Designation: ~~D1480 – 15~~ D1480 – 21

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 D1480; 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. Scope*

1.1 This test method covers 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 80 kPa (600 mm Hg) and viscosities below 40 000 mm^2/s (cSt) at the test temperature. The method is designed for use at any temperature between 20 °C 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 D1217.

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

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous ~~material~~substance that can cause ~~central nervous system, kidney and liver damage.~~ serious medical issues. Mercury, or its vapor, ~~may~~ has been demonstrated to be hazardous to health and corrosive to materials. ~~Caution should be taken~~ Use Caution when handling mercury and ~~mercury containing~~ mercury-containing products. See the applicable product ~~Material~~ Safety Data Sheet ~~(MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware~~ (SDS) for additional information. The potential exists that selling mercury ~~and/or mercury containing products in your state or country may be prohibited by law.~~or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

1.5 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D8278 Specification for Digital Contact Thermometers for Test Methods Measuring Flow Properties of Fuels and Lubricants
E1 Specification for ASTM Liquid-in-Glass Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions:*

3.1.1 *density, n*—mass per unit volume at a specified temperature. **D4052**

3.1.2 *relative density, n*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature. **D4052**

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.

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

6. Apparatus

6.1 *Pycnometer,*[3] ~~Bingham-type~~ Bingham-type of 10 mL capacity (as shown in Fig. 1), constructed of heat-resistant[4] glass.

NOTE 2—Pycnometers having capacities of 2 mL 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~~ (for example ASTM Saybolt Viscosity Thermometers ~~17 °C~~17C to ~~22 °C,~~22C, conforming to the requirements in Specification E1~~, are recommended). For most hydrocarbons~~). Alternative non-mercury-containing liquid-in-glass thermometers such as thermometer S18C and S22C in Specification E2251~~the density coefficient is about 0.0008 units~~ conforming to the temperature range with equal or~~°C, and therefore an error of ± 0.013 °C would cause an error of ± 0.00001 g/mL in density. A standardized platinum~~ better accuracy may be used. A digital contact thermometer meeting the criteria for D02-DCT05 of Specification D8278 ~~resistance thermometer~~ may also be used, and it offers the best means for observing temperature changes in the bath.

NOTE 3—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 g/mL in density.

6.4 *Thermal Shields,* as shown in Fig. 2, to hold the pycnometer and syringe during the filling procedure, constructed of two

[3] There is more than one supplier. If you cannot find a supplier, then contact Subcommittee D02.04.0D on Physical and Chemical Methods for possible suppliers.
[4] Borosilicate glass has been found satisfactory for this purpose.

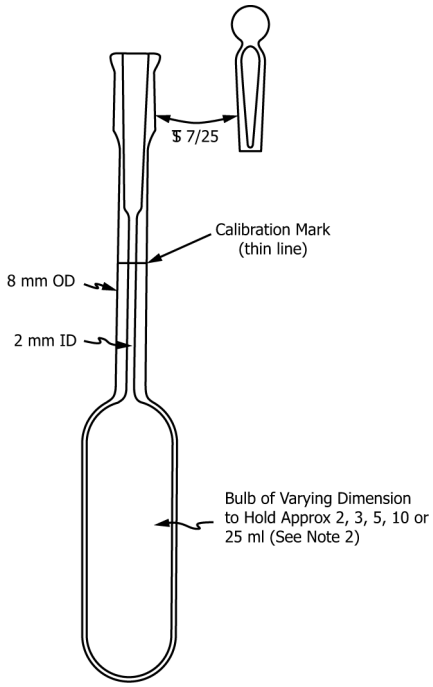


FIG. 1 Bingham-Type Pycnometer

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 mm to 200 mm and swung through 45°.

6.5 *Hypodermic Syringes,* 2 mL to 30 mL capacity, of chemically resistant glass, equipped with a 170 mm, 16 gauge (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 pycnometer) do not cause a significant change in the ratio of the balance arms. The same balance shall be used for all related weighings.

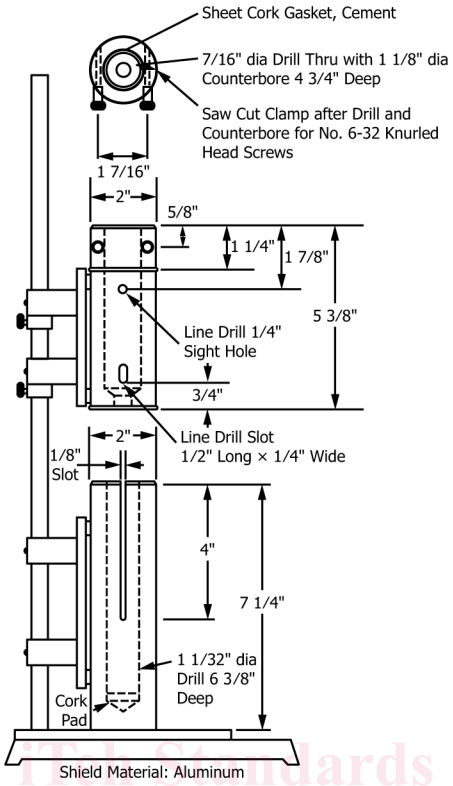
6.10 *Weights,* whose relative values are known to the nearest 0.05 mg or better. Use the same set of weights for the calibration of the pycnometer and the determination of densities.

7. Reagents and Materials

7.1 *Acetone*—(**Warning—**Extremely flammable. Use adequate ventilation.)

7.2 *Isopentane*—(**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—**Causes severe burns. A recognized carcinogen. Do not get in eyes, on skin, or on clothing.)



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	1 7/16	36.5	4¾	121
7/16	11.1	1 1/32	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 gauge Chromel "A" wire: Upper block 1.52 m (60 in.) (25.4 Ω), lower block 2.16 m (85 in.) (35.0 Ω) 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

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 (**Warning**—See 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 °C 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 7.1 and 7.2.)

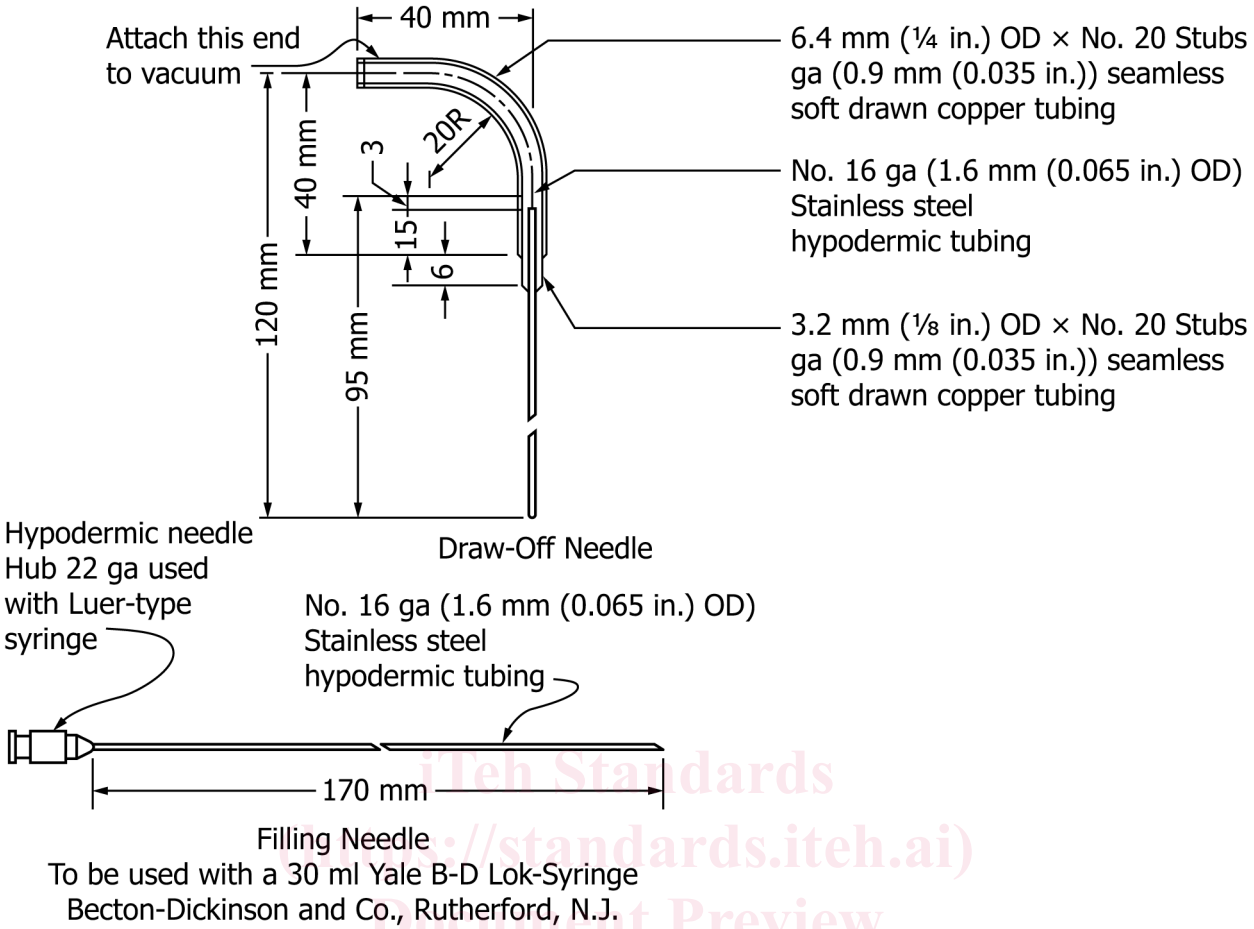


FIG. 3 Accessories for Bingham-Type Pycnometer

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 mL 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 4—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 ~~6~~7). 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.

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 ~~4~~5). 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

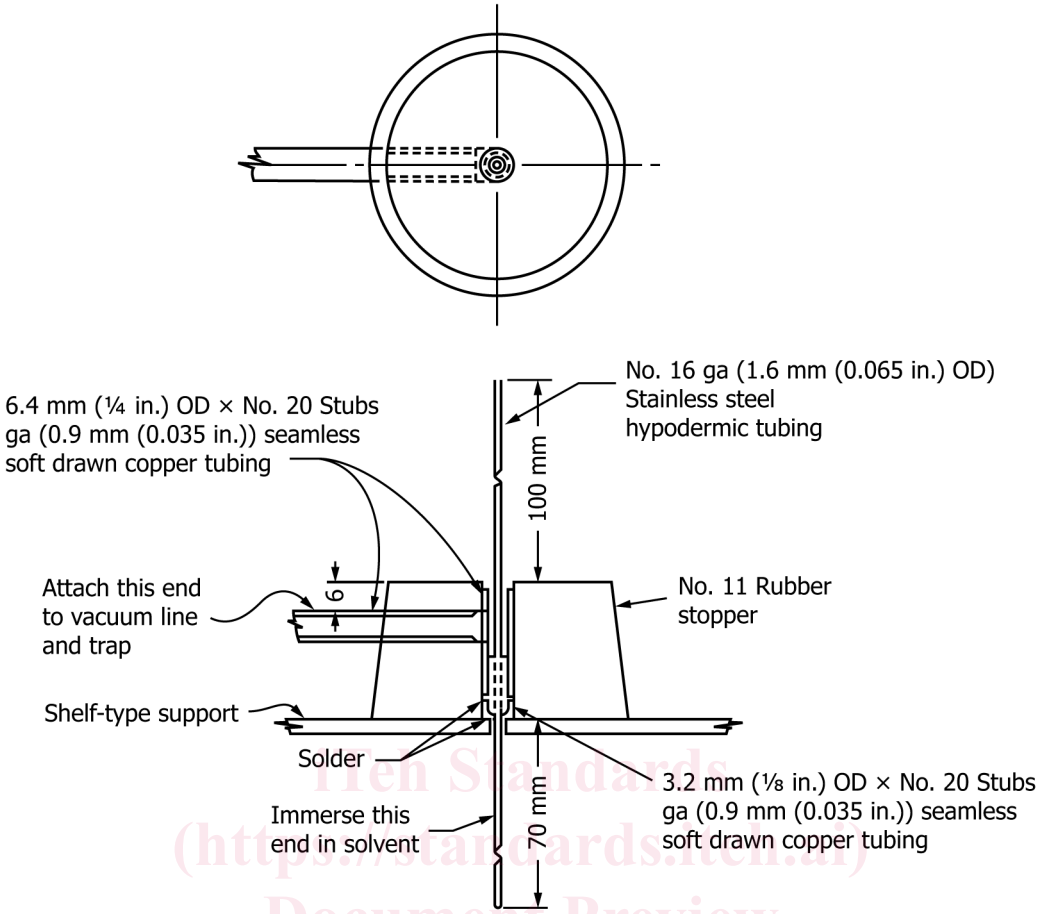


FIG. 4 Cleaner Assembly for Bingham-Type Pycnometer

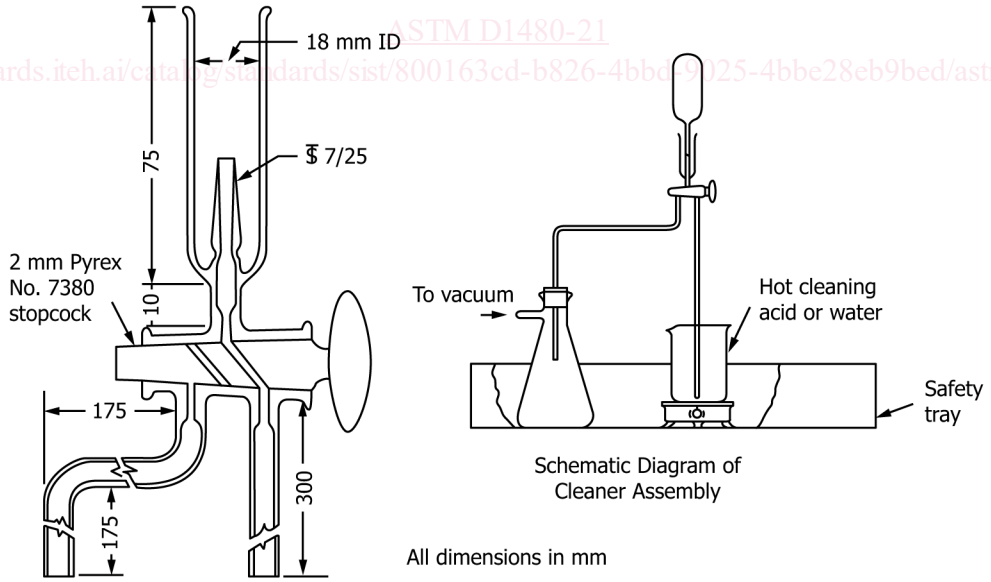


FIG. 5 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

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 the level down exactly to the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility.

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 $C = d_a\,[1 - (F_t W_t)]$, d_a being the density of the air in the balance case in grams per millilitre. See Section 10 of Test Method D1217 for calculating the air density.

Observed Density	Air Density g/mL			
	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

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 5—For temperatures above 80 °C calculate the volume from the coefficient of expansion of the glass observed from calibrations made at 60 °C, 70 °C, 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 6—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 % or higher, aids in reducing weighing difficulties due to static charges.

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

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

$$(\text{weight of water in pycnometer at } t\,°\text{C})$$

See Table 2 for the density of water between 0 °C 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 mm or 20 mm in the expansion chamber above the capillary, depending upon the amount of contraction expected.