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ISO 3838

Second edition
2004-05-01

Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods

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Pétrole brut et produits pétroliers liquides ou solides — Détermination de la masse volumique ou de la densité — Méthodes du pycnomètre à bouchon capillaire et du pycnomètre bicapillaire gradué

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3838 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 3, *Static petroleum measurement*.

This second edition cancels and replaces the first edition (ISO 3838:1983), which has been technically revised.

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Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods

1 Scope

1.1 This International Standard specifies methods for the determination of the density or relative density of crude petroleum and of petroleum products handled as liquids.

1.2 The capillary-stoppered pyknometer method is also for use with solids and this method may also be used for coal tar products, including road tars, creosote and tar pitches, or for mixtures of these with petroleum products. This method is not suitable for the determination of the density or relative density of highly volatile liquids having Reid vapour pressures greater than 50 kPa according to ISO 3007 or having an initial boiling point below 40 °C.

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1.3 The graduated bicapillary pyknometer method is recommended for the accurate determination of the density or relative density of all except the more viscous products, and is particularly useful when only small amounts of samples are available. The method is restricted to liquids having Reid vapour pressures of 130 kPa or less according to ISO 3007 and having kinematic viscosities less than 50 mm^2/s [50 centistokes (cSt)] at the test temperature.

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Special precautions are specified for the determination of the density or relative density of highly volatile liquids.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 91-1:1992, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 °C and 60 °F*

ISO 91-2:1991, *Petroleum measurement tables — Part 2: Tables based on a reference temperature of 20 °C*

ISO 653:1980, *Long solid-stem thermometers for precision use*

ISO 3007:1999, *Petroleum products and crude petroleum — Determination of vapour pressure — Reid method*

ISO 3507:1999, *Laboratory glassware — Pyknometers*

ISO 5024:1999, *Petroleum liquids and liquefied petroleum gases — Measurement — Standard reference conditions*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
density
mass of the substance divided by its volume

NOTE When reporting the density, it is necessary for the unit of density used, together with the temperature, to be explicitly stated, for example kilograms per cubic metre, or grams per millilitre, at t °C.

3.2
apparent mass in air
value obtained by weighing in air against standard masses without making correction for the effect of air buoyancy on either the standard masses or the object weighed

3.3
observed density
value required in order to enter Tables 53A, 53B and 53D, or 59A, 59B and 59D, referred to in ISO 91, determined with soda-lime glass apparatus at a test temperature which differs from the calibration temperature of the apparatus, no correction having been made for the thermal expansion or contraction of the glass

3.4
relative density
ratio of the mass of a volume of a substance at a temperature t_1 to the mass of an equal volume of another substance at a temperature t_2. The temperatures t_1 and t_2 may be equal. For the purpose of this International Standard, the other substance is water, i.e. the relative density is the ratio of the density of the substance at a temperature t_1 to the density of water at a temperature t_2.

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NOTE When reporting the relative density, it is necessary for the temperatures t_1 and t_2 be explicitly stated. ISO 91-1 refers to tables for the reduction of relative density to 60/60 °F. If results are required referred to another reference temperature, the determination should be carried out at that temperature.

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4 Principle

4.1 Capillary-stoppered pyknometer

The masses of equal volumes of the sample and of water are compared. Equal volumes are ensured by the pyknometer being filled so as to overflow when placed in a bath at the test temperature until equilibrium is reached. The calculation (Clause 10) includes corrections for thermal expansion of glass and for buoyancy.

4.2 Graduated bicapillary pyknometer

The graduated arms of the pyknometer are calibrated, using water, in terms of the apparent mass in air of water contained in the pyknometer, and a graph is prepared. The liquid sample is drawn into the dried pyknometer and, after it has reached equilibrium at the test temperature, the liquid levels are noted and the pyknometer weighed. The apparent mass in air of an equal volume of water is read from the graph and the density or relative density of the sample is calculated, with corrections being made as in 4.1.

5 Apparatus

5.1 Capillary-stoppered pyknometer, one of the three types shown in Figure 1 (see 8.1.1).

5.1.1 The pyknometers shall conform to the relevant requirements of ISO 3507.

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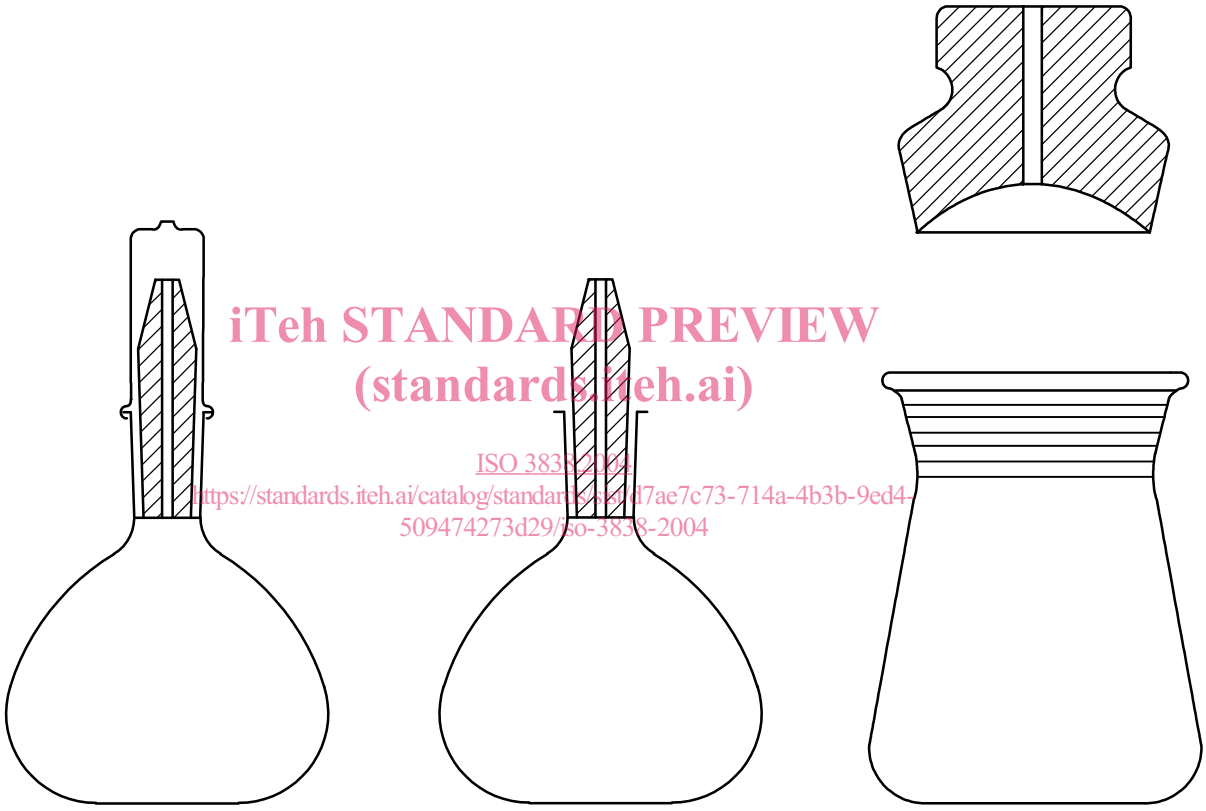
The “warden” form [see a) in Figure 1] is recommended for all except viscous or solid products and should always be used for volatile products. The ground glass cap, or “warden”, greatly reduces expansion and evaporation losses and this form of pyknometer may be used when the test temperature is lower than that of the laboratory.

5.1.2 The form of pyknometer shown in b) in Figure 1, known as the Gay-Lussac type, is suitable for non-volatile liquids except those of high viscosity.

5.1.3 The wide-mouth (Hubbard) form of pyknometer [see c) in Figure 1] is used for very viscous liquids and solids.

5.1.4 As the forms of pyknometer shown in b) and c) in Figure 1 have no “warden” or expansion chamber, they cannot be used when the temperature of the test is so far below that of the laboratory as to cause loss of sample by expansion through the capillary during weighing.



a) b) c)

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Figure 1 — Capillary-stoppered pyknometers

5.2 Graduated bicapillary pyknometer, capacity 1 ml to 10 ml, conforming to the dimensions given in Figure 2 and the characteristics given in Table 1, constructed of borosilicate glass or soda-lime glass, annealed after manufacture, and having a total mass not exceeding 30 g. Any pyknometer conforming with the requirements of the Lipkin pyknometer given in ISO 3507 may be used.

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Table 1 — Characteristics of the graduated bicapillary pyknometer

Nominal capacity, ml	1	2	5	10
Difference between actual capacity and nominal capacity, max, ml	± 0,2	± 0,3	± 0,5	± 1
Maximum mass, g	30			
Overall height, A, mm	175 ± 5			
Height above scale, B, min., mm	40			
Height from bulb to scale, C, min., mm	5			
Distance between centres of vertical limbs, D, mm	28 ± 2			
External diameter of tubing, F, mm	6			
Internal diameter of tubing, G, mm	1 ± 0,1			
Length from bottom of bulb to zero graduation line, H, mm	40			
External diameter of bulb, J, mm	11	14	20	25

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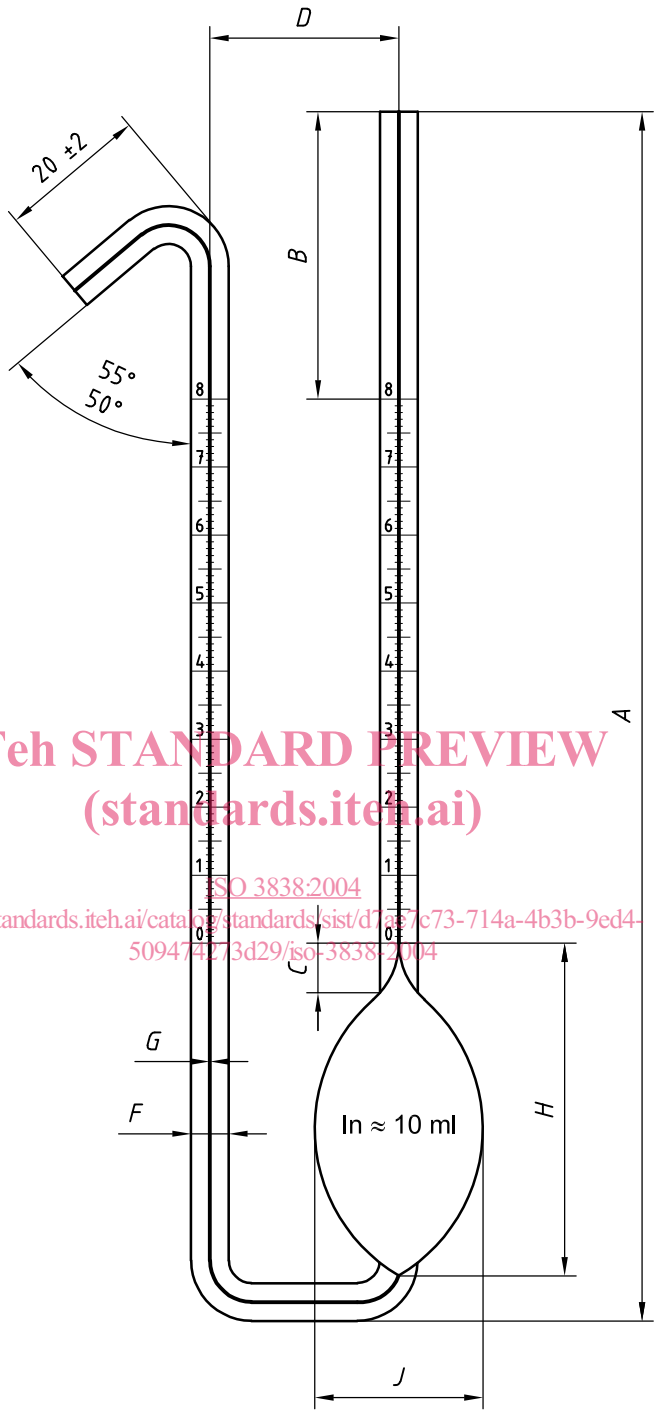
Dimensions in millimetres



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The explanations of symbols *A* to *J* are given in Table 1.

Figure 2 — Graduated bicapillary pyknometer (Lipkin type)

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5.3 Constant-temperature water bath, having a depth greater than that of the pyknometer, capable of being maintained within 0,05 °C (0,1 °F) of the desired temperature.

5.4 Bath thermometer, conforming to the specification ISO 653/STL/0,1/−5/+25. Other total immersion thermometers of suitable range and equal or greater accuracy may also be used.

NOTE For the determinations of relative density 60/60 °F, a Fahrenheit thermometer of suitable range graduated at 0,2 °F intervals may be used or the specified Celsius thermometer may be used at 15,56 °C.

5.5 Pyknometer holder (optional), to hold the pyknometer vertically and at the correct depth in the constant temperature bath. It shall be constructed of any suitable metal that will not corrode in the water bath.

A suitable design of holder for the graduated bicapillary pyknometers is shown in Figure 3.

Several pyknometer holders may be conveniently supported in the water bath by the use of a non-corrodible rectangular metal bar of sufficient length to lie across the rim of the bath. A series of holes of sufficient diameter to accommodate the 6,5 mm rod of the pyknometer holder is drilled in the bar at about 45 mm apart. Each rod is secured in its hole by locking the bar between the hexagon nut, and the winged nut and washer.

5.6 Balance, capable of weighing to the nearest 0,1 mg.

6 Preparation of pyknometer

Thoroughly clean the pyknometer and stopper with surfactant cleaning fluid, rinse well with distilled water, then with a water-soluble volatile solvent such as acetone, and dry. Ensure that all traces of moisture are removed, using a current of filtered air if necessary. Cleaning should be carried out in this manner whenever the pyknometer is to be calibrated or whenever liquid fails to drain cleanly from the internal walls of the pyknometer or the capillary of the stopper. Normally, the pyknometer may be cleaned between determinations by washing with a suitable light petroleum spirit such as 40/60 °C petroleum spirit, followed by vacuum drying.

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NOTE If surfactant cleaning fluids do not give adequate cleaning, chromosulfuric acid cleaning solution may be used.

CAUTION — Chromosulfuric acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr (VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromosulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solution down the drain, but neutralize it with great care owing to the concentrated sulfuric acid present, and dispose of it in accordance with standard procedure for toxic laboratory waste (chromium is highly dangerous to the environment).

7 Calibration of pyknometer

7.1 Conditioning

After drying, allow the pyknometer to reach room temperature. Dissipate any static charge which may have formed on it and then weigh to the nearest 0,1 mg.

If the balance case is not fitted with an electrostatic eliminator, electrostatic charges may be dissipated by breathing on the pyknometer, but ensure that the pyknometer has regained constant mass before recording the mass.

For the greatest accuracy, all the weighings should be made at temperatures within a 5 °C range so as to limit differences in air density.

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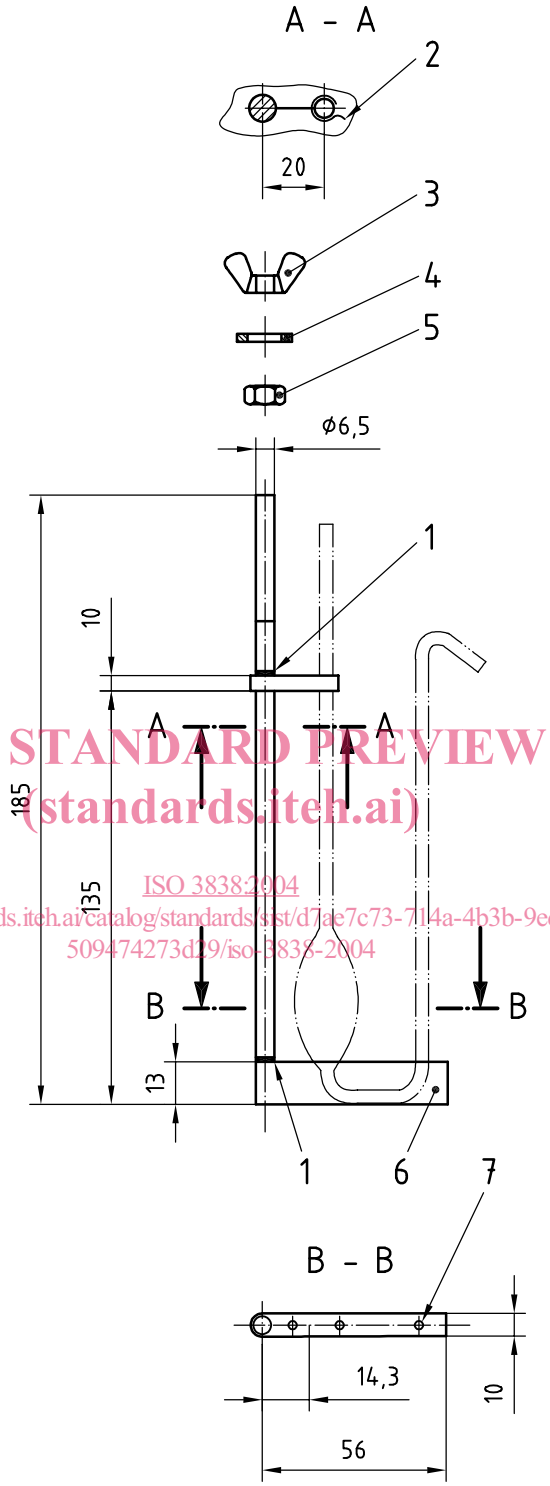
Dimensions in millimetres



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Key

1	solder	4	washer
2	spring clip 0,32 mm sheet metal (brass)	5	hexagon nut
3	wing nut	6	sheet metal tray (brass), sheet thickness 0,315 mm
		7	3 holes, ∅ 3 mm

Figure 3 — A suitable design of holder for graduated bicapillary pyknometer

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