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Liquefied petroleum gases — Determination of vapour pressure — LPG method

Gaz de pétrole liquéfiés — Détermination de la pression de vapeur — Méthode GPL

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

ISO 4256:1978

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Brazil	Italy	Spain
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No member body expressed disapproval of the document.

Liquefied petroleum gases – Determination of vapour pressure – LPG method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the gauge vapour pressures (see note 1) of liquefied petroleum gas products (see note 2) at temperatures of 37,8 °C up to and including a test temperature of 70 °C.

NOTES

1 Information on the gauge vapour pressures of liquefied petroleum gas products under test temperature conditions in the range from 37,8 to 70 °C is pertinent to problems of selecting properly designed storage vessels, shipping containers, and customer utilization equipment to ensure the safe handling of such products.

2 For the purpose of this test method, liquefied petroleum gas products are defined as narrow boiling range hydrocarbon mixtures consisting predominantly of propane or propylene, or both, butanes or butylenes, or both, in which the content of hydrocarbon compounds of boiling point higher than 0 °C is less than 5 % by liquid volume, and whose gauge vapour pressure at 37,8 °C is not greater than approximately 1 550 kPa.¹⁾

2 REFERENCES

ISO 3007, *Petroleum products – Determination of vapour pressure – Reid method.*

ISO 4257, *Liquefied petroleum gases – Determination of sampling characteristics.*²⁾

3 PRINCIPLE

3.1 The test apparatus, consisting of two interconnected chambers and equipped with a suitable pressure gauge, is purged with a portion of the sample which is then discarded. The apparatus is then filled completely with a test portion of the test sample to be tested.

33 1/3 or 40 % by volume of the liquid content of the apparatus is immediately withdrawn to provide adequate free space for product expansion. The apparatus is then immersed in a water bath maintained at the standard test temperature of 37,8 °C or, optionally, at some higher test temperature up to and including a test temperature of 70 °C.

3.2 The observed gauge pressure at equilibrium, after correcting for gauge error and correcting to a standard barometric pressure, is reported as the "LPG vapour pressure" at the selected test temperature.

4 APPARATUS

4.1 Vapour pressure apparatus, constructed as illustrated in the figure, consisting of two chambers, designated as the upper and lower chambers, complying with the requirements in 4.1.1 to 4.1.7.

NOTES

1 CAUTION – To maintain the correct volume ratio between the upper and lower chambers, they shall be matched in pairs and the units shall not be interchanged without recalibrating to ascertain that the volume ratio is within satisfactory limits.

2 The "air chamber" of the method described in ISO 3007 is interchangeable with the "upper chamber" of this method. Similarly, the "liquid chamber" (two-opening type) of ISO 3007 is interchangeable with the "20 % lower chamber" of this method. The apparatus assembly of ISO 3007 must pass, as a safety precaution, the hydrostatic test specified in 4.1.6 before being used for testing liquefied petroleum gas.

1) 1 kPa = 10⁻² bar

2) In preparation.

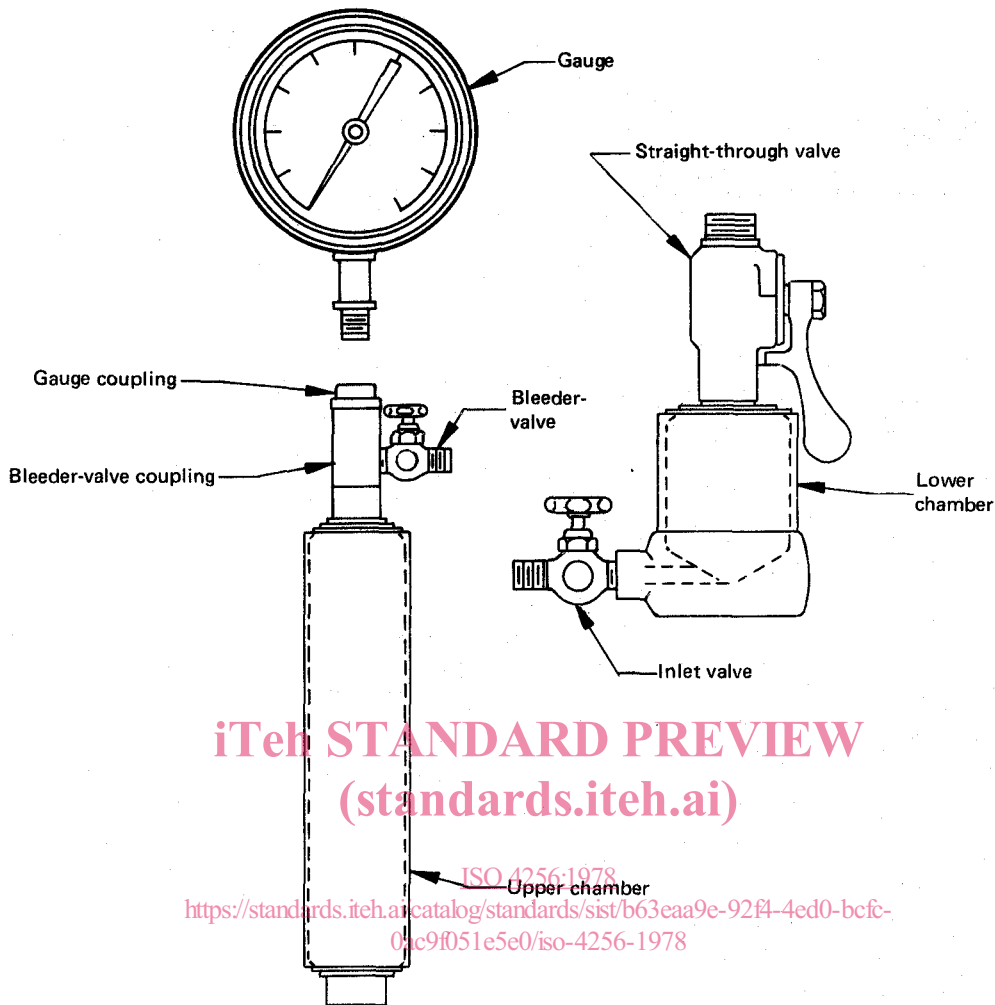


FIGURE — Typical LPG vapour pressure apparatus

4.1.1 Upper chamber

This chamber shall be a cylindrical vessel 51 ± 3 mm in diameter and 254 ± 3 mm in length, inside dimensions, with the inner surfaces of the ends slightly sloped to provide complete drainage from either end when held in a vertical position. At one end of the chamber, a suitable bleeder-valve coupling shall be provided to receive the bleeder-valve assembly and the pressure gauge. At the other end of the chamber an opening approximately 13 mm in diameter shall be provided for coupling with the lower chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

4.1.2 Bleeder-valve assembly

The bleeder-valve for purging the apparatus shall be a nominal 6 mm valve fitted into the side of the bleeder-valve coupling. The lower end shall be threaded to fit into the end fitting of the upper chamber, and the upper end shall be threaded to receive the gauge coupling.

4.1.3 Lower chamber, 33 1/3 %

This chamber shall be a cylindrical vessel of such a volume that the ratio of the volume of the upper chamber to the volume of the lower chamber is $2 \pm 0,03$ (see note 1 to 4.1.4).

4.1.4 Lower chamber, 20 %

This chamber shall be a cylindrical vessel of such a volume that the ratio of the volume of the upper chamber to the volume of the lower chamber is $4 \pm 0,05$ (see notes 1 and 2 below).

NOTES

1 In determining the volumetric capacities of the chambers, the volume of the lower chamber shall be considered as that which is below the "straight-through" valve closure. The volume above the "straight-through" valve closure, including the portion of the coupling attached to the upper chamber, shall be considered as a part of the upper chamber volume. The volume ratios of the chambers shall be determined in accordance with the procedure outlined in the annex of ISO 3007.

2 The apparatus requirements for this method, excluding the bleeder-valve assembly, are identical with those of ISO 3007 with the exception of the 33 1/3 % lower chamber. Although the test procedural details are different, the air and liquid chambers of ISO 3007 may be used in the present method provided that they are of sufficient strength to withstand the higher test pressures (4.1.6).

4.1.5 Valves and method of coupling upper and lower chambers

At one end of the lower chamber, an opening approximately 19 mm in diameter shall be provided to receive a suitable straight-through valve having a minimum internal channel of 13 mm diameter. The other end of the chamber shall be equipped with a nominal 6 mm inlet valve.

Any method of coupling the chambers may be employed provided that the volumetric requirements are met and that the assembly is free from leaks under the conditions of the test.

4.1.6 Hydrostatic test

The assembled chambers shall be certified by the manufacturer to withstand approximately 7 000 kPa gauge hydrostatic pressure without permanent deformation.

4.1.7 Checking for freedom from leaks

Before placing new apparatus in service, and as often as necessary thereafter, the assembled vapour pressure apparatus shall be checked for freedom from leaks by filling it with air, natural gas, nitrogen, or other similar gases, to 3 500 kPa gauge pressure, and then completely immersing it in a water bath. Only apparatus that will withstand this test without leaking shall be used.

4.2 Pressure gauge. Bourdon-type spring gauge of test gauge quality 114 to 140 mm in diameter provided with a nominal 6 mm male thread connection with a passageway not less than 5 mm in diameter from the Bourdon tube to the atmosphere. The range and graduations of the pressure gauge used shall be governed by the vapour pressure of the sample being tested, as shown in table 1.

TABLE 1 – Specifications for pressure gauges

Values in kilopascals

L.P.-Gas vapour pressure	Gauge to be used		
	Scale range	Maximum numbered intervals	Maximum intermediate graduations
655 and under	0 to 700	70	3,5
620 to 1 730	0 to 2 000	175	7
1 660 to 3 460	0 to 3 500	350	35

Only accurate gauges shall be used. When the "gauge error" (see 9.1) exceeds 2 % of the scale range, the gauge shall be withdrawn from use.

4.3 Vapour pressure water bath, of such dimensions that the vapour pressure apparatus may be immersed so as to completely cover the bleeder-valve when the assembly is in an upright position.

4.4 Means for maintaining the bath at the test temperature within the limits as follows :

- test temperature 50 °C and below : $\pm 0,1$ °C;
- test temperature above 50 °C : $\pm 0,3$ °C.

In order to check the bath temperatures, the appropriate bath thermometer (see 4.5) shall be immersed to the test temperature graduation mark on the thermometer scale throughout the vapour pressure determination.

4.5 Thermometers, complying with the appropriate specification in table 2 :

- low range for indicating test temperatures between 35 and 40 °C;
- middle range for indicating test temperatures between 40 and 70 °C;
- high range for indicating test temperatures between 50 and 80 °C.

4.6 Dead-weight tester, of satisfactory range, as a means for checking the accuracy of vapour pressure gauges.

5 SAMPLING AND SAMPLE HANDLING

5.1 Samples shall be obtained and stored in accordance with ISO 4257 unless the test samples can be taken directly from the source of the material to be tested.

5.2 Any convenient method of coupling the vapour pressure apparatus to the sample source may be employed. A minimum length of tubing, 6 to 7 mm in diameter, of grade appropriate to the pressure range involved in the test and made of material corrosion-resistant to the products being sampled, is satisfactory for this purpose. A flexible tubing connection of any satisfactory type greatly facilitates the purging and sampling operations. The tubing shall be made of an electrically conductive material or constructed with a built-in earth (ground) connection to minimize the effect of static electricity.

6 PREPARATION OF APPARATUS

6.1 If the apparatus has been used for testing products other than the type of product to be tested, disassemble, clean thoroughly, and purge the parts in a stream of dry air.

6.2 Assemble the apparatus with the inlet valve of the lower chamber open, the straight-through valve between the two chambers open, the bleeder-valve closed, and with a pressure gauge having a suitable range attached.

TABLE 2 – Specifications for thermometers

Characteristic	Low range	Middle range	High range
Range	34 to 42 °C	40 to 70 °C	50 to 80 °C
Immersion	total	total	total
Graduation at each	0,1 °C	0,1 °C	0,1 °C
Longer lines at each	0,5 °C	0,5 °C	0,5 °C
Figured at each	1 °C (except at 38 °C)	1 °C	1 °C
Scale error not to exceed	0,1 °C	0,1 °C	0,1 °C
Expansion chamber permitting heating to	100 °C	—	130 °C
Overall length	275 ± 5 mm	405 mm	379 ± 5 mm
Stem diameter	6 to 7 mm	5,5 to 7,0 mm	7 to 8 mm
Bulb length	25 to 35 mm	10 to 15 mm	25 to 35 mm
Bulb diameter	> 5 mm; < stem	> 5 mm; < stem	6 to 7 mm
Distance from bottom of bulb to line at	34,4 °C : 135 to 150 mm	40 °C : 100 mm min.	50 °C : 115 to 135 mm
Distance from bottom of bulb to line at	42 °C : 215 to 234 mm	—	80 °C : 324 to 344 mm
Distance from bottom of bulb to top of contraction chamber	60 mm	—	100 mm
Distance from bottom of bulb to bottom of contraction chamber	—	—	80 mm
Stem enlargement diameter	8 to 10 mm	—	—
Stem enlargement length	4 to 7 mm	—	—
Distance from bottom of bulb to bottom of stem enlargement	112 to 116 mm	—	—
Range of ice point scale	ISO 4256:1978	—	-0,5 to + 0,5 °C
Distance from bottom of bulb to ice point	—	—	60 to 70 mm

7 SAFETY PRECAUTIONS

The following safety precautions regarding the handling of liquefied petroleum gases shall be meticulously observed :

- liquefied petroleum gases can cause serious cold burns; care shall be taken, therefore, to prevent the liquid from coming into contact with the skin; when handling liquefied petroleum gases, protective goggles and gloves shall be worn;
- discharge of liquefied petroleum gases can give rise to static electricity, and it is essential to connect apparatus to earth (ground) before discharging;
- care shall be taken to avoid breathing petroleum vapours during the determination of vapour pressure;
- care shall be taken that it is safe to discharge liquefied petroleum gases in the vicinity during ullaging operations;
- the above safety precautions must be supplemented by the note to 8.1 and notes 1 and 3 to 8.2, which give warning of additional hazards involved.

8 PROCEDURE

8.1 Purging

With the assembled apparatus in an upright position, connect the inlet valve of the lower chamber to the sample source with the sampling connection (see 5.2). Open the sample source valve to the apparatus. Cautiously open the bleeder-valve on the upper chamber, permitting the air or vapours, or both, in the apparatus to escape until the apparatus is full of liquid (see note 2 to 8.2). With the sampling line still connected, close the bleeder-valve and the inlet valve in the order given. Quickly invert the apparatus, open the bleeder-valve, and hold the apparatus in this position until all the liquid has been expelled. Allow the residual vapours to escape until the pressure in the apparatus is essentially atmospheric (see note). Then close the bleeder-valve.

NOTE – CAUTION – Safe means for the disposal of vapours and liquids during this operation and in the subsequent sampling operation (8.2) must be provided.

8.2 Introduction of test sample (see note 1)

Return the apparatus, now containing only vapours, to its normal upright position and open the inlet valve. As soon as the apparatus attains essentially the same pressure as at the sample source, momentarily open the bleeder-valve. If liquid does not promptly emerge, repeat the purging step (see 8.1). If liquid appears immediately, close the bleeder and inlet valves in that order (see note 2). Close the valve on the sample source, and disconnect the sampling line. Immediately close the straight-through valve between the two chambers and open the inlet valve, with the apparatus in an upright position. Close the inlet valve as soon as no more liquid escapes, and immediately open the straight-through valve (see note 3).

When using the 33 1/3 % lower chamber (see 4.1.3) proceed to 8.3.

When using the 20 % lower chamber (see 4.1.4) close the straight-through valve and again open the inlet to permit expulsion of the lower chamber contents. As soon as no more liquid escapes from the lower chamber, close the inlet valve and immediately open the straight-through valve.

NOTES

1 **CAUTION** — Materials of high coefficient of liquid expansion, such as propylene, if introduced in the apparatus at temperatures near their boiling point and then warmed to test temperatures, could expand sufficiently to cause the apparatus to become full of liquid with consequent risk to rupture. Therefore, if the 20 % lower chamber is used for such samples, it is imperative that the 40 % ullage procedure be completed promptly.

2 Transfer of the samples, either for purging (see 8.1) or for sampling (see 8.2), can be facilitated by chilling the apparatus with a portion of the material under test. To accomplish this, close the inlet valve and open the bleeder-valve to its wide-open position. Allow the contained sample to evaporate until the apparatus is cooled to well below the temperature of the sample source. Any residual material remaining after this operation can then be expelled from the apparatus through the bleeder-valve by inverting the assembly. The bleeder-valve is then closed, the chilled apparatus is returned to its normal upright position, and the test sample is taken in the manner previously outlined.

3 **CAUTION** — The upper chamber, prior to this operation, is full of liquid at some temperature that is normally below the environmental temperature. Since any warming of the apparatus would cause expansion of the liquid content of the upper chamber, leading to possible rupture of the chamber, it is necessary that the procedural steps of providing free space in the apparatus be completed promptly.

8.3 Vapour pressure determination

8.3.1 Invert the apparatus and shake it vigorously. Return the apparatus to its normal upright position and immerse it in the constant-temperature water bath maintained at the test temperature. The apparatus including the bleeder-valve coupling, but excluding the pressure gauge, must be immersed. Throughout the determination, the temperature of the water bath shall be checked periodically by means of the bath thermometer.

At test temperatures of 50 °C or below, maintain the bath within $\pm 0,1$ °C of the test temperature.

At test temperatures above 50 °C, up to and including 70 °C, maintain the bath within $\pm 0,3$ °C of the test temperature.

Observe the apparatus assembly throughout the test period to ensure freedom from leaks. Discontinue the test and discard the results if at any time a leak is detected.

8.3.2 After 5 min have elapsed, withdraw the apparatus from the water bath, invert it, shake it vigorously, and then return it to the bath; perform the shaking operation quickly to avoid excessive cooling of the apparatus and its contents. Thereafter, at intervals of not less than 2 min, withdraw the apparatus from the bath, invert, shake it vigorously and then return it to the bath. Prior to each removal of the apparatus from the water bath, tap the gauge lightly and observe the pressure reading. These operations will normally require 20 to 30 min to ensure equilibrium. After this time, if consecutive observed gauge readings are constant, record the pressure reading as the "Uncorrected LPG vapour pressure" of the sample at the test temperature.

8.3.3 Without removing the pressure gauge from the apparatus or the apparatus from the bath, attach a test gauge, previously calibrated against a dead-weight tester, to the bleeder-valve outlet and open the bleeder-valve. At the end of 5 min compare the readings of the two gauges. Record any correction thus determined as "gauge error".

9 EXPRESSION OF RESULTS

9.1 Correct the "Uncorrected LPG vapour pressure" for gauge errors.

9.2 Convert the corrected vapour pressure as calculated in 9.1 to a standard barometric pressure of 101,3 kPa by means of the following equation :

$$p_v = p_v' - (101,3 - p_b)$$

where

p_v is the LPG vapour pressure, in kilopascals, converted to a standard barometric pressure of 101,3 kPa;

p_v' is the corrected vapour pressure, in kilopascals (as calculated in 9.1);

p_b is the observed barometric pressure, in kilopascals.

10 PRECISION

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows :

10.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant

operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the value 11,0 kPa only in one case in twenty.

10.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test

method, exceed the value 31,0 kPa only in one case in twenty.

NOTE — These precision values were obtained at 45 °C only; a test program is planned in the near future to obtain precision data at 37,8 °C.

11 TEST REPORT

Report the result in kilopascals as LPG vapour pressure and state the temperature at which the determination was carried out. Indicate the method used by referring to this International Standard.

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