



Designation: D1265 – 23

## Standard Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method<sup>1</sup>

This standard is issued under the fixed designation D1265; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope\*

1.1 This practice covers equipment and procedures for obtaining a representative sample of specification Liquefied Petroleum Gas (LPG), such as specified in Specification **D1835**, GPA 2140, and comparable international standards. This standard is applicable to flow-through cylinders with two valves and is not applicable to single valve cylinders or larger LPG sample containers such as those utilized for barbecue grills and/or forklift cylinders.

1.2 This practice is suitable for obtaining representative samples for all routine tests for LP gases required by Specification **D1835**. In the event of a dispute involving sample integrity when sampling for testing against Specification **D1835** requirements, Practice **D3700** shall be used as the referee sampling procedure.

1.3 This practice may also be used for other Natural Gas Liquid (NGL) products that are normally highly volatile, single phase materials (NGL mix, natural gasoline, field butane, etc.), defined in other industry specifications or contractual agreements, where use of open sample containers would risk the loss of volatile components. It is not intended for non-specification products that contain significant quantities of undissolved gases ( $N_2$ ,  $CO_2$ ), free water or other separated phases, such as raw or unprocessed gas/liquids mixtures and related materials. The same equipment can be used for these purposes, but additional precautions are generally needed to obtain representative samples of multiphase products (see Appendix X1 on Sampling Guidelines in Practice **D3700**).

NOTE 1—Practice **D3700** describes a recommended practice for obtaining a representative sample of a light hydrocarbon fluid and the subsequent preparation of that sample for laboratory analysis when dissolved gases are present. Use of Practice D1265 will result in a small but predictable low bias for dissolved gases due to the liquid venting procedure to establish the 20 % minimum ullage.

1.4 This practice includes recommendations for the location of a sample point in a line or vessel. It is the responsibility of the user to ensure that the sampling point is located so as to obtain a representative sample.

<sup>1</sup> This practice 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.

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1.5 The values stated in SI units are to be regarded as standard.

1.5.1 *Exception*—Non-SI units are shown in parentheses for information only.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- D1835** Specification for Liquefied Petroleum (LP) Gases
- D1838** Test Method for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases
- D3700** Practice for Obtaining LPG Samples Using a Floating Piston Cylinder
- D4175** Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D6849** Practice for Storage and Use of Liquefied Petroleum Gases (LPG) in Sample Cylinders for LPG Test Methods

#### 2.2 Other Regulations:

- Canadian Transportation of Dangerous Goods Regulations<sup>3</sup>
- GPA 2140 Gas Processors Association Liquefied Petroleum Gas Specifications & Test Methods<sup>4</sup>
- IATA Transportation of Dangerous Goods by Air<sup>5</sup>

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from the Canadian General Standards Board, Sales Centre, Gatineau, Canada K1A 1G6, <http://www.ongc-cgsb.gc.ca/>.

<sup>4</sup> Available from Gas Processors Association (GPA), 6526 E. 60th St., Tulsa, OK 74145, <http://www.gpaglobal.org>.

<sup>5</sup> Available from IATA Customer Care, 800 Place Victoria, PO Box 113, Montréal, Quebec H4Z 1M1. [www.iata.org](http://www.iata.org).

\*A Summary of Changes section appears at the end of this standard

U.S. 49 CFR Transportation<sup>6</sup>

2.3 Other Publications:

API RP 2003 Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents

### 3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this practice, refer to Terminology **D4175**.

3.1.2 *high pressure sample cylinder, n*—a container used for storage and transportation of a sample obtained at pressures above atmospheric pressure.

3.1.3 *liquefied petroleum gas (LP Gas, LPG), n*—a narrow boiling range mixture of hydrocarbons consisting of propane, propylene, butanes and butylenes, individually or in specified combinations, with limited amounts of other hydrocarbons (such as ethane) and may contain naturally occurring, petroleum-derived, non-hydrocarbons.

3.1.4 *maximum fill volume (reduced fill volume), n*—the volume of a container that may be safely occupied by the liquid sample, usually expressed as a percentage of the total capacity.

3.1.4.1 *Discussion*—Some regulatory agencies use the expressions “maximum fill density” and “reduced fill density.”

3.1.5 *ou tage tube (internal), n*—a “cut to length” tube placed inside the cylinder used as a way to remove excess liquid sample from the cylinder via manual evacuation after the sample cylinder assembly is removed from the sample point.

3.1.6 *ou taging, n*—practice of removing a portion of liquid contents from a conventional sampling cylinder after filling to provide expansion room.

3.1.7 *u llage (ou tage), n*—in LPG sampling, the volume in a container after filling that remains as vapor phase above the liquid contents to allow for thermal expansion of the liquid.

3.1.8 *u pright, adj*—the vertical orientation of the cylinder where the ou tage tube end is at the top.

### 4. Summary of Practice

4.1 A liquid sample of LPG is transferred from the source into a high pressure sample container by purging the container and filling it with liquid, then providing a minimum 20 % ullage by venting liquid, so that 80 % or less of the liquid volume remains.

NOTE 2—When sampling under very cold conditions, where there is a significant difference between original sample temperature and sample temperature in a laboratory or shipping situation, an ullage volume greater than 20 % may be required to safely account for the significant expansion of LPG with a large rise in temperature.

### 5. Significance and Use

5.1 Samples of liquefied petroleum gases are examined by various test methods to determine physical and chemical characteristics and conformance with specifications.

5.2 Equipment described by this practice may be suitable for transportation of LPG samples, subject to applicable transportation regulations.

### 6. General Information

6.1 Considerable effort is required to obtain a representative sample, especially if the material being sampled is a mixture of liquefied petroleum gases. Consider the following factors:

6.1.1 Obtain samples of the liquid phase only.

6.1.2 When it is definitely known that the material being sampled is composed predominantly of only one liquefied petroleum gas, a liquid sample may be taken from any part of the vessel.

6.1.3 When the material being sampled has been mixed or circulated until it is homogeneous, a liquid sample may be taken from any part of the vessel.

6.1.4 Because of wide variations in the construction details of containers for liquefied petroleum gases, it is difficult to specify a uniform method for obtaining representative samples of heterogeneous mixtures. If it is not practicable to homogenize a mixture to ensure uniformity, obtain liquid samples by a procedure which has been agreed upon by the contracting parties.

6.1.5 Directions for sampling cannot be made explicit enough to cover all cases. They must be supplemented by judgment, skill, and sampling experience. Extreme care and good judgment are necessary to ensure samples which represent the general character and average condition of the material. Because of the hazards involved, liquefied petroleum gases should be sampled by, or under the supervision of, persons familiar with the necessary safety precautions.

6.1.6 Control hydrocarbon vapors vented during sampling to ensure compliance with applicable safety and environmental regulations. Do not vent near ignition sources.

6.1.7 This standard is applicable to flow-through double-ended cylinders with two valves. It is not applicable to single-ended (single valve) cylinders or larger LPG sample containers such as those utilized for barbecue grills and/or forklift cylinders. Additional pertinent information for these applications can be found in Practice **D6849**.

6.1.8 Elimination of potential static charge accumulation and discharge is critical for safety. Provisions for grounding/bonding shall be in place for the sample station and sample cylinders. Having continuous electrical contact to dissipate charge is required, including the transfer lines. Non-conductive materials cannot be utilized unless special grounding provisions are applied. More information on static charge accumulation and grounding/bonding can be obtained from API RP 2003.

### 7. Interferences

7.1 Certain properties of interest can be affected by the material making up the sampling system and sample cylinder assembly. Two examples of analytes commonly associated with adsorption (and desorption) at lower concentrations (for example, <100 ppm by mass) are active sulfur compounds and water. Without the use of properly treated equipment, there is

<sup>6</sup> Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <https://www.ecfr.gov/>.

potential for lower response from analytes than actually present in the sample due to adsorption (or, in the case of desorption, higher response).

7.2 Adsorption and desorption can result from physical attraction or chemical attraction.<sup>7</sup> Due to adsorption, tubing and cylinder components exposed to a sample may need surface treatment (that is, with an inert surface material) in order to collect a representative sample to be analyzed for the property of interest.

7.3 Tubing that is rough on the inside results in a larger surface area than expected. In order to reduce the surface area, the tubing needs to be smooth. Additionally, a surface treatment on the internal wall is needed to reduce the potential for polar molecules to adsorb. For stainless steel tubing and cylinders (which have a high potential for adsorption of polar molecules), this may be achieved by electropolishing and fused silica coating.

7.3.1 This inerting treatment aids in reducing the potential for adsorption of the analyte of interest.

7.3.2 Samples to be tested for the presence of corrosive compounds or sulfur compounds should be taken in containers made inert to such compounds and equipped with valves compatible with chemical, pressure, and temperature requirements; otherwise, determinations of mercaptans and hydrogen sulfide, for example, can be misleading. Internal surfaces of sample containers, valves, outage tubes, and associated lines and fittings may be surface coated to reduce bare metal surfaces reacting with trace reactive components.

NOTE 3—Adsorption is not passivation. With adsorption, molecules are both adsorbing and desorbing. Since each individual action can occur at any time, there is potential for an analysis to see less or more of an analyte than actually present in the sample. Passivation is a permanent or long-term surface treatment that minimizes adsorption of polar compounds.

## 8. Apparatus

8.1 *High Pressure Sample Cylinder*—Use corrosion resistant metal sample containers certified by the authority having jurisdiction for pressure vessels with adequate pressure rating for the product being sampled. Suitable materials include stainless steel, Monel, and possibly other materials. Protective internal coatings or surface treatments to render the internal surface inert are acceptable. The size of the container depends upon the amount of sample required for the laboratory tests to be made. If the container is to be transported, it shall also conform to specifications published in transportation legislation such as U.S. 49 CFR or Canadian Transportation of Dangerous Goods Regulations, and their supplements, reissues, or similar regulations in other jurisdictions.

NOTE 4—It has been common practice to refer to LPG sample containers as “sample bombs.” Use of this term is discouraged because of obvious misunderstanding by many people. Alternate names such as “pressurized sample container” or “high pressure sample cylinder” are recommended.

NOTE 5—DOT 3E cylinders are exempt from requalification in some jurisdictions. Other cylinders may need to be requalified according to local

regulations. See 49 CFR 180.209 for information on requalification requirements for cylinders.

NOTE 6—This standard is not applicable to single valve cylinders or larger LPG sample containers such as those utilized for barbecue grills and/or forklift cylinders.

8.1.1 The sample container should be fitted with an internal outage (ullage) tube to permit release of a minimum 20 % of the container capacity as a liquid. The end of the container fitted with the outage (ullage) tube shall be clearly marked. Typical sample containers are shown in **Figs. 1 and 2**.

8.1.2 The use of sample containers without internal outage (ullage) tubes may be prohibited at some locations. Where sample containers without internal outage (ullage) tubes are acceptable, alternative purging and venting procedures to obtain a minimum 20 % ullage in the container, as described in **11.2.1**, are required.

8.1.3 Leak checks are to be performed prior to first use, after any maintenance, and at the very least annually by pressurizing the cylinder to a minimum of 2758 kPa (400 psig) with inert gas, followed by immersing the cylinder fittings in a bucket of water.

8.1.4 Regulatory and/or local site compliance may require sample cylinder assemblies to incorporate a pressure relief device. The two common types of pressure relief devices are the spring relief valve and the rupture disc.

8.1.4.1 The spring relief valve (also known as a “popet valve”) is a reclosing device. It is designed to open at a preset pressure, where a minimal amount of sample material is released to the immediate vicinity until the pressure drops to a level when the valve re-seats (recloses).

8.1.4.2 The rupture disc is a non-reclosing device. This is often called a burst disc. It contains a bulged disc that is designed to burst or fail when a specified pressure range is reached. The disk is destroyed in the process of relieving pressure, and the entire contents of the sample cylinder will be released to the immediate vicinity.

8.1.4.3 The user is cautioned to understand the hazards associated with the type of relief device being used. Both release sample material when a design pressure is reached. However, a rupture disc releases all contents of the cylinder, thereby creating potentially more risk of exposure or larger incident if an ignition source is encountered. While a spring relief device does not release the entire content, if activated to release some pressure, the sample has been compromised and should be discarded just as if the cylinder had leaked (refer to **15.1**).

8.2 *Sample Transfer Line* made of stainless steel or other compatible metal hose, impervious to the product being sampled, is required. The line can include a flexible hose made of material that is compatible with the chemical, pressure, and temperature encountered during use. The most satisfactory line is one equipped with two valves on the sample-container end, **Fig. 1**, a sampling valve, *A*, and a vent valve, *B*.

8.3 *Sample System*—Consists of sampling line, cylinder, and vent valve. Electrical continuity should be established and maintained across the sample system to decrease risk of static discharge while sampling.

<sup>7</sup> Waters, T., *Industrial Sampling Systems, Reliable Design & Maintenance for Process Analyzers*, Swagelok Company, 2013.

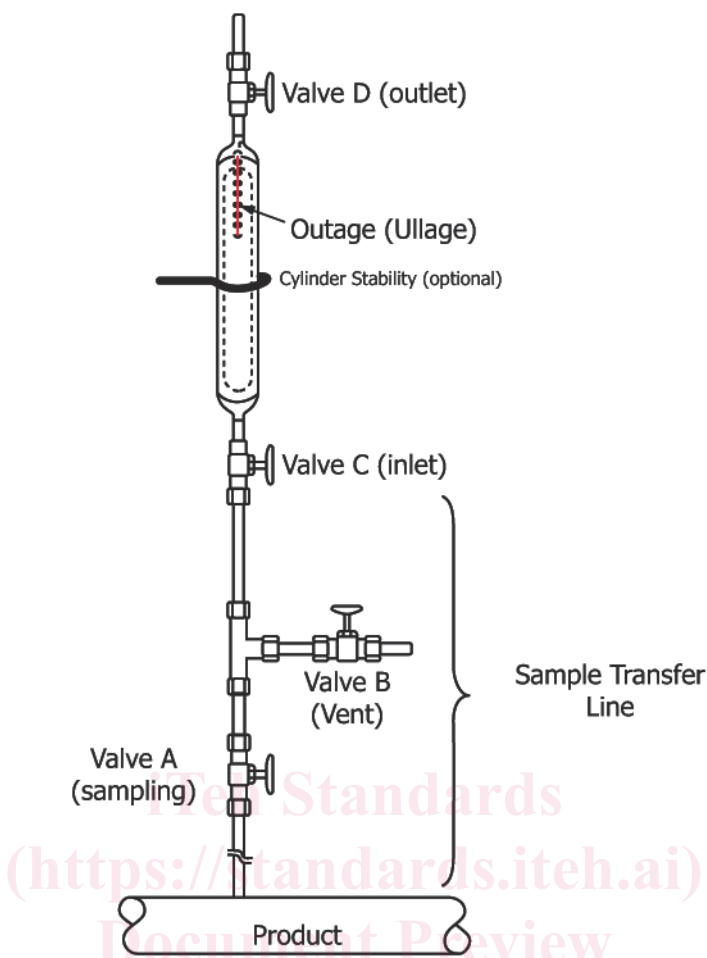


FIG. 1 Typical Sample Container and Sampling Connections (Upright)

**PROCEDURE**

**9. Purging Sample Transfer Line**

9.1 Connect the ends of the sample transfer line securely to the product source and to Valve C (inlet) (Fig. 1) of the container. Close Valve A (sampling), Valve B (vent), and Valve C (inlet). Open the valve at the product source and purge the transfer line by opening Valve A (sampling) and Valve B (vent); capture the vented LPG for safe disposition when possible.

**10. Purging the Sample Container**

10.1 If the history of the sample container contents is not known, or if traces of the previous product could affect the analysis to be carried out, or both, use one of the following two purge procedures:

10.1.1 Ensure that Valve C (Fig. 2) and Valve D on the high pressure sample cylinder are closed. Connect a sample transfer line (with closed Valves A and B) to the cylinder at Valve D and to the sample source. Maintain the cylinder in an upright position such that Valve C is at the top.

10.1.2 Fill sample container by opening Valve A followed by Valve C and Valve D until liquid issues from Valve C. At that time, close Valve C, followed by Valve D and Valve A on the sample transfer line. Vent the sample transfer line by briefly opening Valve B.

10.1.3 Loosen the connection joining the sample container to the sample line and turn container through 180° such that Valve D is at the top. Open Valves C and D and drain out liquid as dictated by environmental requirements.

10.1.4 Return the sample container to position Valve C at the top. Tighten connection to sample transfer line and repeat the purging operation at least three times.

10.2 In a flowing system or a suitable sample loop, the sample cylinder may be flushed inline by connecting the cylinder to allow material to flow from the higher pressure point back to the lower pressure point. Keep the cylinder vertical during flushing. Flush the cylinder with at least 10 times the cylinder volume in a time of less than 5 min to ensure a sufficient flow velocity to obtain turbulent mixing and flushing of the ullage volume area. The sample line shall be equipped with a suitable flow indicator to ensure an adequate flow rate throughout the flushing period.

10.2.1 This procedure is particularly applicable in areas where excessive venting of LPG to the atmosphere is not allowed.

10.3 If the history of the sample container contents is known and would not affect the analysis, use the following purge procedure:

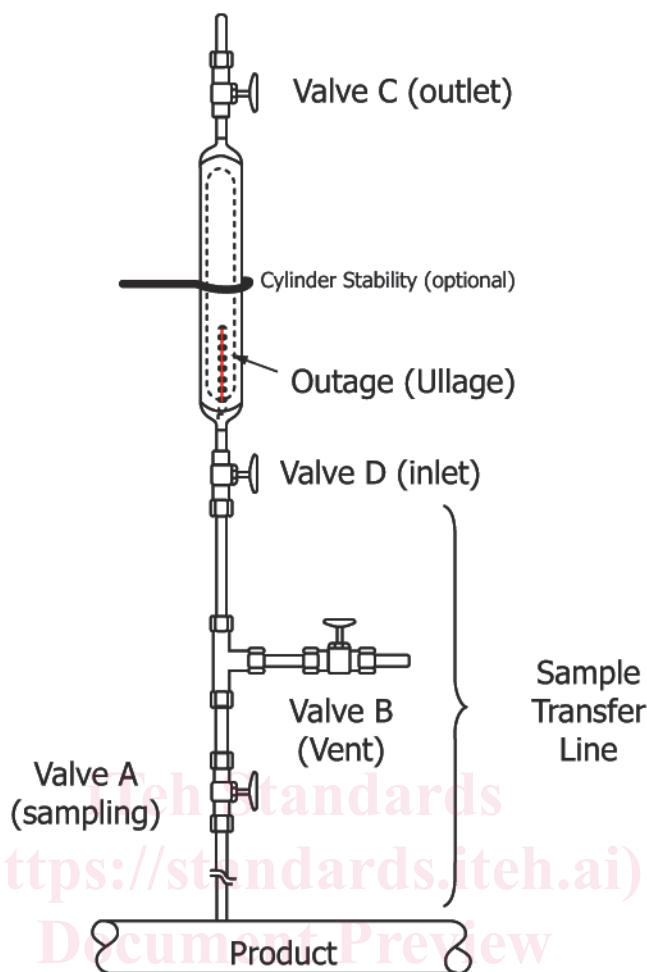


FIG. 2 Typical Sample Container and Alternate Purging Connections (Inverted)

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10.3.1 With the container in an upright position (Fig. 1) and its Valve D (outlet) at the top, close Valve B (vent) and Valve C (inlet) and open Valve A (sampling). Open Valve C (inlet) and partly fill the container with sample by slowly opening the Valve D (outlet). Close the Valve A (sampling) and allow part of the sample to escape in the vapor phase through Valve D (outlet). Close Valve D (outlet) and release the remainder of the sample in the liquid phase by opening Valve B (vent). Repeat the purging operation at least three times.

## 11. Transfer of Sample

11.1 Position the sample container securely in an upright position with Valve D (outlet) at the top (Fig. 1) and both Valves C and D closed.

11.1.1 Close Valve B (vent), open Valve A (sampling), open Valve C (inlet), and fill container with the sample. Close Valve C (inlet) and the valve at the product source. Open Valve B (vent). After the pressure is fully reduced, disconnect sample container from the transfer line. Discard the sample if a leak develops or if either valve is opened during subsequent handling of the sample container before performing the outage (ullage) operations outlined in Section 12.

11.2 The transfer of a sample from one container to another is sometimes desired for a test method (for example, Test

Method D1838). For these transfers, the user needs to consider the physical properties of the sample and ensure the transfer setup maintains the sample's integrity.

11.2.1 For these transfers, connect cylinders together by a suitable system/apparatus, with proper grounding and bonding of the system to prevent static discharge (Fig. 3). The empty secondary cylinder (for example, corrosion cylinder, LPG vapor pressure apparatus, etc.) shall be clean and free of any residues (including moisture) or any solvents that could contaminate the sample.

11.2.2 Cylinder A may need to be pressurized to maintain sample integrity and ensure transfer of liquid sample amount required for testing to the secondary cylinder. Cylinders and associated equipment for transfer should be maintained at the same temperature to aid in preventing flashing of liquid to vapor.

11.2.3 After confirming proper ullage by procedure or by weight, ensure homogeneity with mixing by repeated inverting and uprighting cylinder position several times before transferring sample. Ensure all valves are closed to begin. Workers should also ground and bond themselves when performing the transfer. 6.1.8 contains additional information on grounding/bonding.