Designation: D1265 – 05

Standard Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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.

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 single phase (NGL mix, field butane, etc.), defined in other industry specifications or contractual agreements. 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.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1835 Specification for Liquefied Petroleum (LP) Gases

D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder

2.2 *Other Regulations:*

Canadian Transportation of Dangerous Goods Regulations [3]

GPA 2140 Gas Processors Association Liquefied Petroleum Gas Specifications & Test Methods[4]

IATA Transportation of Dangerous Goods by Air[5]

U.S. CFR 49 Transportation[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *high pressure sample cylinder*—a receptacle used for storage and transportation of a sample obtained at pressures above atmospheric pressure. Also referred to as a "pressurized sample container" or "sample bomb." The term "sample bomb" is not preferred.

3.1.2 *maximum fill density (reduced fill density)*—the volume of a container occupied by the sample, usually expressed as a percentage of the total capacity. Transportation legislation such as U.S. CFR 49, Canadian Transportation of Dangerous Goods Regulations, and IATA regulations limit the percent fill of containers used for shipping LPG and may quote this requirement as a reduced fill density or maximum fill density (normally 80 % maximum liquid fill at 15°C). Lower percent fill (lower fill density) may be required if sampling at lower temperatures.

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum and Products and Lubricants and is the direct responsibility of Subcommittee D02.H0 on Liquefied Petroleum Gas.

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[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.

[3] Available from Government of Canada Publications – PWGSC, Ottawa, ON K1A 0S9.

[4] Available from Gas Processors Association, 6526 E. 60th St., Tulsa, OK 74145.

[5] Available from IATA Customer Care, 800 Place Victoria, PO Box 113, Montréal, Quebec H4Z 1M1. www.iata.org.

[6] Available from U.S. Government Printing Office, 732 N. Capitol Street, NW, Washington, DC 20401.

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

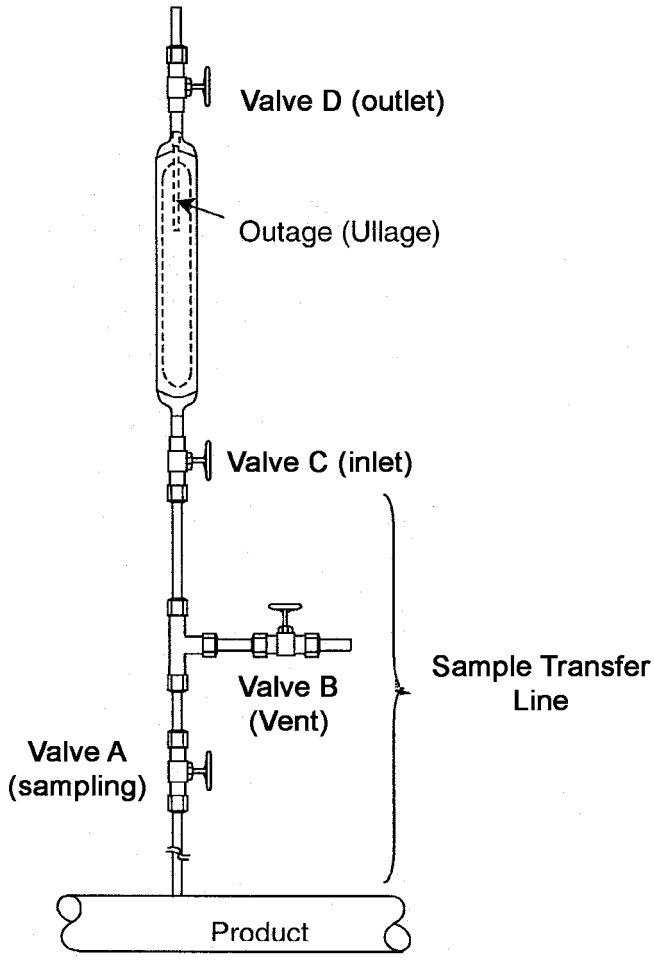

FIG. 1 Typical Sample Container and Sampling Connections

4. Summary of Practice

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

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. The following factors must be considered:

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 variation 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.

NOTE 2—Samples to be tested for presence of corrosive compounds or sulfur compounds should be taken in inert containers equipped with stainless steel valves; otherwise, determinations of mercaptans and hydrogen sulfide, for example, can be misleading. Internal surfaces of sample containers and associated lines and fittings may be surface coated to reduce bare metal surfaces reacting with trace reactive components.

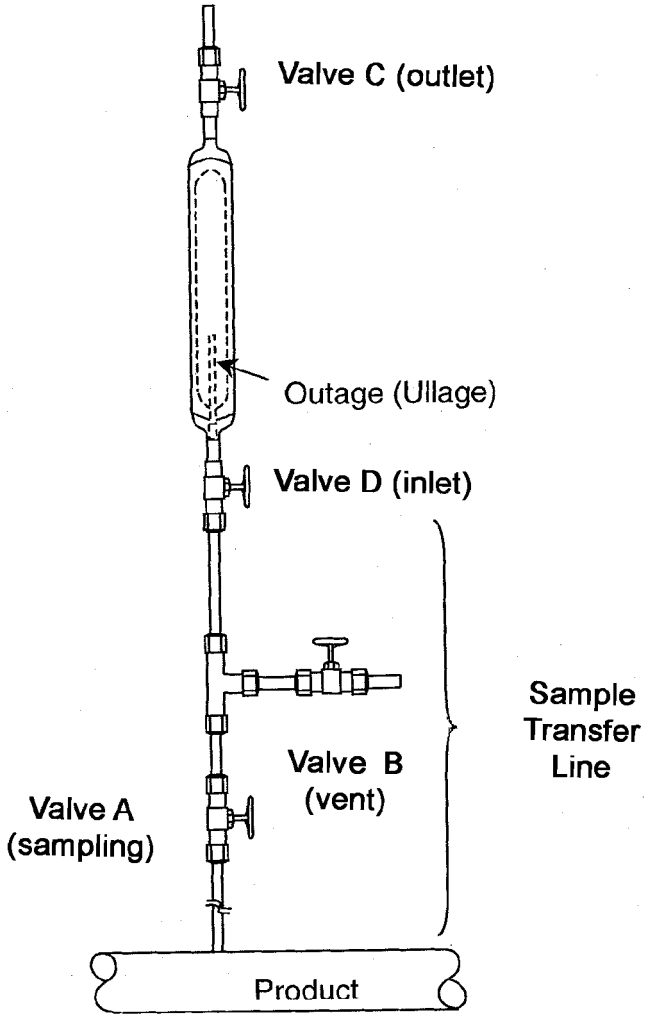

FIG. 2 Typical Sample Container and Alternate Purging Connections

6.1.6 Hydrocarbon vapors vented during sampling must be controlled to ensure compliance with applicable safety and environmental regulations.

7. Apparatus

7.1 *Sample Container*—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. 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 must also conform to specifications published in transportation legislation such as U.S. CFR 49 or Canadian Transportation of Dangerous Goods Regulations, and their supplements, reissues, or similar regulations in other jurisdictions.

7.1.1 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.

7.1.2 Sample containers without an 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.

NOTE 3—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 containers" or "high pressure sample cylinders" are preferred.

7.2 *Sample Transfer Line* made of stainless steel tubing or other flexible metal hose, impervious to the product being sampled, is required. 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*.

PROCEDURE

8. Purging Sample Transfer Line

8.1 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).

9. Purging the Sample Container

9.1 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:

9.1.1 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.

9.1.2 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*.

9.1.3 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.

9.1.4 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.

9.2 In a flowing system or a suitable sample loop, the sample cylinder may be flushed online by connecting the dip-tube end of the cylinder to the higher pressure point, and the other end back to the lower pressure point. Keep the cylinder upright with the dip tube end down to maintain liquid filled 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 by using the dip tube as a venturi mixer. The sample line shall be equipped with a suitable flow indicator to ensure an adequate flow rate throughout the flushing period.

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

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

9.3.1 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.

10. Transfer of Sample

10.1 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.

10.1.1 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 11.

11. Sample Outage (Ullage)

11.1 Immediately after obtaining the sample, place the container in an upright position with the outage (ullage) tube at the top.

11.1.1 Open Valve *D* (outlet) slightly. Allow excess liquid to escape and close the valve at the first sign of vapor, as indicated by the first "sputtering" and change in flow behavior of the vented liquid. If no liquid escapes, discard the sample and refill the container.

NOTE 4—The cylinder must not be stored/transported beyond 80 % of its capacity with sample. Liquid sample must be vented out to the required "reduced fill density" (typically 80 % or less) prior to transport. Where immediate venting is not possible, for example inside hazardous locations or with toxic materials (especially H_2S), provisions shall be made to prevent temperature increase prior to venting in a safe location, transfer to a larger cylinder or immediate analysis or other disposition in accordance with the authority having jurisdiction. It is the responsibility of the user to establish safe procedures for use in permitted facilities that are regulated by site permits or equivalent, separate from transportation regulations.

NOTE 5—Improperly venting LPG vapor to establish the minimum 20 % outage will result in large changes in composition of the remaining liquid due to fractional distillation. It is important that only liquid be vented from the cylinder, and that venting be stopped at the first indication of vapor. Use of proper liquid venting technique will result in only very small changes in composition to the remaining liquid which will not affect product specification results required for compliance with Specification D1835.

NOTE 6—Extreme low temperature sampling. LPG has a larger coefficient of thermal expansion than gasoline or distillate fuels. Sampling at extremely low ambient temperatures or from cryogenic sources may require additional precautions to prevent sample cylinders from becoming liquid full (liquid locked, hydraulically locked) from warming the sample to high ambient temperature.

NOTE 7—Consult authority having jurisdiction for LPG cylinder ullage requirements for transport of LPG sample cylinders.

11.2 For sampling containers without an internal ullage tube, the procedure in 11.2.1 can be used.

11.2.1 One acceptable alternative procedure to use is weighing the sample container immediately after filling, and creating the ullage before analysis. The procedure is to completely fill the sample container using slight overflow indication to ensure complete filling. Without warming the sample, immediately weigh the container plus sample on a balance, and record gross weight. Carefully drain off liquid from the bottom of the vertically oriented container in an environmentally-approved manner. Then weigh the vented container, and using the tare weight of the container, estimate the ullage of the sample in the container. Repeat the venting and weighing procedures to obtain 22 ± 2 % ullage of the sample.

12. Checking for Leaks

12.1 After eliminating the excess liquid sample so that only 80 % or less of the sample remains, immerse in a water bath and check the sample cylinder for leaks. If a leak is detected at any time during the sampling operation, discard the sample.

Repair or replace the leaky container before obtaining another sample. Alternate procedures such as use of soap/water leak detection fluid, leak detection instruments or recording of total cylinder weights may also be used to detect leaks.

13. Care of Samples and Sample Containers

13.1 Place samples in a cool location as soon as possible. Keep them there until all tests have been completed. Discard any samples in containers which develop leaks. Protect the valves on the sample container, either by packing the container in a crate in an approved manner or by using a protective cap, so that accidental unseating of the valve or tampering with it is avoided.

14. Keywords

14.1 liquefied petroleum gases; LPG; sampling

SUMMARY OF CHANGES

Subcommittee D02.H0 has identified the location of selected changes to this standard since the last issue (D1835–04a) that may impact the use of this standard.

(1) Updated 7.1.