IS 1448-32 (1992): Methods of test for petroleum and its products, Part 32: Density and relative density [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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पेट्रोलियम और इसके उत्पाद – परीक्षण पद्धतियाँ

[पी : 32]

घनत्व और आपेक्षिक घनत्व

(दूसरा पुनरीक्षण)

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

[P : 32]

DENSITY AND RELATIVE DENSITY

(Second Revision)

UDC 665·61·7 : 531·756

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 8

FOREWORD

This Indian Standard [P : 32] (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

While preparing the standard considerable assistance has been derived from the standards IP 59/82, IP 189/86 and IP 190/86, issued by the Institute of Petroleum, London (U. K.).

Provisions have been made in this standard for the conversion of density and relative density from the test temperature to 15°C or 20°C by using the ASTM/IP Petroleum Measurement Tables (Clause **3.4.1**). Accordingly, use of various tables viz. Tables 23A, 23B, 23D, 24A, 24B, 24D, 53A, 53B, 53D, 54A, 54B, and 54D of IP 200 and Table A and Table B of IP 250 referred to in API-ASTM-IP Petroleum Measurement Tables (API 2540; ASTM D 1250/IP 200 and IP 250) has been felt necessary for the purpose of corrections for thermal expansion of the pyknometer in the calculation of density from measurements made at a temperature at which the pyknometer has been calibrated (Clauses **9.3** and **17.3**). The Tables mentioned above have, however, not been included in the standard as the same are widely known and in use since long by the various testing laboratories.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

[P : 32]

DENSITY AND RELATIVE DENSITY

(Second Revision)

1 SCOPE

1.1 Four methods are described to determine the density and relative density of solid and liquid petroleum products and similar materials.

1.1.1 The application of these methods is indicated in the following table:

Product	*Recommended Methods*
a) Mobile liquids with Reid vapour pressure up to 1·3 kg/cm², e. g., Stabilized crude oil, Stabilized Gasoline, Naphthas, Kerosine, Gas oils, Lubricating oils, Non-waxy fuel oils	A. Density Balance method B. Bicapillary pyknometer method C. Capillary stoppered pyknometer method
b) Viscous liquids and semi-solids, e. g., Waxy fuel oils, Slack waxes, Soft bitumen	C. Capillary stoppered pyknometer method
c) Solids, e. g., Bitumen	C. (i) Capillary stoppered pyknometer method D. (ii) Displacement method

2 TERMINOLOGY

2.1 Density of Solids and Liquids

The density o a solid or liquid substance at a temperature t is defined as the mass of the substance occupying unit volume at a temperature t. When reporting the density the units of mass and volume used together with the temperature shall be explicitly stated, for example, grams per millilitre at t°C.

2.2 Relative Density of Solids and Liquids

Relative density t_1/t_2 of solid and liquid substance is defined as the ratio of the density of the substance at a temperature t_1 to the density of water at a temperature t_2. When the reference temperature t_2 is 4°C, relative density and density are numerically equal since the density of water at 4°C is unity.

3 METHOD A (RELATIVE DENSITY BALANCE METHOD)

3.1 Outline of the Method

A plummet suspended from the graduated beam of a density or relative density balance (Fig. 1), such as the Westphal balance, is immersed in the liquid which is held at the test temperature. Four rider weights are then disposed on the beam in such a way as to return the latter to its original position. The positions of the tiders on the beam gives a direct indication of the relative density of the liquid. The density of the material is calculated from its relative density. The amount of sample required is small (15 ml), and practically all of it is recoverable.

3.1.1 This method is suitable for determining quickly and accurately the density or relative

density of petroleum products, such as gasoline, kerosine, and gas oil, particularly when the quantities available for test are small.

3.2 Apparatus

3.2.1 *Balance* — Westphal or equivalent balance, capable of determining relative density or density to the forth decimal place, and equipped with a plummet for use at the test temperature.

3.2.2 *Sample Container* — A jacketed container in which the sample temperature, may be kept constant at the test temperature, and from which the sample may be run off at the conclusion of the test.

3.2.3 *Balance Case* — To protect the balance from draughts and dust.

3.2.4 *Support for the Sample Container* — A device for supporting the sample container in a vertical position within the balance case.

3.2.5 *Water-Circulating Pump*

3.2.6 *Constant-Temperature Bath* — A water-bath capable of maintaining the sample at the test temperature to within ± 0·05°C.

3.2.7 *Thermometer* — *See* **3.4.1.1.**

3.3 Buoyancy Corrections

3.3.1 To obtain the true relative density, that is the relative density in vacuum, of a substance from its relative density in air as determined by comparing the mass in air of equal volumes of the substance and water, a correction shall be applied for buoyancy of the air. This correction for the temperature of 15°C is given by the following formula:

The true relative density, B

$= A - 0{\cdot}001\,2\,(A - 1)$

where

A = relative density in air.

For ready reference, buoyancy corrections for various values of [0·001 2 (A — 1)] are given in Table 1.

3.4 Temperature Corrections for Liquid Substances

3.4.1 Whenever possible the substance whose density or relative density is being determined should be brought to the reporting temperature. When this is not practicable the deviation from the reporting temperature should not exceed 10°C for obtaining good results. The conversion of density and relative density from the test temperature to 15°C or 20°C may be done by using the ASTM/IP Petroleum Measurement Tables.

3.4.1.1 For the measurement of temperature, suitable thermometer graduated in tenths of a degree shall be used.

3.5 General Precautions

3.5.1 All apparatus shall be absolutely clean and dry before use. When drying and filtering the liquid samples for the removal of water and suspended impurities, precautions shall be taken to minimize loss of volatile constituents.

3.5.2 When the products are heated to facilitate pouring, changes due to evaporation of volatile constituents shall be avoided.

3.6 Standardization

3.6.1 Assemble the apparatus as shown in Fig. 1 and, using a suitable pump, circulate water from the bath through the annulus of the sample container (1 in Fig. 1) so as to keep the temperature, as measured by the thermometer (2 in Fig. 1) in the container, at the test temperature ± 0·05°C.

3.6.2 Standardize the instrument in the following way:

Adjust the balance to equilibrium in air, and then fill the container with freshly boiled distilled water. When the latter has reached test temperature (10 to 15 minutes) adjust the riders of the beam (3 in Fig. 1) until equilibrium is obtained and read the result to the fourth decimal place. All subsequent results obtained from the balance shall be divided by this figure.

3.6.3 Empty the container by opening the tap (4 in Fig. 1) at the bottom; clean it with acetone, drain it, and dry it with a current of air.

3.7 Procedure

3.7.1 Pour the sample to be tested into the clean sample container without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower-boiling constituents of the lighter oils. In the case of reference determinations, errors arising from changes in the volatility may be avoided by transferring the sample to the sample container by water displacement or by siphoning. If air bubbles are formed, remove them after they have collected on the surface by touching them with a piece of clean blotting paper or filter paper before placing the plummet in the sample.

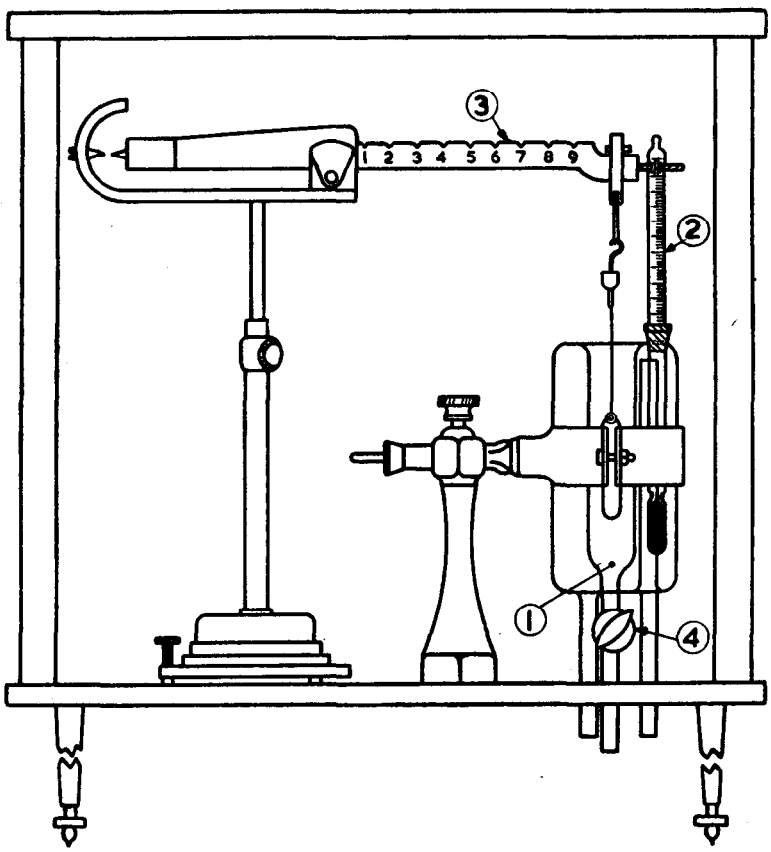


FIG. 1 RELATIVE DENSITY BALANCE

3.7.2 Make the determination on the material under test using the technique described in **3.6.2** and divide the result by the value obtained for distilled water. This gives the relative density in air of the product at the test temperature.

3.8 Calculation and Reporting

3.8.1 To obtain relative density in vacuo, correct the relative density 15°/15°C in air by applying the buoyancy correction from Table 1. Report the value to the forth decimal place as relative density 15°/15°C.

3.8.2 To obtain the value of density in vacuo, for example at 15°C correct the relative density 15°/15°C by applying the buoyancy correction from Table 1, then multiply the figure so obtained by the density of water at 15°C which is 0·999 1 g/ml. Report the result to the fourth decimal place.

3.9 Precision

Results of duplicate tests shall not differ by more than the following amounts:

Size of Plument	*Repeatability*	*Reproducibility*
5 ml	0·000 4	0·000 4

4 METHOD B (GRADUATED BICAPILLARY PYKNOMETER METHOD)

4.1 General

4.1.1 This method covers the determination of the density of crude petroleum and of petroleum products or mixtures of petroleum and non-petroleum products normally handled as liquids. The method is restricted to liquids having Reid vapour pressures of 1·25 bar ($1{\cdot}25 \times 10^5$ Pa) or less and having viscosities less than 50 centistokes at the test temperature.

Table 1 Buoyancy Corrections

(*Clause* 3.3.1)

[Corrections to be added are shown with (+) mark and those to be subtracted with (—) mark]

Relative Density at 15/15°C	0·00	0·01	0·02	0·03	0·04	0·05	0·06	0·07	0·08	0·09
0·40	+0·0007	+0·0007	+0·0007	+0·0007	+0·0007	+0·0007	+0·0006	+0·0006	+0·0066	+0·0006
0·50	+0·0006	+0·0006	+0·0006	+0·0006	+0·0006	+0·0005	+0·0005	+0·0005	+0·0005	+0·0005
0·60	+0·0005	+0·0005	+0·0005	+0·0004	+0·0004	+0·0004	+0·0004	+0·0004	+0·0004	+0·0004
0·70	+0·0004	+0·0003	+0·0003	+0·0003	+0·0003	+0·0003	+0·0003	+0·0003	+0·0003	+0·0003
0·80	+0·0002	+0·0002	+0·0002	+0·0002	+0·0002	+0·0002	+0·0002	+0·0002	+0·0002	+0·0001
0·90	+0·0001	+0·0001	+0·0001	+0·0001	+0·0001	+0·0001	0·0000	0·0000	0·0000	0·0000
1·00	0·0000	0·0000	0·0000	0·0000	0·0000	—0·0001	—0·0001	—0·0001	—0·0001	—0·0001
1·10	—0·0001	—0·0001	—0·0001	—0·0001	—0·0002	—0·0002	—0·0002	—0·0002	—0·0002	—0·0002

4.1.2 Special precautions are described for determining the density of highly volatile liquids.

4.1.3 The method is recommended for the accurate determination of the density of all except the more viscous products, and is particularly useful when only small amounts of sample are available.

4.2 Outline of the Method

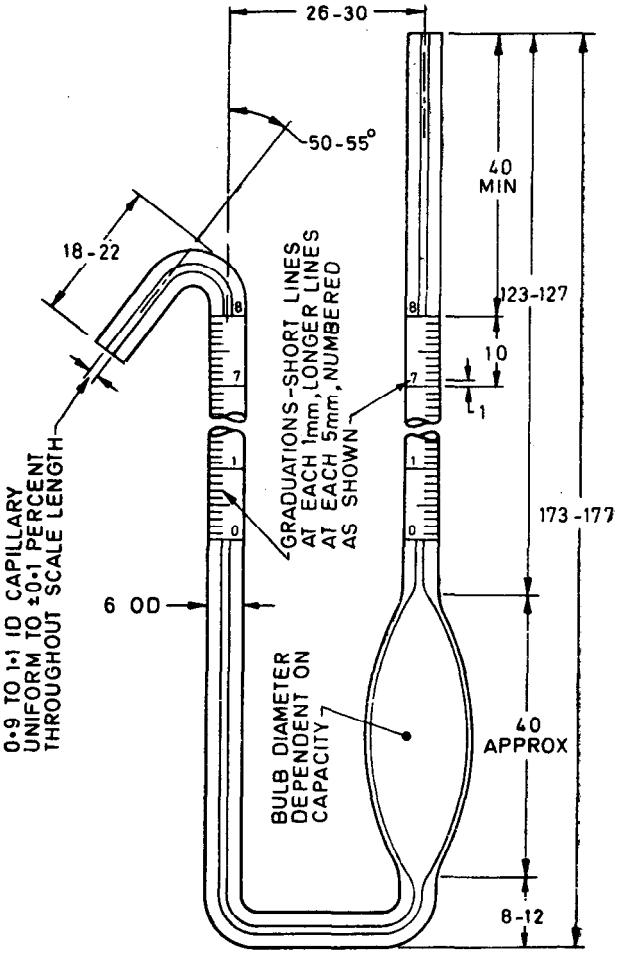
The liquid sample is drawn into the pyknometer and after it has reached equilibrium at the test temperature the liquid levels are noted and the pyknometer is weighed. The density of the sample is then calculated from its weight, a calibration factor proportional to an equal volume of water and a term which corrects for the buoyancy of air.

5 APPARATUS

5.1 Pyknometer — capacity 2 to 10 ml conforming to the dimensions given in Fig. 2 constructed of borosilicate or soda-lime glass, annealed after manufacture, and having a total weight not exceeding 30 g. Any bicapillary pyknometer conforming to these requirements may be used.

5.2 Constant Temperature Bath — water bath having a depth of at least 300 mm, capable of being maintained within ± 0·05°C of the desired temperature.

5.3 Bath Thermometer — *See* **3.4.1.1**. Other total immersion thermometers of suitable range and equal or greater accuracy may also be used.



All dimensions in millimetres.

FIG. 2 GRADUATED BICAPILLARY PYKNOMETER

5.4 Pyknometer Holder (Optional) — to allow the pyknometer to be mounted vertically at the correct depth in the constant temperature bath. A suitable design is shown in Fig. 3; it shall be constructed of metal which will not corrode in the water bath. A series of pyknometer holders may be supported in the constant-temperature 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 wing nut and washer.

Alternatively any other form of pyknometer mounting may be employed which allows the pyknometer to be held vertically in the constant temperature bath at the correct depth of immersion.

5.5 Balance — capable of weighing to the nearest 0·1 mg.

6 PREPARATION OF APPARATUS

6.1 Thoroughly clean the pyknometer with surfactant cleaning fluid, rinse well with distilled water, then with acetone and dry. Ensure that all traces of moisture are removed by drying with a current of filtered air passing slowly through the pyknometer. Wipe the outside of the pyknometer with a clean, lint-free cloth. 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. Normally the pyknometer may be cleaned between determinations by washing with a suitable light petroleum solvent, followed by vacuum drying.

NOTE — If sufficient cleaning fluids do not give adequate cleaning, chromic acid cleaning solution may be used.

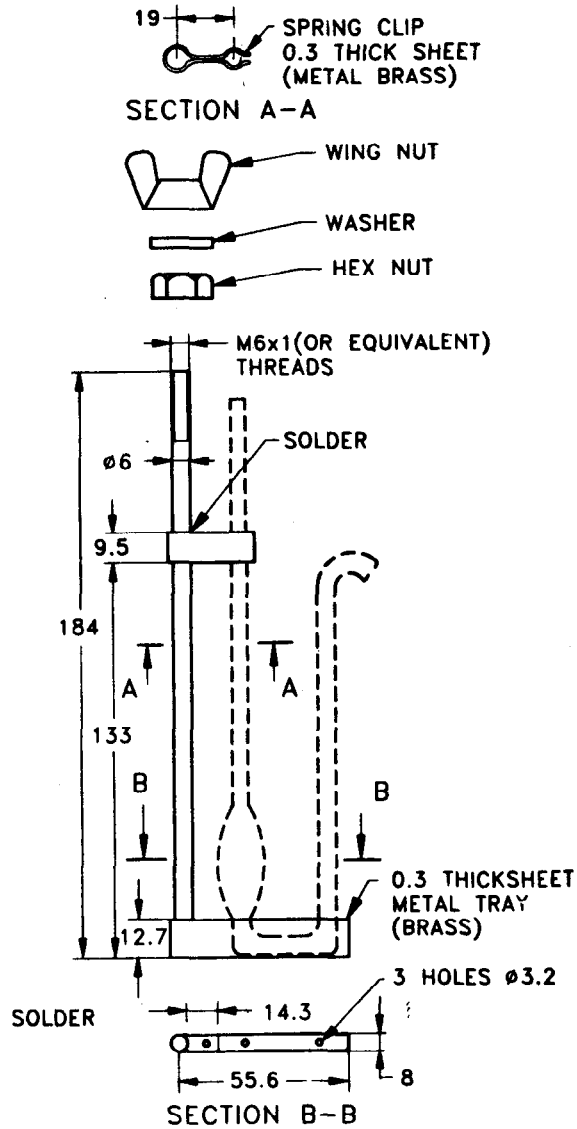
CAUTION : Chromic acid is potentially hazardous in contact with organic materials and is toxic and highly corrosive. If used wear approved face-shield and full-length protecting clothing including gloves.

7 CALIBRATION OF APPARATUS

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

NOTE — If the balance is not fitted with a static eliminator, static charges may be dissipated by breathing on the pyknometer. In such cases ensure that the pyknometer has regained constant mass before recording the weight.

7.2 Fill the pyknometer with sufficient freshly boiled, cooled, distilled water to obtain readings near the top of the graduated capillaries. Filling is readily achieved by placing the hooked tip in the liquid while keeping the pyknometer upright, thus allowing the liquid to be drawn over the bend in the capillary attraction.



All dimensions in millimetres.

FIG. 3 A SUITABLE DESIGN OF PYKNOMETER HOLDER

The pyknometer then fills by syphoning. Place the pyknometer in the constant temperature bath so that the whole of the liquid in the pyknometer is below the level of the bath liquid. Maintain the temperature of the bath at 15° ± 0·05°C or within ± 0·05°C of the required calibration temperature. Keep the pyknometer in the bath for 20 min then read the scale to the nearest small division at the liquid level in each arm.

7.3 Remove the pyknometer from the bath, allow the water on the exterior to drain off and wipe with a clean, dry, lint-free cloth. Allow to come to room temperature, dissipate any static charge, and weigh to the nearest 0·1 mg.

7.4 The difference between the weights of the filled and empty pyknometer gives the weight of water contained by the pyknometer at the test temperature, corresponding to the some of the two scale readings. By removing successive quantities of water repeat the determination so as to obtain a series of at least three pairs of readings, together with the corresponding weights, with the water level at different scale points on the graduated arms. One pair of readings should be at the upper end of the scale and another at the lower end. Plot the sums of the scale readings on the two arms against the corresponding weights. The points should lie on a straight line which gives the weight of water contained by the pyknometer for any combined scale reading. If the points show a scatter of more than two small scale divisions as marked on the pyknometer arms on either side of a straight line drawn through the array of points and subsequent test do not correct this, discard the pyknometer as imperfect.

7.5 If it is desired to determine density at a temperature other than 15°C calibrate the pyknometer at the desired temperature.

7.6 Re-calibrate pyknometer at intervals as dictated by experience.

NOTE — It is reeommended that pyknometer be calibrated at the test temperature at which densities are to be determined, sirce this greatly simplifies the calculation of densities and eliminates any uncertainty as to the coefficient of cubical expansion of the glass used in the manufacture of the pyknometer.

8 PROCEDURE

8.1 Weigh the clean, dry pyknometer to the nearest 0·1 mg, dissipating any static charge if necessary (*see* Note under **7.1**).

8.2 Fill the pyknometer with the sample at approximately the test temperature by the method described in **7.2**, so that the liquid levels are in the graduated portions of the capillaries. When the test temperature is lower than the laboratory temperature low scale readings should be aimed at in order to minimize any losses due to evaporation during weighing. Bring the pyknometer and contents to the test temperature t_1 by immersion for 20 minutes in the constant temperature both as described in **7.2** and obtain readings of the liquid level in the two graduated arms. In the case of the more viscous and/or opaque samples no readings must be taken until ample time for draining has been allowed after any disturbance of the pyknometer. The 20 minutes immersion time is normally sufficient, provided that the pyknometer has not been disturbed during this period.

NOTE — For mixtures of non-petroleum products it is essential to ensure that the test temperature is the same as the final reporting temperature unless an approximate value is acceptable and the volumetric compression of the mixture is known together with the volumetric correction coefficient of the components in the mixture.

8.3 Remove the pyknometer from the bath, allow the water on the exterior to drain off and wipe with a clean, dry, lint-free cloth. Allow to come to room temperature, dissipate any static charge, and weigh to the nearest 0·1 mg.

8.4 When carrying out the determination on highly volatile samples containing appreciable amounts of material boiling below 20°C, or for any sample where there is uncertainty concerning loss which might result from evaporation during the determination, cool the sample and pyknometer to a temperature of 0° to 5°C before filling. If the dew point is sufficiently high to cause condensation of moisture in the pyknometer during the cooling operation, attach a drying tube to the arm of the pyknometer in order to avoid this. With samples of this type it is essential to restrict the filling of the pyknometer to obtain a low scale reading thus minimizing losses due to evaporation. If the total length of unfilled capillary is over 10 cm the rate of diffusion is so low that even with highly volatile compounds such as *iso*-pentane vapour losses during the determination are negligibly low.

9 CALCULATION AND REPORTING

9.1 Symbols

The following symbols are used in the calculations:

t_a — any reference temperature, e.g. 15°C;

t_c — the temperature at which the pyknometer is calibrated by water filling (*see* **7.2** and **7.5**);

t_t — the temperature at which the pyknometer is filled with the liquid under test (*see* **9.2.2**);

m_0 — the apparent mass in air (weight in air) (g) of the empty pyknometer;

m_c — the apparent mass in air (g) of the pyknometer filled with water at the calibration temperature t_c ;

m_t — the apparent mass in air (g) of the pyknometer filled with the liquid under test at the temperature t_t ;

C — the correction for air buoyancy, in g/ml (*see* Table 1);

ρ_c — the density of water, in kg/m³, at the temperature of calibration t_t (*see* Table 2);

α_1 — the coefficient of cubical expansion of borosilicate glass (*see* **9.3.2**);

α_2 — the coefficient of cubical expansion of soda-lime glass (*see* **9.3.3**);

ρ_t — the density of the sample, in g/ml, at the test temperature t_t ;

ρ_s — the density of the sample in g/ml, at any reference temperature t_s ;

ρ_{15} — the density of the sample, in g/ml, at the reference temperature of 15°C;

ρ_{20} — the density of the sample, in g/ml, at the reference temperature of 20°C;

ρt — the apparent density, in g/ml, at the test temperature t_t as determined in soda-lime glass apparatus calibrated at the reference temperature t_s = 15° or 20°C, i.e. the observed density uncorrected for glass expansion — required for entering the API-ASTM — IP Petroleum Measurement Tables.

NOTE — The conventional unit g/ml has been retained for this calibration although the API-ASTM-IP Petroleum Measurement Tables for density are given in kg/cm³.

9.2 Reference, Calibration and Test Temperatures

9.2.1 The pyknometer may be calibrated at any convenient temperature and this may correspond with the reference or test temperatures. It is recomended that pyknometers be calibrated at the test temperature at which densities are to be determined since this greatly simplifies the calculation of density and only the calculations given in **9.4.1.1**, **9.4.2.1** and **9.4.2.2** then apply.

Table 2 Buoyancy Corrections*

(*Clause* 9.1)

$\frac{m_t - m_0}{m_c - m_0}$	Correction, g/ml	$\frac{m_t - m_0}{m_c - m_0}$	Correction g/ml
0·60	0·000 48	0·80	0·000 24
0·61	0·000 47	0·81	0·000 23
0·62	0·000 46	0·82	0·000 22
0·63	0·000 44	0·83	0·000 20
0·64	0·000 43	0·84	0·000 19
0·65	0·000 42	0·85	0·000 18
0·66	0·000 41	0·86	0·000 17
0·67	0·000 40	0·87	0·000 16
0·68	0·000 38	0·88	0·000 14
0·69	0·000 37	0·89	0·000 13
0·70	0·000 36	0·90	0·000 12
0·71	0·000 35	0·91	0·000 11
0·72	0·000 34	0·92	0·000 10
0·73	0·000 32	0·93	0·000 08
0·74	0·000 31	0·94	0·000 07
0·75	0·000 30	0·95	0·000 06
0·76	0·000 29	0·96	0·000 05
0·77	0·000 28	0·97	0·000 04
0·78	0·000 26	0·98	0·000 02
0·79	0·000 25	0·99	0·000 01

*Calculated for standard air of density 0·001 222 g/ml at 15·56°C and at a pressure of 1·013 bar.

These corrections are applicable for air density values between 0·001 1 and 0·001 3 g/ml.

9.3 Corrections for the Thermal Expansion of the Pyknometer

9.3.1 *General*

The calculation of density from measurements made at the temperature t_t which differs from the temperature t_t at which the pyknometer was calibrated, involves a correction for cubical expansion or contraction of the glass from which the pyknometer is made.

If the calculation is based on the density correction tables given in the API - ASTM - IP Petroleum Measurement Tables or IP 250, a similar correction may also be required (*see* **9.3.4**).

9.3.2 *Pyknometer Made of Borosilicate Glass*

9.3.2.1 The coefficient of cubical expansion of borosilicate glasses are known to depend on the glass to fall three main categories having coefficient of cubical expansion of 10×10^{-6}, 14×10^{-6} and 19×10^{-6} per 1°C respectively.

For determination of the highest accuracy when borosilicate pyknometers are used, therefore, either

a) ensure that $t_t = t_c'$ or
b) use a pyknometer for which the coefficient of cubical expansion is known. When the foregoing is not possible and a lower precision is acceptable, the use of 10×10^{-6} per 1°C is recommended.

9.3.3 *Pyknometers, Made of Soda-lime Glass*

9.3.3.1 For pyknometers made of soda-lime glass, the coefficient of cubical expansion may be assumed to be 25×10^{-6} per 1°C.

9.3.4 *API-ASTM-IP Petroleum Measurement Tables (ASTM D 1250 IP and IP 250)*

9.3.4.1 The following tables referred to in API-ASTM-IP Petroleum Measurement Tables are relevant only for determinations of petroleum and petroleum products. The tables must not be used for non-petroleum products for which it must always be ensured that $t_t = t_s$ ρ_s is required.

IP 200 Table 53 Correction of Observed Density to Density at 15°C.

IP 250 Table A Correction of Observed Density to Density at 20°C.

IP 200 Table 54 Correction of Observed Volume to Volume at a reference temperature of 15°C.

IP 250 Table B Correction of Observed Volume to Volume at a reference temperature of 20°C.

9.3.4.2 Enter Tables 53 A, 53B, 53D or Table A (according to the type of sample under test) with densities determined in glass apparatus calibrated at 15° or 20°C as appropriate.

The tables include a correction for the expansion of the glass apparatus used (assumed to be soda-lime glass) for which the conventional value of 23×10^{-6} per 1°C applies.

9.3.4.3 When entering Tables 53A, 53B, 53D or Table A with the results of determinations made with pyknometers constructed from borosilicate glass, a correction will be required for the difference between the coefficient of cubical expansion of the borosilicate glass and the conventional value for soda-lime glass incorporated in the tables.

9.3.4.4 When the pyknometer used is made of soda-lime glass no correction is necessary when using Tables 53A, 53B, 53D or Table A when $t_c = 15°$ or 20°C as appropriate to the table entered.

If t_c does not correspond to the reference temperature t_s for which the table used is constructed, an adjustment is required for the expansion of the pyknometer over the temperature range $t_c \rightarrow t_s$.

9.3.4.5 Alternatively, the corresponding volume correction Tables 54A, 54B, or 54D may be used. These tables do not include any correction for the expansion of glass but must be entered with a density at the appropriate reference temperature. Consequently the use of these tables requires a tedious repetitive calculation. This procedure is not therefore recommended and is not covered in **9.4** and **9.5**.

9.4 Calculation of Density

9.4.1 *Density at any Temperature* t_t

9.4.1.1 When $t_t = t_c$,

$$\rho t = \left[\frac{(m_t - m_o)}{(m_c - m_o)\ 1000} \right] + C$$

9.4.1.2 When $t_t = t_c$,

$$\rho t = \left[\frac{(m_t - m_o)\ \rho_c}{(m_c - m_o)\ 1000\ [\ 1 - \alpha^1\ (t_c - t_t)\ ]} \right] + C$$

where $\alpha^1 = \alpha_1$ or α_2 as appropriate to the pyknometer used.

9.4.2 *Density at Reference Temperature,* t_s

9.4.2.1 When $t_t = t_c = t_s$,

$$\rho_s = \rho t = \frac{(m_t - m_o)\ \rho_c}{(m_c - m_o)\ 1000} + C$$

9.4.2.2 When $t_t = t_c \neq t_s$ ($t_s = 15°$ or 20°C)

The density determined at the test temperature t_t as in **9.4.1.1** is the true density at t_t in soda-lime glass apparatus calibrated at t_s before entering Tables 53A, 53B, 53D or Table A as appropriate. This always applies irrespective of the glass from which the pyknometer is made, i.e. enter Tables 53A, 53B, 53D or Table A with ρt^1 and against t_t extract ρ_{15} or ρ_{20} respectively, interpolating as necessary, where

$$\rho t^1 = \left[\frac{(m_t - m_o)\ \rho_c}{(m_c - m_c)\ 1000} + C \right] [1 + \alpha_2\ (t_s - t_t)]$$

9.4.2.3 When $t_c = t_s \neq t_t$ (t_s = 15° or 20°C)

a) Pyknometer made of soda-lime glass — Tables 53A, 53B, 53D or Table A may be entered directly with ρt^1 and against t_t the corresponding ρ_{15} or ρ_{20} is extracted, interpolating as necessary, where

$$\rho t^1 = \frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C$$

b) Pyknometer made of borosilicate glass — Tables 53A, 53B, 53D or Table A must be entered with ρt^1 adjusted for the differential expansion of the pyknometer glass and against t_t the corresponding ρ_{15} or ρ_{20} is extracted

$$\rho t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right][1 + (\alpha_2 - \alpha_1)(t_s - t_t)]$$

Second-order terms, which are not significant, are ignored.

9.4.2.4 When $t_t \neq t_c \neq t_s$

The density determined at the test temperature t_t as in **9.4.1.2** is the true density at t_t. The result obtained must, therefore, be adjusted to give the apparent density at t_t in soda-lime glass apparatus calibrated at t_s before entering Tables 53A, 53B, 53D or Table A as appropriate. This always applies, irrespective of the glass from which the pyknometer is made, i.e. enter Tables 53A, 53B, 53D or Table A with ρt^1 and against t_t extract ρ_{15} or ρ_{20} as appropriate, interpolating as necessary. The expression for ρt^1 may be rearranged as follows, ignoring second-order terms which are not significant.

a) Pyknometers made of soda-lime glass —

$$\rho t^1 = \left[\frac{(mt_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right][1 + \alpha_s (t_s - t_c)]$$

b) Pyknometers made of borosilicate glass —

$$\rho t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right][1 + \alpha_1 t_s + \alpha_2 t_t - (\alpha_1 + \alpha_2)\,t_c]$$

9.4.2.5 When $t_s = t_t \neq t_c$

Use the expression in **9.4.1.2**.

9.4.3 *Rounding-Off of Calculations and Reporting*

Carry out all calculations to five figures and report the final result to the nearest 0·000 1 as density giving the units of mass and volume and the temperature.

10 PRECISION

10.1 The following criteria should be used for judging the acceptability of results (95 percent confidence).

10.1.1 *Repeatability*

Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Range, g/ml	*Repeatability*
0·777 0 to 0·892 0	0·000 7

10.1.2 *Reproducibility*

The results obtained by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

Range, g/ml	*Reproducibility*
0·777 0 to 0·892 0	0·001 0

The above values were obtained when using 5 ml pyknometers. The use of larger (e.g. 10 ml) pyknometers should give equal or better accuracy.

The method is not restricted to samples having densities within the range 0·777-0·892 g/ml but precision figures are not available for values outside this range.

11 METHOD C (CAPILLARY-STOPPERED PYKNOMETER METHOD)

11.1 General

11.1.1 This method covers the determination of the density or relative density of crude petroleum and of petroleum products handled as liquids or solids. The method may also be used for coal tar products, including road tars, creosote and tar pitches, or for mixtures of these with petroleum products.

11.1.2 The method is not suitable for the determination of the density or relative density of highly volatile liquids having Reid vapour pressure greater than 0·5 bar (5×10^4 Pa) or having initial boiling points below 40°C.

11.2 Summary of Method

11.2.1 The weights of equal volumes of the sample and of water are compared. Equal volumes are ensured by placing the filled pyknometer in a bath at the test temperature until equilibrium is reached.

12 APPARATUS

12.1 Pyknometer — One of the four types shown in Fig. 4 and 5 (*see* **15.1** and **16.1**), constructed of borosilicate glass or soda-lime glass and annealed after manufacture.

The 'warden' form [Fig. 4 (b)] is determined for all except viscous or solid products and should always be used for volatile products. The ground-glass cap, or 'warden', almost eliminates expansion and evaporation losses and this form of pyknometer may be used when the test temperature is lower than that of the laboratory.

The conical (Fig. 5) is recommended for waxy oils and semi-solids.

The form of pyknometer shown in Fig. 4 (a) is suitable for non-volatile liquids except those of high viscosity.

The wide-mouth (Hubbard) form of pyknometer [Fig. 4 (c)] is used for very viscous liquids and solids.

As the forms of pyknometer shown in Fig. 4 (a) and (c) 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.

12.2 Constant-temperature Bath — a 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.

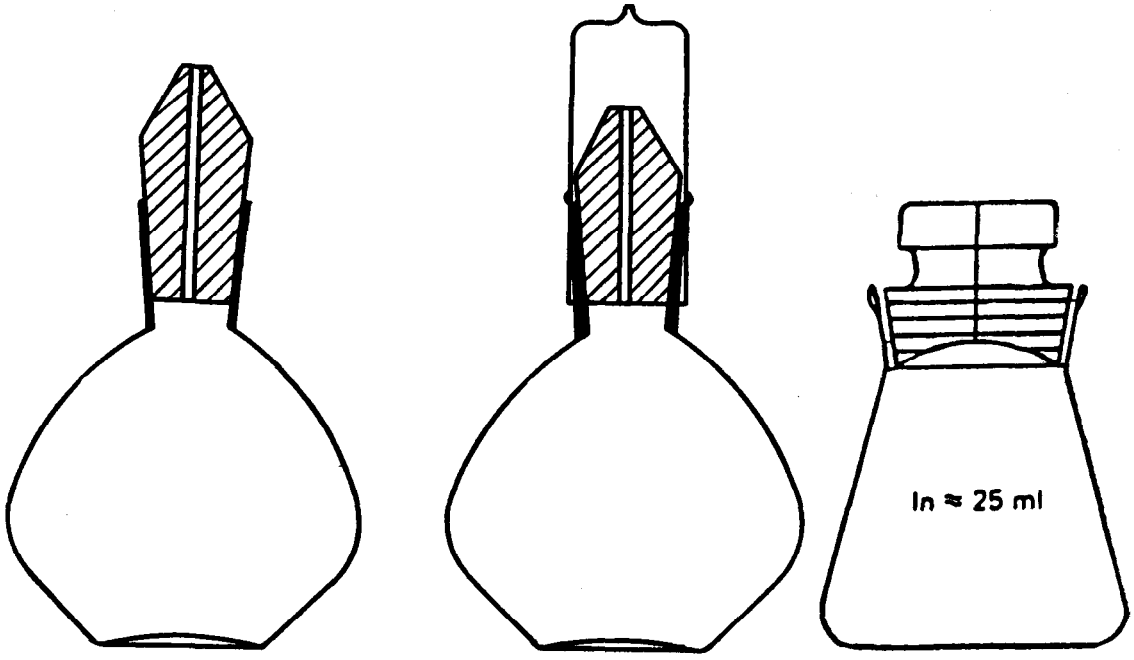
12.3 Bath Thermometer — *See* **3.4.1.1**. Other total immersion thermometers of suitable range and equal or greater accuracy may also be used.

12.4 Pyknometer Holder (Optional) — to hold pyknometer vertically and at the correct depth in the constant-temperature bath, a metal holder is desirable. It may be constructed of any suitable metal which will not corrode in the water bath.

12.5 Balance — capable of weighing to the nearest 0·1 mg.

13 PREPARATION OF APPARATUS

13.1 Thoroughly clean the pyknometer and stopper with a surfactant cleaning fluid, rinse well with distilled water, then with acetone and dry. Ensure that all traces of moisture are removed by drying with a current of filtered air passing slowly through the pyknometer and stopper capillary. Wipe the outside of the pyknometer and stopper with a clean, lint-free cloth.



4A CAPILLARY-STOPPER 4B WARDEN 4C WIDE MOUTH HUBBARD

FIG. 4 PYKNOMETER FORMS

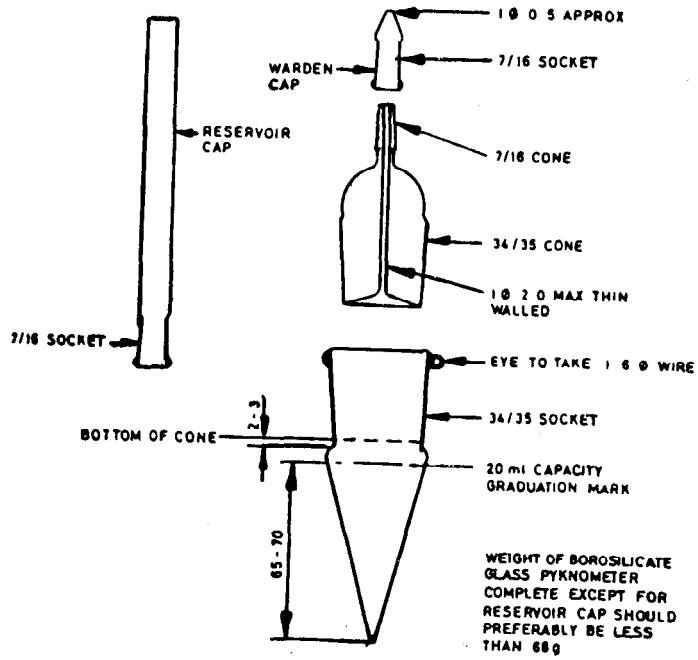


FIG. 5 CONICAL CAPILLARY-STOPPER PYKNOMETER

NOTE — If sufficient cleaning fluids do not give adequate cleaning, chromic acid cleaning solution may be used.

CAUTION — Chromic acid is potentially hazardous in contact with organic materials and is toxic and highly corrosive. If used, wear approved full face-shield and full-length protective clothing including gloves.

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 solvent, followed by vacuum drying.

14 CALIBRATION OF PYKNOMETER

14.1 After drying allow the pyknometer to reach room temperature. Dissipate any static charge which may have formed on it and then weigh, complete with warden if of form 4 (b) or form 5, to the nearest 0·1 mg.

NOTE — If the balance is not fitted with a static eliminator, static charges may be dissipated by breathing on the pyknometer. In such cases ensure that the pyknometer has regained constant mass before recording the weight.

14.2 Fill the pyknometer with freshly-boiled distilled water, cooled to slightly below 15°C, and firmly insert the stopper, taking care to avoid the inclusion of any air bubbles. Immerse the pyknometer to the neck in the constant-temperature bath. When using the conical form of pyknometer, place the reservior cap in the constant-temperature bath. Maintain the bath within $\pm$ 0·05°C ($\pm$ 0·1°F) of the calibration temperature for not less than one hour.

14.3 With the pyknometer and its contents at this temperature wipe the top of the stopper so that it is dry and the meniscus of the water in the capillary is flush with the top of the stopper. Care is necessary during this operation, since capillary action of the cloth can draw liquid out of the stopper. Place the warden firmly on the stopper (if the pyknometer is of this type). When using the conical form, raise the pyknometer in the water bath until the smaller cone is clear of the water. Remove the reservoir cap and wipe the cone so that it is dry and the meniscus of the water in the capillary is flush with the top of the stopper, then place the warden firmly on the stopper.

14.4 Remove the pyknometer from the bath. If not of the 'warden' form, cool the pyknometer and its contents to a temperature slightly below the temperature of the bath.

14.5 Dry the exterior surface of the pyknometer by wiping with a clean, lint-free cloth, paying particular attention to the joint between the stopper and pyknometer, dissipate any static charge and weigh to the nearest 0·1 mg.

14.6 The difference between the weights of the filled and empty pyknometer gives the mass of water contained by the pyknometer at the calibration temperature.

14.7 If it is required to determine density at a temperature other than 15°C or to determine relative density referred to water at a temperature other than (60°F) 15·6°C, calibrate the pyknometer at the required temperature. It is recommended that pyknometer be calibrated at the test temperature at which the densities are to be determined, since this greatly simplifies the calculation of densities and relative densities and eliminates any uncertainty as to the type of glass used in the construction of the pyknometer.

14.8 Re-calibrate pyknometers at intervals as dictated by experience.

NOTE — It is recommended that new pyknometers should be re-calibrated after one year and thereafter at intervals dependent upon the magnitude of any changes found. Soda-lime glass pyknometers may require calibration more frequently than borosilicate glass pyknometers.

15 PROCEDURE FOR LIQUIDS

15.1 Choose an appropriate form and size of pyknometer for the sample to be tested (*see* **12.1**). The 75 ml and 50 ml sizes are normally the most suitable.

15.2 Weigh the clean, dry pyknometer with warden [if of form 4 (b) or 5] and if necessary, disperse any static charge (*see* Note given under **14.1**). Pyknometers of 25 ml or greater capacity should be weighed to the nearest 0·5 mg, and those of smaller capacity to the nearest 0·1 mg.

15.3 Fill the pyknometer with the sample, if necessary warming both sample and pyknometer to assist filling and separation of air bubbles. Bring the pyknometer and its contents to the test temperature (t_t) by immersing pyknometer up to its neck in the constant-temperature bath. Keep the pyknometer in the bath for 20 minutes in order to stabilize the temperature and to permit air bubbles to rise to the surface. If after this time the liquid level is still changing, keep the pyknometer in the bath until the liquid level becomes stable.

NOTE — For mixtures containing non-petroleum products it is essential to ensure that the test temperature is the same as the final reporting temperature unless an appropriate value is acceptable and the volumetric composition of the mixture is known, together with the volumetric correction coefficients of the components in the mixture.

When the temperature is constant, firmly insert the dry capillary stopper, which has also been brought to the test temperature, taking care to avoid trapping air bubbles below the stopper.

NOTE — It is essential to ensure that no air bubbles are left trapped in the liquid and adequate time must be allowed for air bubbles to rise to the surface before inserting the stopper.

Wipe excess liquid from the top of the stopper so that the meniscus of the liquid in the capillary is flush with the top of the stopper. Place the 'warden' over the stopper (if the pyknometer is of this type).

15.4 Remove the pyknometer from the bath and, if not of the 'warden' type cool to a temperature slightly below t_t. Cool the pyknometer and contents to room temperature if the test temperature is high.

15.5 Remove all traces of sample and water from the exterior surface of the pyknometer by wiping with a clean, lint-free cloth, disperse any static charge and weigh to the precision given in **15.2**.

16 PROCEDURE FOR SOLID OR SEMISOLID SAMPLES

16.1 Weigh the clean, dry pyknometer which should be of the wide-mouth type [Fig 4 (c)] or the conical type (Fig. 5) complete with warden to the nearest 0·5 mg.

16.2 Introduce a suitable amount of the sample in the form of small pieces, which should be as regular in order to reduce the possibility of trapping air bubbles. Alternatively, pour the molten sample in to the warmed pyknometer, taking care to avoid the inclusion of air bubbles. Fill the conical pyknometer up to the 20 ml graduation mark. Allow the test portion and pyknometer to cool slowly to ambient temperature. Check that no voids have formed between the test portion sufficiently to remove the voids and again allow to cool.

16.3 Bring the pyknometer and its contents to room temperature and weigh to the nearest 0·5 mg.

16.4 In cases of sample having very high wax contents it may be necessary to weigh the cooled test portion and pyknometer before overcoming the problem of voids. The weighed test portion and pyknometer are then reheated until the voids disappear and allowed to cool. As soon as the melted test portion has set, the surface should be covered with freshly boiled and cooled distilled water to prevent the ingress of air into any voids which may form later at the bottom of the pyknometer.

16.5 Fill the pyknometer with freshly-boiled, cooled distilled water, removing all air bubbles. A fine wire may be used to facilitate the removal of bubbles. Bring the pyknometer and its contents to the test temperature, which must be the same as the calibration temperature of the pyknometer, by immersing the pyknometer up to its neck in the constant-temperature bath. When using the conical form of pyknometer, place the reservoir cap on the capillary stopper and firmly insert it, taking care to avoid trapping air bubbles below the stopper, and immerse to half-way up the reservoir cap in the constant-temperature bath.

Keep the pyknometer in the bath for 20 minutes in order to stabilize the temperature and to permit bubbles to rise to the surface. If after this time the liquid level is still changing, keep the pyknometer in the bath until the liquid level becomes stable.

When the temperature is constant, firmly insert the capillary stopper, which has also been brought to the test temperature, taking care to avoid trapping air bubbles below the stopper. When using the conical pyknometer it is only necessary to raise the pyknometer until the smaller cone is clear of the water and remove the reservoir cap. Wipe excess water from the top of the stopper so that the meniscus of the water in the capillary is flush with the top of the stopper. Place the warden firmly on the stopper if the pyknometer is of the conical form.

16.6 Remove the pyknometer from the bath and if the pyknometer is of the wide-mouth form, cool to a temperature slightly below the tests/calibration temperature. Cool the pyknometer and contents to room temperature if the test temperature is above ambient.

16.7 Dry the exterior surface of the pyknometer by wiping with a clean, lint-free cloth, disperse any static charge and weigh to the nearest 0·5 mg.

17 CALCULATION AND REPORTING

17.1 Symbols — The following symbols are used in the calculations :

t_s — any reference temperature, e. g. 15°C;

t_c — the temperature at which the pyknometer is calibrated by water filling (*see* **16.5** and **17.2**);

t_t — the temperature at which the pyknometer is filled with liquid under test (*see* **17.2.2**);

m_o — the apparent mass in air (weight in air) (g) of the empty pyknometer;

m_c — the apparent mass in air (g) of the pyknometer filled with water at the calibration temperature t_c;

m_t — the apparent mass in air (g) of the pyknometer filled with the liquid under test at the temperature t_t;

m_1 — the apparent mass in air (g) of the pyknometer plus solid or semi-solid (*see* **16.3**);

m_2 — the apparent mass in air (g) of the pyknometer plus solid or semi-solid plus water (*see* **16.7**);

C — the correction of air buoyancy, in g/ml (*see* Table 4);

ρ_c — the density of water, in kg/m^3, at the temperature of calibration;

α_1 — the coefficient of cubical expansion of borosilicate glass (*see* **17.3.2**);

α_2 — the coefficient of cubical expansion of soda-lime glass (*see* **17.3.3**);

ρ_t — the density of the sample, in g/ml, at the test temperature t_t;

ρ_s — the density of sample in g/ml, at any reference temperature t_s;

ρ_{15} — the density of the sample, in g/ml, at the reference temperature of 15°C;

ρ_{20} — the density of the sample, in g/ml, at the reference temperature of 20°C;

ρ_t^1 — the apparent density, in g/ml, at the test temperature t_t as determined in soda-lime glass apparatus calibrated at the reference temperature t_s = 15° or 20°C, i. e., the observed density uncorrected for glass expansion — required for entering the API-ASTM-IP Petroleum Measurement Tables.

d_t — the relevant density at the test temperature t_t;

d_s — the relevant density at reference temperature t_s;

$d_{15\cdot6}$ — the relevant density at the reference temperature of (60°F) 15·6°C;

d_t[1] — the apparent relative density at the test temperature t_t as determined in glass apparatus calibrated at the reference temperature t_s = (60°F) 15·6°C, i. e. the observed relative density uncorrected for glass expansion required for entering the API-ASTM-IP Petroleum Measurement Tables.

17.2 Reference, Calibration and Test Temperatures

17.2.1 The pyknometer may be calibrated at any convenient temperature and this may correspond with the reference or test temperatures. The calculation of density is greatly simplified when the pyknometer is calibrated at the test temperature since only the caculations given in **17.4.1.1**, **17.4.2.1** and **17.4.2.2** then apply.

17.2.2 For qualitative purposes, the test temperature is usually chosen to correspond with the required reference temperature, but for quantitative purposes involving the calculation of the mass or of the apparent mass in air of a given quantity of oil, the density or relative density should be determined within 3°C of the temperature at which the oil is measured by the selected dynamic or static method. However, in the case of very volatile products having a Reid vapour pressure above 0·5 bar, the test should be carried out at a temperature of 15°C or below to minimize the loss of light fractions.

17.3 Correction for the Thermal Expansion of the Pyknometer

17.3.1 *General*

The calculation of density or of relative density from measurements made at a temperature t_t which differs from the temperature t_c at which the pyknometer was calibrated, involves a correction for cubical expansion or contraction of the glass from which the pyknometer is made.

If the calculation is based on the density or relative density correction tables given in the API-ASTM-IP Petroleum Measurement Tables, a similar correction may also be required (*see* **17.3.4**).

17.3.2 *Pyknometers Made of Borosilicate Glass*

17.3.2.1 The coefficient of cubical expansion of borosilicate glass are known to depend on the source of the glass and to fall into three main categories having coefficients of cubical expansion of 10×10^{-6}, 14×10^{-6} and 19×10^{-6} per 1°C respectively.

For determination of the highest accuracy when borosilicate pyknometers are used, therefore, either

a) ensure that $t_t = t_c$ or

b) use a pyknometer for which the coefficient of cubical expansion is known.

When the foregoing is not possible and a lower precision is acceptable, the use of 10×10^{-6} per 1°C is recommended.

17.3.3 *Pyknometers Made of Soda-Lime Glass*

17.3.3.1 For pyknometers made of soda-lime glass, the coefficient of cubical expansion may be assumed to be 25×10^{-6} per 1°C.

17.3.4 *The API-ASTM-IP Petroleum Measurement Tables (API 2540 : ASTM D 250/IP 200 and IP 250*

17.3.4.1 The following tables referred to in the API-ASTM-IP Petroleum Measurement Tables are relevant only for determinations non-petroleum and petroleum products. The tables must not be used for non-petroleum products for which it must always be ensured that $t_t = t_s$ when ρ_s or d_s is required:

IP 200 Tables 53A, 53B and 53D	Correction of Observed Density to Density at 15°C
IP 250 Table A	Correction of Observed Density to Density at 20°C
IP 200 Tables 23A, 23B and 23D	Correction of observed Relative Density (60°/60°F) 15·6°/15·6°C to Relative Density (60°/60°F) 15·6°/15·6°C
IP 200 Tables 54A, 54B and 54D	Correction of Observed Volume to Volume at a reference temperature of 15°C
IP 250 Table B	Correction of Observed Volume to Volume at a reference temperature of 20°C
IP 200 Tables 24A, 24B and 24D	Correction of Observed Volume to Volume at a reference temperature

17.3.4.2 Tables 53A, 53B, A, 53D, 23A, 23B and 23D are entered with densities or relative densities determined in glass apparatus calibrated at a temperature t_c in °C (Tables 53A, 53B 53D or A) or °F (Tables 23A, 23B or 23D).

The tables include a correction for the expansion of the glass apparatus used (assumed to be soda-lime glass) for which the conventional value of (25 ± 2) × 10^{-6} per 1°C applies.

17.3.4.3 When entering Tables 53A, 53B, 53D, A, 23A, 23B, 23D with the results of determinations made with pyknometers constructed from borosilicate glass, a correction will be required for the difference between the coefficient of cubical expansion of the borosilicate glass and the conventional value for soda-lime glass incorporated in the tables.

17.3.4.4 When the pyknometer used is made of soda-lime glass no correction is necessary when using Tables 53A, 53B, 53D, A, 23A or 23B, when t_c=15°C, 20°C or (60°F) 15·6°C as appropriate to the table entered.

If t_c does not correspond to the reference temperature t_s for which the table used is constructed, an adjustment is required for the expansion of the pyknometer over the temperature range $t_c \rightarrow t_s$.

17.3.4.5 Alternatively, the corresponding volume correction Tables 54A, 54B, 54D, 24A, 24B or 24D may be used. These tables do not include any correction for the expansion of glass, but must be entered with a density at the appropriate reference temperature. Consequently the use of these tables requires a tedious repetitive calculation. This procedure is not, therefore, recommended and is not covered in **17.4** and **17.5**.

17.4 Calculation of Density

17.4.1 *Density at any Temperature* t_c

17.4.1.1 When $t_t = t_c$

a) for liquids

$$\rho_t = \frac{(m_1 - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C$$

b) for solids or semi-solids

$$\rho_t = \frac{(m_1 - m_o)\,\rho_c}{(m_c - m_o - m_2 + m_1)\,1\,000} + C$$

17.4.1.2 When $t_t \neq t_c$

$$\rho_t = \frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000\,[\,1 - \alpha^1\,(t_c - t_t)\,]} + C$$

where $\alpha^1 = \alpha_1$ or α_2 as appropriate to the pyknometer used.

17.4.2 *Density at Reference Temperature* t_s

17.4.2.1 When $t_t = t_c = t_s$

a) for liquids

$$\rho_s = \rho_t = \frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C$$

b) for solids or semi-solids

$$\rho_s = \rho_t = \frac{(m_1 - m_o)\,\rho_c}{(m_c - m_o - m_2 + m_1)\,1\,000} + C$$

17.4.2.2 When $t_t = t_c \neq t_s$ = 15° or 20°C

The density determined at the test temperature t_t as in **17.4.1.1**, is the true density at t_t. The result obtained must, therefore, be adjusted to give the apparent density at t_t in soda-lime glass apparatus calibrated at t_s, before entering Table 53 or Table A as appropriate. This always applies irrespective of the glass from which the pyknometer is made, i. e., enter Table 53 or Table A with ρ_t^1 and against t_t extract ρ_{15} or ρ_{20} respectively, interpolating as necessary, where

a) for liquids

$$\rho_t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right][1 + \alpha_2\,(t_s - t_t)]$$

b) for solids or semi-solids

$$\rho_t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o - m_2 + m_1)\,1\,000} + C\right]$$
$$[\,1 + \alpha_2\,(t_s - t_t)\,]$$

17.4.2.3 When $t_c = t_s \neq t_t$ (t_s = 15° or 20°C)

a) Pyknometer made of soda-lime glass — Tables 53A, 53B, 53D or A may be entered directly with ρ_t^1 and against t_t the corresponding ρ_{15} or ρ_{20} is extracted, interpolating as necessary, where

$$\rho_t^1 = \frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1000} + C$$

b) Pyknometers made of borosilicate glass — Tables 53A, 53B, 53D or Table A must be entered with ρ_t^1 adjusted for the differential expansion of the pyknometer glass, and against t_t the corresponding ρ_{15} or ρ_{20} is extracted, interpolating as necessary, where

$$\rho_t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right]$$
$$[\,1 + (\alpha_2 - \alpha_1)\,(t_s - t_t)\,]$$

Second-order terms, which are not again significant, are ignored.

17.4.2.4 When $t_t \neq t_c \neq t_s$

The density determined at the test temperature t_t as in **17.4.1.2** is the true density at t_t. The result obtained must, therefore, be adjusted to give the apparent density at t_t in soda-lime glass apparatus calibrated at t_s before entering Tables 53A, 53B, 53D, or Table A as appropriate. This always applies, irrespective of the glass from which the pyknometer is made, i. e. enter Tables 53A, 53B, 53D, or Table A with ρ_t^1 and against t_t extract ρ_{15} or ρ_{20} respectively, interpolating as appropriate. The expression for ρ_t^1 may be rearranged as follows, ignoring second-order terms which are not significant:

a) Pyknometers made of soda-lime glass

$$\rho_t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right] [1 + \alpha_s\,(t_s - t_c)]$$

b) Pynometers made of borosilicate glass

$$\rho_t^1 = \left[\frac{(m_t - m_o)\,\rho_c}{(m_c - m_o)\,1\,000} + C\right] [1 + \alpha_1 t_s + \alpha_2 t_t - (\alpha_1 + \alpha_2)\,t_c]$$

17.4.2.5 When $t_s = t_t \neq t_c$

Use the expression in **17.4.1.2.**

17.4.3 *Rounding-off of Calculations and Reporting*

Carry out all calculations to five decimal places and report the final result to the nearest 0·000 1 as Density, giving the units of mass and volume and the temperature.

17.5 Calculation of Relative Density

17.5.1 In accordance with the definition, the appropriate values for d_t, d_t^1 and d_s can be derived by dividing the corresponding values for density in **17.4.1** and **17.4.2** by the density of water in the same units at the required reference temperature (*see* Note under **17.5.3**).

17.5.2 When the correction of relative density t/(60°F) 15·6°C of petroleum and petroleum products is required to the reference temperature of (60°F) 15.6°C (*see* **17.4.2.2, 17.4.2.3** and **17.4.2.4**), use Tables 23A, 23B or 23D or IP 200 t_s, t_t and t_c will be in °F and the coefficient of cubical expansion α_1 and α_2 should be stated per 1°F for α_2 the value of 14×10^{-6} per 1°F may be taken.

17.5.3 Carry out all calculations to five decimal places and report the final results to the nearest 0·000 1 as Relative Density giving test and reference temperatures.

NOTE — Since the densities in Table 3 are given in kg/m³, it is essential that these values be divided by 1 000 to convert them to g/ml (kg/l).

18 PRECISION

18.1 With samples which are neither very volatile nor very viscous, and using a pyknometer of at least 25 ml capacity, results of duplicate test should not differ by more than the following amounts:

	Repeatability	*Reproducibility*
Density	0·000 6 g/ml	0·000 6 g/ml
Relative density	0·000 6 g/ml	0·000 6 g/ml

These values are based on estimates of normal good practice and are not derived by statistical analysis.

For volatile or very viscous samples and for solids no specific figures for precision can be given.

19 METHOD D (DENSITY OR RELATIVE DENSITY OF SOLIDS — DISPLACEMENT METHOD

19.1 Outline of the Method

When a solid is weighed first in air and then in water, the difference between the two weighings is equal to the weight of water displaced. The rel. d t/t of the solid may, therefore, be calulated, where t is the temperature of the water. If the solid has a relative density greater than unity a piece of it may be weighed directly in the water. If the relative density is less than unity the solid must be made to sink in the water in some suitable way, for example, by securing it to a metal crucible.

The method may be used for the determination of the relative density of bitumen and similar products which are sufficiently solid to be handled in pieces. The pyknometer method is, however, more precise.

20 APPARATUS

20.1 Thermometer — *See* **3.4.1.1.**

20.2 Balance Straddle — a pan straddle of convenient size to support a beaker and permit determination of the weight of the specimen in water.

20.3 Waxed Thread — a length of fine, waxed silk thread.

20.4 Nickel Crucible — in a modification of the method a thin nickel crucible of 5-25 ml capacity may be used. It should be provided with a three-wire suspension which permits the complete immersion of wire and crucible when determining the weight in water.

21 PROCEDURE FOR SOLIDS OF RELATIVE DENSITY GREATER THAN UNITY

21.1 Suspend a piece of the material, as smooth as possible, free from cracks and dust, and weighing at least 5 g, from the hook on one side of a balance, using a suitable length of waxed silk thread so that the bottom of the specimen is about 25·4 m (1 in) above the straddle. Tare the thread previously by a length of thread on the other pan. Weigh the sample to the nearest mg. Place the sample in a beaker of freshly boiled distilled water maintained at the desired temperature, e. g. 15°C, and allow to remain for sufficient time to ensure that the sample has attained that temperature. Then place the beaker containing distilled water on the pan straddle and suspend the sample from the hook on one side of the balance. Remove with a fine wire any adhering air bubbles and determine the weight of the sample in water.

NOTE — The main source of error is air bubbles either occluded in the sample or adhering to its outer surface during weighing in water. Errors caused by weighing or by temperature variation are usually small in comparison.

22 CALCULATION AND REPORTING

22.1 If a is the weight of the specimen in air, and b is the weight of the specimen in water at t, then

$$\text{relative } d_t/_t \text{ in air} = \frac{a}{a-b}$$

22.2 Report the value so obtained, to the nearest 0·001, as Relative Density $d_t/_t$, Method D. The buoyancy correction in this case is negligible.

22.3 To obtain density multiply the rel d t/t by the density of water at t, obtained from Table 3.

23 PROCEDURE FOR SOLIDS OF RELATIVE DENSITY LESS THAN UNITY

In using the crucible method:

23.1 Suspend the crucible from the hook on one side of the balance by means of a tared length of waxed thread, and determine its weight to the nearest mg.

23.2 Suspend the crucible in a beaker containing freshly boiled distilled water at the desired temperature so that the crucible and wire suspension are completely immersed; remove any air bubbles adhering to the crucible and determine the weight in water.

23.3 Dry the crucible and pour in some of the molten sample, taking care to avoid inclusion of air bubbles, after which allow the crucible and contents to cool, preferably under vacuum to room temperature. Determine the weight in air.

23.4 Place the crucible and content in a beaker of freshly boiled distilled water maintained at the desired temperature and leave until the crucible and contents attain that temperature.

23.5 Place the beaker containing distilled water on the pan straddle and suspend the crucible from the hook on one side of the balance. Remove with a fine wire any air bubbles adhering to the crucible or sample, and determine the weight in water of the crucible and contents.

24 CALCULATION AND REPORTING

24.1 Let W_c = weight of crucible in air;

W_{cw} = weight of crucible in water at t;

W_f = weight of crucible + sample in air;

W_{fw} = weight of crucible + sample in water at t;

then $W_f - W_c$ = weight of sample in air;

and $(W_f - W_{cw}) - (W_{fw} - W_{cw})$ = loss in weight of sample when weighed in water at t.

therefore,

relative density dt°C/t°C in air

$$= \frac{W_f - W_c}{(W_f - W_c) - (W_{fw} - W_{cw})}$$

24.2 Report the result as Relative Density, Method D. The buoyancy correction in this case is negligible.

24.3 To obtain density multiply the relative density $d_t/_t$ by the density of water at t, obtained from Table 3.

25 PRECISION

Results of duplicate tests should not differ by more than the following amounts:

Size of Sample	*Repeatability*	*Reproducibility*
5 g or larger	0·004	0·008
Less than 5 g	No specific permissible variations can be given	

Table 3 Density of Water in kg/m³ Against °C of the International Practical Scale of Temperature (1968)

(*Clause* 9.1)

t_{68}°C	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9	$\Delta p/\Delta t$ kg/m³ 0·1°C	Δp Air-Saturated Minus Air-Free
0	999·839 6	999·846 3	999·852 8	999·859 1	999·865 3	999·871 3	999·877 1	999.882 7	999·888 2	999·893 4	0·005 9	—0·002 6
1	999·898 5	999·903 5	999·908 2	999·912 8	999·917 2	999·921 4	999·925 4	999·929 3	999·933 0	999·936 5	0·004 1	—0·002 7
2	999·939 9	999·943 1	999·946 1	999·948 9	999·951 6	999·954 1	999·956 5	999·958 7	999·960 7	999·962 5	0·002 4	—0·002 8
3	999·964 2	999·965 7	999·967 0	999·968 2	999·969 2	999·970 1	999·970 8	999·971 3	999·971 7	999·971 9	0·000 8	—0·003 0
4	999·972 0	999·971 8	999·971 6	999·971 1	999·970 5	999·969 8	999·968 9	999·967 8	999·966 6	999·965 2	—0·000 8	—0·003 1
5	999·963 7	999·962 0	999·960 2	999·958 2	999·956 0	999·953 7	999·951 3	999·948 7	999·945 9	999·943 0	−0.002 4	—0·003 3
6	999·939 9	999·936 7	999·933 4	999·929 9	999·926 2	999·922 4	999·918 4	999·914 3	999·910 1	999·905 7	—0·003 9	—0·003 4
7	999·901 1	999·896 4	999·891 6	999·886 6	999·881 5	999·876 2	999·870 8	999·865 2	999·859 5	999·853 7	—0·005 3	—0·003 5
8	999·847 7	999·841 6	999·835 3	999·828 9	999·822 3	999·815 7	999·808 8	999·801 9	999·794 7	999·787 5	—0 006 8	—0·003 5
9	999·780 1	999·772 6	999·764 9	999·757 1	999·749 2	999·741 1	999·732 9	999·724 6	999·716 1	999·707 5	—0·008 1	—0·003 4
10	999·698 7	999·689 8	999·680 8	999·671 7	999·662 4	999·653 0	999·643 4	999·633 7	999·623 9	999·614 0	—0·009 5	—0·003 3
11	999·603 9	999·593 7	999·583 4	999·572 9	999·562 3	999·551 6	999·540 8	999·529 8	999·518 7	999·507 4	—0·010 8	—0·003 1
12	999·496 1	999·484 6	999·473 0	999·461 2	999·449 4	999·437 4	999·425 3	999·413 0	999·400 7	999·388 2	—0·012 1	—0·002 9
13	999·375 6	999·362 8	999·350 0	999·337 0	999·323 9	999·310 6	999·297 3	999·283 8	999·270 2	999·256 5	—0·013 3	—0·002 6
14	999·242 7	999·228 7	999·214 6	999·200 4	999·186 1	999·171 7	999·157 1	999·142 4	999·127 6	999·112 7	—0·014 5	—0·002 3
15	999·097 7	999·082 6	999·067 3	999·051 9	990·036 4	999·020 8	999·005 1	998·989 2	998·973 3	998·957 2	—0·015 7	—0·002 0
16	998·941 0	998·924 7	998·908 3	998·891 7	998·875 1	998·858 3	998·841 4	998·824 4	998·807 3	998·790 1	—0·016 8	—0·001 7
17	998·772 8	998·755 3	998·737 8	998·720 1	998·702 3	998·684 5	998·666 5	998·648 3	998·630 1	998·611 8	—0·017 9	—0·001 4
18	998·593 4	998·574 8	998·556 2	998·537 4	998·518 5	998·499 5	998·480 4	998·461 2	998·441 9	998·422 5	—0·019 0	—0·001 1
19	998·403 0	998·383 3	998·363 6	998·343 8	998·323 3	998·303 7	998·283 6	998·263 3	998·242 9	998·222 4	—0·020 1	—0·000 9
20	998·201 9	998·181 2	998·160 4	998·139 5	998·118 5	998·097 3	998·076 1	998·054 8	998·033 4	998·011 9	—0·021 2	—0·000 6
21	997·990 2	997·968 5	997·946 7	997·924 7	997·902 7	997·880 5	997·858 3	997·836 0	997·813 5	997·791 0	—0·022 2	—0·000 4
22	997·768 3	997·745 6	997·722 7	997·699 8	997·676 7	997·653 6	997·630 3	997·607 0	997·583 5	997·560 0	—0·023 2	—0·000 2
23	997·536 3	997·512 6	997·488 7	997·464 8	997·440 8	997·416 6	997·392 4	997·368 0	997·343 6	997·319 1	—0·024 2	—0·000 1
24	997·294 4	997·269 7	997·244 9	997.220 0	997·195 0	997·169 9	997·144 6	997·119 3	997·093 9	997·068 5	—0·025 2	—0·000 0
25	997·042 9	997·017 2	996·991 4	996·965 5	996·939 6	996·913 5	996·887 3	996·861 1	996·834 7	996·808 3	—0·026 1	—0·000 0
26	996·781 8	996·755 1	996·728 4	996·701 6	996·674 7	996·647 7	996·620 6	996·593 4	996·566 1	996·538 8	—0·027 0	—0·000 0
27	996·511 3	996·483 7	996·456 1	996·428 4	996·400 5	996·372 6	996·344 6	996·316 5	996·288 3	996·260 0	—0·028 0	—0·000 0
28	996·231 6	996·203 2	996·174 6	996·146 0	996·117 2	996 088 4	996·059 5	996 030 5	996·001 4	995·972 2	—0·028 9	—0·000 0
29	995·943 0	995·913 6	995·884 2	995·854 6	995·825 0	995·795 3	995·765 5	995·735 6	995·705 6	995·675 6	—0 029 8	—0·000 0
30	995·645 4	995·615 2	995·584 8	995 554 4	995·523 9	995·493 4	995·462 7	995·431 9	995·401 1	995·370 1	—0·030 6	0·000 0
31	995·339 1	995·308 0	995·276 8	995·245 6	995·214 2	995·182 8	995·151 2	995·119 6	995·087 9	995·056 1	—0·031 5	0·000 0
32	995·024 3	994·992 3	994·960 3	994·928 2	994·896 0	994·863 7	994·831 3	994·798 3	994·766 3	994·733 7	—0·032 3	0·000 0
33	994·701 0	994·668 2	994·635 3	994·602 1	994·569 3	994·536 2	994·503 0	994·469 7	994·436 4	994·402 9	—0·033 2	0·000 0
34	994·369 4	994·335 8	994·302 1	994·268 3	994·234 5	994·200 5	994·166 5	994·132 4	994·098 2	994·064 0	—0·034 0	0·000 0
35	994·029 6	993·995 2	993·960 7	993.926 1	993·891 5	993·856 7	990·821 9	993·787 0	993·752 1	993·717 0	—0·034 8	0·000 0
36	993·681 9	993·646 7	993·611 4	993 576 0	993·540 6	993·505 0	993·469 4	993·433 8	993·398 0	993·362 2	—0·035 6	0·000 0
37	993·326 3	993·290 3	993·254 2	993·218 1	993·181 8	993·145 5	993·109 2	993·072 7	993·036 2	993·999 6	—0·036 3	0·000 0
38	992·962 9	992·926 1	992·889 8	992·852 4	992·815 4	992.778 4	992·741 2	992·704 0	992·666 8	992·629 4	—0·037 1	0·000 0
39	992·592 0	992·554 5	992·516 9	992·479 2	992·441 5	992·403 7	992·365 8	992·327 9	992·289 9	992·251 8	—0·037 8	0·000 0
40	992·213 6											

Table 4 Buoyancy Corrections*

(*Clause* 17.1)

$\frac{m_t - m_o}{M}$	Correction, g/ml	$\frac{m_t - m_o}{M}$	Correction, g/ml	$\frac{m_t - m_o}{M}$	Correction, g/ml
0·60	0·000 48	0·80	0·000 24	1·00	0·000 00
0·61	0·000 47	0·81	0·000 23	1·01	— 0·000 01
0·62	0·000 46	0·82	0·000 22	1·02	— 0·000 02
0·63	0·000 44	0·83	0·000 20	1·03	— 0·000 04
0·64	0·000 43	0·84	0·000 19	1·04	— 0·000 05
0·65	0·000 42	0·85	0·000 18	1·05	— 0·000 06
0·66	0·000 41	0·86	0·000 17	1·06	— 0·000 07
0·67	0·000 40	0·87	0·000 16	1·07	— 0·000 08
0·68	0·000 38	0·88	0,000 14	1·08	— 0·000 10
0·69	0·000 37	0·89	0·000 13	1·09	— 0·000 11
0·70	0·000 36	0·90	0·000 12	1·10	— 0·000 12
0·71	0·000 35	0·91	0·000 11	1·11	— 0·000 13
0·72	0·000 34	0·92	0·000 10	1·12	— 0·000 14
0·73	0·000 32	0·93	0·000 08	1·13	— 0·000 16
0·74	0·000 31	0·94	0·000 07	1·14	— 0·000 17
0·75	0·000 30	0·95	0·000 06	1·15	— 0·000 18
0·76	0·000 29	0·96	0·000 05	1·16	— 0·000 19
0·77	0·000 28	0·97	0·000 04	1·17	— 0·000 20
0·78	0·000 26	0·98	0·000 02	1·18	— 0·000 22
0·79	0·000 25	0·99	0·000 01	1·19	— 0·000 23

*Calculated for standard air of density 0·001 222 g/ml at 15·56°C (60°F) and at a pressure of 1 : 013 bar (760 mm mercury).

These corrections are applicable for air density values between 0·001 1 and 0·001 3 g/ml,

NOTE — M = Weight in air water contained by the pyknometer at temperature t.

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