Designation: D3529 – 16

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

This standard is issued under the fixed designation D3529; 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 This test method covers the determination of the fiber content, fiber areal weight, matrix solids content and matrix content of composite material prepregs. Optionally, the matrix solids content can also be determined after a volatiles content has been established. Volatiles content, if appropriate and required, is determined by means of Test Method D3530.

1.2 Procedure A of this test method applies to composite prepreg of primarily thermosetting matrices that can be extracted in organic solvent. The reinforcement and filler must be substantially insoluble in the selected extraction reagent. This procedure may also be used for the same purposes to extract other matrix material types.

1.3 Procedure B of this test method uses ignition loss of a composite prepreg matrix and applies to organic matrix composite systems containing reinforcing fibers that do not change mass when exposed to the matrix combustion method.

1.4 This test method assumes a two-part material system (plus volatiles) and does not distinguish between hybrid reinforcements or matrices. Use with hybrid composites is limited to determination of total reinforcement or total matrix content.

1.5 Matrix solids determination for Procedures A or B uses Test Method D3530 to determine volatiles content.

1.6 Alternate techniques for determining constituent content include Test Methods C613 (resin content by Soxhlet extraction) and D3171 (used principally for consolidated laminates).

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

1.8 *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.* Specific precautionary information is given in Sections 8 and 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C613 Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction
D883 Terminology Relating to Plastics
D3171 Test Methods for Constituent Content of Composite Materials
D3530 Test Method for Volatiles Content of Composite Material Prepreg
D3878 Terminology for Composite Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

2.2 *NFPA Standard:*

NFPA 86 Standard for Ovens and Furnaces[3]

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Practice E177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the 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, that is, fiber volume fraction. See Terminology 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. See Terminology D3878.

3.1.3 *prepreg, n*—the admixture of fibrous reinforcement and polymeric matrix used to fabricate composite materials. Its form may be sheet, tape, or tow. For thermosetting resins, it has

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02269-9101.

been partially cured to a controlled viscosity called "B stage". See Terminology D3878.

3.1.4 *reinforcement content, n*—the amount of reinforcement present in a composite or prepreg expressed either as percent by weight or percent by volume.

3.1.4.1 *Discussion*—If fiber is the sole reinforcement, then fiber content is equal to reinforcement content. See Terminology D3878.

3.1.5 *resin content, n*—see *matrix content*. See Terminology D3878.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dry resin content, n*—a prepreg resin content calculated by subtracting the average mass loss due to volatiles from the initial test mass.

3.2.2 *fiber areal weight, n*—the mass per unit area of the fiberous reinforcement of a composite material.

3.2.3 *matrix solids content, n*—see *dry resin content*.

3.2.4 *wet resin content, n*—a prepreg resin content that includes any volatiles present in the uncured resin. This term is used interchangeably with matrix content and resin content in this test method.

3.2.5 *volatiles content, n*—the amount of volatiles present in a prepreg expressed as percent by weight.

3.3 *Symbols:*

3.3.1 A—specimen area, m^2.

3.3.2 FAW—fiber areal weight; the fiber mass, W_f, divided by the specimen area, A, in g/m^2.

3.3.3 FC—fiber content of specimen, weight percent.

3.3.4 M_c—mass of container, g.

3.3.5 MC—matrix content of specimen, weight percent.

3.3.6 M_f—final mass of specimen and container at end of test, g.

3.3.7 M_I—initial mass of the specimen, g.

3.3.8 MS—matrix solids content of specimen, weight percent.

3.3.9 V_c—average volatiles content test result from Test Method D3530, weight percent.

4. Summary of Test Methods

4.1 Prepreg is sampled and specimens of a specific area are weighed and the matrix removed by appropriate procedure. For Procedure A, the specimen is subjected to suitable solvent that affects the matrix (but not the reinforcement) until the matrix is dissolved and removed. The remaining reinforcement is then dried and weighed. For Procedure B, the specimen is placed in a muffle furnace for a specific time and at a specific temperature (at which the reinforcement remains essentially unchanged) until the matrix can be entirely removed as ash residue. The remaining reinforcement is weighed. For either procedure the change in mass is expressed, both matrix content and fiber content, as a percentage of the specimen initial mass.

4.2 When matrix solids content is requested, adjacent samples are tested for volatiles content according to Test Method D3530. The average mass loss due to volatiles is subtracted from the average resin content result and the result expressed as a percentage of initial specimen mass as matrix solids content. The matrix solids content is applicable mainly to thermosetting matrices.

5. Significance and Use

5.1 This test method can be used to obtain the fiber content, fiber areal weight, matrix content or matrix solids content. Knowledge of these results is useful in specifying material systems and in developing optimum manufacturing processes. Where volatiles content is consistent and not critical to process, matrix content shall be used.

5.2 This test method is limited to reinforcement constituents that do not change mass upon exposure to the matrix removal procedure and limited to matrix constituents that are fully removed by the matrix removal procedure. In the event of experiencing either limitation, an agreement should be reached with the end-user as to how to proceed.

6. Interferences

6.1 *Procedure A:*

6.1.1 *Extent of Cure in Thermosetting Systems*—In Procedure A, the efficiency of matrix dissolution for thermosetting matrix materials is directly related to the extent of cure of the resin system. A resin that has started to cross link (such as B-staged resins) will be increasingly more difficult to extract as the cure advances. If Procedure A is ineffective in removing the matrix, and Procedure B is inappropriate for the fiber, then another test method, such as one of the methods of Test Method D3171, should be consulted.

6.1.2 *Reagent Selection*—The proper reagent, in a suitable quantity, must be selected for the constituents under test. The reagents listed in Section 8 are provided for consideration, particularly with regard to thermosetting materials, but cannot be assured to perform well on all material systems within the scope of this test method.

6.1.3 *Ability of Solvent to Reach Matrix*—Solvents are more efficient if the material is spread as a film, rather than a solid mass. It is recommended that the sample be consolidated as little as possible before introduction into the solvent. A solvent that is satisfactory for a given form may not be adequate with the same material of a different form.

6.1.4 *Solvent Strength*—Solvent strength, or the ability to rapidly dissolve the matrix, may be enhanced by increasing the amount of solvent, increasing agitation, increasing temperature, or by placing the sample under vacuum. These are offered as physical alternatives to use of more hazardous solvents, or combined solvent systems.

6.2 *Procedure B:*

6.2.1 *Applicability of Ignition Loss Approach*—In Procedure B a reinforced resin matrix composite specimen will be exposed to a temperature in excess of 500°C in an air environment for up to 6 h. This test method does not provide a measure of resin content for samples containing reinforcing materials that lose weight under the conditions of the test or contain resins or fillers that do not decompose to volatile materials released by ignition.

6.2.2 *Length of Test*—The sample type and configuration will determine the length of test exposure. The products of matrix combustion will disappear more readily in configurations having a large surface area of resin matrix exposure to the oxidizing atmosphere.

6.2.3 *Pre-Ignition*—Provision is made in this standard for pre-ignition of the specimen and crucible using a Bunsen flame. This step may be helpful to flash-off volatiles and initiation of the burning process, but care should be taken to prevent mechanical loss of sample residue.

6.2.4 *Residual Resin Combustion Products*—Fiber residue should be carefully examined for the presence of adherent resin combustion product before determining the final fiber mass (W_f). *If significant amounts of resin product remain on fiber ("producing a false indication of fiber content") surfaces, the test method should be repeated. Care should be taken during this process to assure that no physical loss in fiber content occurs.*

7. Apparatus

7.1 *Cutting (Grooved) Template or Die*—The specimen area shall be produced with either a cutting templates or a cutting die. The cutting template or die shall be designed to produce the nominal specimen area and shall be capable of limiting the error in area to no more than ±2 %.

NOTE 1—Attainment of 2 % area accuracy with a cutting template and a single-edge blade requires that average cut be made within 0.4 mm of the nominal line. A cutting die is preferred over a cutting template and is typically capable of providing a cut area error of less than 0.05 %.

7.2 *Analytical Balance*—The analytical balance shall be capable of reading to within ±1 mg.

7.3 *Timer,* accurate to nearest minute.

7.4 *Procedure A:*

7.4.1 *Fume Exhaust Hood,*

7.4.2 *Containers (Beakers, Flasks)*—borosilicate or suitable plastic that is not dissolved by solvent. The container shall be of a suitable volume for the sample and solvent (a minimum of 250 mL is suggested). Other sizes of container may be required depending on sample size, and amount of solvent needed to complete the dissolution process.

7.4.3 *Fritted Glass Crucibles or Sample Holder*—The fritted glass crucible filter shall be of a suitable porosity to allow dissolved matrix to pass while retaining reinforcement.

7.4.4 *Agitator*—Mechanical shaker or ultrasonic bath. Minimum shaker speed recommended is 100 agitations or revolutions/minute. Minimum frequency for ultrasonic bath recommended is 40 kHz.

7.4.5 *Hot Plate*—The hot plate shall have adjustable controls suitable for heating the solvent to its boiling point and be capable of controlling the temperature to within ±15°C.

7.4.6 *Vacuum Pump*—Capable of delivering vacuum of at least 50 kPa.

7.5 *Procedure B:*

7.5.1 *Crucible*—Porcelain, or equivalent properties, with a capacity sufficient to completely contain the specimen.

7.5.2 *Electric Furnace*—Muffle design capable of maintaining 565°C +/- 30°C for burn-out of the resin matrix.

7.5.3 *Laboratory Desiccator*—Design for pre- and post-test conditioning of the specimen and crucible.

7.5.4 *Bunsen Flame*—A source of a controlled heat flame for pre-ignition of the specimen.

7.5.5 *Laboratory Safety Equipment*—Heatproof gloves and protective eyewear are specifically recommended. Laboratory exhaust fan and/or ventilation system should be available for use.

8. Reagents

8.1 *Purity of Reagents*—As a minimum, a technical-grade reagent is required to provide accurate results. A reagent-grade reagent will be used when resolving disputes or performing subsequent analysis of extract or residue. Unless otherwise indicated, the reagent shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4]

8.2 *Matrix Dissolution Solvents*—A suitable solvent 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 matrix dissolution solvent. Matrix dissolution solvents found effective for many thermosetting matrices include the following:

8.2.1 *Acetone (2-Propanone)*, CH_3COCH_3.

8.2.2 *1,2 Methyl Pyrrolidinone (NMP)*, $CH_3NOC_3H_6$.

8.2.3 *Methyl Ethyl Ketone (MEK, 2-Butanone)*, $CH_3COC_2H_5$.

8.2.4 *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 3 as "not classifiable as to its carcinogenicity to humans" and is considered a reproductive toxin by the National Toxicology Program. See a recent DMF safety data sheet for more information.)

8.2.5 *Dichloromethane (MECL, methylene chloride)*, CH_2Cl_2. (**Warning—**As of the approval date of this standard, MECL was listed by the International Agency for Research on Cancer in Group 2B as a "possible human carcinogen.")

8.2.6 *Methyl Isobutyl Ketone (MIBK, 4-Methyl-2-pentanone)*, $(CH_3)_2CHCH_2\ COCH_3$.

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 given in Footnote 5.[5] (**Warning—**In addition to other precautions, consult the appropriate material safety data sheet for each material used, including reagent materials and test specimen materials, for specific recommendations on safety and handling.)

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[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.

[5] *Prudent Practices in the Laboratory: Handling and Disposal of Chemicals*, National Academy Press, 1995.

(**Warning—**In addition to other precautions, the extraction and filtering processes should be performed under a suitable vented chemical fume hood.)

(**Warning—**In addition to other precautions, materials that have been exposed to potentially toxic or flammable reagents should be air-dried under a hood before being subsequently oven-dried to eliminate a buildup of a potentially dangerous concentration of vapor in the drying oven. NFPA 86 provides guidelines on amount of flammable materials that may be safely placed in an oven.)

9.2 Use of mixed matrix dissolution solvents with different boiling points are not covered by this test method. (**Warning—**In addition to other precautions, do not use mixed solvents without knowing how the mixed solvent will react under test conditions, which includes drying.)

10. Test Specimens

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

10.2 The specimen size shall be a minimum of 0.0064 m^2 (of nominal size 80 mm x 80 mm). Minimum mass of the specimen shall be 1 g. The area shall include, at a minimum, at least one repeating unit of any textile pattern. If the minimum area provides an insufficient mass or inadequate textile repeating unit the specimen size shall be increased as required.

10.3 When matrix solids content is requested, additional specimens shall be obtained at the same time from adjacent areas for volatiles content according to Test Method D3530. Specimens shall have a minimum total area of 0.0016 m^2.

11. Calibration and Standardization

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

12. Conditioning

12.1 Store composite prepreg with thermosetting resins at low temperatures as recommended by the manufacturer (typically −18°C). Allow sealed packages of material to warm as recommended by manufacturer or controlling specification before seal is opened to ensure that the material does not absorb moisture from the atmosphere.

12.2 To avoid loss of volatile materials, minimize unsealed exposure of the material to ambient temperature. The material may stage (see Section 6) or lose volatiles, producing a false indication of matrix solids content.

12.3 In Procedure B the burn-out crucible should be cleaned by heating to 500°C to 600°C in a muffle furnace and cooled in a desiccator.

13. Procedures

13.1 *Procedure A:* Fiber Content, Fiber Areal Weight and Matrix Content by Solvent Dissolution.

13.1.1 Record the nominal area produced by the cutting template or die as A, in m^2.

13.1.2 Weigh each specimen on an analytical balance to 0.1 mg. Record the mass of each specimen as M_i, in g.

13.1.3 Place each specimen in a separate container using a minimum of 100 mL of solvent for each specimen.

13.1.4 Specimens shall be in the solvent at the steady state condition of the test for a minimum of three minutes. The steady state condition may include one or more of the following:

13.1.4.1 *Agitation*—May be used to speed dissolution of resin into the solvent. When used, a minimum shaker agitation of 60 cycles/minute or a minimum ultrasonic frequency 40 kHz is recommended.

13.1.4.2 *Pressure*—Vacuum may be used to lower solvent pressure and initiate boiling at lower temperatures. This is useful for solvents with high vapor pressure such as MEK or MECL. When used, a vacuum below 50 kPa is recommended.

13.1.4.3 *Temperature*—A raised temperature (not to exceed the boiling point of the solvent) may be used to increase solvent efficiency and reduce resin dissolution times.

13.1.5 Decant solution.

13.1.6 Repeat 13.1.4 and 13.1.5 at least one additional time.

NOTE 2—**Precaution:** A change of solvents or conditions may be made. Also a rinse solvent may be used to make the sample easier to dry. When changing solvents, be sure to assess solvent compatibility and also additional hazards when drying the material.

13.1.7 Place the sample in a weighed, fritted glass crucible of recorded mass, M_c in g. Dry the specimen in a circulating oven at a temperature and time sufficient to remove the solvent (a minimum temperature of 100°C for a minimum of 5 minutes). Cool the crucible with specimen remains in a desiccator to ambient temperature for a minimum of 5 minutes.

13.1.8 Evaluate the completeness of dissolution by examining the dried fiber remains. These should be easily separable, with no evidence of binding action between fibers. If the specimen remains do not meet this condition the test result shall be rejected and the test repeated under a more vigorous condition (one or more of longer time, increased agitation, temperature, or vacuum). The specimen must be dry with no evidence of any residual solvent. If there is evidence of residual solvent then the specimen drying shall be continued (at a higher temperature, or with addition of vacuum, if required) until the solvent is removed.

13.1.9 Weigh the specimen remains in the crucible to 0.1 mg. Record mass as M_f in g.

13.2 *Procedure B*— Fiber Content, Fiber Areal Weight and Matrix Content by Matrix Burn-off.

13.2.1 Record the nominal area produced by the cutting template or die as A, in m^2.

13.2.2 Weigh each specimen on an analytical balance to 0.1 mg. Record the mass of each specimen as M_i , in g.

13.2.3 Weigh a desiccated crucible for each specimen on an analytical balance to 0.1 mg. Record the mass as M_c in g.

13.2.4 Place each specimen in a desiccated crucible and place into a preheated muffle furnace at 500°C, or lower, depending upon the composite system (a temperature below which the matrix system will spontaneously ignite). To avoid spontaneous ignition in the furnace, specimens may be pre-ignited under a controlled heat flame before entrance into the furnace (see 6.2.3 for precautions related to specimen preservation).

13.2.5 Heat the furnace to 565°C +/- 30°C, or other temperature compatible with the composite system that will burn-off the matrix and leave the reinforcement unaltered. The maximum time for burn-off should be 6 h; shorter times are dependent on the system and specimen size.

13.2.6 Remove the crucible from the furnace and evaluate the completeness of dissolution by examining the specimen remains. The reinforcement content should be easily separable, with no evidence of binding action between fibers. If the specimen remains do not meet this condition, the test result shall be rejected and the test repeated under a more vigorous condition (longer time, increased temperature, or both).

13.2.7 Place the crucible in a desiccator and allow the specimen and crucible to reach room temperature.

13.2.8 Weigh the crucible and specimen remains in the crucible to 0.1 mg. Record mass as M_f, in g.

14. Calculation

14.1 *Matrix Content (Wet Resin Content or Resin Content):*

14.1.1 Calculate the matrix content (resin content, or wet resin content) for each replicate as follows:

$$MC = \frac{M_i - (M_f - M_c)}{M_i} \times 100$$

where:

MC = Matrix content of the specimen, weight percent;
M_i = Initial mass of the specimen, g;
M_c = mass of container or crucible, g; and
M_f = mass of container or crucible and reinforcement, g.

14.2 *Matrix Solids Content (Resin Solids Content or Dry Resin Content), when specified:*

14.2.1 Calculate and record the matrix solids content (resin solids content, or dry resin content) for each replicate as follows:

$$MS = \frac{M_i(1 - V_c/100) - (M_f - M_c)}{M_i(1 - V_c/100)} \times 100$$

where:

MS = Matrix solids content, weight percent;
V_c = Average volatiles content, weight percent.

14.3 *Fiber Content:*

14.3.1 Calculate and record the fiber content for each replicate as follows:

$$FC = (M_f - M_c)/M_i \times 100$$

where:

FC = Fiber content, weight percent.

14.4 *Fiber Areal Weight:*

14.4.1 Calculate and record the fiber areal weight for each replicate as follows:

$$FAW = (M_f - M_c)/A$$

where:

FAW = Fiber areal weight of reinforcement, g/m^2; and
A = Nominal area of specimen as produced by the cutting template or die, m^2.

15. Report

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

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

15.1.2 The revision level or date of issue of this test method,

15.1.3 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing,

15.1.4 Complete identification of the material including material specification, material type, material designation, manufacturer, manufacturer's batch number, source (if not from manufacturer), date of certification or prepreg manufacture, expiration of certification, form, filament count, fiber areal weight, and matrix type.

15.1.5 Cutting apparatus used, and nominal specimen area used in calculation,

15.1.6 Matrix dissolution solvent and conditions used in test, such as temperature of solvent, agitation method, time specimen left in solvent.

15.1.7 Observations concerning irregularities in reinforcement fiber appearance, evidence of melting or residual matrix solids.

15.1.8 *Test Result, Fiber Content, Fiber Areal Weight, Matrix Content, or Matrix Solids Content:*

15.1.8.1 Results of the individual specimens and the average value of the replicate set,

15.1.8.2 If matrix solids content is requested, the average results for volatiles content, the matrix solids content for the individual specimens and the average value for matrix solids content of the replicate set.

16. Precision and Bias

16.1 *Precision*—The data required for the development of a precision statement is not yet available for this test method. Committee D30 intends to create a round-robin test series for this test method 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; fiber areal weight; matrix content; matrix solids content; prepreg; resin content; resin solids content

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