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Standard Test Methods for Density Determination of Flax Fiber¹

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1. Scope

1.1 These test methods cover two procedures for determination of the density of flax fiber and are applicable to fibers of any length.

1.1.1 *Test Method A—Gas Pycnometry*—This is the more accurate method of density measurement and is preferred in cases in which specimen conditioning (oven drying) can be applied or outgassing effects can be mitigated by purge settings. The level of accuracy for the test methods can be assessed from the precision and bias tables. Outgassing effects can be assessed by performing a purge cycle, pressurization, and pressure stability check on the sample. If the pressure increases in an isolated sample chamber, outgassing may affect the accuracy of results. Oven dry specimens below 55 °C to equilibrium with a 0 % relative humidity (RH) environment or use longer purge methods, or both, to eliminate outgassing effects.

1.1.2 *Test Method B—Buoyancy (Archimedes) Method*—This is acceptable as an alternative to Test Method A in applications in which less accurate results are sufficient, as represented in the precision and bias tables. Test Method B is preferred in cases in which outgassing effects in Test Method A cannot be addressed by oven drying or purge settings and prevent Test Method A from producing valid results.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Sections 9 and 17.

1.4 *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 Recom-*

mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 *ASTM Standards:*²

D1577 Test Methods for Linear Density of Textile Fibers

D3800 Test Method for Density of High-Modulus Fibers

D3878 Terminology for Composite Materials

D6798 Terminology Relating to Flax and Linen

E12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases (Withdrawn 1996)³

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

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases (Withdrawn 2015)³

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology E12 defines terms relating to density. Terminology D6798 defines terms relating to flax and linen. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over other standards.

3.1.1 *impurity, n*—any material (such as, but not limited to, water, shive, or dirt) present in the sample that, if not removed or accounted for, would obfuscate the density measurement of the targeted material in the sample.

3.2 *Symbols:*

M_1	= weight of suspension wire in air, g.
M_2	= weight of suspension wire in liquid (to immersion point), g.
M_3	= weight of suspension wire plus item whose density is to be determined (in air), g.
M_4	= weight of suspension wire plus item whose density is to be determined (in liquid), g.

¹ These test methods are under the jurisdiction of ASTM Committee D13 on Textiles and are the direct responsibility of Subcommittee D13.17 on Flax and Linen.

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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 The last approved version of this historical standard is referenced on www.astm.org.

$M_3 - M_1$	= weight of item for density to be determined in air, g.
$M_4 - M_2$	= weight of item for density to be determined in liquid, g.
ρ_f	= density of fiber, g/cm ³ .
ρ_i	= density of the impurities, g/cm ³ .
ρ_{if}	= density of the combined fiber and impurities, g/cm ³ .
ρ_s	= density of standard, g/cm ³ .
ρ_l	= density of liquid, g/cm ³ .
ρ_{sur}	= density of surfactant, g/cm ³ .
ρ_w	= density of water, g/cm ³ .
P_1	= equilibrium pressure of the sample chamber after closing the input valve, kPa.
P_2	= equilibrium pressure of the sample and expansion volume chambers after opening the connection valve, kPa.
s	= standard deviation.
V_e	= volume of the calibrated expansion volume chamber, cm ³ .
V_f	= measured volume of the fiber specimen, cm ³ .
V_{min}	= equipment specific minimum volume of solid material required in each test to produce accurate results.
V_s	= volume of the calibrated sample chamber, cm ³ .
W_1	= weight of specimen before testing, g.
W_2	= weight of specimen after testing, g.
ΔW_g	= change in weight of the specimen during the test, g.
x_i	= mass of impurity as a percentage of the total mass of the measured fiber, g.

4. Summary of Test Methods

4.1 *General*—Test Method A is the more accurate method of density measurement in cases in which specimen conditioning (oven drying) can be followed or outgassing can be mitigated by purge settings. Outgassing, such as the release of water vapor from unconditioned fibers when exposed to a dry gas, will reduce the precision of Test Method A by introducing vapor pressure to the gas pressure being used for measurement. In cases in which outgassing cannot be addressed by oven drying the sample or increasing the purge cycle/time, the results from Test Method A are invalid. Test Method B can be used as an alternative to Test Method A in applications in which high precision is not required. Test Method B has a similar method and equipment setup to Test Method D3800, but has been augmented to provide more accurate results for flax fibers.

4.2 Test Method A—Pycnometry Method:

4.2.1 Sample volume is determined by the application of Boyle's Law, which states that the increase in volume of a confined gas results in a proportionate decrease in pressure. The apparatus consists of two chambers of known volume connected by a valve. One of the chambers, the calibrated sample chamber, is accessible for insertion of the test specimen and is connected to a source of high purity (at least 99.99 %) dry gas, such as nitrogen. The pressure in the sample chamber is increased to a predetermined pressure and this value, P_1 , is noted. The valve between the two chambers is then opened and

the second, lower pressure, P_2 , is again noted. The ratio of the pressure change P_1/P_2 , is directly related to the volume of the sample chamber displaced by the specimen. The difference between this volume and the geometric volume of the specimen is a measure of the sample volume.

4.2.2 The sample is weighed to determine the sample mass and the density of the sample is calculated by dividing the sample mass by the sample volume.

4.3 Test Method B—Buoyancy (Archimedes) Method:

4.3.1 The sample is weighed in air and weighed in certified soybean oil that will thoroughly wet the sample and is of a lower density.

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

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 can be used to determine fiber strength and modulus of a single fiber or bundle of fibers. These properties are based on load or modulus slope over an effective area. Fiber density, when used with the lineal mass of the specimen, can calculate an approximate, total effective area for the specimen.

5.3 In composite applications, fiber density is used as 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 *Removal of Impurities*—A bias will exist if impurities are not removed as the measured fiber density will be a combination of the average densities of the fiber and impurity. Eq 1 may be used to calculate the effect of the impurity on the density of the material.

$$\rho_{if} = \frac{(100 - x_i)\rho_f + x_i(\rho_i)}{100} \quad (1)$$

6.2 Test Method A:

6.2.1 The presence of components that can outgas during testing, such as water vapor or volatile organic compounds, interfere with the measurement of the gas pressure and, if not mitigated, can invalidate the results. Typically, oven drying specimens at a temperature higher than the density testing temperature or increasing the amount/time of purge cycles will sufficiently reduce the effect of outgassing in typical samples.

6.2.2 Changes in temperature can cause changes in volume. The equipment and specimens should be given sufficient time to reach a steady state condition.

6.3 Test Method B:

6.3.1 *Temperature*—The temperature of the certified soybean oil shall remain constant within a tolerance of $\pm 1^\circ\text{C}$, since the soybean oil density will change with temperature.

6.3.2 *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.

6.3.3 *Immersion Point*—The distance the sample is lowered into the soybean oil and overall liquid level should be the same throughout determinations for Procedure A. This may be done by putting a line for the desired soybean oil level on the outside of the container.

TEST METHOD A—GAS PYCNOMETRY

7. Apparatus

7.1 Test Method A:

7.1.1 Commercial pycnometer instruments are available from several manufacturers. The commercial instruments or custom laboratory setups should include, at minimum, the following:

7.1.1.1 *Calibrated sample volume chamber*—a chamber having a volume suitable to the sample size and calibrated to the nearest 0.1 cm³. The chamber shall be accessible to the operator such that a sample can be loaded into the chamber and then sealed for testing. The chamber should have a known volume (V_s cm³).

7.1.1.2 *Calibrated expansion volume chamber*; a chamber of known volume (V_e cm³) that is calibrated to the nearest 0.1 cm³.

7.1.1.3 *Pressure transducer*; capable of measuring 0 to 175 kPa with minimum volume displacement and linear within 0.1 %.

7.1.1.4 *Pressure relief valve*, used to avoid the overpressurization of the transducer.

7.1.1.5 *Filter*; prevents powder or dust from contaminating the transducer and valves.

7.1.1.6 *Input flow control valve*, controls the rate of pressurization.

7.1.1.7 *Output flow control valve*, controls the rate of depressurization.

7.1.1.8 *Connection valve*, connects the calibrated sample volume chamber with the calibrated expansion volume chamber.

7.1.1.9 *Nonporous calibration standard(s)*, object(s) of known volume that can be used in different configurations to calibrate the chambers for different volumes of sample.

7.1.1.10 *Digital meter*; used for reading the pressure to 0.007 kPa.

7.1.1.11 *Analytical balance*, capable of weighing to 0.001 g.

7.1.2 The minimum volume of solid material (V_{min}) required to produce accurate measurements is equipment specific and shall be known before conducting testing. The volume can be based on manufacturer recommendations or determined through testing. If done through testing, V_{min} would be the volume in which increasing the volume of the solid material being measured does not change the value of the results beyond what would fall within sample or standard deviation.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Nitrogen, dry and of 99.99 % purity.

NOTE 1—Helium is a commonly used gas in gas pycnometer systems and providing that it is dry and of 99.9 % purity, it can be used instead of nitrogen; however, in some cases, molecules may be sufficiently small enough to penetrate the cell walls of the fibers. This phenomenon can be identified when both of the following occur: P_1 decreases slowly after the sample chamber was sealed (helium entering the solid) and P_2 's value will rise following the initial drop after the valve between the two chambers is opened (helium being released from the solid). If helium penetration occurs, it is recommended that a lower pressure, longer equilibrium time, or nitrogen be used.

9. Hazards

9.1 Appropriate precautions should be taken when handling compressed gases.

10. Sampling, Test Specimens, and Test Units

10.1 Specimens should be chosen to represent the larger bulk sample from which it is taken.

10.2 For statistical purposes, a minimum of five specimens for each sample should be tested.

10.3 Tests should not use a volume less than V_{min} for each specimen. For the best results, the maximum amount of solid volume that can reasonably be packed into the sample chamber should be used for each specimen.

11. Calibration and Standardization

11.1 The apparatus should be calibrated following the manufacturer's recommendations for procedure and frequency. Because of the low packing density of fibers, alternate calibration routines may need to be selected or modified to improve precision when the solid volume of specimen in the chamber is lower than the manufacturer's recommended loading.

11.2 For highest precision results, perform a calibration routine before testing.

11.3 It is recommended that a leak check be done before a series of tests.

12. Conditioning

12.1 Before testing, fibers should be conditioned to equilibrium at 0 % relative humidity (RH) at the temperature the testing will occur. This will remove moisture that can outgas

⁴ *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.

during testing. If the sample is expected to be tested without conditioning, its moisture content should be included in the report and the sample should be evaluated for outgassing and, if required, outgassing mitigation methods used during testing.

12.2 Specimen temperature at the time of testing should be within ± 1 °C of the test temperature.

13. Procedure

13.1 Manufacturers of gas pycnometers may automate or provide specific instructions pertaining to loading/unloading, settings, and operation of their equipment. The procedures provided in this test method are intended to represent the minimum requirements, and users should ensure that their equipment specific procedures meet these minimum requirements.

13.2 Weigh the specimen immediately before testing and record the weight as W_1 g.

13.3 Load the sample chamber with the specimen and seal.

13.4 Vent the sample chamber to atmosphere and zero the pressure transducer. All pressure references in the procedure and calculations refer to gauge pressure.

13.5 Close all vents and the valve separating the sample chamber from the expansion chamber.

13.6 Perform a purge cycle to prepare the specimen before testing. It is recommended that the purge cycle be a series of pulses, a fixed purge time, or exposure to vacuum. For pulses, a minimum of nine pulses should be used and the system should pressurize to 20 kPa before venting and return to less than 3 kPa before starting the next pulse. For purge times, the sample should be exposed to a minimum of 2 min of continuous flow of the test gas. For vacuum purge systems, the sample should be exposed to a minimum of 5 min of vacuum before testing. Report the purge cycle and settings used.

13.7 Pressurize the sample chamber to the target test pressure. The target pressure selected should be between 105 and 125 kPa above ambient and remain consistent for all specimens.

13.8 Close the input valve to the sample chamber and wait a minimum of 10 s for stability. Record the pressure as P_1 kPa and the wait time in seconds. The wait time for stability should be consistent between all specimens.

NOTE 2—If the pressure decreases or increases outside of usual measurement flutter, the system may have a leak, the gas may be penetrating the solid, or outgassing may be an issue. Discontinue testing and consult the manufacturer's documentation regarding leaks, gas penetration, or modifying purge routines.

13.9 Open the valve between the sample chamber and the expansion volume and wait for stability using the same wait time as previous. Record the pressure as P_2 kPa.

13.10 Open the vent valve and allow the chambers to fall to 0 kPa (ambient).

13.11 Repeat 13.7 – 13.10 a minimum of six times to collect multiple determinations on the specimen.

13.12 Remove the specimen from the sample chamber.

13.13 Weigh the specimen and record the weight as W_2 g.

13.14 Repeat the entire procedure for each specimen.

14. Calculation or Interpretation of Results

14.1 For each specimen in the sample:

14.1.1 Calculate and record the weight change (if any) as follows:

$$\Delta W_g = W_2 - W_1 \quad (2)$$

14.1.1.1 Mass loss during testing may indicate outgassing and should be considered when determining the validity of the results.

14.1.2 Calculate the specimen volume for each of the runs (excluding the first three) as follows:

$$V_f = V_s + \frac{V_e}{1 - \left(\frac{P_1}{P_2}\right)} \quad (3)$$

where:

P_1 = Equilibrium pressure of the sample chamber after closing the input valve, kPa, and

P_2 = Equilibrium pressure of the sample and expansion volume chambers after opening the connection valve, kPa.

14.1.2.1 During the first three runs, the equipment may not have reached a steady state condition and small thermal differences between the chambers and gas may cause results with greater deviation than the remaining runs.

14.1.3 Calculate the specimen density using the average specimen volume and the final weight of the specimen as follows:

$$\rho_f = \frac{W_2}{V_f} \quad (4)$$

14.2 Calculate the sample average density from the specimen densities.

TEST METHOD B—BOUYANCY (ARCHIMEDES) METHOD

15. Apparatus

15.1 Bouyancy method kits can be commercially purchased for some balances. The kits or custom laboratory setups should include the comparable or better of the following:

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

15.1.2 *Balance*, analytical, capable of weighing to 0.0001 g (or 0.005 mg from Test Methods **D1577**).

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

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

15.1.5 *Density standard*, a solid piece of borosilicate glass (approximate density 2.2 g/mL) of known density to four significant figures as determined by water immersion (Terminology **E12**).