



Standard Test Method for Determination of Residue Composition in Liquefied Petroleum Gas (LPG) Using Automated Thermal Desorption/Gas Chromatography (ATD/GC)¹

This standard is issued under the fixed designation D7828; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of residue in LPG by automated thermal desorption/gas chromatography (ATD/GC) using flame ionization detection (FID).

1.2 The quantitation of residue covers a component boiling point range from 69°C to 522°C, equivalent to the boiling points of C₆ through C₄₀ n-paraffins.

1.2.1 The boiling range covers possible LPG contaminants such as gasoline, diesel fuel, phthalates and compressor oil. Qualitative information on the nature of the residue can be obtained from this test method.

1.2.2 Materials insoluble in LPG and components which do not elute from the gas chromatograph or which have no response in a flame ionization detector are not determined.

1.2.3 The reporting limit (or limit of quantitation) for total residue is 6.7 µg/g.

1.2.4 The dynamic range of residue quantitation is 6.7 to 3300 µg/g.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1265 Practice for Sampling Liquefied Petroleum \(LP\) Gases, Manual Method](#)

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.H0 on Liquefied Petroleum Gas.

Current edition approved Nov. 1, 2012. Published March 2013. DOI: 10.1520/D7828-12.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[D1835 Specification for Liquefied Petroleum \(LP\) Gases](#)
[D2158 Test Method for Residues in Liquefied Petroleum \(LP\) Gases](#)

[D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder](#)

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

[E355 Practice for Gas Chromatography Terms and Relationships](#)

2.2 *Environmental Protection Agency:*³

[EPA Method TO-17 Determination of Volatile Organic Compounds in Ambient Air using Active Sampling onto Sorbent Tubes](#)

3. Terminology

3.1 *Definitions:*

3.1.1 For the definitions of common chromatographic terms, refer to Practice [E355](#).

3.1.2 *quality control sample, QC sample, n*—for use in quality assurance program to determine and monitor the precision and stability of a measurement system; a stable and homogenous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system. The material is properly stored to ensure sample integrity, and is available in sufficient quantity for repeated long-term testing. **D6299**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *residue, n*—soluble contaminants in LPG with boiling points from 69°C to 522°C, covering the boiling point range of C₆ to C₄₀ n-paraffins.

3.2.2 *sorbent tube, n*—a tube that contains commercially available adsorbents whose composition is specific to retain the components of interest (residues).

3.2.3 *thermal desorber, n*—a sample introduction technique used in gas chromatography in which residue adsorbed on an adsorbent material is desorbed into a gas chromatograph.

³ Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

3.2.3.1 *Discussion*—Figs. 1-3 illustrate the flow path and function of the thermal desorber used in this test method.

3.2.4 *thermal desorption, n*—the process of using heat, temperature, gas flow and time to remove adsorbed components captured in a sorbent tube for introduction into a gas chromatograph for analysis.

3.2.5 *time group, n*—in gas chromatography, a set time window used in determining the response (area).

3.2.5.1 *Discussion*—In this test method, the time for response begins after the elution of pentane and stops after the elution of C₄₀ or of the heavier oil being used for this range, with the sum of the integrated peaks within this time providing a total area of the residue.

3.3 *Acronyms:*

3.3.1 *ATD*—automated thermal desorption

3.3.2 *IS*—internal standard

3.3.3 *LPG*—liquefied petroleum gas

4. Summary of Test Method

4.1 Liquefied Petroleum Gas (LPG) is sampled by Practice D1265 or Practice D3700.

4.2 A single phase sample of LPG is then captured on a fixed volume sample loop (Sampling Cell). The sample is maintained within the sample loop at a pressure above its bubble point as it is released directly onto the hydrocarbon-selective absorbent tube material, thereby trapping the C₆ plus hydrocarbons (residue). The C₅ minus components pass through the sorbent(s). The loop weight containing the sample provides the sample mass.

4.3 The sorbent tube is placed onto the thermal desorber, which desorbs the residue from the tube directly onto the analytical column of the gas chromatograph for separation and quantitation.

4.4 The data handling system acquires the raw data from the flame ionization detector. The processing method, which contains the response factor (RF) and integration parameters from

standards previously analyzed, is applied to the sample, and the mass of residue in the sample is calculated.

5. Significance and Use

5.1 Residue in LPG is a contaminant that can lead to operational problems in some end use applications. Engines, micro-turbines, fuel cells and other equipment may be sensitive to residue levels as low as 10 mg/kg.

5.2 Contamination of LPG can occur during production, transport, delivery, storage and use. A qualitative indication of the contaminants can help track down the source of the contamination from manufacture, through the distribution system, and to the end user.

5.3 This test method is designed to provide a lower detection limit, wider dynamic range, and better accuracy than gravimetric methods like Test Method D2158.

5.4 This test method can be performed with little or no discharge of LPG vapors, compared to Test Method D2158 which requires evaporation of 100 mL of sample per test.

5.5 Sampling for residue in LPG using sorbent tubes can be performed in the field, and the sorbent tubes sent to a laboratory for analysis. This saves significant costs in shipping (weight of tube is approximately 10 grams), and is much safer and easier than transporting LPG cylinders.

5.6 This test method determines total residues from C₆ to C₄₀, compared to a thermal gravimetric residue method such as Test Method D2158 which heat the residue to 38°C, resulting in a lower recovery due to loss of lighter residue components.

5.7 If there is a need to decrease the detection limit of residue or individual compounds of interest below 10 µg/g, the procedures in this test method can be modified to achieve 50 times enhanced detection limit, or 0.2 µg/g.

6. Apparatus

6.1 *Sampling Apparatus*—Refer to Appendix X1 for the typical apparatus and configuration for sampling LPG onto sorbent tubes.

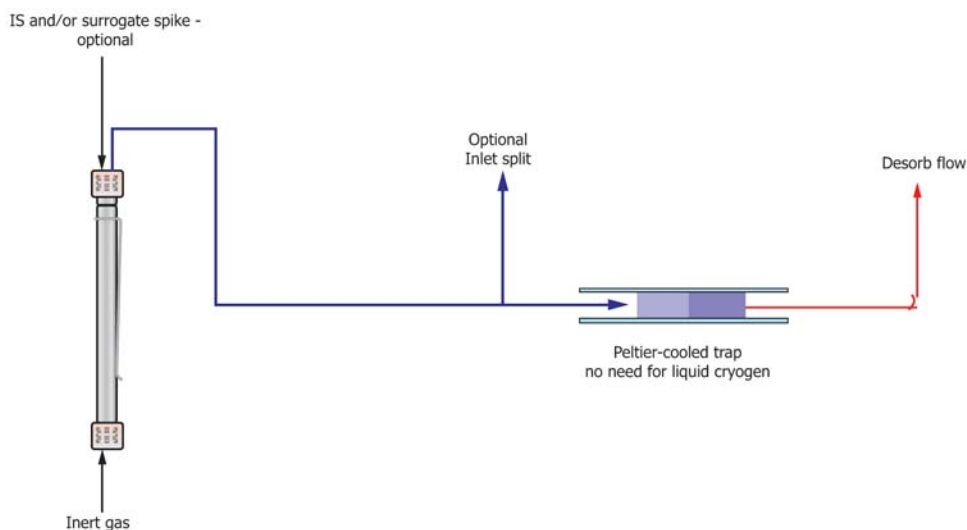


FIG. 1 Sample Tube Desorption (Primary Desorption)

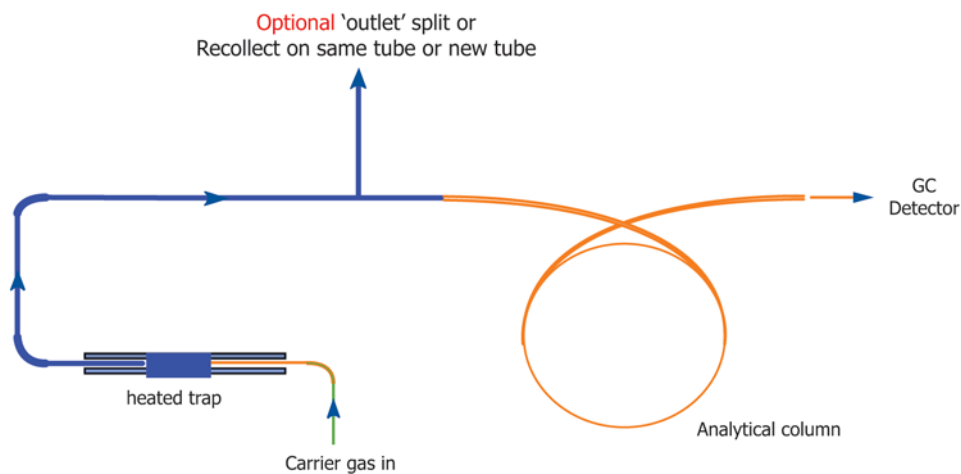


FIG. 2 Cold Trap Desorption (Secondary Desorption)

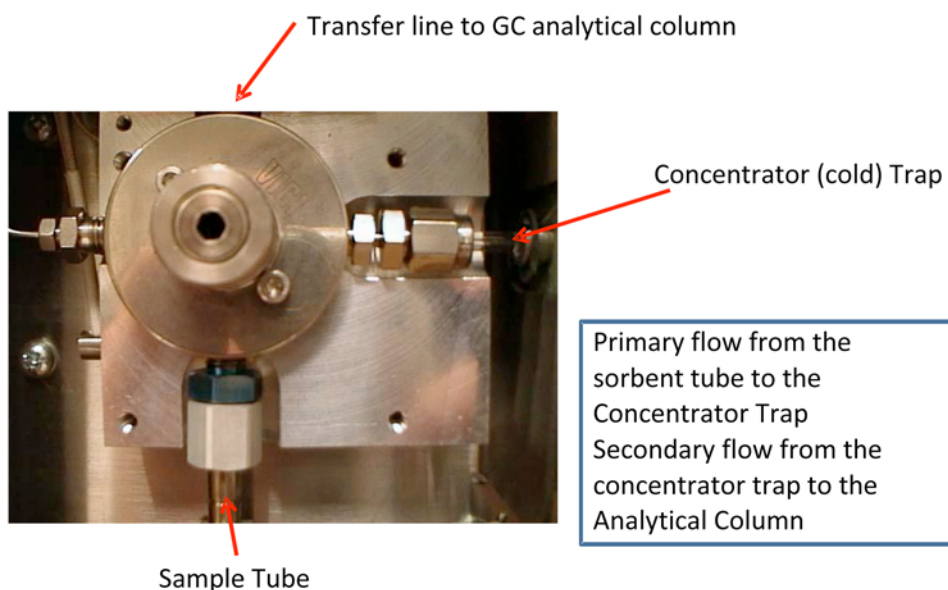


FIG. 3 Valving and Connections in a Thermal Desorber

6.2 Thermal Desorber:

6.2.1 A thermal desorber is necessary to remove the residue from the sorbent tube and introduce it to a gas chromatograph. It shall be designed to hold the sorbent tubes described in 7.4, and be capable of the parameters of temperature and flow as detailed in Table 1.

TABLE 1 Thermal Desorber Parameters

Sorbent Tube	Desorb for 18 min at 375°C at 30 mL/min
Concentrator Trap	Trap Low 5°C; Trap high 380°C; Trap Hold 14 min
Pneumatics	Inlet split 50 mL/min; Outlet split 30 mL/min; analytical column flow 0.8 mL/min
Ambient Purge ^A	Purge for 3 min at ambient temp at 50 mL/min
Transfer Line ^B	290°C
Valve Temp ^B	260°C
GC Cycle Time	34 min

^A A purge is used to rid the tube of residual oxygen and C₅ minus prior to primary desorption.

^B Manufacturer specific parameter. Refer to manufacturer's guidelines for these parameters.

6.2.2 Sorbent Tube Autosampler (Optional)—An autosampler may be used, but is not required. The instrument used to design this test method had a 50-tube automated sampler.

6.2.3 Sample Re-collection (Optional)—Most of the sample being analyzed may be re-collected via the split vent onto the same sorbent tube or onto a new sorbent tube. Sample re-collection allows re-analysis of the same sample if needed, and preservation of the sample if required for further analytical testing or legal retention, etc.

6.2.4 Transfer line configuration to install the thermal desorber to the GC or GC column is vendor specific. Consult with the manufacturer.

6.2.5 Appendix X2 contains the apparatus required for spiking standards onto standard sorbent tubes.

6.3 Gas Chromatograph (GC), configured with a split/splitless injector and a flame ionization detector that is capable of establishing the method parameters as detailed in Table 2 is required.