



Designation: D 3529/D 3529M – 97 (Reapproved 2003)^{ε1}

Standard Test Method for Matrix Solids Content and Matrix Content of Composite Prepreg¹

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.

^{ε1} 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 Furnaces³

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*.

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 $\pm 15^\circ\text{C}$.

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