Designation: D3800 – 99 (Reapproved 2010)

Standard Test Method for Density of High-Modulus Fibers[1]

This standard is issued under the fixed designation D3800; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the density of high-modulus fibers and is applicable to both continuous and discontinuous fibers.

1.2 The values stated in SI units are to be regarded as standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. 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.* See Section 9 for additional information.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D3878 Terminology for Composite Materials

D5229/D5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

D6308 Guide for Identification of Composite Materials in Computerized Material Property Databases[3]

E12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology E12 defines terms relating to density. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over other standards.

3.2 *Symbols:*

ρ_s	=	density of standard
ρ_l	=	density of liquid
ρ_f	=	density of fiber
ρ_{mf}	=	density of the measured fiber containing sizing
ρ_{ml}	=	density of the measured liquid containing surfactant
ρ_{sur}	=	density of surfactant
ρ_{sz}	=	density of sizing
ρ_w	=	density of water
s	=	standard deviation
M_1	=	weight of suspension wire in air
M_2	=	weight of suspension wire in liquid (to immersion point)
M_3	=	weight of suspension wire plus item whose density is to be determined (in air)
M_4	=	weight of suspension wire plus item whose density is to be determined (in liquid)
$M_3 - M_1$	=	weight of item for density to be determined in air
$M_4 - M_2$	=	weight of item for density to be determined in liquid

4. Summary of Test Method

4.1 *General*—Using random selection techniques, a suitable size sample of high-modulus fiber can be tested by any of the three procedures described in this test method. Procedure A using water with a surfactant as the liquid medium is preferred due to environmental and safety considerations. The other methods shall not be used if Procedure A is adequate. Interim

[1] This test method is under the jurisdiction of ASTM Committee D30 onComposite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

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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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

use of Procedures B or C is allowed while a comparison is made to results using Procedure A.

4.2 *Procedure A—Buoyancy (Archimedes) Method*:

4.2.1 The sample is weighed in air and weighed in a liquid that will thoroughly wet the sample and is of a lower density.

4.2.2 The difference in weight of the sample in the two media is the buoyancy force. This force is converted to sample volume by dividing it by the liquid density. The sample weight in air divided by the sample volume equals the sample density.

4.3 *Procedure B—Sink-Float Technique*:

4.3.1 The sample is placed in a container containing a liquid that will thoroughly wet the sample and is of a lower density. A liquid of higher density than the sample and miscible with the first liquid is then added slowly to the container under constant gentle mixing until the sample is suspended in the mixture.

4.3.2 The density of the resulting mixed liquid is determined using either a hydrometer or a pycnometer. The density of the sample is equal to the density of the liquid in which the sample is suspended.

4.4 *Procedure C*—For an alternative method, which may be used, see Test Method D1505.

5. Significance and Use

5.1 Fiber density is useful in the evaluation of new materials at the research and development level and is one of the material properties normally given in fiber specifications.

5.2 Fiber density is used to determine fiber strength and modulus both of a fiber bundle and an individual filament. These properties are based on load or modulus slope over an effective area. Fiber density may be used with lineal mass of the fiber to give an approximation of effective tow area. Tow area divided by the average number of filaments in a tow gives an approximation of the effective area of an individual filament.

5.3 Fiber density is used as a constituent property when determining reinforcement volume and void volume based on reinforcement mass and laminate density.

6. Interferences

6.1 *General (All Methods)*:

6.1.1 *Temperature*—The temperature of the liquid shall remain constant within a tolerance of ±1°C, since liquid density changes with temperature.

6.1.2 *Sample Wetting (Entrapped Air)*—Since this test method is very dependent on buoyancy, any entrapped air in the sample will change the measured density and not give a true material density. Ensure visually that the sample does not contain entrapped air bubbles.

6.1.3 *Homogenous Mixture*—The density of the liquid shall be uniform, through suitable agitation.

6.1.4 *Removal of Sizing*—A bias will exist if sizing is not removed. In this case, the measured fiber density is a combination of the density of the fiber and the sizing. The following equation may be used to calculate the effect of the sizing on the density of the material.

$$\rho_{mf} = \frac{(100 - x)\,\rho_f + x(\rho_{sz})}{100} \quad (1)$$

where

x = mass of sizing as a percentage of the total mass of the measured fiber.

6.1.5 *Effect of Surfactant Density*—The addition of a surfactant to a liquid may produce bias if not considered. The effect may be shown by the following equation:

$$\rho_{ml} = \frac{(100 - x)\,\rho_l + x(\rho_{sur})}{100} \quad (2)$$

where

x = mass of surfactant as a percentage of total mass of the measured liquid.

6.2 *(Method A)*:

6.2.1 *Immersion Point*—The distance the sample is lowered into the liquid and the overall liquid level should be the same throughout determinations for Procedure A. This may be done by putting a line for the desired liquid level on the outside of the container. The sample size should be within a few grams from one sample to another.

7. Apparatus

7.1 *General*:

7.1.1 *Thermometer*, capable of reading the test temperature during the test to 0.1°C.

7.1.2 *Agitator*—Stirrer or mixing propeller capable of slowly agitating solution without test interference.

7.2 *Procedure A*:

7.2.1 *Balance*, analytical, capable of weighing to 0.0001 g, adapted for suspension weighing.

7.2.2 *Balance Stand*, depending on the type of balance used; two recommended stands are shown in Figs. 1 and 2.

7.2.3 *Laboratory Jack*, heavy-duty precision.

7.2.4 *Suspension Wire*, nickel or stainless steel, approximately 0.4 mm in diameter, cut and shaped to match the system used.

7.2.5 *Vacuum Desiccator (with Pump)*—An airtight container in which a low vacuum (less than 75 kPa [22 in. Hg]) can be maintained.

7.2.6 *Density Standard*—A solid piece of borosilicate glass (density approximately 2.2 g/mL) of known density to four significant figures as determined by water immersion.[4] A NIST standard of this type (SRM 1825) is recommended.

7.2.7 *Vacuum Pump or Aspirator*, used to provide vacuum-to-vacuum desiccator.

7.2.8 *Container*, glass or other transparent container resistant to a liquid medium is recommended.

7.2.9 *Immersion Liquid*—The liquid used shall not dissolve or otherwise affect the specimen, but should wet it and have a specific gravity less than that of the specimen.[5] The specific gravity of the immersion liquid shall be determined shortly before and after each use.

7.3 *Procedure B*:

7.3.1 *Container*, glass or other transparent container resistant to liquids used is recommended.

[4] A No. 19 "Pyrex" glass stopper with a 3.175-mm diameter hole bored through the top for suspension purposes has proved satisfactory.

[5] One suitable surfactant to use with water is Triton X manufactured by Rohm and Haas, Philadelphia, PA.

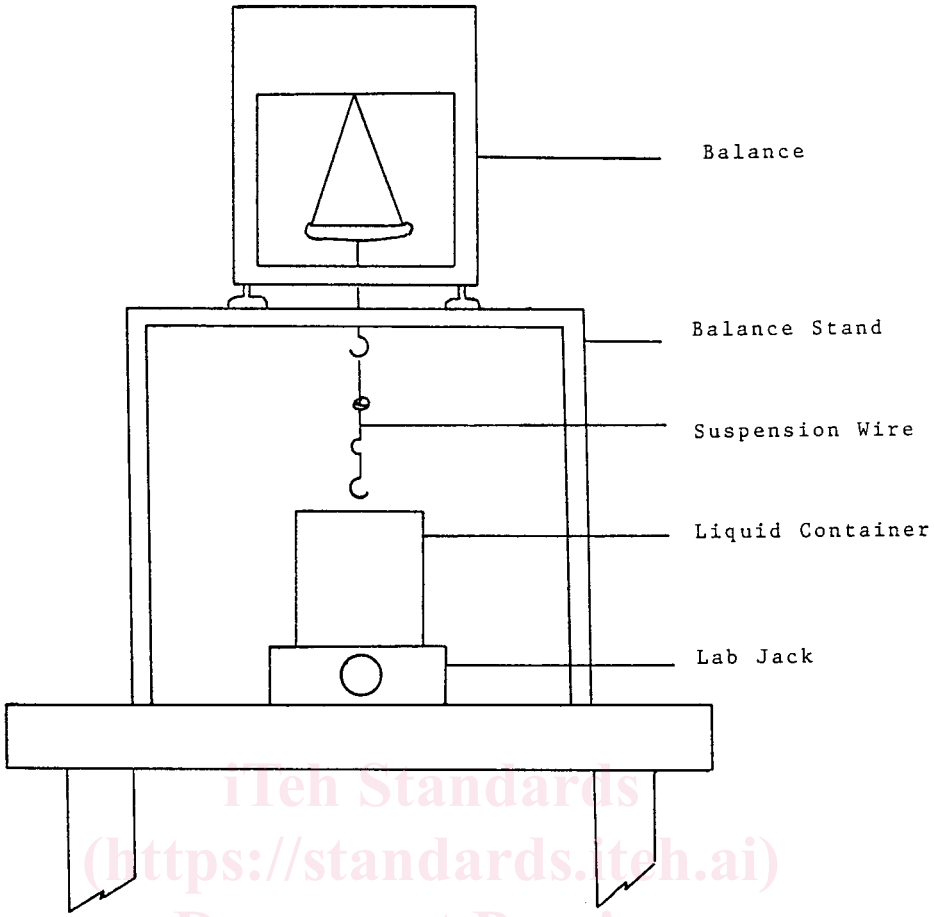

FIG. 1 Density Apparatus (Alternative)

7.3.2 *Immersion Liquids*—See Notes 1 and 2. One liquid should have a density less than the fiber, and the other greater, so when mixed they have the same density as the fiber. Two suitable liquids are trichloroethylene and dibromomethane (having densities of 1.464 and 2.477 g/mL). Both of these liquids pose hazards (see Section 8).

7.3.3 *Hydrometer*, capable of reading liquid density.

7.4 *Procedure C*—Use the apparatus described in Test Method D1505.

NOTE 1—Standard deionized or distilled water need not be measured, but can be taken as a value from standard tables.[6] For the determination of the specific gravity of the liquid, the use of a standard plummet of known volume (Note 3) or Test Method A, C, or D of Test Methods D891, using the modifications required to give specific gravity at 23°C is recommended. One suggested procedure is the following: If a constant temperature water bath is not available, determine the weight of the clean, dry pycnometer with the thermometer to the nearest 0.1 mg on an analytical balance. Fill the pycnometer with water cooler than 23°C. Insert the thermometer stopper causing excess water to be expelled through the side arm. Permit the filled bottle to warm in air until the thermometer reads 23°C. Remove the drop of water at the tip of the side arm with a bit of filter paper, taking care not to draw any liquid from within the capillary. Place the cap over the side arm, wipe the outside carefully, and weigh the filled bottle again to the nearest 0.2 mg. Empty the pycnometer, dry, and then fill and weigh with the other liquid in the same manner as was done with the water. Calculate the specific gravity at 23°C of the liquid, ρ_f, as follows:

$$\rho_l = (b - e)/(w - e) \quad (3)$$

where:

e = apparent weight of empty pycnometer,
w = apparent weight of pycnometer filled with water at 23°C, and
b = apparent weight of pycnometer filled with liquid at 23°C.

If a constant-temperature bath is available, a pycnometer without a thermometer may be used.

NOTE 2—One standard, which has been found satisfactory for this purpose, is the Reimann Thermometer Plummet. These are normally calibrated for measurements at temperatures other than 23/23°C, so that recalibration is necessary for the purpose of these test methods. Calibrations at intervals of one week are recommended.

8. Reagents

8.1 *Purity of Reagents*—As a minimum, a technical grade reagent is required to provide accurate results. However, when resolving disputes or performing subsequent analysis of extract or residue, a reagent grade reagent shall be used. Unless otherwise indicated, it is intended that the reagents conform to

[6] One such reference is in *CRC Handbook of Chemistry and Physics*, CRC Press Inc., Boca Raton, FL.