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**Liquefied petroleum gases — Assessment  
of the dryness of propane — Valve freeze  
method**

*Gaz de pétrole liquéfiés — Évaluation de la siccité du propane — Méthode  
de givrage de vanne*

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13758 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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# Liquefied petroleum gases — Assessment of the dryness of propane — Valve freeze method

**WARNING** — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems 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.

## 1 Scope

This International Standard describes a procedure for the assessment of whether liquefied petroleum gas (LPG) hydrocarbons consisting predominantly of propane and/or propene are sufficiently dry to avoid malfunctions in pressure-reducing systems installed in domestic, industrial and automotive LPG applications.

The test is normally used as a functional pass/fail test in which the behaviour of the product is assessed in a specially designed and calibrated regulator valve.

### NOTES

- 1 If excessive dissolved water is contained in the LPG under test, it will cause freezing and blockage of a test regulator valve, which is an indication that the product could cause malfunctions in commercial pressure-reducing systems.
- 2 If the product under test contains an antifreeze agent, the time taken for the test valve to freeze is not necessarily a function of dryness, but may provide an indication of the tendency of the product to cause freezing in pressure-reducing regulators under service conditions.

## 2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4257:1988, *Liquefied petroleum gases — Method of sampling*.

## 3 Definition

For the purposes of this International Standard, the following definition applies:

**3.1 valve freeze time; freeze-off time:** Time, in seconds, from the start of the test until the test product ceases to flow through a standard test valve under specified conditions.

## 4 Principle

A liquid-phase aliquot of the sample to be tested is allowed to flow through the wide-open test valve under its own vapour pressure, in order to cool the valve body by vaporization. After cooling, the test valve is partially closed to a small pre-set orifice and the time required for the valve to freeze, and thus interrupt the normal flow, is recorded. The average time measured for a number of successive observations is recorded as the valve freeze time.

## 5 Apparatus

**5.1 LPG freeze test valve**<sup>1)</sup>, a precision instrument specially constructed and calibrated solely for this test procedure.

The valve has two open positions, a wide-open position for purging and cooling, and a small pre-set orifice for testing.

The valve shall not be dropped, strained or disassembled, except to clean the filter in accordance with the manufacturer's instructions.

NOTE — Valves suspected of being defective should be returned to the manufacturer for inspection, reconditioning and recalibration.

**5.2 Stopwatch**, mechanical or electronic, with an accuracy of at least 0,2 s.

**5.3 Pressurized sample container** [if required (see 6.2)], of minimum capacity 11,5 l.

**5.4 Wipe cloth**, absorbent, clean and dry, of natural or synthetic material.

## 6 Sampling

**6.1** Moisture test measurements are extremely sensitive to uncontrollable sampling errors, and thus, wherever possible, the tests shall be carried out at the LPG supply source rather than on samples taken from bulk supply. Likewise during testing, do not expose the test valve to direct strong sunlight, high levels of radiant heat, or precipitation.

**6.2** If the test cannot be carried out by connecting the apparatus directly to the bulk LPG supply, a sample shall be taken into a suitable sample container (5.3) in accordance with the procedure described in ISO 4257 or an equivalent national standard.

## 7 Procedure

**7.1** Connect the test valve (5.1) to the liquid line of the bulk product source, or to the liquid-phase connection of the sample container (5.3) by means of clean, dry metallic pipework and fittings. The body of the valve shall be horizontal with the outlet opening aimed vertically upwards. Position the valve so that the internal surfaces of the outlet opening are clearly visible to the operator.

**7.2** Sample pressure at the inlet to the valve shall be not more than 700 kPa above the vapour pressure of the product at the temperature of the sample container. When the sample source pressure is above this limit, insert a liquid propane pressure regulator upstream of the test valve in order to comply with this requirement.

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1) Details of suppliers may be obtained from the Secretariat of ISO/TC 28.

Open the main valve on the sample source and set the valve on the test apparatus to the purge position. Purge the sample line and the apparatus for 15 s. Close the test valve for 2 s to 3 s, open it for 2 s to 3 s, and continue this intermittent opening and closing until a uniform frost cover has accumulated on the housing around the test valve. Turn the valve quickly to the test position and simultaneously start the stopwatch (5.2). Stop the watch at the instant the liquid ceases to flow through the valve (freeze-off time).

NOTE — The instant liquid stops flowing through the valve, frost will form on the internal surfaces of the valve outlet. The watch should be stopped at this instant which can be foreseen by observing the frost line climb the valve body and roll over the lip of the valve outlet.

**7.3** Disregard the observed freeze-off time for the initial run. Quickly wipe the test valve outlet threads with the cloth (5.4). Open the test valve to the purge position for approximately 15 s to ensure the removal of ice from the pre-set opening. Repeat the operations in 7.2 until three consecutive freeze-off times are identical within  $\pm 2$  s. If the freeze-off time in three consecutive test runs exceeds 180 s, discontinue the test.

#### NOTES

- 1 For freeze-off times of less than 60 s, up to eight test runs may be required.
- 2 Failure to purge the apparatus with the valve open to the purge position for approximately 15 s between tests will give erroneous results. Purging ensures that ice formed in the pre-set opening in the preceding test will be removed..

## 8 Expression of results

**8.1** Report to the nearest 1 s, the average result of three consecutive tests giving freeze-off times of less than 180 s.

**8.2** If the results of three consecutive test runs exceed 180 s, report the result as “over 180 s”.

**8.3** In the normal operation of this test, where it is used for specification purposes as a pass/fail criterion at a level of 60 s, report either “pass” (freeze-off time > 60 s) or “fail” (freeze-off time < 60 s).

NOTE — Data obtained in 1967 indicated that at moisture levels of 14 mg/kg and 26 mg/kg, the freeze-off time can be expected to exceed 180 s, at 49 mg/kg the freeze-off time can be expected to be below 18 s, and at 93 mg/kg the expected freeze-off time reduces to below 5 s.

## 9 Precision

In the case of pass/fail data or results from qualitative tests, no generally accepted method for assessing precision is currently available.

## 10 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the result of the test (see clause 8);
- d) any deviation, by agreement or otherwise, from the procedures specified;
- e) the date of the test.

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