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Designation: D 1480 – 02

An American National Standard

Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer[1]

This standard is issued under the fixed designation D 1480; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[1] This test method is under the jurisdiction of ASTM Committee ~~D-2~~ D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee ~~D02.04on Hydrocarbon Analysis.~~ D02.04.0D on Physical and Chemical Methods.

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~~In 1962, this test method was adopted~~ approved in 1997 as ~~standard without revision.~~ D 1480–93(1997).

1. Scope

1.1 This test method describes two procedures for the measurement of the density of materials which are fluid at the desired test temperature. Its application is restricted to liquids of vapor pressures below 600 mm Hg (80 kPa) and viscosities below 40 000 cSt (mm^2/s) at the test temperature. The method is designed for use at any temperature between 20 and 100°C. It can be used at higher temperatures; however, in this case the precision section does not apply.

NOTE 1—For the determination of density of materials which are fluid at normal temperatures, see Test Method ~~D 941 or where greater precision is desired see Test Method~~ D 1217.

1.2 This test method provides a calculation procedure for converting density to specific gravity.

1.3 The values stated in acceptable SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Note ~~1, Note 2,~~ 1 and Note ~~3.~~ 2.

2. Referenced Documents

2.1 *ASTM Standards:*

D ~~94~~1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by ~~Lipkin Bicapillary~~ Bingham Pycnometer[2]

~~D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[2]~~

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in a vacuum (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in a vacuum) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or it is the ratio of the density of the material at t_1 to the density of water at t_2. When the reference temperature is 4°C (the temperature at which the relative density of water is unity), relative density (specific gravity) and density are numerically equal.

4. Summary of Test Method

4.1 The liquid sample is introduced into the pycnometer, equilibrated to the desired temperature, and weighed. The density or specific gravity is then calculated from this weight and the previously determined calibration factor, and a correction is applied for the buoyancy of air.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and to assess the quality of crude oils.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperatures of 15°C.

5.3 The determination of densities at the elevated temperatures of 40 and 100°C is particularly useful in providing the data needed for the conversion of kinematic viscosities in centistokes (mm^2/s) to the corresponding dynamic viscosities in centipoises (mPa·s).

6. Apparatus

6.1 *Pycnometer*,[4] Bingham-type of 10-mL capacity (as shown in Fig. 1), constructed of heat-resistant[5] glass.

NOTE 2—Pycnometers having capacities of 2 to 25 mL are available but have not been cooperatively evaluated.

6.2 *Constant-Temperature Bath*, provided with suitable pycnometer holders and means for maintaining temperatures constant to ±0.01°C in the desired range. Water-glycerin mixtures can be used for temperatures up to 100°C.

6.3 *Bath Thermometer*, graduated in 0.1°C subdivisions and standardized for the range of use to the nearest 0.01°C (ASTM Saybolt Viscosity Thermometers 17C to 22C, conforming to the requirements in Specification E 1, are recommended). For most hydrocarbons the density coefficient is about 0.0008 units/°C, and therefore an error of ±0.013°C would cause an error of ±0.00001 in density.

6.4 *Thermal Shields*, as shown in Fig. 2, to hold the pycnometer and syringe during the filling procedure, constructed of two aluminum shells with suitably spaced viewing ports, the upper bored to hold a 30-mL hypodermic syringe and the lower bored to hold a 25-mL Bingham pycnometer. A winding of No. 26 Chromel "A" wire, insulated from the shields with mica, covered with insulating tape, and having resistances connected in series of 25 Ω on the upper shield and 35 Ω on the lower produces controlled heat to the shields by means of a variable transformer. A stand is necessary to support the shields in such a manner that the center of the wells may be aligned, and the upper shield raised 180 to 200 mm and swung through 45°.

6.5 *Hypodermic Syringes*, 2 to 30-mL capacity, of chemically resistant glass, equipped with a 170-mm, 16-gage (0.065 in.) filling needle made from stainless-steel tubing, as shown in Fig. 3.

6.6 *Draw-off Needle*, made of stainless-steel tubing, as shown in Fig. 3.

6.7 *Solvent Cleaning Assembly*, as shown in Fig. 4.

6.8 *Chromic Acid Cleaning Apparatus*, similar to that shown in Fig. 5.

6.9 *Balance*, capable of reproducing weighings within 0.1 mg when carrying a load of 30 g. The balance shall be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled

[4] ~~Available from~~

[4] The sole source of supply of the pycnometer known to the committee at this time is Reliance Glass Co., 220 Gateway Rd., Bensonville, IL 60106-0825. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[5] Borosilicate glass has been found satisfactory for this purpose.

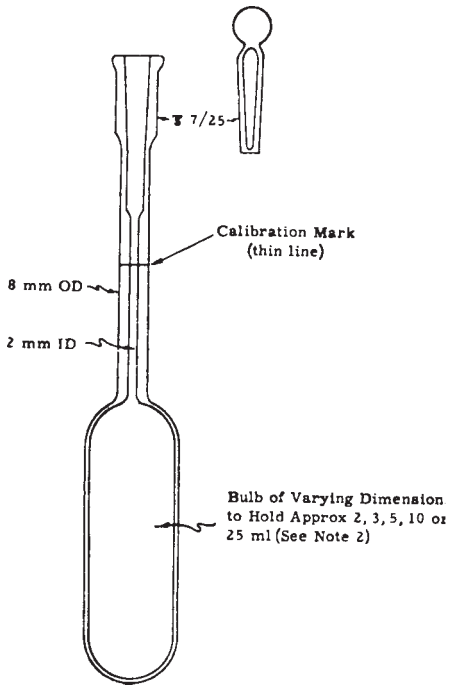
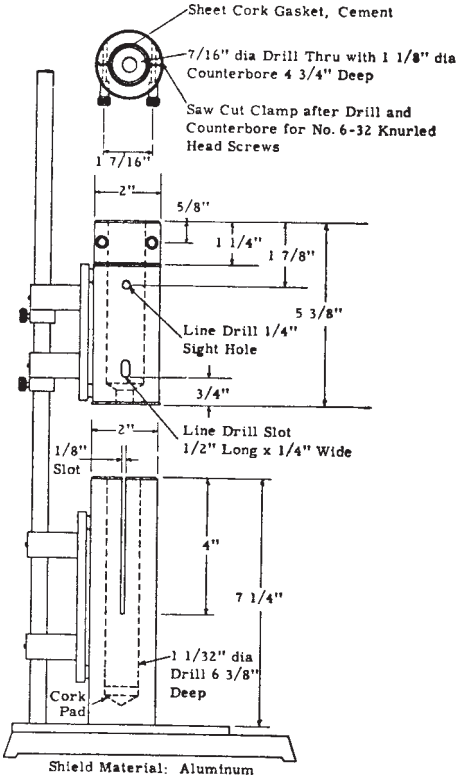


FIG. 1 Bingham-Type Pycnometer



Metric Equivalents

in.	mm	in.	mm	in.	mm	in.	mm
⅛	3.2	⅝	15.9	1¼	31.8	4	102
¼	6.4	¾	19.1	1⁷⁄₁₆	36.5	4¾	121
⁷⁄₁₆	11.1	1³⁄₃₂	26.2	1⅞	47.6	5⅜	136
½	12.7	1⅛	28.6	2	50.8	6⅜	162
						7¼	184

NOTE 1—Cover shields with mica or insulating cement. Wind with No. 26 gage Chromel "A" wire: Upper block 60 in. (1.52 m) (25.4Ω), lower block (85 in. (2.16 m) (35.0Ω) wound vertically. Cover with insulating tape or insulating cement and connect heaters in series. Insulate shields from stand with ¼-in. Transite.

FIG. 2 Details of Thermal Shields for 30-mL Syringe and 25-mL Pycnometer

pycnometer) do not cause a significant change in the ratio of the balance arms. The same balance shall be used for all related weighings.

6.10 *Weights*, whose relative values are known to the nearest 0.05 mg or better. Use the same set of weights for the calibration of the pycnometer and the determination of densities.

7. Reagents and Materials

7.1 *Acetone*—~~(**Warning —See Note 3).**~~

~~NOTE 3—**Warning:**~~ **Warning**—Extremely flammable. Use adequate ventilation.~~)~~

7.2 *Isopentane*—~~(**Warning**—See Note 4).**~~

~~NOTE 4—**Warning:**~~ **Warning**—Extremely flammable. Avoid build up of vapors and remove all sources of ignition, especially non-explosion proof electrical apparatus.~~)~~

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—~~(**Warning**—See Note 5).**~~

~~NOTE 5—**Warning:**~~ **Warning**—Causes severe burns. A recognized carcinogen. Do not get in eyes, on skin or clothing.~~)~~

8. Preparation of Apparatus

8.1 Clean the pycnometer thoroughly with hot chromic acid cleaning solution by means of the assembly shown in Fig. 5

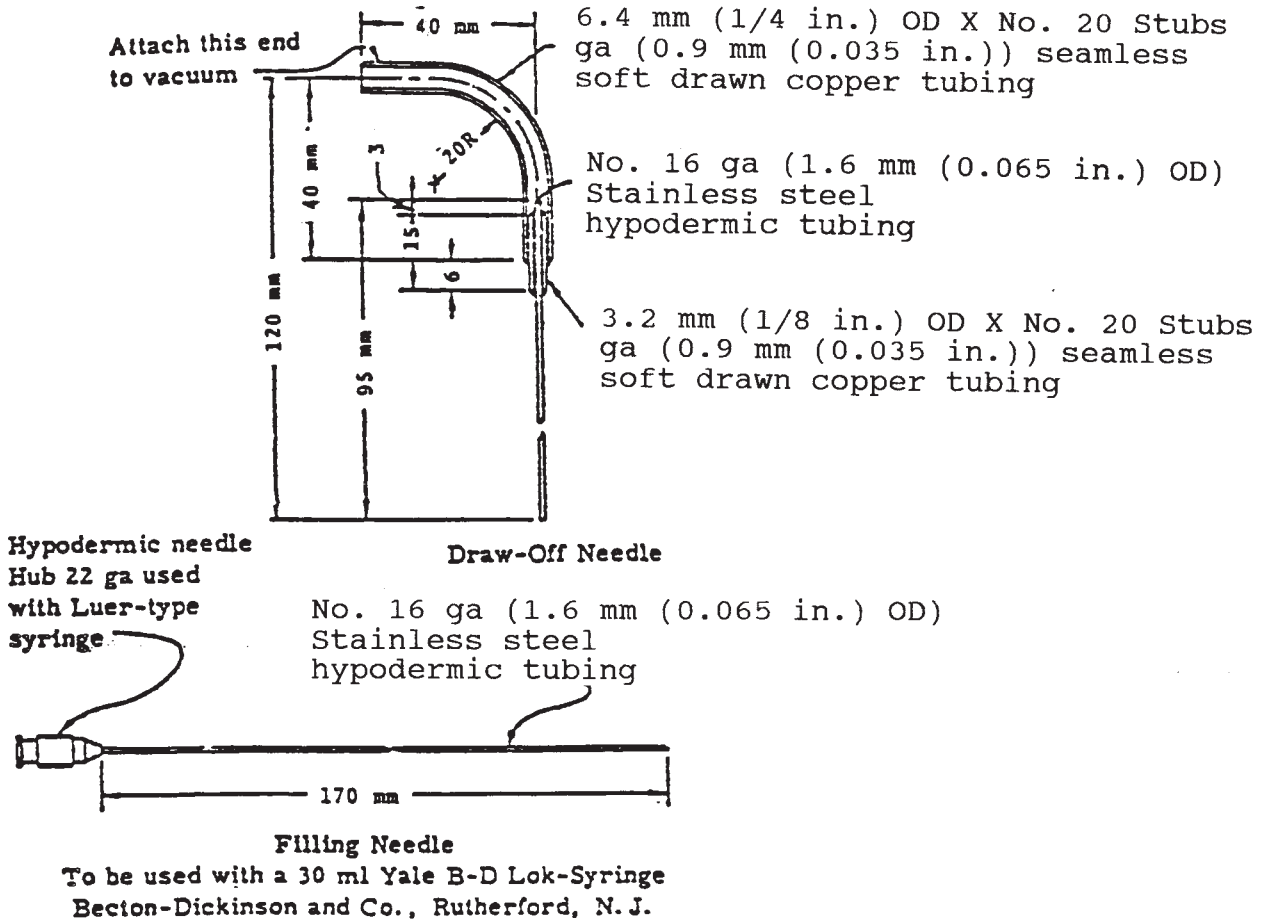


FIG. 3 Accessories for Bingham-Type Pycnometer

(**Warning**—See ~~Note 5).~~ 7.3.) Chromic acid solution is the most effective cleansing agent. However, surfactant cleansing fluids have also been used successfully. Mount the apparatus firmly and connect the trap to the vacuum. Warm the necessary amount of cleaning acid in the beaker, place the pycnometer on the ground joint, and evacuate by opening the stopcock to vacuum. Fill the pycnometer with acid by turning the stop-cock, and either repeat several times, or remove the filled pycnometer and allow it to stand for several hours at 50 to 60°C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer with distilled water. Clean in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, rinsing with pure, dry acetone, followed by isopentane, and vacuum drying. (**Warning**—See ~~Note 3~~ 7.1 and ~~Note 4.)~~ 7.2.)

8.2 Transfer the pycnometer to the cleaner assembly shown in Fig. 4, with vacuum line and trap attached to the side tube as indicated. Place the pycnometer on the cleaner with the upper hypodermic needle extending upward into the pycnometer, and press the edge of the ground joint on the rubber stopper until the vacuum holds it in place. Draw out all the liquid or sample. Immerse the lower end of the hypodermic tube in a suitable solvent and draw 20 to 25 mL through the pycnometer. Leaving the pycnometer in place, draw air through it until it is dry. Clean the hypodermic syringe with the same apparatus.

9. Calibration of Pycnometers

9.1 Weigh the clean, dry pycnometer to 0.1 mg and record the weight.

NOTE 6~~3~~—It is convenient to use the lightest of a set of pycnometers as a tare. For best results the treatment and environment of both pycnometer and tare should be identical for some time prior to weighing.

9.2 With a syringe of suitable size, transfer freshly boiled and cooled distilled water to the pycnometer through the filling needle (Note ~~9).~~ 6). Avoid trapping air bubbles in the bulb or capillary of the pycnometer, removing bubbles, as they form, with the syringe, when possible. Also remove any water above the calibration mark and dry the overflow chamber and capillary with a cotton-fiber pipe cleaner or cotton swab which has been moistened slightly with acetone. Do not touch the plunger of the syringe or hypodermic needle with fingers as minute quantities of oil transferred this way would cause faulty drainage in the capillary neck of the pycnometer.

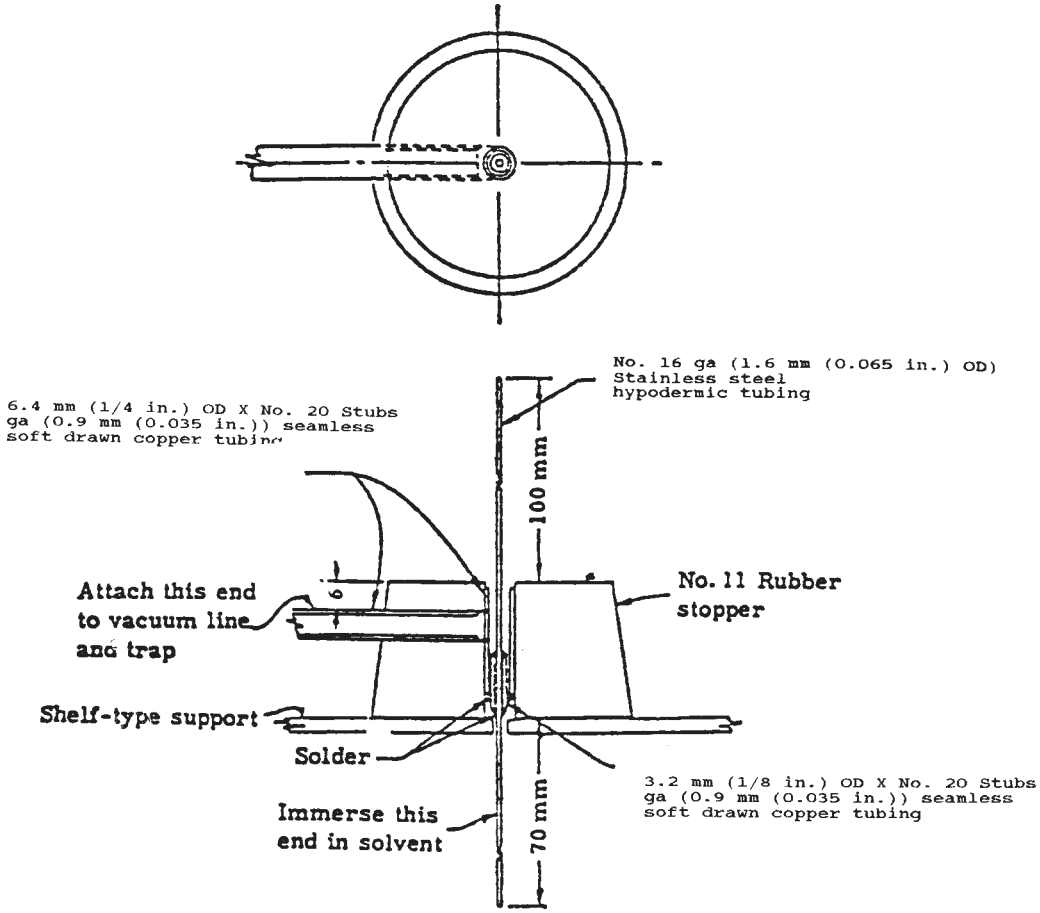


FIG. 4 Cleaner Assembly for Bingham-Type Pycnometer

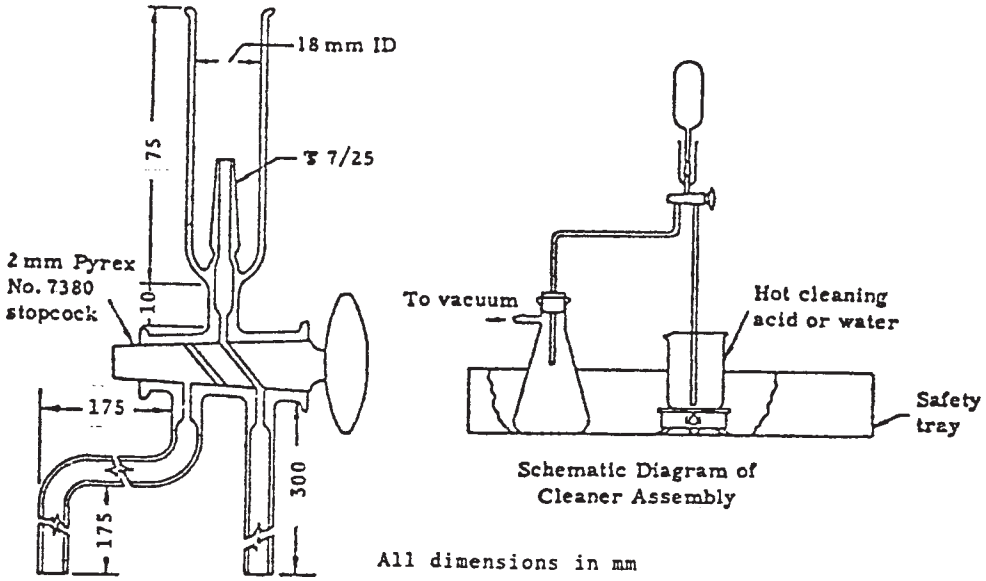


FIG. 5 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

9.3 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath adjusted to a constancy of ±0.01°C at the desired temperature (Note ~~7~~. 4). Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid through the steel draw-off needle until expansion has stopped, indicating that the liquid has reached the temperature of the thermostat. To minimize errors caused by faulty drainage, do not allow the liquid to expand more than 10 mm above the calibration mark at any time. Allow the contents to equilibrate an additional 10 min and draw

TABLE 1 Vacuum Corrections to be Applied to Densities Observed in Air of Various Densities

NOTE 1—Interpolate linearly for intermediate sample densities.
NOTE 2—For air densities outside this table the vacuum correction shall be calculated from the equation $C = d_a[1 - (F_tW_t)]$, d_a being the density of the air in the balance case in grams per millilitre. See Section 10 of Test Method D 1217 for calculating the air density.

Observed Density	Air Density			
	0.00116	0.00118	0.00120	0.00122
	Corrections to be Added			
0.60	0.00046	0.00047	0.00048	0.00049
0.65	0.00040	0.00041	0.00042	0.00042
0.70	0.00034	0.00035	0.00036	0.00036
0.75	0.00029	0.00029	0.00030	0.00030
0.80	0.00023	0.00024	0.00024	0.00024
0.85	0.00017	0.00018	0.00018	0.00018
0.90	0.00011	0.00012	0.00012	0.00012
0.95	0.00005	0.00006	0.00006	0.00006
1.00	0	0	0	0
	Corrections to be Subtracted			
1.05	0.00005	0.00006	0.00006	0.00006
1.10	0.00011	0.00012	0.00012	0.00012
1.15	0.00017	0.00018	0.00018	0.00018
1.20	0.00023	0.00024	0.00024	0.00024

the level down exactly to the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark, with the draw-off needle or pipe cleaner, depending upon the volatility of the sample. Portions in the overflow bulb can be removed with a cotton swab moistened with acetone.

NOTE ~~7~~4—For temperatures above 80°C calculate the volume from the coefficient of expansion of the glass observed from calibrations made at 60, 70, and 80°C.

9.4 Replace the glass stopper, remove the pycnometer from the bath, wash the outside surface with acetone, and dry thoroughly with a chemically clean, lint-free, slightly damp cloth. Place the pycnometer in or near the balance case for 20 min and weigh to the nearest 0.1 mg.

NOTE ~~8~~5—In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg in the weight of the pycnometer. This charge may not be completely dissipated in less than 30 min. The use of about 0.1 mg of radium bromide- or polonium-coated foil in the balance case, or maintaining the relative humidity at 60 percent or higher, aids in reducing weighing difficulties due to static charges.

9.5 Calculate the pycnometer calibration factor, F_t, from the equation:

$$F_t = (\text{density of water at } t°\text{C})/(\text{weight of water in pycnometer at } t°\text{C}) \quad (1)$$

See Table 2 for the density of water between 0 and 100°C.

9.6 Duplicate determinations should not show a variation greater than ±0.2 mg in the net weight of the water in the pycnometer.

10. Procedure for Viscous Liquids

10.1 Weigh the pycnometer as directed in Section 8.

10.2 Warm, in an oven or convenient warming chamber, the pycnometer, syringe with needle, and sample to a convenient working temperature consistent with the fluidity and volatility of sample. Draw the requisite amount of sample into the syringe and immediately fill the warmed pycnometer taking care to avoid occluding air bubbles in the pycnometer bulb or capillary. Continue the addition of sample, withdrawing the filling needle gradually so that the tip remains immersed in the sample, until the sample has been added to a depth of 10 or 20 mm in the expansion chamber above the capillary, depending upon the amount of contraction expected.

10.3 Immerse the pycnometer bulb in the constant-temperature bath. As the sample contracts continue sample addition before the level recedes into the capillary or until a sufficient amount has been added to maintain the meniscus slightly above the calibration mark at the reference temperature. Allow to equilibrate to reference temperature.

NOTE ~~9~~6—Equilibration time depends upon the viscosity and temperature of the sample at the time of filling. Usually this is three to four times that required for a fluid sample. A safe criterion is to allow 15 min more equilibration time after the meniscus remains stationary.

10.4 Remove excess sample with the 16-gage needle attached to a vacuum line, warming the needle if necessary. Swab the capillary above the calibration mark and the overflow chamber several times with a pipe cleaner or small cotton swab slightly moistened with a suitable solvent. Follow with a dry swab. Final adjustment to the mark may be done by picking out sample with a small probe, splinter, or wire.

TABLE 2 Density of Water[A]

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999840	21	0.997991	40	0.992212
3	0.999964	22	0.997769	45	0.990208
4	0.999972	23	0.997537	50	0.988030
5	0.999964	24	0.997295	55	0.985688
10	0.999699	25	0.997043	60	0.983191
15	0.999099	26	0.996782	65	0.980546
15.56	0.999012	27	0.996511	70	0.977759
16	0.998943	28	0.996231	75	0.974837
17	0.998774	29	0.995943	80	0.971785
18	0.998595	30	0.995645	85	0.968606
19	0.998404	35	0.994029	90	0.965305
20	0.998203	37.78	0.993042	100	0.958345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

10.5 Remove the pycnometer from the bath, wash the outer surface with a suitable solvent followed by acetone and dry thoroughly with a clean, lint-free, slightly damp cloth. Observe the same cleaning procedure as used in calibrating the pycnometer in the bath. Allow the pycnometer to come to room temperature and weigh to the nearest 0.1 mg.

11. Procedure for Melted Solids at High Temperature

11.1 Place the sample in a heat-resistant container and bring to a temperature 8 to 12°C above its melting point in an explosion-proof oven.

11.2 Insert the pycnometer, previously weighed to the nearest 0.1 mg, in the lower chamber of the thermal shield and lightly clamp the syringe in the upper chamber so that the filling needle is inside the pycnometer. Apply power to the shields until the temperature is 2 to 3°C above the melting point of the sample, then reduce the voltage until the shield temperature increases less than 0.5°C/min.

NOTE—~~10—In~~ 7—In the absence of a thermal shield, an oven can be fitted with a rack to support the pycnometer and hypodermic, and the whole operation of charging the syringe and filling the pycnometer performed in the oven. Weights applied to the syringe plunger reduce the filling time. An internal light and glass door for the oven are aids in this procedure.

11.3 After thermal equilibrium of sample, pycnometer, and syringe has been established, raise the upper shield, swing to one side, and quickly charge the syringe.

11.4 Quickly wipe the needle, swing the syringe over, and lower into the pycnometer. Fill the pycnometer in the usual manner, as given in 10.2. Remove the syringe and needle and place the pycnometer in the bath for temperature equilibration. Remove excess sample with a thin strip of filter paper or heated draw-off needle, taking care not to remove sample below the calibration mark.

11.5 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath, adjusted to the desired temperature within ±0.01°C. Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid with thin strips of filter paper. Continue in this manner until expansion has stopped, indicating that the liquid has reached the temperature of the bath. To minimize errors caused by faulty drainage, do not allow the liquid to expand more than 10 mm above the calibration mark at any time. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to the top of the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark with filter paper or a pipe cleaner, barely moistened with a suitable solvent if necessary.

11.6 Replace the glass stopper, and proceed as directed in 10.5.

12. Calculation

12.1 Calculate the density of the sample, corrected to vacuum, by the following equation:

$$\text{Density in vacuum } (d_t), \text{ g/mL} = (F_t)(W_t) + C \quad (2)$$

where:

F_t = calibration factor of the pycnometer at t°C,
W_t = weight of sample, g, in pycnometer at t°C, and
C = vacuum correction, obtained from Table 1.

12.2 Calculate the relative density (specific gravity) of the sample by dividing the density, as obtained in 12.1, by the density of water at the reference temperature obtained from Table 2.

13. Precision and Bias

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

13.1.1 *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:

Pycnometer Volume, mL	Repeatability, g/mL
10	0.00005

13.1.2 *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:

Pycnometer Volume, mL	Reproducibility, g/mL
10	0.00014

NOTE ~~11—If~~ 8—If pycnometers of other than 10 mL in volume are used, or if the temperature of test exceeds 100°C, this precision statement may not apply.

13.1.3 *Bias*—The difference of results from the established value when compared to pure reference materials is not expected to be more than ±0.00014 g/mL. Specific bias has not been established by cooperative testing.

14. Keywords

14.1 density; gravity; pycnometer; relative density; specific gravity