Designation: D2713 – 20

Standard Test Method for Dryness of Propane (Valve Freeze Method)[1]

This standard is issued under the fixed designation D2713; 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 measurement of the dryness of propane products that do not contain antifreeze agents such as, but not limited to, commercial propane and special duty propane (see Specification D1835).

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *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.*

1.4 *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:*[2]

D1265 Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method

D1835 Specification for Liquefied Petroleum (LP) Gases

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *freeze-off time, n*—the time, in seconds, for a propane water test valve to freeze under the conditions of this test method.

3.1.2 *propane water test valve, n*—a specific valve designed and manufactured for performing in this test method.

3.1.3 *valve freeze, adj*—relating to the procedure for determining the dryness of propane for this test method.

4. Summary of Test Method

4.1 A liquid-phase sample of the product to be tested is flowed through the propane water test valve to cool the valve body. After cooling, the test valve is partially closed to a small preset flow rate and the time required for the valve to freeze, due to water dissolved in the sample and thus interrupt the normal flow, is recorded. Higher dissolved water concentrations will result in shorter freeze-off times. The average observed time for several successive observations is recorded as the observed valve freeze time.

5. Significance and Use

5.1 This test is a functional test in which the water concentration in the product is related to product behavior characteristics in a pressure-reducing system of special design to arrive at a measure of product acceptability in common use applications. Experience has demonstrated that excessive water content (dissolved water) will cause freeze-up difficulties in pressure reducing systems.

6. Interferences

6.1 Antifreeze agents will interfere with this test method and can indicate that the product is drier than it really is. However, the relative freeze-off times of such materials tested by this procedure can be an indication of the tendency of these products to cause freezing in pressure-reducing regulators.

7. Apparatus

7.1 *Propane Water Test Valve*[3]—A specially constructed and calibrated valve manufactured solely for this test (Note 1). The valve has two open positions, a wide open position for flushing, and a small preset flow position for testing.

NOTE 1—The propane water test valve is a precision instrument and it should be so treated. It should not be dropped, strained in any way, or disassembled, except to clean the filter in accordance with the manufacturer's instructions. Valves suspected of being defective should be

[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.

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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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1423. Contact ASTM Customer Service at service@astm.org.

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

returned to the manufacturer for inspection, reconditioning or recalibration.

7.2 *Stop Watch or Timer,* measuring in seconds.

7.3 *LP Gas Sample Cylinder,* of suitable volume. In order to perform multiple tests, a container having a minimum capacity of 11 L (3 U.S. gal) is typically required.

7.4 *Cloth,* dry, clean.

7.5 *Connection Tubing,* clean pipe or metallic tubing for connecting the propane water test valve to the sample point or sample cylinder. Do not use a rubber hose or plastic-lined hose.

8. Hazards

8.1 The primary hazards of handing propane are fire, explosion, and freezing "burns" due to the autorefrigeration of LP gas as it expands.

9. Sampling

9.1 Moisture test measurements on propane samples should be performed by connecting the test valve directly to the bulk supply if possible, rather than on samples taken from the bulk supply, due to the risk of uncontrollable sampling errors that can affect the sensitivity of moisture test measurements. Referee tests should be conducted on the bulk supply.

9.2 If the test cannot be run by connecting the apparatus directly to the bulk propane supply, a sample may be taken into a clean, dry sample cylinder (7.3). In such cases, the sample shall be collected strictly in accordance with directions given in Practice D1265.

9.2.1 When using a sample cylinder, be careful not to vent LPG vapor from the sample cylinder.

NOTE 2—Venting vapor from a sample cylinder would result in loss of water from the LPG sample. The partition coefficient of water in LPG results in a higher concentration of water in the vapor phase than in the liquid phase. Further, after some liquid LPG has been withdrawn and there is a significant volume of vapor phase relative to the volume of liquid phase in the sample cylinder, the concentration of water in the liquid phase can decrease. Either venting vapor or withdrawing a liquid test specimen from a sample cylinder with a relatively large vapor volume could give an erroneously low concentration of water being measured in the liquid LPG.

10. Procedure

10.1 Sample pressure, at the inlet to the test valve, shall not be more than 690 kPa (100 psi) above the vapor pressure of the product at the sample temperature. When sample source pressure is above this limit, use a liquid propane pressure regulator to hold the pressure, at the inlet to the test valve, within this limit.

10.2 Connect (see 7.5) the propane water test valve to the liquid line of the bulk product source or to the liquid phase connection of the sample cylinder (7.3), so that the body of the valve is horizontal and the outlet opening is aimed vertically upward. The valve shall be positioned so that the internal surfaces of the outlet opening are clearly visible to the operator.

10.2.1 Ensure that the lighting is sufficient to observe the end point of the test.

10.3 Open the main valve on the sample source and set the valve on the test apparatus in the purge position. Purge the sample line and the apparatus for 15 s.

10.4 Close the test valve for 2 s or 3 s, open it for 2 s or 3 s, close it for 2 s or 3 s, and continue this intermittent opening and closing until a uniform frost cover has accumulated on the housing around the outlet of the test valve.

10.5 Snap the valve to the test position and simultaneously start the stop watch.

10.6 Stop the watch at the instant the liquid propane ceases to flow through the valve (Note 3). Any release of gas through the valve, typically accompanied by a "hissing" sound, shall not be interpreted as liquid flow.

NOTE 3—For information, liquid propane stops flowing through the test valve when frost forms on the internal surface of the valve outlet, plugging it. The higher the concentration of water in propane, the quicker the test valve will freeze off and plug. Under good lighting conditions, an experienced operator can sometimes be forewarned of this instant of liquid propane stoppage by watching the frost line climb and roll over the lip of the valve outlet. Regardless of the observation of a 'climbing frost line,' the end of the test is defined in 10.6 by the instant the liquid propane ceases to flow through the test valve.

10.7 Disregard the observed time for the initial freeze-off run. Quickly wipe the test valve outlet threads with a clean, dry cloth.

10.8 Open the test valve to the purge position for about 15 s to ensure the removal of ice from the preset opening.

NOTE 4—Failure to purge the apparatus with the valve open to the purge position for about 15 s between tests will give erroneous results. Purging assures that ice formed in the preset opening in the preceding test will be removed.

10.9 Repeat the operations as described in the trial run (beginning at 10.4) until three successive freeze-off times agree with each other to within ±5 s.

NOTE 5—For freeze-off times of less than 1 min, this may require as many as seven or eight test runs.

10.9.1 If the freeze-off time on two consecutive test runs is 90 s or greater, the test on the product may be discontinued and the freeze-off time recorded as greater than 90 s.

10.9.2 For freeze-off times shorter than 90 s, record the average time for three consecutive determinations as the freeze-off time, to the nearest whole second.

11. Report

11.1 If the valve does not freeze off within 60 s, report the result as "pass" in accordance with this test method. If the valve does freeze off within 60 s, report the result as a "fail" in accordance with this test method.

12. Precision and Bias

12.1 In the case of pass–fail data or results from other qualitative tests, no generally accepted method for determining precision or bias is currently available.

NOTE 6—Data from a series of tests conducted in 1967 indicate the following: At moisture levels of 14 ppm and 26 ppm, all valves can be expected to give freeze times over 3 min; at a moisture level of 49 ppm it can be expected that none of the valves will give freeze times over 18 s; at a moisture level of 93 ppm it can be expected that none of the valves will give freeze times over 5 s.

NOTE 7—The moisture concentrations in Note 6 are believed to be in parts per million (ppm) by mass, mg/kg, based on the solubility of water