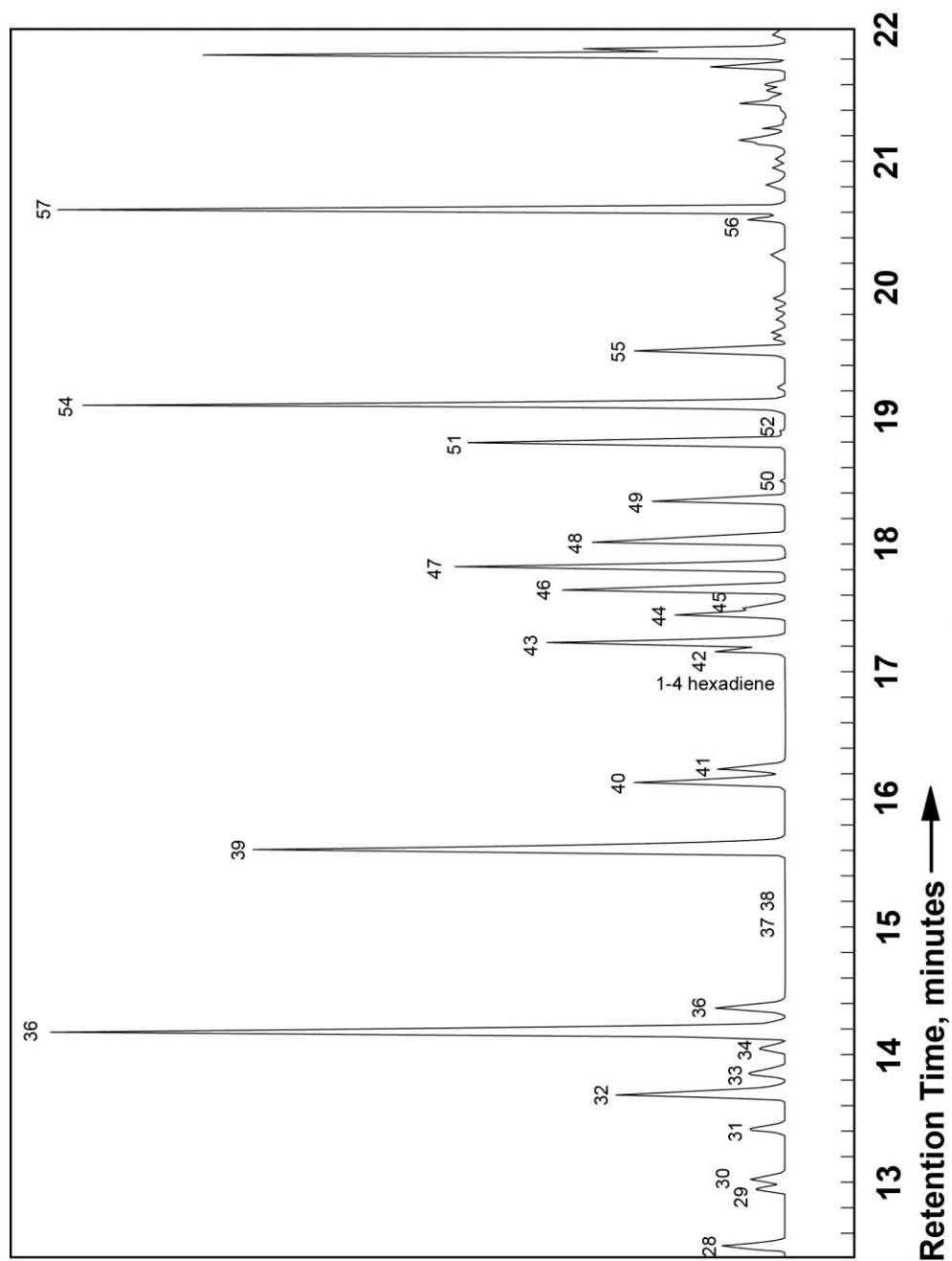


Figure 2

Typical Chromatogram (12.5- 22 minutes)

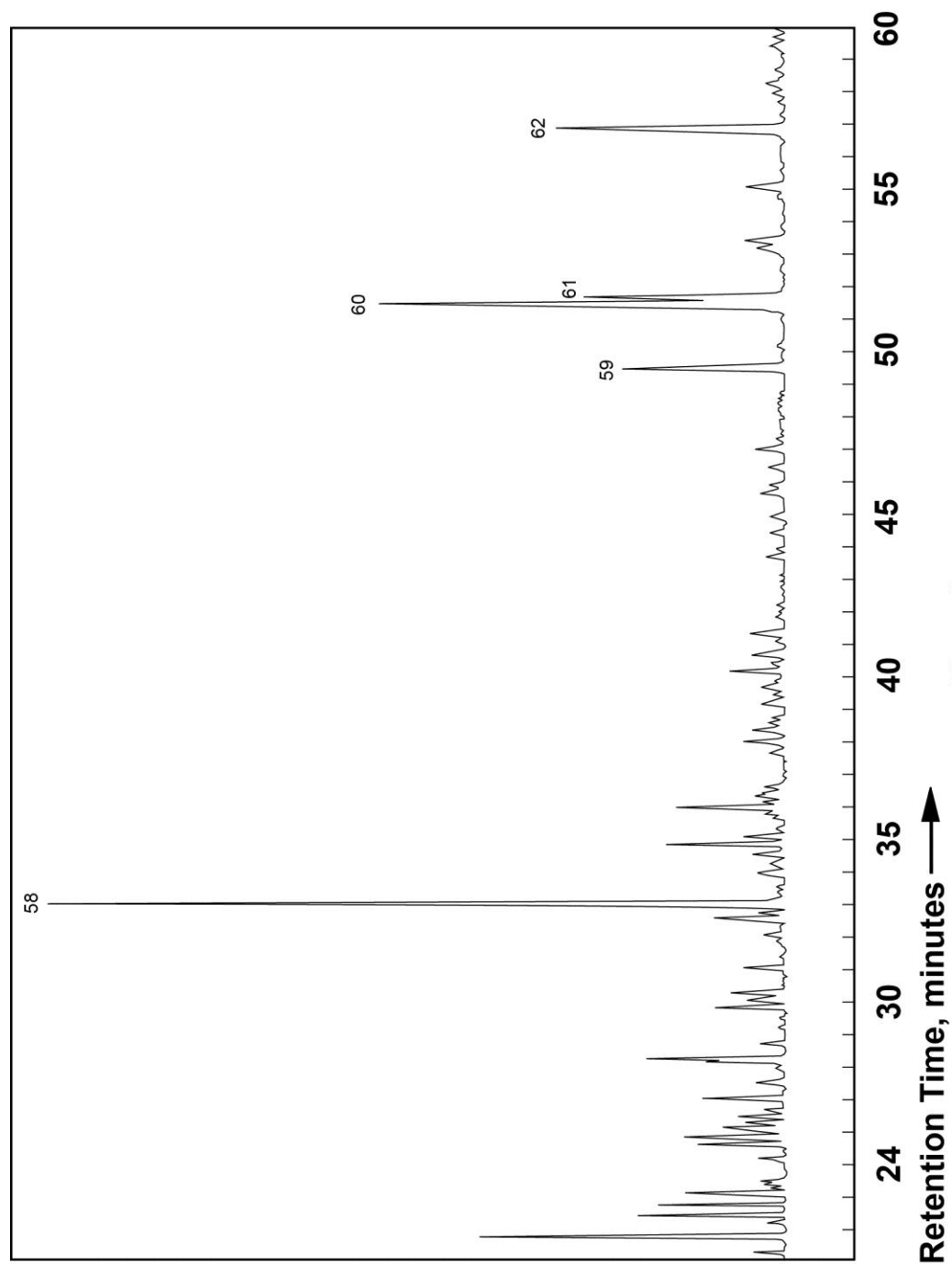


Figure 3
Typical Chromatogram (22 – end minutes)

Appendix

Analysis of Liquefied Petroleum Gas (LPG)

Scope

This appendix describes the extension of the method to analyze LPG samples in addition to liquid hydrocarbon samples. This method is not optimized to provide complete isomer distribution of all the olefinic compounds that may be present in LPG, but may be useful in determining the heavier (C₅ plus) components that are sometimes present in LPG, either by specific component or by grouping. For a more complete isomer distribution of C₃ and C₄ components, see ASTM Method D 2163, “Analysis of Liquefied Petroleum (LP) Gases and Propene Concentrates by Gas Chromatography”, UOP Method 373, “Composition of C₂ through C₅ Hydrocarbon Mixtures by Gas Chromatography”, or UOP Method 539, “Refinery Gas Analysis by Gas Chromatography”.

References

- ASTM Practice D 1265, “Sampling Liquefied Petroleum (LP) Gases (Manual methods),” www.astm.org
- ASTM Method D 2163, “Analysis of Liquefied Petroleum (LP) Gases and Propene Concentrates by Gas Chromatography,” www.astm.org
- UOP Method 373, “Composition of C₂ through C₅ Hydrocarbon Mixtures by Gas Chromatography,” www.astm.org
- UOP Method 516, “Sampling and Handling of Gasolines, Distillate Fuels, and C₃-C₄ Fractions,” www.astm.org
- UOP Method 539, “Refinery Gas Analysis by Gas Chromatography,” www.astm.org

Outline of Method

The LPG sample is injected as a liquid under pressure. The remainder of the method is unchanged.

Apparatus

The following apparatus is required in addition to the apparatus listed in the body of the method.

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Injection apparatus, LPG, for injection of LPG samples in the liquid phase, see Figure A1:

Fitting, internal union, for connecting injection port to 4-port sampling valve, Valco Instruments, Cat. No. ZU1T

Fitting, reducing union, 3.2- mm (1/8- inch) to 1.6- mm (1/16- inch), Swagelok, Cat. No. SS-200-6-1

Tubing, stainless steel, 1.6-mm (1/16-inch) OD, 0.76- mm (0.30- inch) ID, SGE Analytical Science, Cat. No. 062416

Tubing, translucent, FEP Teflon, 3.2-mm (1/8-inch) OD, 1.6- mm (1/16- inch) ID, 3450 kPa (500 psi), Thomas Scientific, Cat. No. 9567K20

Valve, 4-port, Stainless Steel, 0.5-ul internal sample volume, 1.6- mm (1/16- inch) fittings, manual with standoff, Valco Instruments, Cat. No. CI4UWE.5

Valve, vent shut-off, Swagelok, Cat. No. SS-ORS2

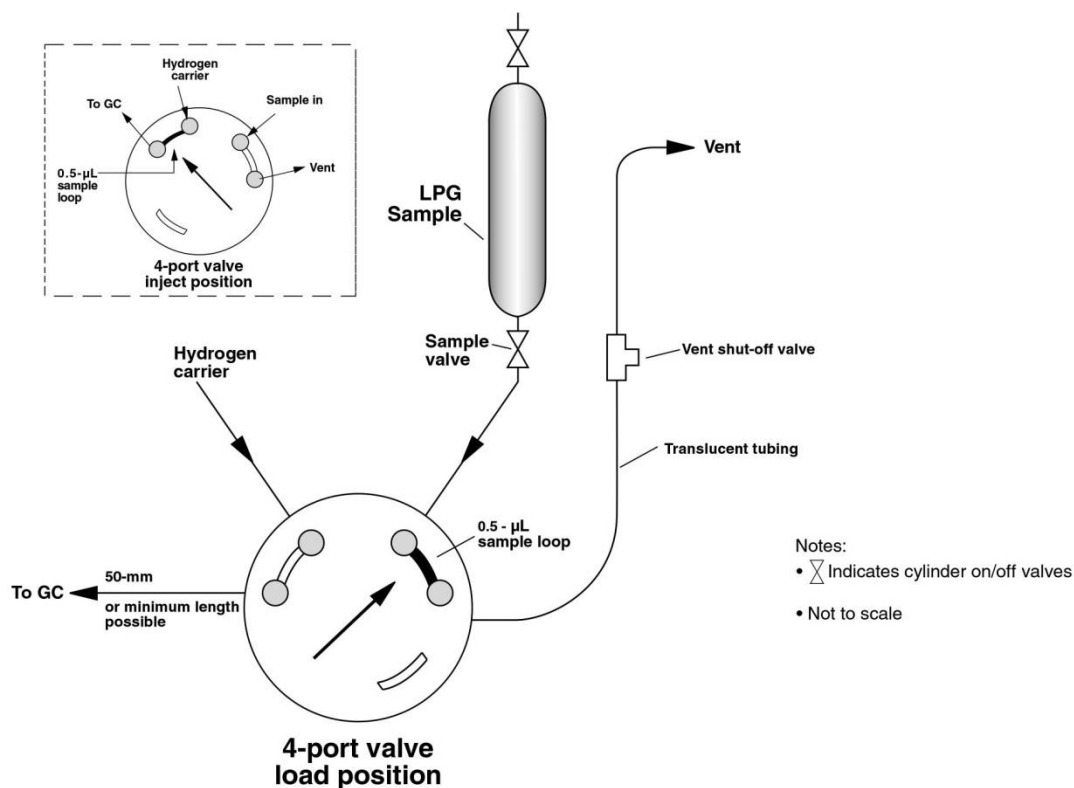


Figure A1
LPG Sampling System

Procedure

Sampling

Obtain the sample by following the procedures described in ASTM Practice D 1265, “Sampling Liquefied Petroleum (LP) Gases (Manual Methods),” UOP Method 516, “Sampling and Handling of Gasolines, Distillate Fuels, and C3-C4 Fractions,” or other reliable technique. The specially coated cylinders described in UOP Method 516 may be used but are not required for this application.

Chromatographic Technique

1. Assemble the apparatus as described in *Procedure*, Steps 1 and 2, in the body of the method.
2. Install the LPG injection valve in the carrier gas line just ahead of the injection port and connect the valve ports according to the manufacturer’s instructions. Refer to Figure A1.
 - An injection valve is required for LPG samples.
 - The line from the injection valve to the injection port should be as short as possible.
 - The injection valve should be mounted so that it maintains ambient temperature at the start of the run to keep liquid from bubbling in the valve or injection lines prior to injection.
 - Route the vent line away from personnel, to a hood or other exhaust, in accordance with local safety procedures.

3. Establish the recommended operating conditions as described in *Procedure*, Steps 3 through 5, in the body of the method and stabilize the instrument at the initial column temperature.
4. If the sample may contain water or sediment, or is of unknown origin, it must be checked for these contaminants before analysis by releasing a small amount of the sample from the bottom of the cylinder. Place the LPG sample cylinder in a vertical position in a hood or well-vented area. If the cylinder has an outage tube, the outage tube must be at the top. Briefly open the bottom valve into a suitable container in a fume hood to check that no water or sediment is present in the LPG. If water or sediment is determined to be present, discontinue the analysis and obtain a clean sample.
5. Pressurize the LPG cylinder containing the sample (or blend) to approximately 1400 to 2000 kPa gauge (200 to 300 psig) with nitrogen or helium.
6. Mount the cylinder in a vertical position. If the cylinder has an outage tube, the outage tube fitting must be at the top. Connect the bottom valve to the sample injector valve inlet tubing.
 - If the cylinder is fitted with an eductor tube, connect the eductor tube outlet to the sample injection inlet tubing in such a manner that the eductor tube is sampling liquid LPG. See Figure A2 for diagram of common LPG sample cylinders.
 - The connecting tubing between the cylinder and the injector valve must be as short as possible.
7. Place the injection valve in the fill position.
8. Ensure that the vent shut-off valve is closed.
9. Fully open the bottom valve, or eductor valve of the sample cylinder.
10. Partially open the vent shut-off valve to permit LPG flow through the sampling system.
11. Continue the flow until entrained bubbles are no longer observed through the translucent tubing, and inlet lines are adequately flushed of previous sample residuals.
 - CAUTION: Inspect the translucent tubing regularly. Replace at first signs of wear or kinking. Pressure on the translucent tubing must never exceed 2000 kPa (300 psig).
12. Stop the sample flow by closing the vent shut-off valve.
13. Inject the sample immediately by switching the injection valve to the inject position, and start the integrator and the column temperature programming sequence.
 - The injection valve remains in the inject position for the duration of the sample run.
14. Close the LPG cylinder valve and immediately open the vent shut-off valve to vent the sampling system.
15. Identify each component and group of components and calculate their concentrations as described in *Procedure*, Step 8, and *Calculations* in the body of the method.

Precision

Precision statements were determined using UOP Method 999.

Repeatability and Site Precision

A nested design was carried out for determining components in one LPG sample with two analysts in one laboratory. Each analyst carried out tests on two separate days, performing three tests each day. The total number of tests for each component was 12. The precision data are summarized in Table A1. Two tests performed by the same analyst on the same day should not differ by more than the repeatability allowable difference with 95% confidence. Two tests performed in one laboratory by

different analysts on different days should not differ by more than the site precision allowable difference with 95% confidence.

The data in Table A1 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Table A1
Repeatability and Site Precision, mass-%

<u>Component</u>	<u>Repeatability</u>			<u>Site Precision</u>	
	<u>Mean</u> <u>Conc.</u>	<u>Within-</u> <u>Day esd</u>	<u>Allowable</u> <u>Difference</u>	<u>Within-</u> <u>Lab esd</u>	<u>Allowable</u> <u>Difference</u>
Propylene	25.15	0.190	0.60	0.194	0.63
Propane	14.39	0.101	0.31	0.109	0.35
1-Butane	16.18	0.054	0.18	0.056	0.18
1-Butene & Isobutylene	17.55	0.077	0.24	0.081	0.26
1,3-Butadiene	0.24	0.005	0.02	0.005	0.02
<i>n</i> -Butane	9.51	0.045	0.14	0.047	0.15
<i>trans</i> -2-butene	9.12	0.052	0.16	0.053	0.17
Ethylacetylene & neopentane	0.01	0.004	0.01	0.007	0.04
<i>cis</i> -2-butene	6.52	0.037	0.12	0.039	0.13
3-Methyl-1-butene	0.31	0.002	0.01	0.002	0.01
Isopentane	0.76	0.006	0.02	0.006	0.02
1,4-Pentadiene	0.02	0.004	0.01	0.004	0.01
1-Pentene	0.07	0.001	0.01	0.001	0.01
2-Methyl-1-butene	0.10	0.001	0.01	0.001	0.01
<i>n</i> -Pentane	0.02	0.001	0.01	0.001	0.01
<i>trans</i> -2-pentene	0.02	0.001	0.01	0.001	0.01
<i>cis</i> -2-Pentene	0.01	0.001	0.01	0.001	0.01
2-Methyl-2-butene	0.02	0.0004	0.01	0.001	0.01

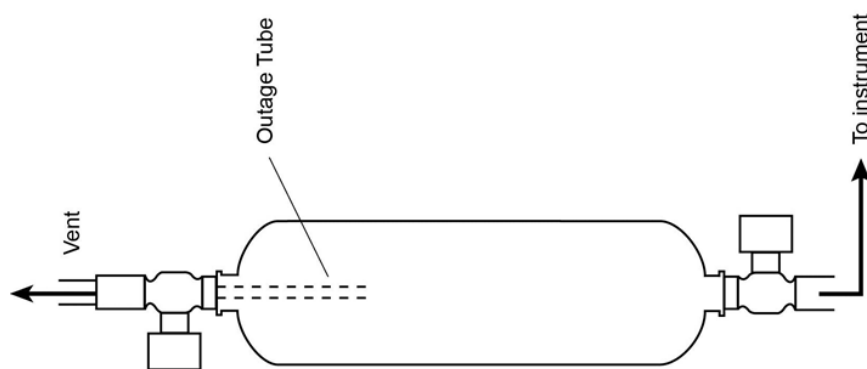
Reproducibility

There is insufficient data to calculate reproducibility of the test at this time.

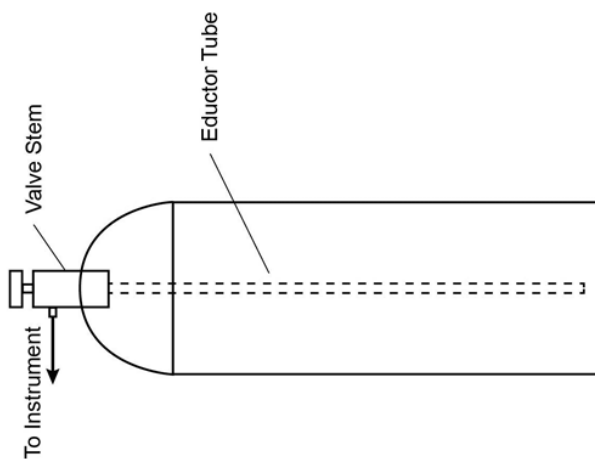
Suggested Suppliers

Swagelok Company, 29500 Solon Rd., Solon, OH 44139, USA, 1-440-248-4600,
www.swagelok.com

Valco Instrument Co., Inc. (VICI), P. O. Box 55603, Houston, TX 77255, USA (713-688-9345)
www.vici.com



Sample Cylinder with Outage Tube



Sample Cylinder with Eductor Tube

Figure A2
Common Sample Cylinder Diagrams