

Liquefied petroleum gases — Determination of gauge vapour pressure — LPG method

The European Standard EN ISO 4256:1998 has the status of a
British Standard

ICS 75.160.30

National foreword

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Liquefied petroleum gases — Determination of gauge vapour pressure — LPG method (ISO 4256:1996)

Gaz de pétrole liquéfiés - Détermination de la pression de vapeur relative - Méthode GPL (ISO 4256:1996)

Flüssiggase - Bestimmung des Dampfdruckes - LPG - Verfahren (ISO 4256:1996)

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Foreword

The text of the International Standard from Technical Committee ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

This European Standard replaces EN ISO 4256:1995.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 1998, and conflicting national standards shall be withdrawn at the latest by December 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 4256:1996 has been approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

Liquefied petroleum gases — Determination of gauge vapour pressure — LPG 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 International 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 method for the determination of gauge vapour pressures of liquefied petroleum gas products (see clause 3) at temperatures within the approximate range of 35 °C to 70 °C.

NOTES

1 Information on the vapour pressure of liquefied petroleum gases is required for the selection of properly designed storage vessels, shipping containers and customer utilization equipment, to ensure the safe handling of these products, and to ensure that maximum operating design pressures are not exceeded under the foreseen ambient operating conditions.

2 The vapour pressure of liquefied petroleum gases is an indirect measure of the lowest temperature at which initial vaporization can be expected to occur. It may also be considered to be an indirect indication of the most volatile constituent present in the product.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members

of IEC and ISO maintain registers of currently valid International Standards.

ISO 3007:1986, *Petroleum products — Determination of vapour pressure — Reid method*.

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

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 vapour pressure: Pressure exerted by the vapour of a liquid when in equilibrium with the liquid.

In this International Standard, the term “vapour pressure” shall be understood as gauge vapour pressure, which is absolute vapour pressure minus atmospheric pressure.

3.2 liquefied petroleum gas (LPG): Hydrocarbon gas that can be stored and/or handled in the liquid phase under moderate conditions of pressure and at ambient temperature. It consists essentially of C₃ and C₄ alkanes or alkenes, or a mixture of these, contains generally less than 5 % by liquid volume of material of higher carbon number, and has a gauge vapour pressure not exceeding approximately 1 600 kPa at 40 °C.

4 Principle

The test apparatus, equipped with a pressure gauge, is purged and then filled completely with an aliquot of the test sample. A given volume of the liquid content of the apparatus is withdrawn, and the apparatus immersed in a water bath maintained at the test temperature. The observed gauge pressure at equilibrium, corrected for gauge error and ambient barometric pressure, is recorded.

5 Apparatus

5.1 Vapour pressure apparatus, constructed as illustrated in figure 1, consisting of two chambers,

designated as the upper and lower chambers, complying with the requirements of 5.1.1 to 5.1.7. If the "air chamber" and the "20 % lower chamber" of the method described in ISO 3007 are used, which are interchangeable with the corresponding chambers in this method, the apparatus assembly shall pass a hydrostatic test (7.3) before being used for testing liquefied petroleum gases.

The volume ratios of the chambers shall be determined in accordance with annex A of ISO 3007:1986.

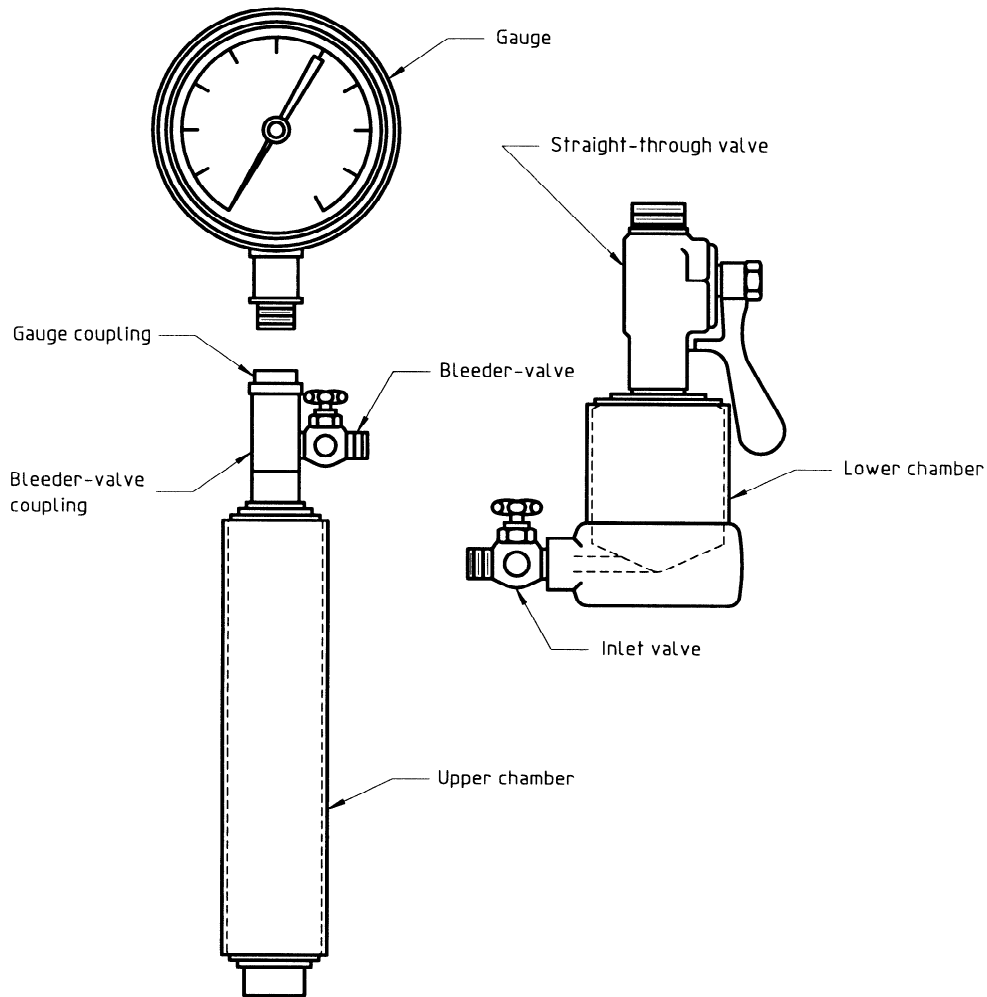


Figure 1 — Typical apparatus for measuring LPG vapour pressure

5.1.1 Upper chamber, consisting of a cylindrical vessel of inside dimensions $51 \text{ mm} \pm 3 \text{ mm}$ in diameter and $254 \text{ mm} \pm 3 \text{ mm}$ in length, 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 upper 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.

5.1.2 Lower chamber, 33 1/3 %, a cylindrical vessel of volume such 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, 5.1.3).

5.1.3 Lower chamber, 20 %, a cylindrical vessel of volume such 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).

NOTES

1 In determining the volumetric capacities of the chambers, the volume of the lower chamber is 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, is considered as part of the upper chamber volume.

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 procedure 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 (7.3).

5.1.4 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.

5.1.5 Valves and coupling.

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.

NOTE — 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.

5.2 Pressure gauge

A Bourdon-type spring gauge of test gauge quality 114 mm 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.

The observed gauge readings shall be corrected for "gauge error" in accordance with 9.2, either by direct calibration against a dead-weight tester (5.5), or by the use of a second gauge certified by a recognized official body.

Table 1 — Specifications for pressure gauges

LPG vapour pressure	Gauge specifications		
	Scale range	Maximum numbered intervals	Maximum intermediate graduations
kPa	kPa		
≤ 655	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

5.3 Vapour pressure bath, of dimensions such that the bleeder-valve assembly is completely immersed when the vapour pressure apparatus is inserted in an upright position.

The bath shall be capable of maintaining the test temperature within the following limits:

- for test temperature $\leq 50 \text{ }^\circ\text{C}$: $\pm 0,1 \text{ }^\circ\text{C}$;
- for test temperature $> 50 \text{ }^\circ\text{C}$: $\pm 0,3 \text{ }^\circ\text{C}$.

5.4 Temperature sensor, either a thermometer conforming to the specifications given in annex A, or a suitably calibrated electronic measuring device of equivalent accuracy.

The thermometer shall be positioned in the bath in such a manner that it can be viewed throughout the test and adjusted so that it is immersed to the test temperature graduation mark.

5.4.1 Low-range thermometer, for indicating test temperatures between 35 °C and 40 °C.

5.4.2 Middle-range thermometer, for indicating test temperatures between 41 °C and 50 °C.

5.4.3 High-range thermometer, for indicating test temperatures between 51 °C and 80 °C.

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

NOTE — If the second gauge (5.2) is certified by a recognized official body, this apparatus is unnecessary.

6 Sampling and sample handling

6.1 Obtain and store samples in accordance with ISO 4257 unless the test samples are taken directly from the source of the material to be tested.

6.2 Use any convenient method of coupling the vapour pressure apparatus to the sample source.

A minimum length of tubing, 6 mm 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. If a flexible tubing connection is used, 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.

6.3 Additional safety precautions for the handling of liquefied petroleum gases, given in annex B, shall be meticulously observed during all the following operations.

7 Preparation of apparatus

7.1 Cleaning

Disassemble the apparatus, clean thoroughly, and purge the parts in a stream of dry air.

7.2 Assembling

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 the pressure gauge (5.2) attached.

7.3 Hydrostatic test

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

7.4 Leak test

Before placing new apparatus in service, and each time the apparatus is used after a period of at least a week of nonutilization, 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.

8 Procedure

8.1 General

For specific hazard statements, see annex B.

If necessary, chill the apparatus with a portion of the material under test to facilitate transfer, either for purging (8.2) or for introduction of test sample (8.3). 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. Expel any residual material remaining after this operation from the apparatus through the bleeder-valve by inverting the assembly. Close the bleeder-valve. Return the chilled apparatus to its normal upright position, and take the test sample in the manner outlined below.

8.2 Purging

With the assembled apparatus (7.2) in an upright position, connect the inlet valve of the lower chamber to the sample source using the sampling connection (6.2). Open the sample source valve to the apparatus. Cautiously open the bleeder-valve on the upper chamber, permitting the apparatus to fill with liquid (see 8.1). With the sampling line still connected, close the bleeder-valve and the inlet valve in that order. 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, then close the bleeder-valve.

WARNING — All practical work should be carried out without stoppage time to avoid excessive pressure buildup.

8.3 Introduction of test sample

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 (8.2). If liquid appears immediately, close the bleeder-valve and inlet valves in that order (see 8.1). 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.

When using the 33 1/3 % lower chamber (5.1.2), proceed to 8.4.

When using the 20 % lower chamber (5.1.3), close the straight-through valve and for a second time 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.

8.4 Vapour pressure determination

8.4.1 Invert the apparatus and shake it vigorously. Return the apparatus to its normal upright position. Immerse the apparatus including the bleeder-valve coupling, but excluding the pressure gauge, in the constant-temperature bath maintained at the test temperature.

Throughout the determination, take the temperature of the water bath periodically by means of the bath thermometer. At test temperatures ≤ 50 °C, maintain the bath within $\pm 0,1$ °C of the test temperature; at test temperatures > 50 °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.

8.4.2 After 5 min have elapsed, withdraw the apparatus from the water bath, invert it, shake it vigorously along the vertical axis, 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 it, 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. Continue these operations until two consecutive gauge readings do not differ by more than 5 kPa. After this time, record the pressure reading as the "uncorrected

gauge vapour pressure" of the sample at the test temperature.

NOTE — Under normal operating conditions, constant consecutive gauge readings are achieved after a period of 20 min to 30 min.

8.4.3 Record the observed barometric pressure (p_b) required for the calculation in clause 9.2.

9 Determination of gauge error

9.1 If a noncalibrated gauge has been used to obtain the observed uncorrected gauge vapour pressure, the "gauge error" shall be determined at the end of the test by the procedure described in 9.2. The readings obtained shall not be regarded as test results, as the conditions of measurement differ.

9.2 Without removing the pressure gauge from the apparatus or the apparatus from the bath, attach a test gauge (5.2) 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".

10 Calculation

10.1 Correct the "uncorrected vapour pressure" for gauge errors (see 9.2).

10.2 Convert the corrected vapour pressure (10.1) to a standard barometric pressure of 101,3 kPa by means of the following equation.

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

where

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

p_o is the corrected gauge vapour pressure (10.1), in kilopascals;

p_b is the observed absolute barometric pressure (8.4.3), in kilopascals.

11 Expression of results

Report the gauge vapour pressure result to the nearest 5 kPa, together with the test temperature.

12 Precision

12.1 Repeatability, r

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 normal and correct operation of the test method, exceed the value below only in one case in 20:

$$r = 12 \text{ kPa}$$

12.2 Reproducibility, R

The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in 20:

$$R = 19 \text{ kPa}$$

13 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) all details necessary for complete identification of the product tested;
- c) the result of the test (see clause 10);
- d) any deviation, by agreement or otherwise, from the standard procedures specified;
- e) the date of the test.

Annex A (normative)

Specifications for thermometers

A.1 The thermometers specified in 5.4 shall meet the requirements given in table A.1.

Table A.1 — Specifications for thermometers

Characteristics	Low-range	Middle-range	High-range
Range °C	34 to 42	25 to 55	50 to 80
Immersion	Total	Total	Total
Graduation at each °C	0,1	0,1	0,1
Longer lines at each °C	0,5	0,5	0,5
Figured at each °C	1	1	1
Scale error not to exceed °C	0,1	0,1	0,1
Expansion chamber permitting heating to °C	100	105	130
Overall length mm	275 ± 5	379 ± 5	379 ± 5
Stem diameter mm	6 to 7	7 to 8	7 to 8
Bulb length mm	25 to 35	25 to 35	25 to 35
Bulb diameter mm	> 5 and < stem	6 to 7	6 to 7
Distance from bottom of bulb to line at °C: mm	34,4: 135 to 150	25: 115 to 135	50: 115 to 135
Distance from bottom of bulb to line at °C: mm	42: 215 to 234	55: 324 to 344	80: 324 to 344
Distance from bottom of bulb to top of contraction chamber (max.) mm	60	100	100
Distance from bottom of bulb to bottom of contraction chamber (min.) mm	–	80	80
Stem enlargement diameter mm	8 to 10	–	–
Stem enlargement length mm	4 to 7	–	–
Distance from bottom of bulb to bottom of stem enlargement mm	112 to 116	–	–

The following thermometers comply with the specifications given in table A.1:

Low-range: ASTM 18 C / IP 23 C
 Middle-range: ASTM 64 C
 High-range: ASTM 65 C

Annex B
(normative)

Safety precautions

B.1 General precautions

B.1.1 The safety precautions regarding the practice of handling liquefied petroleum gases in the laboratory shall be meticulously observed during the determination specified in this International Standard.

B.1.2 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.

B.1.3 Discharge of liquefied petroleum gases can give rise to static electricity, and it is essential to connect apparatus to earth (ground) before discharging.

B.1.4 Take care to avoid breathing petroleum vapours during the determination of vapour pressure.

B.1.5 Care shall be taken that it is safe to discharge liquefied petroleum gases in the vicinity during ullaging operations.

B.2 Specific precautions for this International Standard

B.2.1 Re: subclause 8.2

Safe means for the disposal of vapours and liquids during this operation and in the subsequent sampling operation (8.3) shall be provided.

B.2.2 Re: subclause 8.3

- a) Materials of high liquid coefficient of expansion, such as propene, if introduced in the apparatus at temperatures near their boiling point and then warmed to test temperatures, could expand sufficiently to cause the apparatus full of liquid to rupture. Therefore, if the 20 % lower chamber is used for such samples, it is imperative that the 40 % ullage procedure be completed promptly.
- b) 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.

Annex ZA (normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 4257	1988	Liquefied petroleum gases - Method of sampling	EN ISO 4257	1995

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