Designation: C 613/C 613M – 97

Standard Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction[1]

This standard is issued under the fixed designation C 613/C 613M; 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.

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 D 3530/D 3530M.

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 D 3171 (matrix digestion), D 2584 (matrix burn-off/ignition), and D 3529 (matrix dissolution). Test Method D 2584 is preferred for reinforcement materials, such as glass, quartz, or silica, that are unaffected by high-temperature environments.

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

1.5 *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 statements are given in Section 9 and Note 1 and Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]
D 2584 Test Method for Ignition Loss of Cured Reinforced Resins[3]
D 3171 Test Method for Fiber Content of Resin-Matrix Composites by Matrix Digestion[4]
D 3529/D 3529M Test Method for Matrix Solids Content and Matrix Content of Composite Prepreg[4]
D 3530/D 3530M Test Method for Volatiles Content of Composite Material Prepreg[4]
D 3878 Terminology of High-Modulus Reinforcing Fibers and Their Composites[4]
E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[5]
E 177 Practice for Use of Terms Precision and Bias in ASTM Test Methods[5]
E 456 Terminology Relating to Quality and Statistics[5]
E 1309 Guide for Identification of Composite Materials in Computerized Material Property Databases[4]
E 1471 Guide for Identification of Fibers, Fillers, and Core Materials in Computerized Material Property Databases[4]

2.2 *NFPA Standard:*

NFPA 86 Standard for Ovens and Furnaces[6]

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines terms relating to composite materials. Terminology D 883 defines terms relating to plastics. Terminology E 456 and Practice E 177 define 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 *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.

3.1.2 *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 matrices it

---

[1] This test method is under the jurisdiction of ASTM Committee D-30 on High Modulus Fibers and Their Composites and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

Current edition approved Sept. 10, 1997. Published May 1998. Originally published as C 613 – 67. Last previous edition C 613 – 67(1990)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.02.

[4] *Annual Book of ASTM Standards*, Vol 15.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from National Fire Protection Association Standards Council, 1 Batterymarch Park, PO Box 9101, Quincy, MA 02269-9101.

has been partially cured to a controlled viscosity called "B stage". See Terminology D 3878.

3.1.3 *resin content, n*—see matrix content. See Terminology D 3878.

3.1.4 *sample, n*—a small part or portion of a material or product intended to be representative of the whole. See Terminology D 883.

3.1.5 *test result, n*—the value obtained for a given property from one test unit.[7]

3.1.5.1 *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.

3.1.6 *test specimen, n*—a test unit or portion of a test unit upon which a single or multiple observation is to be made.[7]

3.1.7 *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.

3.1.7.1 *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 *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 *reinforcement content, n*—the amount of reinforcement 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, reinforcement volume fraction.

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

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

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

3.3 *Symbols:*

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

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

3.3.3 c—percent reinforcement mass change due to reagent exposure.

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

3.3.5 M_a—additional mass of filler in the test specimen.

3.3.6 M_e—mass of the test specimen extraction residue.

3.3.7 M_i—initial mass of the test specimen.

3.3.8 M_r—mass of reinforcement in the test specimen.

3.3.9 n—number of replicates in the sample population.

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

3.3.11 W_f—weight percent of filler in prepreg.

3.3.12 W_m—weight percent of matrix in prepreg.

3.3.13 W_r—weight percent of reinforcement in prepreg.

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

3.3.15 $\bar{x}$—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 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

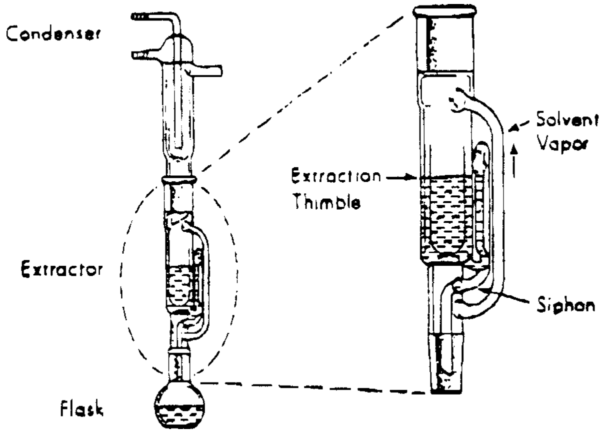

FIG. 1 Schematic of Soxhlet Extraction Apparatus

---

[7] See *Form and Style for ASTM Standards*.

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 D 3530/D 3530M. 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 D 3171 or D 2585 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 reproducible 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 ± 15°C.

7.2.3 *Air-Circulating Drying Oven*—The drying oven shall be capable of maintaining a temperature of 163 ± 3°C.

NOTE 1—**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°C and shall be capable of controlling the required reagent temperature within ±15°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.

7.4 *For Determining Filler Content*:

7.4.1 *Vacuum Filter System*—The vacuum filter system shall be suitable for filtering material from the filtering crucible and holder.

7.4.2 *Filtering Crucible*—The filtering crucible shall be of fritted glass and of suitable pore size and of appropriate volume (30 mL is suggested).

NOTE 2—Filter porosity should be sized to filter the smallest expected filler size from the reinforcement. If there is any doubt about the filter pore-size selection, evaluate, with the material under test, filters of successively different porosity size until confidence is established in the filter size selected. While the glass fiber filter is used in concert with the fritted filter to reduce any tendency to clog, note that certain materials, particularly those containing filler of a broad range of particle size and shape, may nevertheless clog the filter pores without visible sign. The filter tare mass should be monitored for change as a result of the test. A change in the filter tare mass indicates a potentially incorrect determination of reinforcement to filler proportion, and therefore, incorrect reinforcement and filler content test results.

7.4.3 *Crucible Holder*—The crucible holder shall be capable of holding the filtering crucible.

7.4.4 *Glass Fiber Filter*—A glass fiber filter of suitable porosity and of appropriate diameter to fit in the filtering crucible.[8]

7.5 *Miscellaneous Common Laboratory Items*—Other commonly available laboratory items may be needed including: scissors or knife, beakers or flasks, flexible tubing, equipment connectors, wash bottles, aluminum foil, and lint-free wipes.

8. Reagents and Materials

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.[9] Other equivalent grades may be used, provided the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Extraction Reagents*—A suitable extraction reagent shall be selected that is compatible with the material system under test and the apparatus. Read and understand the procautions listed in Section 9 before selecting an extraction reagent. Extraction reagents that have been found effective for many thermosetting matrices include the following:

8.2.1 *Dimethylformamide (DMF)*, $(CH_5)_2NCHO$.

NOTE 3—**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.

8.2.2 *Ethanol (Ethyl Alcohol)*, C_2H_5OH.

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 the following:

8.3.1 *Acetone (2-Propanone)*, CH_8COCH_8.

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 given in Footnote 10.[10]

NOTE 4—**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.

NOTE 5—**Precaution:** In addition to other precautions, the extraction and filtering processes should be performed under a suitable vented chemical fume hood.

NOTE 6—**Precaution:** In addition to other precautions, materials that have been exposed to potentially toxic or flammable reagents must be air-dried under a hood before being subsequently oven-dried, to eliminate build-up of a potentially dangerous concentration of vapor in the drying oven. Useful guidelines for estimating the safe volatiles mass for a given oven size are given in NFPA Standard 86.

9.2 Use of mixed extraction reagents with different boiling points are not covered by this test method.

NOTE 7—**Precaution:** In addition to other precautions, do not use mixed extraction reagents with different boiling points. Use of mixed extraction reagents with different boiling points can result in an explosion if the low-boiling fraction siphons into the extraction chamber while the high-boiling fraction is being heated; the low-boiling fraction may then superheat and overpressure the apparatus.

10. Sampling, Test Specimens, and Test Units

10.1 *Test Units*—Unless otherwise specified, the test unit shall consist of a single test specimen upon which a single observation is to be made.

10.2 *Sampling*—Unless otherwise specified, at least three test specimens (test units) per sample shall be evaluated. For statistically significant data the procedures outlined in Practice E 122 should be consulted. The method of sampling shall be reported.

10.3 *Test Specimen Geometry*—The mass of each individual test specimen shall be at least 1.0 g and, unless otherwise specified, shall be 2.0 to 3.0 g.

10.4 *Test Specimen Preparation*:

10.4.1 *Labeling*—Label each test specimen container so that they will be distinct from each other and traceable back to the sampled material. Report the labeling scheme and method.

11. Preparation of Apparatus

11.1 Clean the extraction thimbles and filter crucibles. Dry the filter crucibles (including the glass fiber filters) and extraction thimbles in the drying oven (unless otherwise specified, dry at 163 ± 3°C) until there is no perceptible mass change with time. Remove from the oven and cool in the desiccator. Determine and record, to within 0.1 mg, the initial tare mass of each. The tare mass of the filter crucibles shall include the installed glass-fiber filter.

11.2 After cleaning, drying, and taring, store all extraction thimbles and filter crucibles in the desiccator until use.

12. Calibration and Standardization

12.1 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.

---

[8] A Reeve Angle Grade 934 AH or equivalent is suggested.

[9] *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.

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

13. Conditioning

13.1 No preconditioning or conditioning of the test specimen is required.

13.2 Unless otherwise specified, conduct the tests at 23 ± 3°C and 50 ± 10 % relative humidity. Record and report the actual laboratory environment.

14. Procedures

14.1 *Selection of Test Parameters*—Specify the following test parameters, as applicable, prior to test.

14.1.1 *Test Results*—Specify the properties to be determined by this test method, including: volatiles content, matrix content (dry, wet, or both), and reinforcement content. If no properties are specified, determine and report only matrix content (wet resin content) and reinforcement content, and filler content if appropriate.

14.1.2 *Reagent Selection*—Specify the reagents (Section 8) to be used for extraction and washing. If appropriate reagents are not specified, or not known, contact the material manufacturer for guidance on reagent selection.

14.1.3 *Sampling Method*—Specify the material sampling method, if sampling is to be conducted by the testing laboratory.

14.2 *General Instructions*:

14.2.1 Report any deviations from this test method, whether intentional or inadvertent.

14.2.2 Shield the balance from air drafts and isolate it from vibrations that could affect its accuracy.

14.2.3 Determine mass to the nearest 0.1 mg.

14.2.4 Process each test specimen separately.

NOTE 8—Reagent quantities and container volumes in this test method are estimates based on common material systems and the standard test specimen mass. Reagent quantities and equipment sizes may need to be adjusted as a function of material system, coupon size, or both.

14.3 *Volatiles Content*—If volatiles content is to be determined, on the same number of additional and separate test specimens, each sampled immediately adjacent to one of the extraction test specimens, determine and report volatiles content in accordance with Test Method D 3530/D 3530M. If the dry resin content is required, the average volatiles content from these tests will be used to correct the extraction results.

NOTE 9—Volatiles content is conducted on separate test specimens since the process of determining volatiles content tends to advance thermosetting resins, making subsequent resin extraction by this test method difficult or impossible.

14.4 *Extraction*:

14.4.1 *Cutting*—Cut the prepreg test specimen into small pieces (nominally 10 to 15-mm squares), place the pieces into a clean, dry, tared extraction thimble, and blend the pieces thoroughly. Take care during cutting and blending to avoid losing even small quantities of matrix or reinforcement.

14.4.2 *Initial Mass*—Weigh the test specimen and thimble, subtract the tared mass of the thimble, and record this result as M_i, the initial test specimen mass.

14.4.3 *Refluxing*:

14.4.3.1 Setup the extraction apparatus under a suitably vented chemical fume hood.

14.4.3.2 Position the thimble within the extractor. Add extraction reagent to the extraction tube sufficient to immerse the test specimen and fill about ⅔ of the thimble (typically 35 mL of reagent).

14.4.3.3 Assemble the Soxhlet extractor to the reservoir flask containing additional reagent (typically 55 mL of reagent, for a total of about 90 mL of reagent in the assembly). Attach the condenser to the top of the extractor, and provide supports for the entire assembly as needed.

14.4.3.4 Turn on the condenser cooling water.

14.4.3.5 Set the hot plate temperature control to a temperature appropriate for the selected reagent and turn on the hot plate. When condensation of the reagent occurs adjust the hot plate temperature to effect 3 to 10 reflux changes per hour. Continue to reflux for a minimum of 4 h or 20 reflux changes, whichever comes first.

NOTE 10—If the extractor volume is too large compared to the volume of the liquid in the reservoir, with each cycle the siphoning liquid may cool the reservoir below the reagent boiling point, and a stable process may be difficult to obtain. A magnetically driven stirrer placed in the reservoir may help.

NOTE 11—For high boiling reagents it may be necessary to wrap the extraction chamber with aluminum foil to reduce heat loss.

14.4.3.6 When extraction is completed, turn off the hot plate and allow the apparatus to cool until safe to handle. Remove the extraction thimble from the extraction assembly, drain any remaining reagent, and air-dry under a hood until any flammable or toxic materials have evaporated. Complete the drying to essentially constant mass in the forced-air drying oven at 163± 3°C. Examine the residue for signs of incomplete extraction. If matrix material remains, repeat the extraction until extraction is complete, either with the original reagent or with an alternate reagent.

14.4.3.7 Weigh the test specimen and thimble. Subtract the tare mass of the thimble from this result and record as M_e, the extracted test specimen residual reinforcement and filler mass.

14.4.4 *Filler Content*—If filler is known or suspected to exist, or is visibly present in the extracted residue, filter the extract to separate the filler from the reinforcement. Otherwise record the residual mass M_e as the reinforcement mass, M_r.

14.4.4.1 Under a suitable vented chemical fume hood, wash the extraction tube, reservoir flask, and extraction thimble (including the extraction residue), using a wash bottle and appropriate solvent wash, and saving all washings in a flask. All visible filler must be washed from the apparatus in order for filler content to be determined.

NOTE 12—If washing the extraction apparatus with solvent fails to remove filler that is caked on the apparatus, the following procedure has been used to remove carbonaceous filled phenolic resins. Similar procedures may be developed for other material systems, if needed. Place the air-dried apparatus in a muffle furnace at 400 ± 15°C for a minimum of 1 h. Cool in a desiccator and then weigh. If the apparatus was pretared, subtract the tare mass from these results and record the total as additional filler mass, M_a, to be added to the filler mass. If the apparatus was not pretared, determine the tare masses by inserting the apparatus into the muffle furnace at 540 ± 15°C for a minimum of 1 h; cool in a desiccator, then weigh, recording these weighings as the tared masses.

14.4.4.2 Place a tared filter crucible in a vacuum filtration system. Filter the washings with this system through the filter crucible, washing the residue clean of filler with a suitable solvent until only the reinforcement remains.

14.4.4.3 Air-dry the filter crucible under a chemical fume hood, then complete the drying process in the drying oven at 163 ± 3°C, for a minimum of 1 h or until an essentially constant mass is achieved. Cool the filter crucible in a desiccator and weigh. Subtract the crucible tare mass from the result and record the resulting reinforcement mass as M_r.

14.4.5 *Correction for Reinforcement Mass Change*—If the reagent is known (or observed) to create a significant change (> ±3 %), or inconsistent change, or both, in the mass of the reinforcement material, then replace the reagent with another that has little to no effect on the reinforcement. Correct small, consistent reinforcement mass changes by the following process.

NOTE 13—Certain reinforcements may, when exposed to ambient humidity, contain adsorbed or absorbed water up to a few weight percent. Steps should be taken during this evaluation to control this adsorbed or absorbed moisture, so that an accurate assessment of reinforcement change due to reagent exposure may be made.

14.4.5.1 Measure out dry (no sizing or finish) reinforcement material equal to the mass of reinforcement in the nominal test specimen. Record this value as *A*, the initial mass of the dry reinforcement.

14.4.5.2 Duplicate the extraction procedure used on the prepreg, with the dry reinforcement material, exposing the dry reinforcement to the reagent for the same length of time that the prepreg was exposed.

14.4.5.3 Weigh the exposed dry reinforcement in the thimble. Subtract the tared mass of the thimble from this result and record this value as *B*, the final mass of the exposed dry reinforcement. A correction to the final results is made by calculation in 15.1, based on any change in reinforcement mass resulting from this exposure.

15. Calculation

15.1 *Reinforcement Mass Change Due to Reagent Exposure*—Calculate the reinforcement mass change (loss or gain) due to reagent exposure in accordance with Eq 1, reporting the test result to the nearest 0.1 %.

$$c = \frac{A - B}{A} \times 100 \tag{1}$$

where:

c = percent reinforcement mass change due to reagent exposure, %,
A = initial mass of dry reinforcement, g, and
B = mass of dry reinforcement after exposure to reagent, g.

NOTE 14—A positive value for *c* indicates a mass loss due to reagent exposure, while a negative value indicates a mass gain due to reagent exposure.
Do not use the mass change, *c*, in subsequent calculations if *c* is greater than −0.5 % but less than +0.5 %.

15.2 *Reinforcement Mass*—Calculate the reinforcement mass of the test specimen in accordance with Eq 2, reporting the test result to the nearest 0.001 g.

$$M_{ro} = \frac{100\, M_r}{100 - c} \tag{2}$$

where:

M_{ro} = original reinforcement mass, g, and
M_r = measured remainder of reinforcement, g.

15.3 *Reinforcement Content*—Calculate the reinforcement content (weight percent) of the test specimen in accordance with Eq 3, reporting the test result to the nearest 0.1 %.

$$W_r = 100 \times \frac{M_{ro}}{M_i} \tag{3}$$

where:

W_r = weight percent reinforcement, %,
M_{ro} = original mass of reinforcement, g, and
M_i = initial mass of test specimen, g.

15.4 *Matrix Content*—Calculate the matrix content (wet resin content) (weight percent) of the test specimen in accordance with Eq 4, reporting the test result to the nearest 0.1 %.

$$W_m = 100 - \left(\frac{M_e}{M_i} \times 100\right) \tag{4}$$

where:

W_m = weight percent of matrix, %, and
M_e = mass of extracted residue, g.

15.5 *Dry Resin Content*—Where appropriate and required, calculate the dry resin content (weight percent) of the test specimen in accordance with Eq 5, reporting the test result to the nearest 0.1 %.

$$W_m\,(\text{dry}) = \frac{100 - W_r - VC}{1 - VC/100} \tag{5}$$

where:

W_m(dry) = weight percent of matrix, %, and
VC = average volatiles content (weight percent) from 14.3, %.

15.6 *Filler Content*—Calculate the filler content (weight percent) of the test specimen in accordance with Eq 6, reporting the test result to the nearest 0.1 %.

$$W_f = 100 - W_r - W_m + \frac{100 \times M_a}{M_i} \tag{6}$$

where:

W_f = weight percent of filler, %, and
M_a = additional filler mass cleaned from equipment, g.

16. Report

16.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable. Guides E 1309 and E 1471 may be helpful to those reporting material descriptions, constituent descriptions, or both.

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

16.1.2 The revision level or date of issue of this test method.

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

16.1.4 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from manufacturer), date of certification or prepregging, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, reinforcement areal weight, and matrix type.

16.1.5 Method of preparing the test specimen, including test specimen labeling scheme and method, test specimen geometry, sampling method, and specimen cutting method.

16.1.6 Calibration dates and methods for all measurement and test equipment, or a suitable reference to the same.

16.1.7 The type of apparatus used and the nominal and actual test temperatures.

16.1.8 Relative humidity and temperature of the testing laboratory.

16.1.9 Number of test specimens tested.

16.1.10 The matrix content (wet, dry, or both) of the test specimen by weight percent;

16.1.11 The reinforcement content of the test specimen by weight percent;

16.1.12 The filler content of the test specimen by weight percent;

16.1.13 The volatiles content of the prepreg by weight percent;

16.1.14 The percent reinforcement mass change due to exposure to the extraction reagent.

16.1.15 The average value $(\bar{x})$, standard deviation (s_n−1), and percent coefficient of variation (% CV) for the matrix content, reinforcement content, filler content, and volatiles content, for sample populations of three or more.

16.1.16 The date(s) and location(s) of the test(s); and

16.1.17 The name of the test operator(s).

17. Precision and Bias

17.1 Committee D-30 is currently planning a round-robin test series for this test method for the purpose of defining the precision. Bias cannot be determined for this test method as no reference material exists.

18. Keywords

18.1 composite materials; filler content; matrix content; prepreg; reinforcement content; resin content; resin matrix content; volatiles content

APPENDIX

(Nonmandatory Information)

X1. TEST DATA REPORTING FORM (Fig. 2)

Composite Material Designation:
Reinforcement Designation:
Extraction Reagent:

Reinforcement Change Due to Medium

Mass Measurements (g)			
Filter Tare Wt.	Initial Dry Reinforcement *A*	Final Dry Reinforcement *B*	Loss (or Gain) Due to Medium $c = \frac{A-B}{A} \times 100$

Extraction Measurement Data

	Mass Measurements (g)								
Test Specimen ID	Thimble Tare Mass	Thimble + Specimen Mass	M_i Specimen Mass	M_e Residue Mass	Filter Tare Mass	M_r Reinf. Mass	Matrix Content (w/o)	Reinf. Content (w/o)	Filler Content (w/o)

Average			
Standard Deviation			
Coefficient of Variation, %			

FIG. X1.1 Example Test Data Reporting Form

NOTICE: This standard has either been superceded and replaced by a new version or discontinued. Contact ASTM International (www.astm.org) for the latest information.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (http://www.astm.org).