~~Designation:D1480–02ε1~~ Designation: D 1480 – 07

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.

~~ε[1] NOTE—Original footnote 4 was deleted editorially in June 2006, since there is no longer a sole supplier of the pycnometer.~~

1. Scope*

1.1 This test method ~~describes~~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 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 1217.

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

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

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 and Note 2.~~

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1217~~Practice for Classification of Computed Radiology Systems~~ Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

E 1~~Practice for Classification of Computed Radiology Systems~~ Specification for ASTM Liquid-in-Glass Thermometers

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.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.**

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*,[3] Bingham-type of 10-mL capacity (as shown in Fig. 1), constructed of heat-resistant[4] 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.~~ , 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. A standardized platinum resistance thermometer may also be used, and it offers the best means for observing temperature changes in the bath.

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

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

[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.

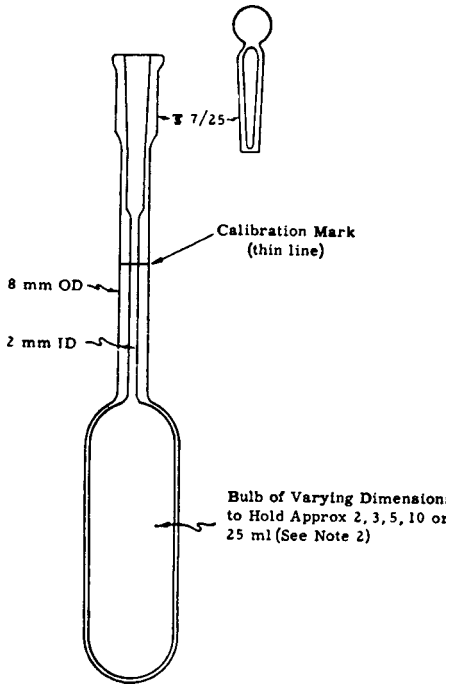
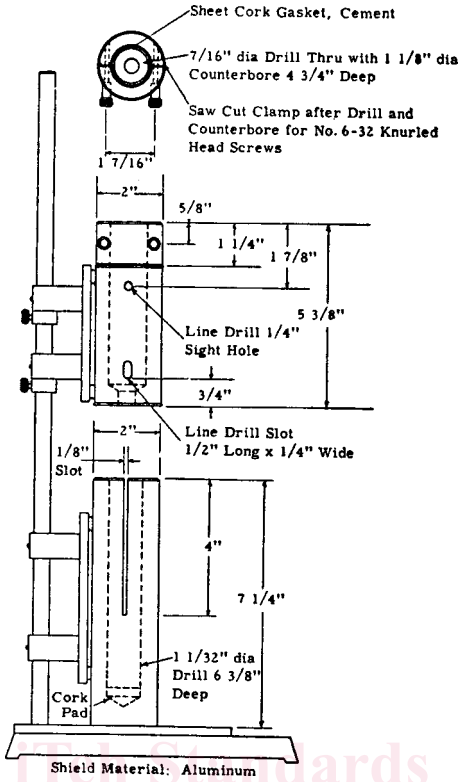


FIG. 1 Bingham-Type Pycnometer



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	1³²⁄₃₂	26.2	1⅞	47.6	5⅜	136
½	12.7	1⅛	28.6	2	50.8	6⅜	162
						7¼	184

NOTE—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

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 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 (**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 to 60°C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer

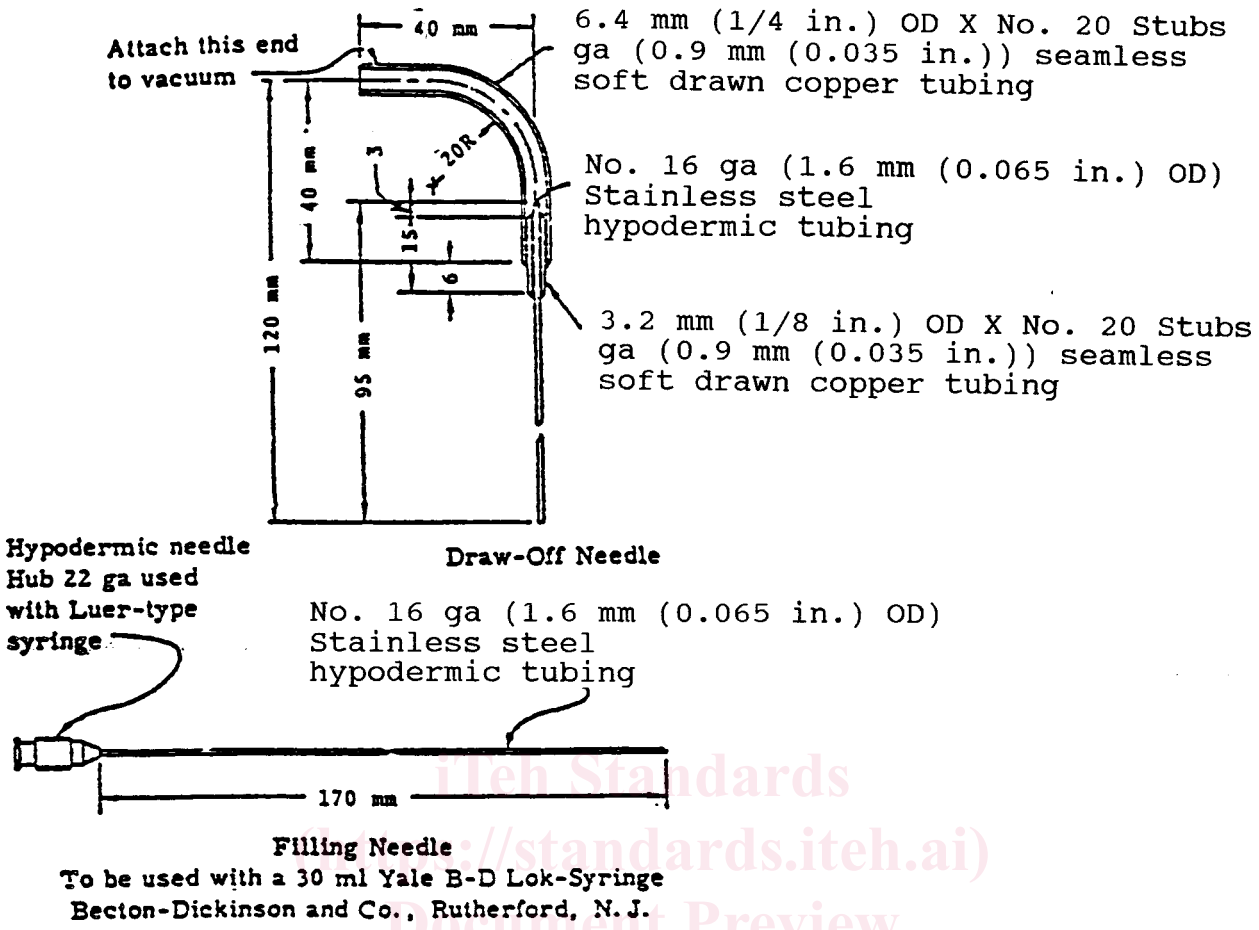


FIG. 3 Accessories for Bingham-Type Pycnometer

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.)

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 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 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.

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). 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