International Standard 3993

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Liquefied petroleum gas and light hydrocarbons — Determination of density or relative density — Pressure hydrometer method

Gaz de pétrole liquéfiés et hydrocarbures légers — Détermination de la masse volumique ou de la densité relative — Méthode de l'aréomètre sous pression

First edition — 1984-08-01

UDC 665.725 : 531.754.3 **Ref. No. ISO 3993-1984 (E)**

Descriptors : petroleum products, liquefied petroleum gases, hydrocarbons, tests, physical tests, determination, density (mass/volume), hydrometers, test equipment.

Price based on 6 pages

Foreword

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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.

International Standard ISO 3993 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in March 1983.

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

Austria	Iran	Sri Lanka
Australia	Iraq	Sweden
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The member body of the following country expressed disapproval of the document on technical grounds :

France

Printed in Switzerland

Liquefied petroleum gas and light hydrocarbons — Determination of density or relative density — Pressure hydrometer method

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of density or relative density of liquefied petroleum gases and other light hydrocarbons. The prescribed apparatus shall not be used for materials having gauge vapour pressures higher than 1,4 MPa[1] (14 bar) (absolute vapour pressure 1,5 MPa) at the test temperature.

CAUTION — Attention is drawn to the hazards encountered when working with liquefied petroleum gas or light hydrocarbons. The requirements of any national, local or domestic safety code should always be strictly observed.

1.2 Alternative calibration procedures are described, but only the one using a certified hydrometer is suitable for the determination of density to be used in calculations of quantities for custody transfer or fiscal purposes.

NOTE — An alternative method for calculating the density of liquefied petroleum gases from gas chromatographic analysis is described in ISO 6578[2].

1.3 Provision is made in the annex for the use of thermohydrometers.

2 Definitions

For the purpose of this International Standard, the following definitions shall apply.

2.1 density : The mass of the liquid divided by its volume.

When reporting the density, the unit of density used, together with the temperature, shall be explicitly stated, for example kilogram per metre cubed or gram per millilitre at t °C (see the note). The standard reference temperature for international trade in petroleum and its products is 15 °C (see ISO 5024); but other reference temperatures may be required for legal metrology or other special purposes.

NOTE — In this International Standard, the preferred unit is the kilogram per metre cubed, but provision is also made for the use of the gram per millilitre.

2.2 relative density (this term now replaces the former term specific gravity) : The ratio of the mass of a volume of the liquid at a temperature t_1 to the mass of an equal volume of pure water at a temperature t_2, i.e. the ratio of the density of the liquid at a temperature t_1 to the density of pure water at a temperature t_2.

When reporting the relative density, the temperatures t_1 and t_2 shall be explicitly stated, for example relative density 60/60 °F. The standard reference temperature is 15 °C, but 20 °C and 60 °F are also in general use for t_1 and t_2 and other temperatures may be employed for t_1.

3 Principle

The apparatus is purged with a portion of the sample before filling with the portion to be used for testing. The pressure cylinder is filled to a level at which the enclosed hydrometer floats freely. The hydrometer reading and the temperature of the sample are noted.

1) The SI unit of pressure is the pascal : 1 Pa = 1 N/m^2; 10^5 Pa = 1 bar = 1,019 72 kgf/cm^2

2) At present at the stage of draft.

4 Apparatus

4.1 Hydrometers, made of glass, graduated in density or relative density, with the appropriate range and conforming to the dimensions given in table 1.

NOTE — For the use of thermohydrometers see the annex.

Use a certified hydrometer, or calibrate the hydrometers in accordance with clause 7. Calibration corrections should be applied if the scale errors are in excess of 0,5 of a subdivision of the scale.

4.2 Thermometer, having a sensitivity of at least 2,7 mm/1 °C (1,5 mm/1 °F) calibrated for total immersion and of suitable dimensions to fit inside the hydrometer cylinder (4.3).

A thermometer conforming to ISO/R 653, STL/0.2/ − 15/ + 45 is recommended.

The thermometer shall be held firmly inside the hydrometer cylinder by means of a suitable clip in such a position as not to interfere with the free motion of the hydrometer.

4.3 Hydrometer cylinder, constructed of glass or transparent plastics, for example polymethylmethacrylate or equivalent material, conforming to the design and dimensions given in the figure. The ends shall be tightly sealed by means of chloroprene gaskets and metal end-plates as shown.

CAUTION — As a precautionary measure a protective shield shall be placed around the plastics or glass cylinder. Replace any cylinders that show any fogging, crazing, cracking, or etching.

NOTE — Certain compounds attack plastics and cloud the inner surface of the cylinder, making it difficult or impossible to read the hydrometer. Tests have shown no attack by ethane, ethylene, propane, propylene, butane, isobutane, normal butylenes, isobutylene, pentane and isopentane and no attack is expected from butadiene. Users are cautioned, however, always to clean the cylinder thoroughly after each determination. Ketones and alcohols must not be used for cleaning as they attack and weaken plastics whilst aromatics also tend to attack the surface of plastics and should similarly not be used.

The liquid inlet valve and the liquid outlet valve shall be tightly connected to a base plate which shall be so bored as to give both valves a common inlet to the cylinder. The vapour vent valve shall be similarly connected to the top plate. All valves shall be 6 mm or equivalent needle valves.

The cylinder shall not be operated at a gauge pressure greater than 1,4 MPa (14 bar).

4.4 Water bath, fitted with a thermostat or other means of maintaining the bath at a constant temperature of 15 ± 0,2 °C or 20 ± 0,2 °C or 60 ± 0,5 °F, and of such dimensions that the hydrometer cylinder (4.3) can be completely immersed.

5 Reference liquids

The following reference liquids are required for calibration of the hydrometer if a certified hydrometer is not available.

5.1 Pure propane, having a certified density or relative density.

NOTE — Propane of density 507,6 kg/m^3 (0,507 6 g/ml) at 15 °C or 500,0 kg/m^3 (0,500 0 g/ml) at 20 °C or a relative density 60/60 °F of 0,507 3 is suitable.

5.2 Pure butane, having a certified density or relative density.

NOTE — Butane of density 584,5 kg/m^3 (0,584 5 g/ml) at 15 °C or 578,8 kg/m^3 (0,578 8 g/ml) at 20 °C or a relative density 60/60 °F of 0,584 4 is suitable.

Table 1 — Ranges and dimensional specification for pressure hydrometers

Alternative ranges	500 to 580 kg/m^3 570 to 650 kg/m^3	0,500 to 0,580 g/ml 0,570 to 0,650 g/ml	0,500 to 0,580 0,570 to 0,650
Subdivisions	1 kg/m^3	0,001 g/ml	0,001
Figured every	5 or 10 kg/m^3	0,005 or 0,010 g/ml	0,005 or 0,010
Overall length	330 mm max.		
Bulb diameter	18 to 20 mm		
Bulb wall thickness	0,4 to 0,6 mm		
Stem diameter	8 to 9 mm		
Stem wall thickness	0,3 to 0,35 mm		
Scale length	110 to 130 mm		

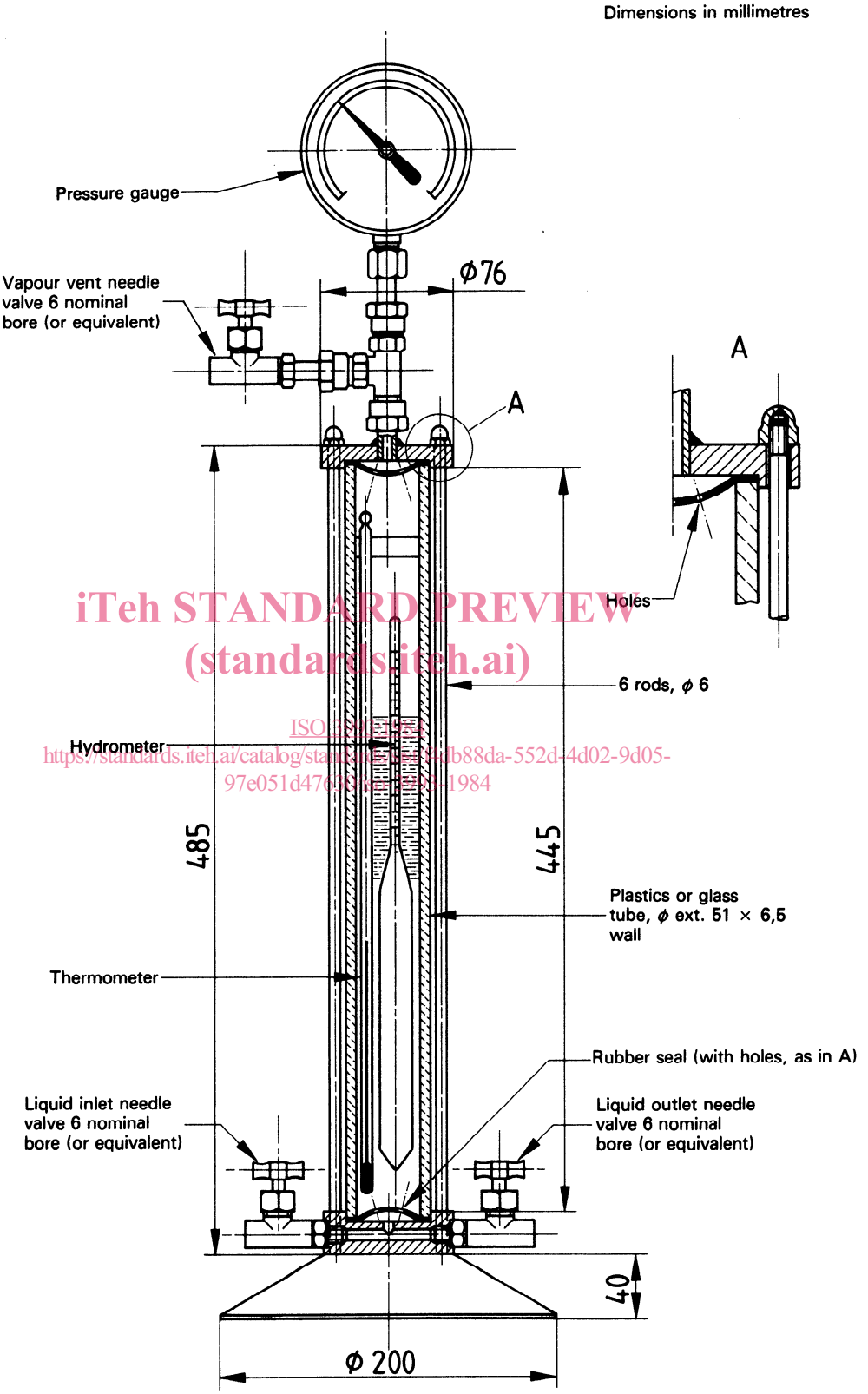

Figure — Pressure hydrometer cylinder

6 Sampling

The procedure for sampling for calibration of the apparatus and for subsequent testing is described below.

6.1 Carefully clean and dry the hydrometer (4.1), the thermometer (4.2) and the inside wall of the pressure cylinder (4.3). Insert the hydrometer in the pressure cylinder and attach the thermometer and cover plate.

6.2 Connect the source of supply of the liquid to be tested to the inlet valve by suitable fittings so that a representative sample can be introduced into the cylinder (4.3); ascertain that these connections are free from leaks. Open the outlet valve and purge the sampling connections by opening the inlet valve slightly, permitting the product to flow through the outlet valve at the bottom of the cylinder.

6.3 When the connections have been purged, close the outlet and vent valves and open the inlet valve, permitting the liquid to enter the cylinder until it is full. If necessary, the vent valve may be opened slightly to permit complete filling of the cylinder, after which it shall be closed. At no time shall the pressure in the cylinder be allowed to rise above a gauge pressure of 1,4 MPa (14 bar).

6.4 When the cylinder has been filled, close the inlet valve and open the outlet valve, permitting the contents of the cylinder to be withdrawn completely and the pressure inside the cylinder to be reduced to that of the atmosphere.

6.5 Close the outlet valve and open the inlet valve, filling the cylinder to a level at which the enclosed hydrometer floats freely. If it is necessary to accomplish this filling by venting vapour through the vent valve, repeat the purging to cool the cylinder sufficiently to permit its being filled without the necessity of venting.

6.6 With all valves closed, examine the apparatus for leaks; if leaks are detected, discard the sample, reduce the pressure to atmospheric and repair the leaks. Repeat the sampling procedure.

7 Calibration of apparatus

7.1 If a hydrometer having a calibration certificate issued by an approved laboratory is not used, the hydrometer shall be calibrated using one of the alternative procedures specified in 7.2 or 7.3 before densities are determined.

7.2 Take three or more samples having different densities in the range of the hydrometer to be tested. Using the procedure specified in clause 8 determine in duplicate for each sample the hydrometer reading on a certified hydrometer. Average the two values for each sample if these do not differ by more than 0,5 kg/m^2, or the equivalent. If the duplicate results differ by more than 0,5 kg/m^2, or the equivalent, repeat the determinations. Then obtain the average of duplicate hydrometer readings on the hydrometer being calibrated following the same procedure. Ensure that the maximum difference in temperature for all these determinations is not more than 0,4 °C (1 °F).

Compare the hydrometer readings obtained on the two hydrometers and record any differences as corrections to be applied to hydrometer readings on the hydrometer being calibrated.

7.3 Use the procedure in clause 8 to determine the density of one of the reference liquids of certified density (see clause 5) at a temperature within ± 0,2 °C (0,5 °F) of the certification temperature. Make duplicate determinations and average the two results if these do not differ by more than 0,5 kg/m^3, or the equivalent. Deduct the average from the certified density of the reference liquid to obtain the correction to be applied. If the two results differ by more than 0,5 kg/m^3, or the equivalent, repeat the determinations.

NOTE — This method provides a check of the hydrometer at only one point on the scale, but this is often acceptable for routine testing.

8 Procedure

8.1 Prepare the apparatus and take a sample of the liquid to be tested as specified in clause 6.

8.2 Disconnect the cylinder from the source of supply of liquid and place it in the water bath (4.4) maintained at 15 ± 0,2 °C, 20 ± 0,2 °C or 60 ± 0,5 °F until thermal equilibrium has been obtained. To accelerate thermal adjustment, occasionally remove the cylinder from the water bath, swirl it gently a few times to ensure mixing, and replace it in the water bath. Exercise care to prevent damage to the hydrometer and thermometer.

Measure the temperature of the water bath using a thermometer outside the cylinder in order to avoid the effects of pressure on the thermometer inside the cylinder. Use the thermometer inside the cylinder to ensure that the liquid under test has reached constant temperature and that the temperature of the liquid does not vary significantly during the determination.

8.3 Remove the cylinder from the water bath (see the note), stand it on a firm level surface and, while the hydrometer is floating freely, take the hydrometer reading as promptly as possible in the following manner :

Observe a point slightly below the plane of the liquid surface and then raise the line of vision until this surface, seen as an ellipse, becomes a straight line. The point where this line cuts the hydrometer scale is the reading of the instrument. A white card held behind the cylinder just below the liquid level will improve the visibility of the surface. Estimate the hydrometer reading to the nearest one-fifth of a subdivision of the scale.

NOTE — The cylinder may be left in the water bath, provided that the hydrometer reading can be clearly observed in accordance with the remainder of this sub-clause.

Read the temperature in the cylinder to the nearest 0,2 °C (0,5 °F) immediately before and after reading the hydrometer. If these temperatures differ by more than 0,4 °C (1 °F), repeat the determination.

8.4 Immediately after each determination, empty the liquid from the cylinder and vent to reduce the pressure to atmospheric. Highly volatile liquids and liquefied petroleum gases must not be left in the apparatus since, at high ambient temperatures, they might generate sufficient pressure to burst the cylinder.

8.5 This method may be used for measurements in the field at ambient temperatures, with the realisation that the results are less precise and the precision data in clause 9 may not apply.

CAUTION — If so used, the cylinder must be vented and the test discontinued if the pressure in the cylinder rises above a gauge pressure of 1,4 MPa (14 bar).

8.6 Apply hydrometer corrections, if any, to the result. If determinations are not carried out at the reference temperature correct the results in accordance with the petroleum measurement tables referred to in ISO 91[1)] (see notes 1 and 2).

NOTES

1 Table 53A should be used for the correction of densities to 15 °C for corrected densities down to 612 kg/m^3 and table 23A for the correction of relative densities to 60/60 °F for corrected relative densities down to 0,612; for values below these ranges and down to the equivalent of 500 kg/m^3 (0,500 g/ml) at 15 °C, reference should be made to the corresponding tables in the 1952 edition of API 2540, ASTM D 1250 and IP 200, and to tables 33 and 34 of ASTM D 1250-80; table A in the Addendum to ISO/R 91[2)] should be used for the correction of density to 20 °C.

2 Suitable conversions of corrected hydrometer readings with butadiene from test temperature (−20 to 60 °C) to reference temperature are given by the equation :

$$\varrho_t = \text{hydrometer reading} + \alpha(t_1 - t) + \beta(t_1 - t)^2$$

or

$$d_t = \text{hydrometer reading} + \alpha(t_1 - t) + \beta(t_1 - t)^2$$

where

ϱ_t is the density in kilograms per cubic metre at 15 °C or 20 °C;

t is the reference temperature of 15 °C, 20 °C or 60 °F;

t_1 is the temperature at which the hydrometer reading was taken;

α and β are the coefficients taken from table 2, corresponding to the type of soda-lime[3)] glass hydrometer used;

d_t is the relative density 60/60 °F.

Table 2 — Coefficients for conversion of hydrometer readings to density or relative density for butadiene

Hydrometer type	α	β
Density at 15 °C	1,215 7	$1{,}911 \times 10^{-3}$
Density at 20 °C	1,234 6	$1{,}910 \times 10^{-3}$
Relative density 60/60 °F	$6{,}753\,9 \times 10^{-4}$	$5{,}898 \times 10^{-7}$

9 Precision

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

9.1 Repeatability

The difference between successive 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 following value only in one case in twenty :

density 1 kg/m^3 or 0,001 g/ml
relative density 0,001

9.2 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 following value only in one case in twenty :

density 3 kg/m^3 or 0,003 g/ml
relative density 0,003

10 Test report

The test report shall contain the following information

a) reference to this International Standard;

b) the value of the corrected reading, to the nearest 1 kg/m^3, or equivalent;

c) whether the value reported is density or relative density;

d) if density, the unit and temperature (see 2.1);

e) if relative density, the temperatures t_1 and t_2 (see 2.2);

f) the method used for hydrometer calibration (see clause 7).

1) ISO 91, *Petroleum measurement tables*.

2) In course of revision as ISO 91/2.

3) Calculated for glass having a thermal cubic expansion coefficient of $25 \times 10^{-6}\ K^{-1}$.

Annex

Use of thermohydrometers

(Forms part of the Standard.)

In certain circumstances, in particular for measurements in the field, thermohydrometers may be more convenient to use. Thermohydrometers of suitable range and of dimensions such that they float freely within the pressure cylinder (minimum clearances : at walls 5 mm, at top and bottom 25 mm) may be used.

No precision figures can be given for results obtained when using thermohydrometers, and the test report should state that a thermohydrometer was used.