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Petroleum products — Fuels (class F) — Liquefied petroleum gases — Specifications

*Produits pétroliers — Combustibles (classe F) — Gaz de pétrole liquéfiés —
Spécifications*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9162 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Annex A of this International Standard is for information only.

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Petroleum products — Fuels (class F) — Liquefied petroleum gases — Specifications

HEALTH AND SAFETY WARNING — The use of this International Standard involves potentially hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to consult the relevant safety and health regulations, determine the applicability of regulatory limitations, and establish appropriate safety and health practices, prior to use.

1 Scope

This International Standard specifies required characteristics and additional information to be supplied to the purchaser by the vendor of those products commonly referred to as liquefied petroleum gases (see ISO 8216-3) and is intended to apply to international transfers of commercial propane and commercial butane. This International Standard is neither intended nor expected to replace or encroach upon national standards of any country, since each country is subject to different regulations, codes, industry practices and market utilization. Required characteristics for specific uses should be formulated to meet the specific needs of those uses in accordance with applicable national standards and safety codes.

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 3993 : 1984, *Liquefied petroleum gas and light hydrocarbons — Determination of density or relative density — Pressure hydrometer method.*

ISO 4256 : 1978, *Liquefied petroleum gases — Determination of vapour pressure — LPG method.*

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

ISO 4259 : 1979, *Petroleum products — Determination and application of precision data in relation to methods of test.*

ISO 4260 : 1987, *Petroleum products and hydrocarbons — Determination of sulfur content — Wickbold combustion method.*

ISO 6251 : 1982, *Liquefied petroleum gases — Corrosiveness to copper — Copper strip test.*

ISO 7941 : 1988, *Commercial propane and butane — Analysis by gas chromatography.*

ISO 8216-3 : 1987, *Petroleum products — Fuels (class F) — Classification — Part 3: Family L (Liquefied petroleum gases).*

ISO 8819 : 1987, *Liquefied petroleum gases — Detection of hydrogen sulfide — Lead acetate method.*

ISO 8973 : — ¹⁾, *Liquefied petroleum gases — Calculation method for density and vapour pressure.*

3 Definitions

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

3.1 commercial propane: A hydrocarbon product composed predominantly of propane and/or propene; the remaining part may consist mainly of ethane/ethene and butane/butene isomers.

3.2 commercial butane: A hydrocarbon product composed predominantly of butanes and/or butenes; the remaining part may consist mainly of propane/propene and pentane/pentene isomers.

4 Requirements

4.1 General

When determined in accordance with the methods given in table 1, the properties of commercial butane and commercial

1) To be published.

propane shall be in accordance with the limiting requirements given in that table.

NOTE — Individual contractual agreements, national standards, national safety codes and/or requirements of distribution systems may prescribe other limits.

4.2 Water content

Commercial butane and commercial propane shall not contain free or suspended water detectable by visual inspection.

5 Precision and interpretation of test results

Most of the methods of test given in table 1 contain a statement of the precision, i.e. the repeatability and reproducibility, to be expected from them but, in cases of dispute, the procedure described in ISO 4259, which uses precision data in the interpretation of test results, shall be used.

6 Additional information to be supplied by the vendor to the purchaser

The vendor of the commercial butane or propane shall supply the purchaser with the following additional information.

- a) Density: The density, in kilograms per cubic metre at 15 °C, determined by the method in ISO 3993 or ISO 8973.
- b) C₂ hydrocarbon content: The molar percentage of C₂ hydrocarbons and the method used to carry out the determination.

Physical limitations of transport and storage facilities shall be considered for refrigerated transport; a common limit is 2 % (molar) maximum.

- c) Unsaturated hydrocarbons: The molar percentage of unsaturated hydrocarbons, determined in accordance with ISO 7941.

- d) Residual matter: The residual matter, in milligrams per kilogram, and the method used to carry out the determination.

NOTE — The attention of the user of this International Standard is directed to the introductory discussion and proposed test method given in Annex A.

7 Documentation

The documentation supplied to the purchaser by the vendor shall include at least the following:

- a) a reference to this International Standard;
- b) the type of liquefied petroleum gas supplied, i.e. commercial propane or commercial butane;
- c) the brand name of the product;
- d) the ISO designation code, i.e. ISO-F-LP or ISO-F-LB (see ISO 8216-3);
- e) the supplier's production code and the date of production;
- f) precautionary and safety advice.

If a transportable container is supplied with the liquefied petroleum gas, it shall also be clearly marked with this information.

8 Sampling

A representative sample of non-refrigerated commercial butane or commercial propane shall be taken in accordance with the procedure given in ISO 4257.

For refrigerated liquefied petroleum gases, a sampling procedure shall be agreed between the parties concerned.

NOTE — Proper sampling of the liquefied petroleum gases is extremely important if the test results are to be significant.

Table 1 — Specifications for liquefied petroleum gases

Characteristics	Method of test	Commercial propane ISO-F-LP	Commercial butane ISO-F-LB
Gauge vapour pressure at 40 °C, kPa, max.	ISO 4256 ²⁾ or ISO 8973	1 550 ¹⁾	520 ¹⁾
Volatility			
C ₂ hydrocarbons		Report value ³⁾	
C ₄ hydrocarbons, % (molar), max.	ISO 7941	7,5 ¹⁾	
C ₅ hydrocarbons, % (molar), max.	ISO 7941	0,2 ⁶⁾	2,5 ⁶⁾
Unsaturated hydrocarbons, % (molar)	ISO 7941	Report value ⁴⁾	Report value ⁴⁾
Dienes, % (molar), max.	ISO 7941	0,5 ⁶⁾	0,5 ⁶⁾
Residual matter	5)	5)	5)
Corrosiveness to copper, max.	ISO 6251	1	1
Sulfur, mg/kg, max.	7)	50 ⁶⁾	50 ⁶⁾
Hydrogen sulfide	ISO 8819	Pass	Pass
Free water content		None ⁸⁾	None ⁸⁾
<p>1) Certain national standards and/or regulations may prescribe other limits.</p> <p>2) In case of dispute about the vapour pressure, ISO 4256 shall prevail.</p> <p>3) See clause 6, item b). Physical limitations of transport and storage facilities shall be considered for refrigerated transport; a common limit is 2 % (molar) maximum.</p> <p>4) See clause 6, item c). Certain national standards may prescribe a limitation on unsaturated hydrocarbons.</p> <p>5) It has not been possible to take a decision on a value for inclusion in this specification because there is no suitable ISO test method and no satisfactory correlation between the test results obtained using methods developed in different countries. Limits will be specified when a suitable test method has been adopted. Until a suitable test method has been adopted, the user of this specification is referred to the introductory discussion and proposed test method appended as annex A. Report the value for the sample concerned, and the method used to determine it, as additional information [see clause 6, item d)].</p> <p>6) Certain national standards may prescribe other limits.</p> <p>7) An ISO method specific to liquefied petroleum gas is being developed. Until such an ISO method is available, ISO 4260 is recommended as the referee method. Other methods specified in national standards, and having a valid precision statement, are acceptable for quality control purposes.</p> <p>8) The presence of water shall be determined by visual inspection. For shipments of refrigerated liquefied petroleum gases at their atmospheric boiling point, it is essential that the water content is below the saturation level at this temperature as determined by a method agreed between buyer and seller.</p>			

Annex A (informative)

Determination of the evaporation residue of liquefied petroleum gases

A.1 General

Liquefied petroleum gases may contain trace amounts of non-volatile residues. These contaminants can include a variety of hydrocarbons derived from the distillation process, compressor lubricating oils, valve greases and rubber and elastomer extenders from hoses.

In the majority of applications, the liquid is evaporated and the fuel is fed to the appliance in the gaseous phase.

In those cases where the natural evaporation capacity of the storage tank is not sufficient to provide the required quantity of gas, vaporizers are widely used. In this case, excessive non-volatile residues can be very detrimental.

A distinction can be made between industrial and automotive vaporizers and a variety of energy sources may be applied.

Automotive vaporizers use, in the majority of installations, the engine coolant as a heat source. With the engine fully warmed up, the coolant will have a temperature of about 80 °C to 100 °C. Taking this temperature into account as well as the more complicated construction of the automotive vaporizer/pressure regulator relative to the industrial installation, it can be concluded that the automotive equipment is the most sensitive with respect to the content of residual matter.

A commonly used method for the determination of the content of residual matter is that specified in ASTM D-2158 (IP 317), *Standard test method for residues in liquefied petroleum (LP) gases*. In this case, the volatile part is evaporated at a temperature of 100 °F (37,8 °C) and the residual part is quantified. However, this evaporation temperature is much below the level at which industrial and automotive vaporizers function, which means that more volatile hydrocarbons are collected, which are in fact not harmful.

There is therefore a need for a high-temperature method for the determination of residual matter in liquefied petroleum gases that will correlate with deposit formation in vaporizers, in both automotive and industrial applications. The standard conditions for such a method should provide for the residues being subjected to a final temperature in the region of 100 °C; the determination should be carried out on a mass basis and the result expressed accordingly, i.e. in milligrams per kilogram.

The levels of accuracy needed are as follows:

- Values up to 20 mg/kg should be reported to the nearest 1 mg.
- Values from 20 mg/kg to 100 mg/kg should be reported to the nearest 5 mg.
- Values over 100 mg/kg should be reported to the nearest 10 mg.

A suggested method is given in clause A.2 in the expectation that it may eventually become a standard method when sufficient experience of its use has been accumulated.

A.2 Proposed method for determination of evaporation residue of liquefied petroleum gases (high-temperature evaporation method)

A.2.1 Scope

This method describes a procedure for the determination of the evaporation residue of liquefied petroleum gas.

A.2.2 Principle

A known mass of sample is concentrated by evaporation on a steam bath. The concentrate is passed through a membrane filter to remove any particulate matter. The filtrate is then transferred to a basin, with the aid of dichloromethane, and evaporated, after which the evaporation residue is dried and weighed.

A.2.3 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

A.2.3.1 Acetone.

A.2.3.2 Ammonium persulfate/sulfuric acid, 8 g/l solution.

Prepare a sufficient volume of solution, as required, by carefully dissolving an amount of ammonium persulfate (ammonium peroxodisulfate) in concentrated sulfuric acid (ρ 1,84 g/ml) to obtain the specified concentration.

A.2.3.3 Carbon dioxide, solid, for the cooling bath (A.2.4.6).

A.2.3.4 Dichloromethane, evaporation residue maximum 5 mg/l.

WARNING — Dichloromethane is an irritant and presents a health hazard if incorrectly handled. Avoid inhalation. Extract vapour by working in a fume cupboard.

A.2.3.5 Laboratory detergent*).

A.2.3.6 Pentane.

*) Information on suitable products is available from the Secretariat of TC 28.

A.2.4 Apparatus

Ordinary laboratory apparatus and

A.2.4.1 Evaporation basins, made of borosilicate glass, capacity 150 ml, diameter 90 mm.

A.2.4.2 Filter holder, made of glass, for 47 mm membrane filters, consisting of a 300 ml funnel and funnel base with filter support screen.

A.2.4.3 Membrane filter discs, white plain, pore size 0,80 μm , diameter 47 mm.

A.2.4.4 Sample cylinder, made of stainless steel, with two valves, maximum working pressure 2,5 MPa (25 bar), capacity 0,5 litres, 1 litre or 2,5 litres depending on the size of the test portion (see A.2.6).

A.2.4.5 Cooling coil, made of stainless steel, internal diameter 4 mm, length 2 m, with coil diameter to fit the cooling bath and with a suitable connection to the sample cylinder (A.2.4.4).

A.2.4.6 Cooling bath, temperature below $-60\text{ }^{\circ}\text{C}$, containing a suitable liquid, e.g. acetone, which is cooled by means of solid carbon dioxide (A.2.3.3) and which is capable of accommodating the cooling coil (A.2.4.5).

A.2.4.7 Oven, capable of being maintained at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

A.2.4.8 Balance, capacity 10 kg, accurate to 1 g or better.

A.2.4.9 Analytical balance, accurate to 0,1 mg.

A.2.4.10 Thermometer, capable of measuring a temperature of $-60\text{ }^{\circ}\text{C}$.

A.2.5 Preparation of apparatus

A.2.5.1 Clean new evaporation basins (A.2.4.1) by immersion in laboratory detergent (A.2.3.5) for at least 12 h before use. In cases of dispute, or where anomalous results have been obtained, soak basins in ammonium persulfate/sulfuric acid solution (A.2.3.2) for 12 h. Remove the basins from the cleaning solution using forceps and handle only with forceps thereafter. Wash the basins with tap water, then deionized water and dry in the oven (A.2.4.7) for 30 min at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. Place the basins in a desiccator without drying agent for 30 min prior to use.

A.2.5.2 Remove any organic material from used basins by thoroughly rinsing with pentane (A.2.3.6), if necessary wiping with a clean, lint-free cloth or tissue. Rinse with acetone and

dry in the oven for 30 min at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, handling the basins with forceps only. Place the basins in a desiccator without drying agent for 30 min prior to use. If the basins are still not sufficiently clean, or in cases of dispute, or where anomalous results have been obtained, clean the basins as described in A.2.5.1, after first removing any organic material by rinsing with pentane.

A.2.5.3 Clean sample cylinders (A.2.4.4) by rinsing with acetone and pentane, and dry with a stream of dry air or nitrogen. It is desirable to reserve separate cylinders for use only in this determination.

A.2.6 Test portion

Depending on the expected evaporation residue, take as the test portion the amount of sample given in table A.1, using a clean sample cylinder (A.2.4.4) of appropriate capacity.

Table A.1 — Size of test portion

Expected evaporation residue mg/kg	Mass of test portion g
less than 10	1 000
10 to 20	400
greater than 20	200

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A.2.7 Procedure

A.2.7.1 Using balance A.2.4.8, weigh the sample cylinder with sample to the nearest 1 g. Wearing suitable protective gloves, connect the bottom valve of the sample cylinder to the cooling coil (A.2.4.5) maintained in the cooling bath (A.2.4.6) at $-60\text{ }^{\circ}\text{C}$. Transfer the entire contents of the cylinder, via the cooling coil, to a beaker containing 50 ml of dichloromethane (A.2.3.4). (**WARNING — See A.2.3.4.**)

Disconnect the cylinder from the cooling coil and reweigh, again to the nearest 1 g.

A.2.7.2 Place the beaker on a steam bath in a fume cupboard and evaporate the test portion until the level of liquid in the beaker is approximately 5 mm from the bottom. Adjust the steam supply to the bath so that the liquid in the beaker simmers gently.

NOTE — The test portion should not be allowed to evaporate too vigorously, in order to prevent losses by entrainment.

A.2.7.3 Place a filter disc (A.2.4.3) in the filter holder (A.2.4.2) and draw the test portion concentrate obtained in A.2.7.2 carefully through by applying a slight vacuum. Transfer the filtrate to a clean basin (see A.2.5), previously weighed, using the analytical balance (A.2.4.9), to the nearest 0,1 mg, place the basin on a steam bath and begin to evaporate its contents.

Rinse the beaker with 100 ml of dichloromethane, in three portions, passing each portion through the filter disc and slowly transferring these rinsings to the basin.

NOTE — The filtration step described above can be omitted if a 0,8 µm membrane filter disc is incorporated in the sample line. However, in this case, special care should be taken to ensure that the cooling coil and the evaporation beaker are scrupulously clean.

A.2.7.4 Allow the contents of the basin to evaporate. Remove the basin from the steam bath, wipe the underside of the basin with a clean lint-free cloth or tissue to remove any residual moisture, and place in the oven, maintained at 105 °C ± 5 °C, for 30 min. Remove the basin from the oven, allow to cool in the desiccator and then weigh, using the analytical balance (A.2.4.9), to the nearest 0,1 mg.

A.2.7.5 Perform a simultaneous blank determination following the procedure described in A.2.7.3 and A.2.7.4, using 150 ml of dichloromethane instead of the test portion concentrate and omitting rinsing of the beaker.

A.2.8 Expression of results

The total residue on evaporation of the test portion, expressed in milligrams per kilogram, is given by the formula

$$\frac{(m_2 - m_3) \times 10^3}{m_1}$$

where

m_1 is the mass, in grams, of the test portion ;

m_2 is the mass, in milligrams, of residue obtained in the sample determination ;

m_3 is the mass, in milligrams, of residue obtained in the blank determination.

A.2.9 Test report

The test report shall include the following particulars :

- a) the method used (a reference to annex A of ISO 9162) ;
- b) the results and the units in which they have been expressed ;
- c) any unusual features noted during the determination ;
- d) any operation not included in this International Standard, or regarded as optional.

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