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

This standard is issued under the fixed designation D3171; 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 U.S. Department of Defense.

1. Scope

1.1 These test methods determine the constituent content of composite materials by one of two approaches. Test Method I physically removes the matrix by digestion or ignition or carbonization by one of eight 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. Test Method II, applicable only to laminate materials of known fiber areal weight, calculates reinforcement or matrix content (by weight or volume), and the cured ply thickness, based on the measured thickness of the laminate. Test 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 Test Method I assumes that the reinforcement is essentially unaffected by the digestion or ignition medium or carbonization. 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. Procedure H carbonizes the matrix in a furnace.

1.1.4 Test 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 15 and 16 assume zero void content to perform the calculation.

1.2 *Units*—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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* See Section 9 for additional information.

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

[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. Referenced Documents

2.1 *ASTM Standards:*[2]

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D883 Terminology Relating to Plastics
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
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 D883 defines terms relating to plastics. Terminology E12 defines terms relating to specific gravity. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 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. **D3878**~~

~~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. **D3878**~~

~~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. **D3878**~~

~~3.1.4 *resin content, n*—See matrix content. **D3878**~~

~~3.1.5 *void volume, n*—the volume in the specimen without mass, that is identified as neither matrix nor reinforcement. **D3878**~~

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Terminology E12 defines terms relating to specific gravity. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over other documents.

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.~~23 °C.

3.2.1.1 *Discussion*—
The ~~definition~~definitions of specific gravity and density are essentially equivalent to the definitions of apparent specific gravity and apparent density in Terminology E12, 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~~23 °C referenced to the standard unit volume weight of water at ~~23°C.~~23 °C.

3.3 *Symbols:*

A = area of the specimen.
A_r = calculated mass of one layer of reinforcement/unit area.
~~CR = carbonization ratio of the neat resin~~
CR_m = carbonization ratio of the neat resin.
ρ_c = density of the composite specimen.

[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.

ρ_m = density of the cured matrix.
ρ_r = density of the reinforcement or fiber.
h = thickness of the specimen.
h_p = cured ply thickness, mm.
~~m_c = mass of the dry crucible for neat resin carbonization~~
m_c = mass of the dry crucible for neat resin carbonization.
~~m_{cr} = mass of the dry crucible with carbonized neat resin~~
m_{cr} = mass of the dry crucible with carbonized neat resin.
~~m_d = final mass of the neat resin residue after carbonization~~
m_d = final mass of the neat resin residue after carbonization.
~~m_i = initial mass of the neat resin specimen~~
m_i = initial mass of the neat resin specimen.
M = mass 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_d = final residue mass of the composite specimen~~
M_d = final residue mass of the composite specimen.
~~M_i = initial mass of specimen before digestion or combustion.~~
M_i = initial mass of composite specimen before digestion, combustion, or carbonization.
~~M_f = final mass of specimen after digestion or combustion.~~
M_f = final mass of composite specimen after digestion, combustion, or carbonization.
~~M_m = mass of the resin matrix in the composite specimen~~
M_m = mass of the resin matrix in the composite specimen.
N_p = number of plies in the laminate.
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 (13.1.2 and 13.1.3), 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.

4.1.1.8 *Procedure H,* for any reinforcements, particularly carbon, that are not affected at high temperature in a nitrogen atmosphere, and any resin matrix systems. The correction for the retention of the matrix (13.1.3) is not necessary for this procedure. See Annex A8.

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, 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~~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*[4,5]—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/cm^3. 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

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

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

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.~~±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~~3 °C or other target temperature within ~~±3°C.~~±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~~30 °C minimum. Used exclusively for Procedure G (see Annex A7).

7.3.1.5 *Nitrogen-Purging Furnace,* capable of maintaining a temperature where the polymer matrix is carbonized under nitrogen environment, but the reinforcement is unaffected. This is typically 560 ± ~~40°C~~40 °C minimum. Used exclusively for Procedure H (see Annex A8).

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 (375-mm~~50 kPa (375 mm 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.

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~~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.[6] 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,* $HOCH_2CH_2OH$.

[6] *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.

8.2.3.3 *Solid 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 %~~ 5 to 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 (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 D792 or D1505) 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~~625 mm^2 on one laminate face. The specimen shall be roughly cylindrical or cuboid in shape.

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 D5229/D5229M. 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 ± ~~5°C~~5 °C and less than 65 % RH. Test specimens for density within ~~10 min~~ 10 min of removal from desiccator.

13. Procedure

13.1 *Test 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.

13.1.1.8 *Procedure H*—Perform Procedure H in accordance with Annex A8.

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 or 14.2.3.

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 *Test Method II:*

13.2.1 Weigh each specimen to the nearest 0.0001 g.

13.2.2 Determine the density of each specimen in accordance with Test Methods D792 or D1505, or use the calculation shown in 14.3~~,~~ if fiber volume percent or void percent, or both, are required. The preconditioning for D792 or D1505 specimens is to be in accordance with D3171 Section 12.

13.2.3 A micrometer with a 4 to ~~7~~8 mm ~~[0.16~~(0.16 to ~~0.28 in.]~~0.32 in.) nominal diameter ball interface shall be used to measure the specimen thickness when at least one surface is irregular (such as the bag-side of a laminate). A micrometer with a 4 to ~~7~~8 mm ~~[0.16~~(0.16 to ~~0.28 in.]~~0.32 in.) nominal diameter ball interface or with a flat anvil interface shall be used to measure the specimen thickness when both surfaces are smooth (such as tooled surfaces). Measure thickness in at least ten locations. Take the average measurement to determine the specimen thickness.

13.2.4 A micrometer or caliper, with a flat anvil interface, shall be used to measure the length and the width of the specimen. The accuracy of the instruments shall be suitable for reading to within 1 % of the sample dimensions. For typical specimen geometries, an instrument with an accuracy of ±0.0025 mm ~~[±0.0001 in.]~~(±0.0001 in.) is adequate for the thickness measurement, while an instrument with an accuracy of ±0.025 mm ~~[±0.001 in.]~~(±0.001 in.) is adequate for the length and the width measurement. Measure the specimen ~~width~~length and ~~length~~width in at least three locations. Take the average of the measurements to determine the length and width.

14. Calculation

14.1 *Test Method I, Procedures A through G:*

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

$$W_r = (M_f/M_i) \times 100 \quad (1)$$

where:

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

M_i = initial mass of the composite specimen before digestion or combustion, g and
M_f = final mass of the composite 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.

$$V_r = (M_f/M_i) \times 100 \times \rho_c/\rho_r \quad (2)$$

where:

ρ_r = density of the reinforcement, g/cm^3 and
ρ_c = density of the specimen, g/cm^3.

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

$$W_m = (M_i - M_f)/M_i \times 100 \quad (3)$$

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

$$V_m = (M_i - M_f)/M_i \times \rho_c/\rho_m \times 100 \quad (4)$$

where:

ρ_m = density of the matrix, g/cm^3.

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

$$V_v = 100 - (V_r + V_m) \quad (5)$$

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 *Test Method I, Procedure H:*

14.2.1 *Carbonization Ratio of the Neat Resin*—Calculate the carbonization ratio of the neat ~~resin,~~ resin sample, ~~CR,~~ CR_m, in accordance with Eq 6:

~~$$CR = m_d/m_i \quad (6)$$~~

$$CR_m = m_d/m_i \quad (6)$$

where:

m_d = final mass of the neat resin residue after carbonization, g, and
~~m_i = initial mass of the neat resin specimen, g~~
m_i = initial mass of the neat resin specimen, g.

NOTE 6—The mass of the neat resin residue after carbonization may be obtained by taking the mass of the crucible with residue minus the crucible mass ($m_d = m_{cr} - m_c$). If there is no residue from neat resin specimen after carbonization, CR_m is equal to zero since m_d is equal to zero.

14.2.2 *Mass of the Resin Matrix in the Composite Specimen*—Calculate the mass of the resin matrix in the composite specimen, M_m, in grams, in accordance with Eq 7:

$$M_m = (M_i - M_d)(1 - CR) \quad (7)$$

$$M_m = (M_i - M_d)(1 - CR_m) \quad (7)$$

where:

M_i = initial mass of the composite specimen, g, and
M_d = final residue mass of the composite specimen, g

M_i = initial mass of the composite specimen before carbonization, g, and
M_d = final residue mass of the composite specimen, g.

NOTE 7—The mass of the composite specimen residue after carbonization may be obtained by taking the mass of the crucible with residue minus the crucible mass ($M_d = M_{cr} – M_c$). "*M*" represents any mass associated with fiber reinforced composite specimens, while "*m*" the mass related to neat resin specimens.

14.2.3 *Reinforcement Content, Weight Percent*—Calculate reinforcement content in the composite specimen, in weight percent, in accordance with Eq 8:

$$W_r = (M_i - M_m)/M_i \times 100$$
$$= (M_d - M_i \times CR)/(M_i \times (1 - CR)) \times 100 \quad (8)$$

$$W_r = (M_i - M_m)/M_i \times 100$$
$$= (M_d - M_i \times CR_m)/(M_i \times (1 - CR_m)) \times 100 \quad (8)$$

14.2.4 *Reinforcement Content, Volume Percent*—Calculate reinforcement content, in volume percent, in accordance with Eq 9.

$$V_r = ((M_i - M_m)/M_i) \times 100 \times \rho_c/\rho_r \quad (9)$$

where:

ρ_r = density of the reinforcement, g/cm^3 and
ρ_c = density of the composite specimen, g/cm^3.

14.2.5 *Matrix Content, Weight Percent*—Calculate matrix content, in weight percent, in accordance with Eq 10.

$$W_m = M_m/M_i \times 100 \quad (10)$$

14.2.6 *Matrix Content, Volume Percent*—Calculate matrix content, in volume percent, in accordance with Eq 11.

$$V_m = (M_m/M_i) \times 100 \times \rho_c/\rho_m \quad (11)$$

where:

ρ_m = density of the matrix, g/cm^3.

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

NOTE 8—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.

14.3 *Test Method II:*

14.3.1 *Specimen Density, g/cm^3*—Calculate specimen density in grams per cubic centimeter with Eq 12 or use equivalent test methods such as Test Methods D792 or Test Method D1505.

$$\rho_c = M_i/(A \times h \times 1000) \quad (12)$$

$$\rho_c = M/(A \times h \times 1000) \quad (12)$$