Designation: D 3800 – 99

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

This standard is issued under the fixed designation D 3800; 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 the determination of the density of high-modulus fibers and is applicable to both continuous and discontinuous fibers.

1.2 *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.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[2]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[3]

D 3878 Terminology of High-Modulus Reinforcing Fibers and Their Composites[4]

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[4]

D 6308 Guide for Identification of Composite Materials in Computerized Material Property Databases[4]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[5]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[6]

E 1471 Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases[4]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to composite materials. Terminology E 12 defines terms relating to density. Practice E 177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 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 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

[1] This test method is under the jurisdiction of ASTM Committee D-30 on Composite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

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[2] *Annual Book of ASTM Standards*, Vol 15.05.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 15.03.

[5] Discontinued; see *1995 Annual Book of ASTM Standards*, Vol 15.05. Replaced by Terminology E 1547.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

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 D 1505.

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.[7] 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.[8] 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.

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

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

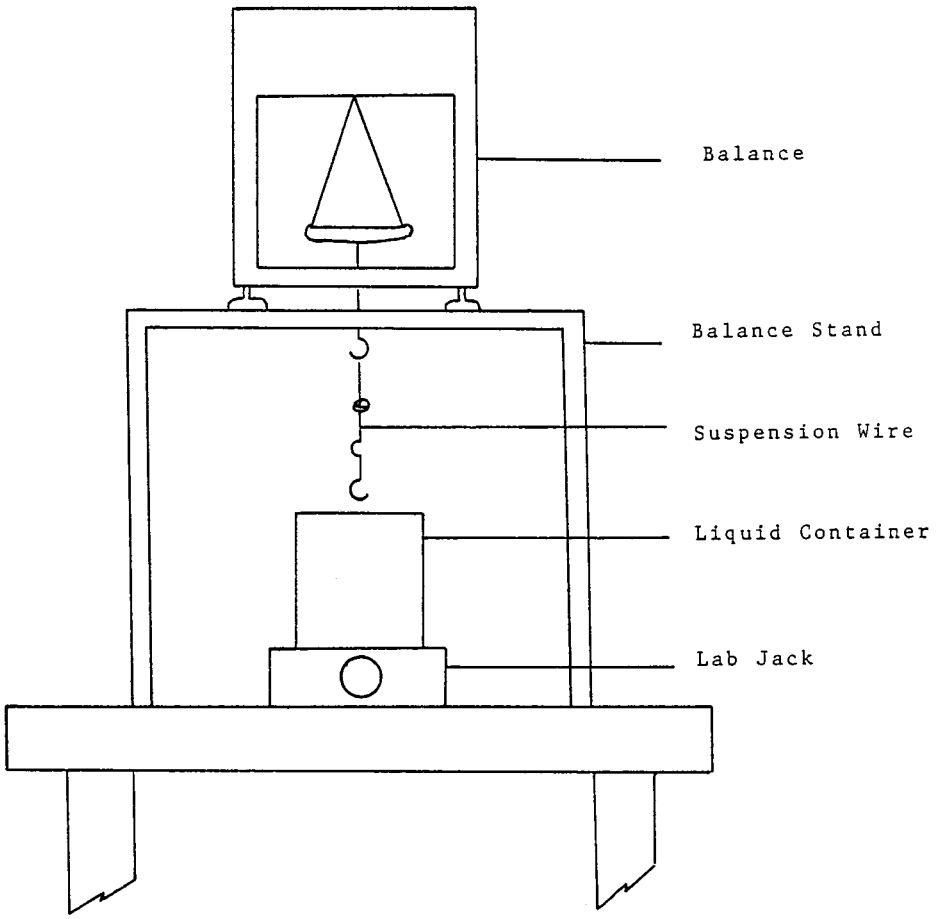

FIG. 1 Density Apparatus (Alternative)

7.3.3 *Hydrometer*, capable of reading liquid density.

7.4 *Procedure C*—Use the apparatus described in Test Method D 1505.

NOTE 1—Standard deionized or distilled water need not be measured, but can be taken as a value from standard tables.[9] 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 D 891, 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_l, 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 the specifications of the Committee on Analytical Reagents of

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

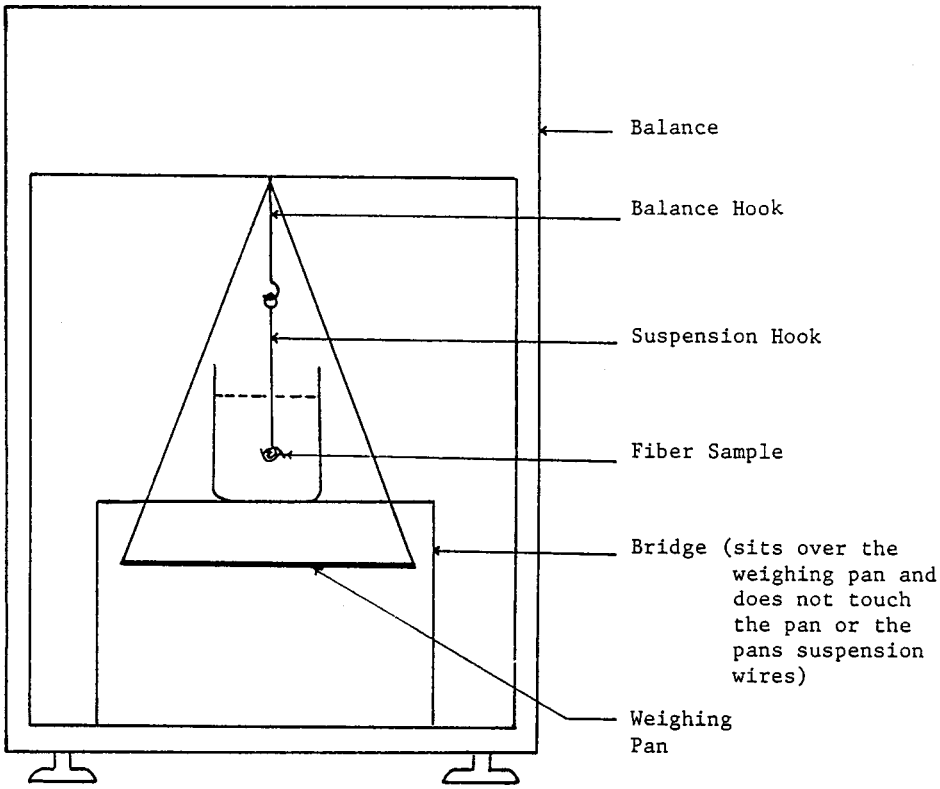

FIG. 2 Density Apparatus (Alternative)

the American Chemical Society where such specifications are available.[10]

8.1.1 *Water*, H_2O, (deionized or distilled and degassed), may contain a wetting agent such as glycerin or surfactant. This is the safest reagent found, and recommended for Procedure A.

NOTE 3—Reagents in 8.1.2-8.1.6 should be used if water is found to be unsatisfactory to accurately determine the density of the fiber. Other reagents are listed in order of known hazard or toxicity.

8.1.2 *Acetone (2-Propanone)*, CH_3CHOCH_3.

8.1.3 *Methanol (Methyl Alcohol)*, CH_3OH.

8.1.4 *o-dichlorobenzene*, CH_4Cl_2. (**Warning**—*o*-dichlorobenzene has been identified as toxic and an irritant.)

8.1.5 *Dibromomethane*, CH_2Br_2. (**Warning**—As of the approval date of this test method, dibromomethane was listed by the International Agency for Research on Cancer in Group 2C as "toxic.")

8.1.6 *Trichloroethylene*, $CHClCCl_2$. (**Warning**—As of the approval date of this test method, trichloroethylene was listed by the International Agency for Research on Cancer in Group 2D as a "cancer suspect agent" and mutagen.)

9. Hazards

9.1 This test method should be used only by laboratory workers with general training in the safe handling of chemicals. A source of useful information is *Prudent Practices in the Laboratory: Handling and Disposal of Chemicals*, National Academy Press, 1995, 448 pp., ISBN 0-309-05229-7. (**Warning**—In addition to other warnings, consult the appropriate material safety data sheet for each material used, including reagent materials and test specimen materials, for specific recommendations on safety and handling.)

10. Test Specimen

10.1 A minimum of three test specimens shall be tested for each sample.

10.2 The test specimen weight shall be a minimum of 0.5 g.

11. Calibration and Standardization

11.1 All measuring equipment shall have certified calibrations that are current at the time of use of the equipment. The calibration documentation shall be available for inspection.

12. Conditioning

12.1 Test Method D 5229/D 5229M may be used to determine equilibrium dryness of a fiber. In general, no special conditioning is needed for carbon fiber, less than 1 h at 100°C is needed for glass fibers, and approximately 4 h at 100°C is needed for aramid fibers.

12.2 Condition liquids to a test temperature, typically 23°C.

13. Procedure

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

13.1.1 *Equipment Assembly*:

13.1.1.1 The assembly of the apparatus is shown in Fig. 1 or Fig. 2. The balance stand must be firmly secured to a stable

[10] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

surface with the balance resting on the stand directly over the hole provided for the suspension system. Place the immersion fluid container on the laboratory jack directly under the suspension hook.

13.1.1.2 To prevent stray air currents between the bottom of the balance and the top of the stand, it is advisable to shield this area. If excessive vibration is observed while weighing, vibration damping pads must be used.

13.1.2 *Density Standard Calibration*—Fill the immersion fluid container ¾ to ⅞ full with freshly boiled water, which has reached equilibrium at room temperature. Place the container on a collapsed laboratory jack and zero the balance. Attach the suspension wire and weigh. Record as M_1, g. Raise the laboratory jack to the immersion point of the suspension wire and record the weight as M_2, g. Rinse the wire with acetone and let air dry. Attach the wire plus glass standard and weigh. Record as M_3, g. Again raise the jack to the immersion point and weigh. Record as M_4, g. Lower the jack, remove the standard plus wire, and rinse them with acetone to dry. Measure the temperature of the water to 0.1°C and record the water density at that temperature.

13.1.3 *Immersion Fluid Standardization*—Fill the clean and dry fluid container ¾ to ⅞full with liquid (water with surfactant, methanol, or 1,2 dichlorobenzene for example) and allow to come to temperature equilibrium. Proceed by weighing the suspension wire. Record as M_1, g. Raise the laboratory jack to the immersion point of the suspension wire and record the weight as M_2, g. Rinse the wire with acetone and let air dry. Attach the wire plus glass standard and weigh. Record as M_3, g. Again raise the jack to the immersion point and weigh. Record as M_4, g. Remove wire and standard and rinse with acetone. This step should be done at the beginning of each series of density determinations.

13.1.4 *Yarn Density*—Select a suitable representative length of yarn, weighing at least 0.15 g. Wrap the sample (the end of a small winding cone is suitable) and intertwine the ends of the fiber tow or yarn to prevent unraveling. Proceed by weighing the suspension wire. Record as M_1, g. Raise the laboratory jack to the immersion point of the suspension wire and record the weight as M_2, g. Rinse the wire with acetone and let air dry. Attach the sample to the wire and weigh. Record as M_3, g. Remove the wire and soak in the liquid and vacuum degas. Reattach the sample and wire to the balance. Again raise the jack to the immersion point and weigh. Record as M_4, g. Remove wire and standard and rinse with acetone.

13.2 *Procedure B—Sink Float Technique*:

13.2.1 In a suitable container, prepare a mixture of two miscible liquids that, when thoroughly mixed, will yield a liquid with a density less than the density of the fiber to be tested.

13.2.2 Cut approximately 150 mm of material to be tested and tie a loose overhand knot.

13.2.3 Put the sample into the solution prepared in 13.2.1.

13.2.4 Put the container with the sample and liquid into a vacuum desiccator and pull vacuum to approximately 10-mm Hg or until boiling of the solution begins. Hold vacuum for a minimum of 2 min or until all trapped air on the fiber surface is removed.

13.2.5 Remove the container from the vacuum desiccator and allow to equilibrate to room temperature. The sample should be near the bottom of the container.

13.2.6 Add small increments of the heavier liquid used to prepare the mixture in 8.2.1. After each addition, gently swirl the contents of the container. Continue adding increments of the heavy liquid until the fiber sample is suspended at an intermediate point.

13.2.7 Wait 5 min. If the fiber sinks, repeat 13.2.6. If the fiber rises, repeat 13.2.6, but with a light liquid. If the fiber remains stationary proceed to 13.2.8.

13.2.8 Measure the density of the liquid with a hydrometer that reads to the appropriate limits. For example, if the liquid has a density of 1.5 g/mL, use an hydrometer with a range from 1.4 to 1.6 g/mL. More accurate results can be obtained from hydrometers having a narrow range. Read the hydrometer to two decimal places and interpolate the third decimal place. The hydrometer reading is the density of the fiber.

14. Calculation (Procedure A)

14.1 *Density of Glass Standard,* g/mL:

$$\rho_s = (M_3 - M_1)\,\rho_w/((M_3 - M_1) - (M_4 - M_2)) \quad (4)$$

where: the liquid is water and the sample is the glass standard.

The density of the standard should remain consistent to ±0.0001 g/mL.

14.2 *Density of Immersion Liquid,* g/mL:

$$\rho_l = \rho_s\,((M_3 - M_1) - (M_4 - M_2))/(M_3 - M_1) \quad (5)$$

where: the liquid is the test liquid and the sample is the glass standard.

14.3 *Density of Fiber Sample,* g/mL:

$$\rho_f = (M_3 - M_1)\rho_l/((M_3 - M_1) - (M_4 - M_2)) \quad (6)$$

where: the liquid is the test liquid and the sample is the fiber.

15. Report

15.1 Report the following information:

15.1.1 Reporting of items that are beyond the control of a given test laboratory, such as material details shall be the responsibility of the requestor.

15.1.2 Complete identification of the material in accordance with Guide D 6308, including fiber type, surface treatment, and fiber manufacturer.

15.1.3 This test method (D 3800 and Procedures A-C).

15.1.4 Complete test parameters including test temperature in degrees Celsius and immersion liquid(s) used.

15.1.5 Each measured density and average, g/mL. A measure of the degree of variation in the density such as standard deviation.

16. Precision and Bias

16.1 *Precision*—The data required for the development of a precision and bias statement is not available for this test method. Committee D-30 is currently planning a round-robin test for this test method to determine precision.

17. Keywords

17.1 Archimedes method; buoyancy method; density; fiber density