Designation: D 3529/D 3529M – 97 (Reapproved 2003)$^{\epsilon 1}$

Standard Test Method for Matrix Solids Content and Matrix Content of Composite Prepreg[1]

This standard is issued under the fixed designation D 3529/D 3529M; 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.

ϵ^1 NOTE—Warning/precaution notes were moved into text editorially in October 2003.

1. Scope

1.1 This test method covers the determination of the matrix solids content and matrix content of composite material prepregs. Volatiles content, if appropriate and required, is determined by means of Test Method D 3530/D 3530M.

1.2 This test method focuses on thermosetting matrix material systems for which matrix may be extracted in organic solvent. However, other, unspecified reagents may be used with this test method to extract other matrix material types for the same purposes.

1.3 This test method does not distinguish between hybrid reinforcements or matrices. Its use with hybrids is limited to a determination of total reinforcement or matrix, or the use of a standard factor to be used as the weight of a particular reinforcement or matrix constituent.

1.4 Alternate techniques for determining matrix and reinforcement content include Test Methods C 613/C 613M (Soxhlet extraction) and D 3171 (meant typically for laminates; covering matrix digestion or combustion).

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

1.6 *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 8.2.4 and 8.2.5

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 613/C 613M Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction

D 883 Terminology Relating to Plastics

D 3171 Test Method for Constituent Content of Composite Materials

D 3530/D3530M Test Method for Volatiles Content of Composite Material Prepreg

D 3878 Terminology of Composite Materials

E 177 Practice for Use of Terms Precision and Bias in ASTM Test Methods

E 1309 Guide for Identification of Composite Materials in Computerized Property Databases

2.2 *NFPA Standard:*

NFPA 86 Standard for Ovens and Furnaces[3]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to composite materials. Terminology D 883 defines terms relating to plastics. Practice E 177 defines terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 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. See Terminology 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. See Terminology D 3878.

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. See Terminology D 3878.

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 D 3878.

3.1.5 *resin content*, *n*—see *matrix content*.

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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.

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

3.2.3 *wet resin content*, *n*—a prepreg resin content that does not account for mass loss due to volatiles. This is used interchangeably with matrix content and resin content in this test method.

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

3.3 *Symbols:*

3.3.1 M_c—mass of container.

3.3.2 M_i—initial mass of the specimen.

3.3.3 M_f—final mass of specimen and container at end of test.

3.3.4 W_m—matrix content of specimen in weight percent.

3.3.5 W_{ms}—matrix solids content of specimen in weight percent.

4. Summary of Test Method

4.1 Specimens of the prepreg are weighed and subjected to solvent, that may be heated, until the matrix is dissolved. The dissolved matrix is rinsed from the reinforcement residue which is then dried and weighed. The loss in mass is expressed as matrix content, a percentage of the specimen mass.

4.2 Where applicable, adjacent samples are tested for volatiles content according to D 3530/D 3530M. A calculation of amount of resin in the sample after volatiles content is subtracted is expressed as matrix solids content, a percentage of specimen mass. 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 matrix content or matrix solids content. Knowledge of the matrix content or matrix solids content is useful 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 lose or gain weight upon exposure to solvent and matrix constituents that do not dissolve in the solvent. Alternative methods or solvents should be used or an agreement should be made with the end user for how to consider the effect of incomplete matrix dissolution or reinforcement weight change for reporting purposes.

6. Interferences

6.1 *Extent of Cure in Thermosetting Systems*—The efficiency of matrix dissolution for thermosetting matrix materials is directly related to the extent of cure of the resin system. Resins that have started to cross link (such as B-staged resins) will be increasingly more difficult to extract as the cure advances. This test method may not be appropriate for such materials; one of the methods of Test Method D 3171 is then recommended.

6.2 *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.

6.3 *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.

6.4 *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.

7. Apparatus

7.1 *Cutting (Grooved) Template*—Cutting templates shall vary no more than 0.4 mm from center line when used. This gives a maximum error of 2 %. This is not required if a cutting die is used.

7.2 *Cutting Blade*—Die cutting blades are recommended to provide a consistent cut area error from sample to sample of no greater than 0.05 % of the target area. If a die is not available, a single edged blade will be used.

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

7.4 *Timer*, accurate to nearest minute.

7.5 *Fume Exhaust Hood*.

7.6 *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.

7.7 *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.

7.8 *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.

7.9 *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 ±15°C.

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

8. Reagents

8.1 *Purity of Reagents*—As a minimum, a technical-grade reagent is required to provide accurate results. However, when resolving disputes or performing subsequent analysis of extract or residue, a reagent-grade reagent shall be used. Unless otherwise indicated, it is intended that the reagent 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 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.)

8.2.5 *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 2A and 2B as a "probable human carcinogen.")

8.2.6 *Methyl Isobutyl Ketone (MIBK, 4-Methyl-2-pentanone),* $(CH_3)_2CHCH_2COCH_3$.

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 8.[5] (**Precaution—**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.) (**Precaution—**In addition to other precautions, the extraction and filtering processes should be performed under a suitable vented chemical fume hood.) (**Precaution—**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. (**Precaution—**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 6400 mm^2. Minimum mass of the specimen shall be 1 g.

11. Calibration and Standardization

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

12. Conditioning

12.1 Store composite prepreg with thermosetting resins at low temperatures as recommended by the manufacturer (typically approximately −18°C (0°F)). 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 Do not expose the material, that usually has some volatile content, at ambient temperature for long periods of time before testing is begun. The material may stage (see 6.1) or lose volatiles, that gives a false indication of matrix solids content.

13. Procedure

13.1 *Matrix Content*:

13.1.1 Weigh each specimen on an analytical balance to 0.1 mg. Record the mass of each specimen as *W*1.

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

13.1.3 Sample shall be in the solvent at the steady state condition of the test for a minimum of three min. The steady state condition may be one or more of the following:

13.1.3.1 *Agitation*—May be used to speed dissolution of resin into the solvent. Minimum shaker agitation of 60 cycles/minute. Recommended minimum ultrasonic frequency 40 kHz.

13.1.3.2 *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. Recommended vacuum is below 50 kPa.

13.1.3.3 *Temperature*—A raised temperature may be used to increase solvent efficiency and reduce resin dissolution times. Recommended temperatures are in the range above room temperature up to the boiling point of the solvent.

13.1.4 Decant solution.

13.1.5 Repeat 13.1.3 and 13.1.4 at least one additional time.

NOTE 1—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.

13.1.6 Place the sample in a weighed, fritted glass crucible or other suitable container of recorded mass, *W*2. Dry the sample in a circulating oven at a temperature and time satisfactory to remove the solvent (a minimum temperature of 100°C for a minimum of 5 min). Cool the crucible or container with sample in a desiccator to ambient temperature for a minimum of 5 min.

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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.

13.1.7 Weigh the specimen and the container or crucible to 0.1 mg. Record mass as *W*3.

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 sample does not meet this condition, the test is not accurate. The test should be repeated under a more vigorous condition (longer time, increased temperature or vacuum). The sample should also be dry, with no evidence of any residual solvent. If there is evidence of residual solvent, *W*3 is invalid and the sample should be returned to the oven for a longer time, or at a higher temperature.

13.2 *Volatiles Content*.

13.2.1 Determine volatiles content V_c in accordance with Test Method D 3530/D 3530M. Volatiles content specimens shall be sampled from an area adjacent to the matrix content specimens if matrix solids content is to be determined.

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, W_t, percent by weight, as follows:

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

where:

M_i = initial mass of the specimen, g;
V_c = volatiles content, weight percent;
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)*:

14.2.1 Calculate the matrix solids content, resin solids content, or dry resin content, W_{ms}, as a weight percent as follows:

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

where:

V_c = volatiles content, weight percent.

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. Use Guide E 1309 as a guide,

15.1.5 Cutting apparatus used, and apparatus tolerance conditions or sample dimensions, and

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

15.1.7 *Test Result, Matrix Content, or Matrix Solids Content*:

15.1.7.1 Matrix content expressed as a weight percent of three specimens and the average value for the three specimens (if matrix content reported),

15.1.7.2 The test method used to determine the average volatiles content and time and temperature used to remove volatiles (if matrix solids content is reported), and

15.1.7.3 Matrix solids content expressed as a weight percent of three specimens and the average value for the three specimens (if matrix solids content is reported).

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 D30 is currently planning 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; matrix content; matrix solids content; prepreg; resin content; resin solids content