



Designation: C613 – 23

Standard Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction¹

This standard is issued under the fixed designation C613; 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 a Soxhlet extraction procedure to determine the matrix content, reinforcement content, and filler content of composite material prepreg. Volatiles content, if appropriate, and required, is determined by means of Test Method [D3530](#).

1.1.1 The reinforcement and filler must be substantially insoluble in the selected extraction reagent and any filler must be capable of being separated from the reinforcement by filtering the extraction residue.

1.1.2 Reinforcement and filler content test results are total reinforcement content and total filler content; hybrid material systems with more than one type of either reinforcement or filler cannot be distinguished.

1.2 This test method focuses on thermosetting matrix material systems for which the matrix may be extracted by an 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 Alternate techniques for determining matrix and reinforcement content include Test Methods [D3171](#) (matrix digestion), [D2584](#) (matrix burn-off/ignition), and [D3529](#) (matrix dissolution and ignition loss). Test Method [D2584](#) is preferred for reinforcement materials, such as glass, quartz, or silica, that are unaffected by high-temperature environments.

1.4 The technical content of this standard has been stable since 1997 without significant objection from its stakeholders. As there is limited technical support for the maintenance of this standard, changes since that date have been limited to items required to retain consistency with other ASTM D30 Committee standards. The standard therefore should not be considered to include any significant changes in approach and practice

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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since 1997. Future maintenance of the standard will only be in response to specific requests and performed only as technical support allows.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 9 and [7.2.3](#) and [8.2.1](#).

1.7 *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.*

2. Referenced Documents

- 2.1 *ASTM Standards*:²
 - [D883 Terminology Relating to Plastics](#)
 - [D2584 Test Method for Ignition Loss of Cured Reinforced Resins](#)
 - [D3171 Test Methods for Constituent Content of Composite Materials](#)
 - [D3529 Test Methods for Constituent Content of Composite Prepreg](#)
 - [D3530 Test Method for Volatiles Content of Composite Material Prepreg](#)
 - [D3878 Terminology for Composite Materials](#)
 - [E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process](#)

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.

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

2.2 NFPA Standard:³

NFPA 86 Standard for Ovens and Furnaces

3. Terminology

3.1 *Definitions*—Terminology D3878 defines terms relating to composite materials. Terminology D883 defines terms relating to plastics. Terminology E456 and Practice E177 define terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other documents.

3.2 *Definitions of Terms Specific to This Standard:*

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

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

3.2.2.1 *Discussion*—In this test method the reinforcement is separated from the remainder of the material, which includes the matrix and the filler. If the filler is not then separated from the matrix to determine the proportion of each, then the filler content is included in the matrix content.

3.2.3 *replicate, n*—a test specimen tested under nominally identical conditions as other test specimens from the same sample.

3.2.4 *test result, n*—the value obtained for a given property from one test unit.⁴

3.2.4.1 *Discussion*—A test result may be a single observation or a combination of a number of observations when two or more test specimens are measured for each test.

3.2.5 *test specimen, n*—a test unit or portion of a test unit upon which a single or multiple observation is to be made.⁴

3.2.6 *test unit, n*—a unit or portion of a material that is sufficient to obtain a test result(s) for the property or properties to be measured.

3.2.6.1 *Discussion*—A test unit may be a subunit of a primary (first stage) sampling unit or it may be a subunit of a composite of primary sampling units or of increments from these primary sampling units.

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

3.2.8 *wet resin content, n*—prepreg resin content determined by considering volatiles as part of the resin mass.

3.3 *Symbols:*

A—initial mass of dry reinforcement during a reagent exposure evaluation

B—final mass of dry reinforcement during a reagent exposure evaluation

c—percent reinforcement mass change due to reagent exposure

CV—coefficient of variation statistic of a sample population for a given property

M_a—additional mass of filler in the test specimen

M_e—mass of the test specimen extraction residue

M_i—initial mass of the test specimen

M_r—mass of reinforcement in the test specimen

n—number of replicates in the sample population

s_{n-1}—standard deviation statistic of a sample population for a given property

W_f—weight percent of filler in prepreg

W_m—weight percent of matrix in prepreg

W_r—weight percent of reinforcement in prepreg

x_i—test result for an individual test specimen from the sample population for a given property

\bar{x} —average value of a sample population for a given property

4. Summary of Test Method

4.1 The exposed surface area of the prepreg material test specimen is increased by cutting the test specimen into smaller pieces. The test specimen is weighed and the matrix material removed by means of Soxhlet extraction. The extracted residue is dried and weighed. If a filler is present in the residue, in addition to reinforcement, the two components are separated by filtering the residue. From mass measurements of the initial test specimen, and of the residue taken at various stages in the process, the matrix content, reinforcement content, and filler content are calculated and reported in weight percent.

4.1.1 *Soxhlet Process*—While described in detail in common quantitative chemical analysis textbooks, the Soxhlet process is summarized as follows. The test specimen is loaded into a filtering extraction thimble, which is placed into the extraction chamber of a Soxhlet extraction assembly (see Fig. 1) containing an appropriate extraction reagent. The porous thimble allows the liquid extraction reagent to pass while retaining the test specimen. Freshly distilled liquid reagent enters from the top of the extraction chamber, filling it until the liquid reaches the highest level of the reagent-return tube. At this moment the tube operates as a siphon, draining the extraction chamber completely as it returns the liquid reagent

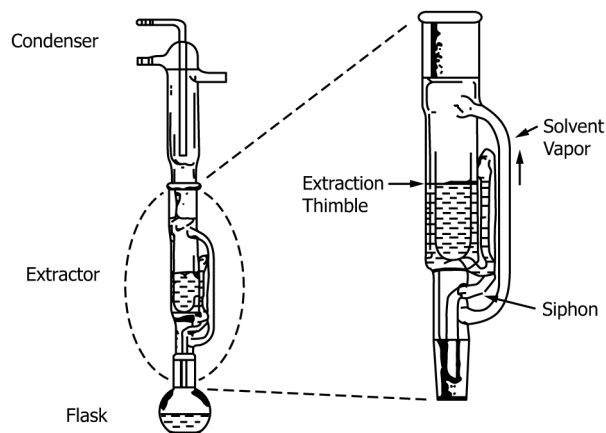


FIG. 1 Schematic of Soxhlet Extraction Apparatus

³ Available from National Fire Protection Association (NFPA), 1 Batterymarch Park, Quincy, MA 02169-7471, <http://www.nfpa.org>.

⁴ See *Form and Style for ASTM Standards*.

and any extracted material to a reservoir beneath the extraction chamber. The heated reservoir boils the reagent, the vapor of which is led to a condenser placed above the extraction chamber. The distilled condensate then drips down into the thimble, starting once again the process of filling the extraction chamber. The Soxhlet operation is not a continuous operation, but rather a sequence of fillings and siphonings, each cycle of which is called a reflux change. The heat input and reagent volume are adjusted to cause the boiling reagent to return to the extraction flask from the condenser at 3 to 10 reflux changes per hour, with the extraction continuing for a minimum of 4 h or 20 reflux changes, whichever comes first.

4.1.2 *Volatiles Content*—Volatiles content is primarily applicable to thermosetting materials, and, if required, is determined by Test Method **D3530**. Volatiles content determination requires different test specimens than those used in the extraction process, since the process of determining volatiles content renders thermosetting material specimens unsuitable for subsequent organic solvent extraction.

5. Significance and Use

5.1 The prepreg volatiles content, matrix content, reinforcement content, and filler content of composite prepreg materials are used to control material manufacture and subsequent fabrication processes, and are key parameters in the specification and production of such materials, as well as in the fabrication of products made with such materials.

5.2 The extraction products resulting from this test method (the extract, the residue, or both) can be analyzed to assess chemical composition and degree of purity.

6. Interferences

6.1 *Extent of Cure in Thermosetting Systems*—The efficiency of extraction 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; Test Methods **D3171** or **D2584** may be better test method choices.

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 *Thimble Contamination*—If the extract is to undergo further analysis, the thimble must be clean to avoid a significant source of contamination.

6.4 *Reinforcement Mass Change as a Result of Reagent*—The calculations of this test method assume that the reinforcement mass (or filler, if filler content is being determined) is not significantly affected (whether mass increase or mass loss) by exposure to the reagent. Small, consistent changes in the reinforcement mass caused by exposure to the reagent can be corrected by the process described in 14.4.5. The resulting correction may be used if this change is sufficiently reproduc-

ible under the conditions of the test, and if this change has the same value for the reinforcement alone as for the reinforcement in the matrix. Otherwise, a different reagent, or another test method, must be selected.

7. Apparatus

7.1 General Requirements:

7.1.1 *Container Volume*—A suggested volume is shown for each container. However, other sizes may be required depending upon the test specimen size, the amount of reagent needed to complete the extraction process, and the relative sizes of related equipment.

7.1.2 *Thermal Shock*—Laboratory equipment that is subjected to non-ambient temperatures (hot or cold) shall be of tempered-glass or PTFE materials.

7.1.3 *Post-Test Elemental Analysis*—If a post-test elemental analysis of the extract or residue is to be performed, laboratory equipment contacting the test specimen shall be constructed of PTFE and test specimen cutting shall be limited to tools that do not leave an elemental trace.

7.2 General Equipment:

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

7.2.2 *Muffle Furnace*—The muffle furnace used to condition glass extraction thimbles shall be capable of maintaining a temperature of $510\text{ }^{\circ}\text{C} \pm 15\text{ }^{\circ}\text{C}$.

7.2.3 *Air-Circulating Drying Oven*—The drying oven shall be capable of maintaining a temperature of $163\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$. (**Warning**—For safety purposes listed in NFPA 86, take care to limit volatile concentration in the oven by controlling sample quantity, temperature, and ventilation.)

7.2.4 *Desiccator*—The desiccator shall be capable of containing the required test specimens.

7.3 Extraction Assembly:

7.3.1 *Extraction Thimbles*—The extraction thimbles shall be deep, narrow filtering cups, of either borosilicate glass in an appropriate pore size, or fat-extracted cellulose paper, suitable for use in the extraction chamber.

7.3.2 *Hot Plate*—The hot plate shall have adjustable controls suitable for heating the reagent within the reservoir flask to $260\text{ }^{\circ}\text{C}$ and shall be capable of controlling the required reagent temperature within $\pm 15\text{ }^{\circ}\text{C}$.

7.3.3 *Reservoir Flask*—The reservoir flask shall be of borosilicate glass, of suitable volume (125 mL is suggested) for the reagent quantity and extraction chamber volume, and shall have a ground tapered joint capable of connection with the remainder of the assembly.

7.3.4 *Soxhlet Extraction Chamber*—The extraction chamber shall be of borosilicate glass, with an automatic recycling siphon that recycles at a suitable liquid volume (50 mL is suggested), and with a ground tapered joint at each end capable of connecting with the remainder of the assembly.

7.3.5 *Condensing Chamber*—The condensing chamber shall be of borosilicate glass, shall be water cooled, and shall have a ground tapered joint capable of connecting with the remainder of the assembly.