Standard Test Methods for
Constituent Content of Composite Materials[1]

This standard is issued under the fixed designation D 3171; 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 These test methods determine the constituent content of composite materials by one of two approaches. Method I physically removes the matrix by digestion or ignition by one of seven procedures, leaving the reinforcement essentially unaffected and thus allowing calculation of reinforcement or matrix content (by weight or volume) as well as percent void volume. Method II, applicable only to laminate materials of known fiber areal weight, calculates reinforcement or matrix content (by weight or volume) based on the measured thickness of the laminate. Method II is not applicable to the measurement of void volume.

1.1.1 These test methods are primarily intended for two-part composite material systems. However, special provisions can be made to extend these test methods to filled material systems with more than two constituents, though not all test results can be determined in every case.

1.1.2 The procedures contained within have been designed to be particularly effective for certain classes of polymer or metal matrices. The suggested applications are discussed in Section 4, as well as at the start of each procedure.

1.1.3 Method I assumes that the reinforcement is essentially unaffected by the digestion or ignition medium. A procedure for correction of the results for minor changes in the reinforcement is included. Procedures A through F are based on chemical removal of the matrix while Procedure G removes the matrix by igniting the matrix in a furnace.

1.1.4 Method II assumes that the fiber areal weight of the reinforcement material form is known or controlled to an acceptable tolerance. The presence of voids is not measured. Eq 9 and 10 assume zero void content to perform the calculation.

1.2 The SI units shown are considered 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 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:*
D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[2]
D 883 Terminology Relating to Plastics[2]
D 1505 Test Method for Density of Plastics by the Density Gradient Technique[2]
D 3878 Terminology of High-Modulus Reinforcing Fibers and Their Composites[3]
D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[3]
E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]
E 171 Specification for Standard Atmospheres for Conditioning and Testing Flexible Barrier Materials[5]
E 177 Practice for Use of Terms Precision and Bias in ASTM Test Methods[6]
E 1309 Guide for the Identification of Composite Materials in Computerized Material Property Databases[3]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to composite materials. Terminology D 883 defines terms relating to plastics. Terminology E 12 defines terms relating to specific gravity. Practice E 177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over other documents.

3.1.1 *fiber content, n*—the amount of fiber present in a composite or prepreg expressed either as percent by weight or percent by volume. This is sometimes stated as a fraction. If no fillers exist, this is equivalent to reinforcement content.

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[1] These test methods are under the jurisdiction of ASTM Committee D30 on Composite Materials and are the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.
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[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 15.03.
[4] Discontinued; see *1995 Annual Book of ASTM Standards*, Vol 15.05. Replaced by Terminology E 1547.
[5] *Annual Book of ASTM Standards*, Vol 15.09.
[6] *Annual Book of ASTM Standards*, Vol 14.02.

D 3878

3.1.2 *matrix content, n*—the amount of matrix present in a composite or prepreg expressed either as percent by weight or percent by volume. For polymer matrix composites this is resin content. **D 3878**

3.1.3 *reinforcement content, n*—the amount of nonmatrix material (fiber and filler) in a composite or prepreg expressed either as percent by weight or percent by volume. **D 3878**

3.1.4 *resin content, n*—See matrix content. **D 3878**

3.1.5 *void volume, n*—the volume in the specimen without mass, that is identified as neither matrix nor reinforcement. **D 3878**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *density, $\rho^{23°C}$*—the weight per unit volume measured in air, of the impermeable portion of a material at 23°C.

3.2.1.1 *Discussion*—The definition of specific gravity and density are essentially equivalent to the definitions of apparent specific gravity and apparent density in Terminology E 12, because no correction is made for buoyancy of the material in air. However, this difference is insignificant for most engineering purposes.

3.2.2 *specific gravity, $SG^{23°C}$*—the ratio of the weight in air of a unit volume of the impermeable portion of a material at 23°C referenced to the standard unit volume weight of water at 23°C.

3.3 *Symbols:*

A = area of the specimen.
A_r = calculated mass of one layer of reinforcement/unit area.
ρ_c = density of the composite specimen.
ρ_m = density of the cured matrix.
ρ_r = density of the reinforcement or fiber.
h = thickness of the specimen.
M_c = mass of the dry crucible or sintered glass filter.
M_{cr} = mass of the dry crucible or sintered glass filter with reinforcement residue.
M_i = initial mass of specimen before digestion or combustion.
M_f = final mass of specimen after digestion or combustion.
V_m = volume percent of matrix in specimen.
V_r = volume percent of reinforcement in the specimen.
V_v = void volume percent in the specimen.
W_m = weight percent of matrix in the specimen.
W_r = weight percent of reinforcement in the specimen.

4. Summary of Test Method

4.1 *Test Method I*—The matrix portion of a material specimen of known mass is removed in a hot liquid medium (for dissolution) or furnace (for combustion). When dissolving in a hot liquid medium, the remaining residue, containing the reinforcement, is then filtered, washed, dried, cooled, and weighed. The weight percent of the reinforcement is calculated, and from this value, and if densities of both the composite and the reinforcement are known, the volume percent is calculated. An additional calculation for void volume may be made if the density of the matrix is known or determined.

4.1.1 A correction for weight change of the reinforcement or retention of the matrix may be made (13.3 and 13.4), if this change is sufficiently reproducible under the conditions of the test and has the same value for the reinforcement or matrix alone as for the constituents in the composite.

4.1.1.1 *Procedure A*, for matrices such as epoxy resin, steel, copper, or others digestible by concentrated nitric acid.

NOTE 1—Many reinforcements are attacked by nitric acid. If reinforcement is attacked, an alternative method is recommended, depending on the matrix. See Annex A1.

4.1.1.2 *Procedure B*, for matrices such as epoxy, phenolic, polyamide, or thermoplastic resin, or others digestible by an aqueous mixture of sulfuric acid and hydrogen peroxide. See Annex A2.

4.1.1.3 *Procedure C*, for matrices such as epoxy resin and others digestible by a mixture of ethylene glycol and potassium hydroxide. See Annex A3.

NOTE 2—Procedure C is especially applicable to anhydride-cured epoxy systems containing aramid or carbon reinforcement.

4.1.1.4 *Procedure D*, for matrices such as aluminum, brass, or others digestible by sodium hydroxide solution. See Annex A4.

4.1.1.5 *Procedure E*, for matrices such as steel, titanium, copper, aluminum, or others digestible by hydrochloric acid. See Annex A5.

4.1.1.6 *Procedure F*, a version of Procedure A for microwave-aided heating. See Annex A6.

4.1.1.7 *Procedure G*, for reinforcements such as glass, or ceramic that are not affected by high-temperature environments, or reinforcements such as carbon where temperature is adequately controlled so that reinforcement does not char. See Annex A7.

4.2 *Test Method II*—The thickness of a relatively flat panel made with reinforcement of known and consistent areal weight is measured. By the thickness of the panel, the reinforcement and matrix content is calculated.

5. Significance and Use

5.1 A constituent content of a composite material must be known in order to analytically model the material properties (mechanical, physical, thermal, or electrical) of the composite which are affected by the reinforcement or matrix. Also, knowledge of the constituent content is required for evaluation of the quality of a fabricated material and the processes used during fabrication.

5.2 The void volume of a composite material may significantly affect some of its mechanical properties. Higher void volumes usually mean lower fatigue resistance, greater susceptibility to moisture penetration and weathering, and increased variation or scatter in strength properties. Knowledge of the void volume of a composite material is desirable as an indication of the quality of a composite.

5.3 Reinforcement content may be used to normalize mechanical properties affected by amount of reinforcement in the coupon.

6. Interferences

6.1 *Density of Constituents*—Calculation of the void volume assumes that reinforcement density and matrix density obtained on a lot or material basis are held in the laminate sample. There is a normal variation in reinforcement and matrix densities that is dependent on the constituent material. This assumption used by the void calculation equations is typically minor, changing the void calculation by less than 0.2 %. One indication of this variation is the possibility of obtaining a negative void volume in low-void volume composites. If procedural errors can be ruled out, then it is reasonable to believe that constituent density variation is responsible. Negative void content is a physical impossibility, but a possibility in these calculations. It is useful to report negative void contents to assess if constituent density values are incorrect or within a typical range of material variation. The negative void value then sets an upper bound on error of this test method for any material.

6.2 *Coupon Size*—Ability to estimate void content is also determined by coupon size and limitations of measuring apparatus. For example, with just limitations of the analytical balance (accurate to 0.2 mg), a coupon of 0.2 g with a void volume of 1.0 % would have an uncertainty of 10 % (reported void volume in the range of 0.9 to 1.1 %) on the void volume calculation as a result of possible balance error. A 1-g sample would have an uncertainty of 2 % in the void volume calculation (reported void volume in the range of 0.98 to 1.02 %) because of possible balance error for the same 1.0 % void volume.

6.3 *Error in Previous Measures*[7,8]—Ability to estimate void content is also determined by the accuracy of previous measures. Density measures of constituents and laminate have some limitations. Good measures of these properties should have an uncertainty of less than 0.0005 g/mL. For a typical carbon/epoxy laminate, uncertainty in the void volume because of the limitation of the constituent density measurement would be approximately 1 %.

6.4 *Mass Change of Reinforcement*—Fibers may lose mass by any of the techniques in Test Method I. This may be investigated by subjecting the reinforcement without matrix to the test conditions of the composite. Once the technique is established for a material, no significant changes are expected between samples unless the product or test conditions vary significantly.

6.5 *Residual Matrix Retained*—Matrix may be retained by any of the techniques of Test Method I. This may be investigated quantitatively by subjecting the matrix to the test conditions of the composite. Qualitatively, matrix appears as hardened pieces in the sample at the end of the test. Once the technique is established for a material, no significant changes are expected between samples unless the product or test conditions vary significantly.

6.6 *Micrometer Interface*—The thickness of the laminate continuously changes, particularly for surfaces with a release cloth or irregular surface. Test Method II measures the laminate at certain areas. The micrometer gives an indication of the thickness of the material at a point. The micrometer thickness measure is dependent on (*1*) variation in thickness of the panel, (*2*) type and diameter of thickness measuring device, (*3*) ability to hold panel perpendicular to the measurement device, and (*4*) sensitivity of the measurement device.

6.6.1 Ball micrometer geometry tends to give a thickness measure for Test Method II that more closely approximates fiber volume if there is a rough surface texture than a flat-faced micrometer that tends to overstate laminate thickness. For some material forms, such as open weaves, the ball geometry is not practical, so that a flat face micrometer is recommended.

7. Apparatus

7.1 *General Requirements*:

7.1.1 *Thermal Shock*—Laboratory equipment, which is subjected to nonambient temperatures (hot or cold), shall be of tempered glass or polytetrafluoroethylene (PTFE) materials.

7.1.2 *Post-Test Elemental Analysis*—If a post-test elemental analysis of the reinforcement residue is to be performed, laboratory equipment contacting the specimen shall be constructed of PTFE, and specimen cutting performed only by diamond-tipped tools.

7.2 *General Usage*:

7.2.1 *Analytical Balance*—The analytical balance shall be capable of reading to within ±0.1 mg.

7.2.2 *Laboratory Desiccator*.

7.3 *Test Method I*:

7.3.1 *Heating Equipment*:

7.3.1.1 *Constant Heat Source*—Heating mantle, hot plate, or controlled temperature bath, capable of heating material to the required temperature for the particular digestion medium and shall be capable of maintaining the temperature to ±10°C.

7.3.1.2 *Microwave*, capable of maintaining a constant power output. The microwave setup shall include an overpressure fail-safe device. Used exclusively for Procedure F (see Annex A6).

7.3.1.3 *Drying Oven*, air circulating, capable of maintaining a temperature of 100 ± 3°C or other target temperature within ±3°C.

7.3.1.4 *Muffle Furnace*, capable of maintaining a temperature where the polymer matrix is removed, but the reinforcement is unaffected. This is typically 600 ± 30°C minimum. Used exclusively for Procedure G (see Annex A7).

7.3.2 *Miscellaneous Equipment*:

7.3.2.1 *Sample Container*, beaker, sealed vessel, or flask of borosilicate glass or PTFE, minimum size 50 mL.

7.3.2.2 *Vacuum Source*, capable of 50-kPa (15-in. Hg) pressure.

7.3.2.3 *Static Control Device*, capable of eliminating static charge from beaker walls.

7.3.2.4 *Filtering Apparatus*, this may consist of a filtering flask with crucible holder and sintered glass filter or some other apparatus.

[7] "The interface region in glass fiber-reinforced epoxy resin composites: 1. Sample Preparation, Void Content and Interfacial Strength," *Composites*, 26, 1995 pp. 467–475.

[8] "A Comparison of Void Measurement Methods for Carbon/Epoxy Composites," U.S. Army Materials Technology Laboratory (US Army Research Laboratory) MTL TR91–13.

NOTE 3—Filter porosity should be sized to filter the smallest expected reinforcement size. This is particularly important for discontinuous reinforcements or for materials which have been ground before digestion (Note 4). If any doubt exists about the filter size selection, successively finer filters shall be evaluated with the material being tested until confidence is established in the filter size selected. Resin fillers or other constituent materials not destroyed by digestion may be retained both within the reinforcement and due to filter size. An estimation of this "trapped" matrix may be needed to adjust fiber content. Used in Procedures A-F (see Annex A1-Annex A7).

7.3.2.5 *Reflux Condenser*, capable of preventing loss of digestion medium by allowing volatilized vapors to recondense into the container. Used in Procedures A and C (see Annex A1 and Annex A3).

7.3.2.6 *Other Common Equipment*—Other generally available laboratory terms may be needed for the various procedures such as beakers, pipettes, watchglasses, and lint-free wipes.

7.4 *Items for Test Method II*:

7.4.1 *Thickness Measuring Device*—Micrometer or digital indicator (with 6-mm diameter ball/ball measuring ends). Device is capable of reaching the center of the laminate test specimen surface. Capable of reading to 0.001 mm.

7.4.2 *Calipers*, capable of reading length or width of the specimen to 0.1 % accuracy. Caliper reading length may vary depending on specimen size from 75 to over 1500 mm. Optical devices may be used for larger specimens.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[9] 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 *Digestion Reagents*—A suitable digestion reagent shall be selected that is compatible with the material system, test method, and apparatus. Read and understand the precautions listed in Section 9 before selecting an extraction reagent. Extraction reagents that have been found effective for many matrices include:

8.2.1 *Procedures A and F*:

8.2.1.1 *Nitric Acid*, HNO_3, 70 % aqueous.

8.2.2 *Procedure B*:

8.2.2.1 *Hydrogen Peroxide*, H_2O_2, 30 to 50 % aqueous. (**Warning**—As of the approval date of this standard, H_2O_2 was classified by the international agency for Research on Cancer as "unknown" (meaning the possibility of this material causing cancer in humans is unknown). There is limited evidence of a cancer risk associated with laboratory animals.)

8.2.2.2 *Sulfuric Acid*, H_2SO_4, 96 to 98 % aqueous.

8.2.3 *Procedure C*:

8.2.3.1 *Dimethylformamide* (DMF), $(CH_3)_2NCHO$. (**Warning**—As of the approval date of this standard, DMF was listed by the international agency for Research on Cancer in Group 2B as a "possible human carcinogen" and is considered a reproductive toxin by the National Toxicology Program. See a recent DMF material safety data sheet for more information.)

8.2.3.2 *Ethylene Glycol*, $HOCH_2CH_2OH$.

8.2.3.3 *Potassium Hydroxide*, KOH.

8.2.4 *Procedure D*:

8.2.4.1 *Sodium Hydroxide*, NaOH 40 to 80 % aqueous.

8.2.5 *Procedure E*:

8.2.5.1 *Hydrochloric Acid*, HCl 5-10 % aqueous.

8.3 *Washing Reagents*—A suitable washing reagent(s) shall be selected that is compatible with the material system under test and the apparatus. Read and understand the precautions listed in Section 9 before selecting a washing reagent. Washing reagents that have been found effective include:

8.3.1 *Acetone*, CH_3COCH_3.

8.3.2 *Water*, distilled or demineralized.

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, 449 pp., ISBN 0-309-05229-7. (**Warning**—In addition to other precautions, consult the appropriate material safety data sheet for each material used, including caustics, oxidizers, and composite materials for specific recommendations on safety and handling.) (**Warning**—In addition to other precautions, the digestion or combustion process should be performed under a suitably vented fume hood. Chemical processes and combustion processes shall not be performed in the same fume hood.)

9.2 Carry out all oxidations behind a safety shield or hood sash while using a ventilation hood.

9.3 Use standard procedures for handling acids and caustics.

9.4 Store materials by type. Acids especially need to have a containment area by acid type.

9.5 Store 30 to 50 % hydrogen peroxide in a freezer, or in a cool safety hood, in the original container with a vented cap. Do not allow contact with any organic material. Flush spills with copious amounts of water.

9.6 If incidental skin contact is made with any reagent (except water), wash with copious amounts of water.

9.7 No attempt should ever be made to purify hydrogen peroxide by distillation. Explosive decomposition is said to occur with boiling solution containing 65 % hydrogen peroxide.

9.8 Ensure that all digestion medium is removed before placing the sample in the oven.

9.9 Use of mixed digestion reagents, apparatus, or conditions not covered by these test methods may increase hazards as a result of splashing, toxic fumes, overpressurization, or explosion. If conditions from these test methods are altered, follow good laboratory practice for new test method development.

9.10 The combination of nitric acid and acetone is specifically cautioned against in the use of these test methods

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[9] *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.

(Procedures A and F) since it may form an explosive media. It is good practice to rinse thoroughly so that reactive constituents are not combined. Also reactive constituents shall not be placed in the same (waste) container.

9.11 Additional nonchemical hazards such as high heat and possibility of glass implosion under vacuum or other glass breakage exist in these test methods. Follow good laboratory practice to minimize risks.

10. Test Specimen

10.1 *Test Method I*:

10.1.1 *Sampling of Test Specimens*—The minimum number of recommended test specimens is three. Use the same specimen sized and cut in accordance with 10.1.2 and 10.1.3 for density (as determined by Test Methods D 792 or D 1505) and reinforcement volume/void content, except in Procedure G, in which the specimen may need additional dicing to maximize surface area for combustion.

10.1.2 *Test Specimen Geometry*—The specimen shall have a minimum mass of 0.5 g for constituent volume only, and 1.0 g if void content is to be obtained, and any shape not restricted by the apparatus. The specimen should contain a representative volume of the material being evaluated. The same specimen may be used for density and reinforcement volume determination.

10.1.3 *Specimen Cutting*—The specimen shall be free from oil, grease, or other foreign matter. If volume percent is to be calculated by measuring the density of the specimen, that cutting must not cause the specimen to fray or delaminate. An improperly cut specimen may trap air during submersion, giving a false density.

10.1.4 *Specimen Conditioning*—After cutting, the sample shall be conditioned in accordance with Section 12.

10.2 *Test Method II*:

10.2.1 *Sampling of Test Specimens*—The minimum number of recommended test specimens is one.

10.2.2 *Test Specimen Geometry*—The specimen may be the dimensions of the laminate panel. Minimum specimen surface area is 625 mm^2. The specimen shall be roughly rectangular in dimensions.

10.2.3 *Specimen Cutting*—The specimen shall be free from oil, grease, or other foreign matter.

10.2.4 *Specimen Conditioning*—After cutting, the sample shall be conditioned in accordance with Section 12.

11. Calibration and Standardization

11.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

12. Conditioning

12.1 Dry the test specimens to an equilibrium condition in accordance with Procedure D of Test Method D 5229/ D 5229M. If it may be shown that specimens are dry to within 1 % error, within a shorter period of time, the reduced drying time may be used.

12.2 Unless otherwise specified, conduct the tests at 23 ± 3°C and 50 ± 10 % RH. Test specimens for density within 10 min of removal from desiccator.

13. Procedure

13.1 *Method I*:

13.1.1 *General Procedures*

13.1.1.1 *Procedure A*—Perform Procedure A in accordance with Annex A1.

13.1.1.2 *Procedure B*—Perform Procedure B in accordance with Annex A2.

13.1.1.3 *Procedure C*—Perform Procedure C in accordance with Annex A3.

13.1.1.4 *Procedure D*—Perform Procedure D in accordance with Annex A4.

13.1.1.5 *Procedure E*—Perform Procedure E in accordance with Annex A5.

13.1.1.6 *Procedure F*—Perform Procedure F in accordance with Annex A6.

13.1.1.7 *Procedure G*—Perform Procedure G in accordance with Annex A7.

13.1.2 *Correction for Fiber Weight Change During Test*:

13.1.2.1 Weigh one blank consisting only of reinforcement. The blank mass shall roughly equal the mass of reinforcement in the test specimens to the nearest 0.0001 g.

13.1.2.2 Perform the full test procedure on blank.

13.1.2.3 The mass difference between the original blank mass and the mass after digestion or combustion divided by the original blank mass is the reinforcement fractional loss or gain.

13.1.2.4 This fractional loss (or gain), if reproducible, may be added (or subtracted) to the equation in 14.1.1.

13.1.2.5 Only fractional differences larger than 0.005 (0.5 %) shall be considered significant.

13.1.3 *Correction for Matrix Weight Change*:

13.1.3.1 Weigh one blank consisting only of cured matrix. The blank mass shall roughly equal the mass of matrix in the test specimens to the nearest 0.0001 g.

13.1.3.2 Perform the full test procedure on blank.

13.1.3.3 Since all matrix should be removed, the mass after digestion or combustion divided by the original blank mass is the matrix residue. This represents matrix material not removed by digestion or combustion.

13.1.3.4 This fractional gain, if reproducible, may be added to the equation in 14.1.3.

13.1.3.5 Only fractional differences larger than 0.005 (0.5 %) shall be considered significant.

13.2 *Method II*:

13.2.1 Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 or use the calculation shown in 14.2, if fiber volume percent or void percent, or both, are required.

13.2.2 Weigh each specimen to the nearest 0.0001 g.

13.2.3 Determine the thickness of the laminate with a micrometer or dial indicator (use a 6-mm diameter ball probe for uniformity, unless otherwise indicated (see 6.6). Measure thickness in at least ten locations. Take the average measurement to determine the specimen thickness.

13.2.4 Measure the specimen width and length with calipers or other suitable device in at least three locations. Take the average of the measurements to determine the length and width.

14. Calculation

14.1 *Method I*:

14.1.1 *Reinforcement Content, Weight Percent*—Calculate reinforcement content, in weight percent in accordance with Eq 1.

$$W_r = (M_f/M_i) \times 100 \tag{1}$$

where:
M_i = initial mass of the specimen, g and
M_f = final mass of the specimen after digestion or combustion, g.

NOTE 4—The mass of the fiber may be obtained by taking the mass of the crucible with reinforcement minus the crucible mass ($M_f = M_{cr} - M_c$).

14.1.2 *Reinforcement Content, Volume Percent*—Calculate reinforcement content, in volume percent, in accordance with Eq 2.

$$V_r = (M_f/M_i) \times 100 \times \rho_c/\rho_r \tag{2}$$

where:
ρ_r = density of the reinforcement, g/mL and
ρ_c = density of the specimen, g/mL.

14.1.3 *Matrix Content, Weight Percent*—Calculate matrix content, in weight percent, in accordance with Eq 3.

$$W_m = (M_i - M_f)/M_i \times 100 \tag{3}$$

14.1.4 *Matrix Content, Volume Percent*—Calculate matrix content, in volume percent, in accordance with Eq 4.

$$V_m = (M_i - M_f)/M_i \times \rho_c/\rho_m \tag{4}$$

where:
ρ_m = density of the matrix, g/mL.

14.1.5 *Void Volume*—Calculate void volume, in percent, in accordance with Eq 5.

$$V_v = 100 - (V_r + V_m) \tag{5}$$

NOTE 5—Negative values of V_v shall be reported. Values of void volume more negative than –0.2 % indicate a potential testing problem; input values and test procedure shall be investigated. See 6.1.

14.2 *Method II*:

14.2.1 *Specimen Density, g/mL*—Calculate specimen density in grams per millilitre with Eq 6 or use equivalent test methods such as Test Methods D 792 or Test Method D 1505.

$$\rho_c = M_i/(A \times h \times 0.001) \tag{6}$$

where:
M_i = mass of the specimen, g;
A = area of the specimen, m^2; and
h = thickness of the specimen, mm.

14.2.2 *Reinforcement Content, Weight Percent*—Calculate the reinforcement content in weight percent in accordance with Eq 7.

$$W_r = A_r \times N \times 0.01/(\rho_c \times h) \tag{7}$$

where:
A_r = weight of one sheet of reinforcement/unit area, g/m^2 and
N = number of sheets in the test specimen.

14.2.3 *Reinforcement Content, Volume Percent*—Calculate the reinforcement content in volume percent in accordance with Eq 8.

$$V_r = (A_r \times N)/(\rho_r \times h) \tag{8}$$

14.2.4 *Matrix Content, Weight Percent*—Calculate the matrix content in weight percent in accordance with Eq 9.

$$W_m = 100 - (A_r \times N \times 0.01/(\rho_c \times h) \tag{9}$$

14.2.5 *Matrix Content, Volume Percent*—Calculate the matrix content in volume percent in accordance with Eq 10.

$$V_m = W_m \times \rho_c/\rho_r \tag{10}$$

15. Report

15.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable:

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

15.1.2 Revision level or date of issue of this test method.

15.1.3 Actual test procedure used.

15.1.4 Any variations to this test method, anomalies noticed during testing, correction factors, or any equipment problems that occurred during testing.

15.1.5 Complete identification of the material tested including type, source, form, and dimensions (see Guide E 1309).

15.1.6 Fiber density values used, and their source.

15.1.7 Individual and average values and standard deviations of reinforcement and matrix weight and reinforcement and matrix volume percent.

15.1.8 Individual and average value and standard deviation for void percent.

15.1.9 Matrix density value used and source of value.

15.1.10 Drying times and oven temperature for both original specimen drying (before density determination and after digestion).

15.1.11 For Test Method II, measurement equipment used, individual and average thickness width and length measures.

16. Precision and Bias

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

16.2 *Bias*—Bias cannot be determined for this test method as no accepted reference standard exists.

17. Keywords

17.1 composite materials; fiber content; matrix content; reinforcement content; resin content; void volume

ANNEXES

(Mandatory Information)

A1. PROCEDURE A—MATRIX DIGESTION USING NITRIC ACID

Warning—Understand the hazards described in Section 9 before working with acids.

A1.1   Determine the density of each specimen in accordance with Test Methods D 792 or Test Method D 1505 if fiber volume percent or void percent, or both, are required.

A1.2   Weigh each specimen, M_i, to the nearest 0.0001 g.

NOTE A1.1—Specimens may require further dicing or grinding before digestion. After machining, specimens should be reweighed. Care should be taken that the diced or ground specimen represents original sample composition.

A1.3   Place each specimen in a separate flask or beaker containing at least 30 mL of 70 % nitric acid.

A1.4   Constant heating with heating mantle, hot plate, or controlled temperature bath is recommended. Temperature requirements depend on the system to be digested. Generally, temperatures should not exceed 80°C. (**Warning**—Reflux condensers are recommended if the temperature exceeds 40°C.)

A1.5   The maximum time for digestion should be 6 h. Shorter times are dependent on the composite system. The matrix is considered fully digested if no trace of the reinforcement/matrix laminate combination remains. Unfortunately, this is difficult to detect until the specimen has been removed from the digestion media.

A1.6   Filter the contents into tared or preweighed (M_c) sintered glass filters (or filter-lined crucibles) under a vacuum of 17 kPa or better. Wash the fiber three times with distilled water. An acetone wash may be used as the final wash after nitric acid is removed from the specimen to improve drying times.

A1.7   Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A1.8   Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A2. PROCEDURE B—MATRIX DIGESTION USING SULFURIC ACID/HYDROGEN PEROXIDE

Warning—Understand the hazards described in Section 9 before working with acids.

A2.1   Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A2.2   Weigh each specimen (M_i) to the nearest 0.0001 g.

A2.3   Place each specimen into a 100-mL [minimum] beaker.

A2.4   Add a minimum of 20-mL sulfuric acid. (**Warning**—Do not use less than 20-mL acid.) Place the beaker on a hot plate and heat until the mixture starts to fume.

A2.5   After the solution is dark (with no appreciable change in color for 5 min) add 50 or 30 % hydrogen peroxide down the side of the beaker to oxidize the matrix. (**Warning**—This procedure increases acid fumes. Wear rubber gloves, laboratory coat, and eye protection.) Typically, about 35 mL or more of hydrogen peroxide are added. The fibers float to the top of the solution, and the solution appears clear or color intensity dramatically fades.

A2.6   Remove the beaker from the hot plate and allow the solution to cool. Cooling may be facilitated by a water or ice bath. Any nondigested laminate should be clearly visible at this point. If the matrix is not digested, the solution may be filtered, then reintroduced into the beaker. Steps A2.4-A2.6 may be repeated.

A2.7   Filter the contents into tared or preweighed, M_c, sintered glass filters (or filter-lined crucibles) under a vacuum of 17 kPa or better. Wash the fibers three times with distilled water. An acetone wash may be used as the final wash after sulfuric acid and hydrogen peroxide is removed from the specimen to improve drying times.

A2.8   Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A2.9   Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A3. PROCEDURE C—MATRIX DIGESTION USING ETHYLENE GLYCOL/POTASSIUM HYDROXIDE

Warning—Understand the hazards described in Section 9 before working with caustics.

A3.1 Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A3.2 *Preparation of Reagent*—Prepare a 10 % solution of potassium hydroxide in ethylene glycol. (For example: Place 100 g of potassium hydroxide into a 1500-mL volumetric flask. Add 500 mL of ethylene glycol. Heat gently to dissolve the solid. Then add another 500 mL of ethylene glycol for a total volume of 1 L.) Label and store flask.

A3.3 Weigh each specimen, M_i, to the nearest 0.0001 g.

A3.4 Place each specimen into a separate 250-mL flask and add 100 mL of the potassium hydroxide-ethylene glycol solution to each flask.

A3.5 Connect the flask to a reflux condenser. Start the cooling water flow and adjust the temperature of the hot plate or heating mantle to bring the solution to a gentle boil.

A3.6 Boil until the matrix is completely digested. An indicator is when fibers separate and appear to float freely in the solution.

A3.7 Remove the beaker from the reflux condenser.

NOTE A3.1—Because potassium hydroxide will attack the glass filter, it is advisable to precondition the filter by pouring a small amount of potassium hydroxide-ethylene glycol solution onto the filter.

A3.8 Filter the contents into tared or preweighed (M_c) sintered glass filters under a vacuum of 17 kPa or better. Wash the fiber two times with dimethylformamide, and three additional times with distilled water. An acetone wash may be used as the final wash after potassium hydroxide-ethylene glycol solution is removed from the specimen to improve drying times.

A3.9 Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A3.10 Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A4. PROCEDURE D—MATRIX DIGESTION USING SODIUM HYDROXIDE

Warning—Understand the hazards described in Section 9 before working with caustics.

A4.1 Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A4.2 Weigh each specimen, M_i, to the nearest 0.0001 g.

A4.3 Place each specimen into a suitable beaker.

A4.4 Add 20 to 25 mL of 40 to 80 % sodium hydroxide solution to the beaker and heat specimen on a hot plate to 80°C maximum. Heat used depends on the required heat to digest the matrix for the system.

A4.5 When the matrix digestion is complete, remove the beaker from the hot plate and allow to cool.

A4.6 Filter the contents into tared or preweighed, M_c, sintered glass filters (or crucible with glass filter) under a vacuum of 50 kPa or better. Wash three times with distilled water. An acetone wash may be used as the final wash after the sodium hydroxide is removed from the specimen to improve drying times.

A4.7 Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A4.8 Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A5. PROCEDURE E—MATRIX DIGESTION USING HYDROCHLORIC ACID

Warning—Understand the hazards described in Section 9 before working with acids.

A5.1 Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A5.2 Weigh each specimen, M_i, to the nearest 0.0001 g.

A5.3 Place each specimen into a suitable beaker.

A5.4 Add 20 to 25 mL of 5 to 10 % hydrochloric acid

solution to the beaker and heat specimen on a hot plate. Heat used depends on required heat to digest the matrix for the system.

A5.5 When the matrix digestion is complete, remove the beaker from the hot plate and allow to cool.

A5.6 Filter the contents into tared or preweighed, M_c, sintered glass filters (or crucible with glass filter) under a vacuum of 17 kPa or better. Wash three times with distilled water. An acetone wash may be used as the final wash after the

sodium hydroxide is removed from the specimen to improve drying times.

A5.7   Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A5.8   Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A6. PROCEDURE F—MATRIX DIGESTION WITH NITRIC ACID IN A MICROWAVE OVEN

Warning—Understand the hazards described in Section 9 before working with acids.

A6.1   Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A6.2   Weigh each specimen, M_i, to the nearest 0.0001 g.

NOTE A6.1—Specimens may require further dicing or grinding before digestion. Specimens in the state to be digested should be reweighed after density determination in this case. Care should be taken that the diced or ground specimen represents original sample composition.

A6.3   Place each specimen in a specially designed pressure vessel, which encloses the solution and sample, but allows pressure release.[10] Add at least 30 mL of 70 % nitric acid solution.

A6.4   Place lid on the vessel and attach tubing so that nitric acid vapor will be off-gassed to release pressure. Set time and

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[10] A device that has been found suitable is available from CEM Corp., Matthews, NC.

power requirements of the microwave in accordance with the size of specimen and composite system.

A6.5   The maximum time for digestion should be 30 min. Shorter times are dependent on the system. The matrix is considered fully digested if no trace of the reinforcement/ matrix laminate combination remains. Unfortunately, this is difficult to detect until the specimen has been removed from the digestion media.

A6.6   Filter the contents into tared or pre-weighed, M_c, sintered glass filters (or filter-lined crucibles) under a vacuum of 17 kPa or better. Wash the fiber three times with distilled water. An acetone wash may be used as the final wash after nitric acid is removed from the specimen to improve drying times.

A6.7   Place the specimen in an oven at 100°C minimum until the sample is dried (approximately 1 h at 100°C).

A6.8   Cool the filter or crucible to room temperature in a desiccator. Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.

A7. PROCEDURE G—MATRIX BURNOFF IN A MUFFLE FURNACE

A7.1   Determine the density of each specimen in accordance with Test Methods D 792 or D 1505 if fiber volume percent or void percent, or both, are required.

A7.2   Weigh each specimen, M_i, to the nearest 0.0001 g.

NOTE A7.1—Specimens may require further dicing or grinding before burn off. Reweigh specimens after machining. Care should be taken that the diced or ground specimen represents original sample composition.

A7.3   Place each specimen in a desiccated preweighed crucible, M_c. The crucible should be cleaned by heating to 500 to 600°C or more in a muffle furnace and cooled in a desiccator before weighing.

A7.4   Place the crucible into a preheated muffle furnace at 500°C or lower depending on the composite system (a temperature below the temperature at which samples will spontaneously ignite). Heat to 565 ± 30°C, or other temperature

compatible with the composite system, that will burn off the matrix and leave the reinforcement.

NOTE A7.2—Materials placed in such a hot of environment may ignite. Specimens may be ignited under a controlled heat flame before entrance into a muffle furnace. If specimens are preignited, the muffle furnace may be set at the actual burn-off temperature.

A7.5   The maximum time for burn off should be 6 h. Shorter times are dependent on the system and specimen size. The matrix is considered combusted if no matrix/reinforcement block exists. Ash and reinforcement should be the only items visible.

A7.6   Place the specimen and crucible in a desiccator and allow to cool to room temperature.

A7.7   Weigh the specimen in its holder, M_{cr}, to the nearest 0.0001 g.