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Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content using calcination methods

Plastiques renforcés de verre textile — Préimprégnés, compositions de moulage et stratifiés — Détermination des taux de verre textile et de charge minérale par des méthodes calcination

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 1172:1996), which has been technically revised.

The main changes are as follows:

- more detailed definitions of procedures in 7.2 (Method A) have been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content using calcination methods

WARNING — This document does not give details of the precautions that should be taken to meet health and safety requirements. The test methods described require the use of high temperatures and concentrated acids. It is the responsibility of the user of this document to follow the appropriate health and safety procedures.

1 Scope

This document specifies two calcination methods for the determination of the textile glass and mineral filler content of glass-reinforced plastics:

- Method A: for the determination of the textile glass content when no mineral fillers are present.
- Method B: for the determination of the textile-glass and mineral-filler content when both components are present.

This document is applicable to the following types of material:

- prepregs made from yarns, rovings, tapes or fabrics;
- SMC, BMC and DMC moulding compounds;
- textile-glass-reinforced thermoplastic moulding materials and granules;
- filled or unfilled textile-glass laminates made with thermosetting or thermoplastic resins.

The methods do not apply to the following types of reinforced plastic:

- those containing reinforcements other than textile glass;
- those containing materials which do not completely burn off at the test temperature (for example, those based on silicone resin);
- those containing mineral fillers which degrade at temperatures below the minimum calcination temperature.

NOTE For these materials, ISO 11667 can be used.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 8604, *Plastics — Prepregs — Definitions of terms and symbols for designations*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 8604 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Principle

A test specimen is weighed and subsequently calcinated at a specified temperature. The specimen is then reweighed and the non-combustible matter content (glass + filler) obtained by determining the difference in mass of a test specimen before and after calcination in one of the following ways:

a) in the case of materials containing no fillers, the glass content is calculated directly from the difference in mass (method A);

b) in the case of materials containing both glass and filler, the glass and filler remaining after calcination are separated by dissolution of the filler in hydrochloric acid. The difference between the mass of the specimen before calcination and the mass of the dried specimen after reaction with acid is used to measure the glass content. The filler content is obtained by calculating the difference between the mass of the specimen after calcination and the mass of the dried specimen after reaction with acid (method B).

The test method requires that all weighings be made at constant mass after repeated calcination and/or drying. In those cases where known materials are being tested regularly, a minimum time for the calcination and drying stages may be determined by experiment to ensure that constant mass has been reached.

NOTE 1 If the material tested contains a resin which is combustible under the test conditions and/or fillers which do not degrade by calcination, then the loss on ignition is equal to the resin content. The resin content calculated in this way includes the combustible part of the other components in the composition (glass size, pigments, etc.) but this is usually small compared to the resin content.

NOTE 2 In cases where fillers are present that are degraded at the test temperature, it is not possible to obtain an accurate determination of the glass, resin or filler content.

5 Sampling

5.1 The determination of the glass and filler contents shall be carried out in parallel on two specimens which are as near identical as possible. The result of the test is the average of the measurements on the two specimens, provided that the difference between the two measurements is less than 5 %. If this is not the case, a third specimen shall be tested which is as near identical to the other two as possible. The three values shall then be used to calculate the test result.

5.2 In order to carry out an evaluation test, the result of which is as representative as possible of the glass and filler content of the elementary unit or laboratory sample, this test procedure may need to be repeated a certain number of times, at specific locations in the elementary unit examined. The number of times and the actual locations will be specified either in the product specification or by the person requesting the analysis. In the latter case, the number and location will be decided by experience or as the result of previous work.

5.3 For all tests other than those on elementary units, take specimens that are as representative of the material under test as circumstances allow.

6 Preparation of test specimens

The test specimens shall be fully representative of the piece or batch examined. They shall be obtained in accordance with Clause 5.

Unless otherwise specified, it is recommended that the specimens be cut out in a shape which allows them to fit into a silica boat or porcelain crucible.

The mass of each specimen shall be within the range:

- 2 g to 20 g for prepregs and moulding compounds;
- 2 g to 10 g for laminates.

For each test result, use a minimum of two specimens (see 5.1).

In the case of prepregs and moulding compounds which contain solvents or free monomer, care shall be taken to avoid loss of volatile matter. For SMC, the protective release film shall not be removed from the laboratory sample or the test specimen until just before commencing the test procedure. All prepreg and moulding-compound laboratory samples, including SMC, shall be sealed in a vapour proof plastic bag immediately after the laboratory sample has been taken.

7 Determination

7.1 General

The choice of method used to determine the glass and filler content will depend on the presence or absence of filler.

7.2 Method A

7.2.1 Reagents

No reagents are required for method A.

7.2.2 Apparatus

Normal laboratory apparatus, plus the following:

7.2.2.1 Balance, maximum permissible error of 0,1 mg.

7.2.2.2 Silica boat or porcelain crucible, of a suitable size to contain a specimen.

7.2.2.3 Muffle furnace, capable of maintaining the chosen temperature (see 7.2.3.2) to within ±20 °C.

7.2.2.4 Desiccator, containing a suitable drying agent (e.g. silica gel).

7.2.2.5 Ventilated drying oven, set at 105 °C ± 3 °C.

7.2.3 Procedure

7.2.3.1 Preparation of the boat or crucible

Weigh the clean, dry boat or crucible (7.2.2.2) to the nearest 0,1 mg on the balance (7.2.2.1). Place in the muffle furnace (7.2.2.3) set to the chosen temperature (see 7.2.3.2), and leave for 10 min. After cooling to ambient temperature in the desiccator (7.2.2.4) verify that the mass has not changed. Constant mass is reached when the difference in mass of two successive weighing is less than 0,5 % of the specimen mass.

7.2.3.2 Calcination

Weigh the clean, dry boat or crucible, prepared as indicated in 7.2.3.1. Record the mass in grams as m_1.

Place the test specimen to the boat or crucible and dry at 105 °C for at least 10 min in the ventilated drying oven (7.2.2.5) to constant mass. Constant mass is reached when the difference in mass of two successive weighing is less than 0,5 % of the specimen mass.

Cool to ambient temperature in the desiccator and reweigh. Record the mass in grams as m_2.

In the case of specimens which contain volatile matter, care is needed to avoid loss of the volatile matter. Omit the drying stage therefore. Remove the specimen from the vapour-proof bag and remove any release film. Place the specimen in the boat or crucible, weigh and record the mass in grams as m_6.

Place the boat or crucible containing the test specimen in the muffle furnace, preheated to a temperature of 625 °C and heat to constant mass. Constant mass is reached when the difference in mass of two successive weighing is less than 0,5 % of the specimen mass.

For reinforced products with glass or filler which will not withstand this calcination temperature, a temperature between 500 °C and 600 °C may be used, in accordance with the specification for the glass or filler. It is essential to maintain the chosen temperature constant to ±20 °C.

Allow the boat or crucible, together with the residue, to cool in the desiccator to ambient temperature and reweigh. Record the mass in grams as m_3.

7.2.4 Expression of results

Calculate, for each specimen, the glass content M_{glass}, expressed as a percentage of the initial mass, using Formula (1):

$$M_{\text{glass}} = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \tag{1}$$

where

m_1 is the initial mass, in grams, of the dry boat or crucible;

m_2 is the initial mass, in grams, of the dry boat or crucible plus dried specimen;

m_3 is the final mass, in grams, of the boat or crucible plus residue after calcination.

In the case of specimens containing volatile matter, when the drying stage was omitted, replace m_2 in Formula (1) by m_6, where m_6 is the initial mass, in grams, of the dry boat or crucible plus undried test piece.

If the results of the individual measurements differ by more than 5 % in relative value, carry out an additional determination on a third specimen taken from the same location in the elementary unit or laboratory sample.

Express the result of the test as the average of the two (or three) individual measurements.

7.3 Method B

7.3.1 Reagents

The following reagents are required for method B.

7.3.1.1 Hydrochloric acid, with a volume fraction of 35 % concentration, commercial grade.

7.3.1.2 Denatured ethanol.

7.3.1.3 Chromic acid mixture, for cleaning.

The recommended composition of the mixture is 7 % sodium dichromate in concentrated sulfuric acid.

7.3.2 Apparatus

The apparatus given in 7.2.2, plus the following:

7.3.2.1 40 mm diameter sintered-glass filter, porosity P 160. i.e. 100 µm to 160 µm (see ISO 4793).

7.3.2.2 250 ml beaker.

7.3.2.3 Suction flask.

7.3.2.4 Glass rod.

7.3.2.5 Tweezers.

7.3.3 Procedure

7.3.3.1 Preparation of boats or crucibles

Follow the procedure given in 7.2.3.1.

7.3.3.2 Preparation of sintered-glass filter

Before each test, clean the sintered-glass filter (7.3.2.1) by soaking in the chromic acid mixture (7.3.1.3). Place the filter on the suction flask (7.3.2.3), apply the suction, and rinse the filter first with warm water, then with denatured ethanol (7.3.1.2). Dry the filter in the drying oven (7.2.2.5) to constant mass. Constant mass is reached when the difference in mass of two successive weighing is less than 0,5 % of the specimen mass. Record the mass in grams as m_4.

7.3.3.3 Calcination

Follow the procedure given in 7.2.3.2.

7.3.3.4 Separation of glass and filler when filler completely dissolves in hydrochloric acid

Separate the filler from the glass as follows:

Place into the 250 ml beaker (7.3.2.2), 5 ml of hydrochloric acid (7.3.1.1) per gram of residue remaining in the boat or crucible after calcination.

Using the glass rod, slowly add the residue in the boat or crucible to the acid in the beaker. Stir carefully to ensure that all the residue has reacted with the acid, taking care that the effervescence caused by the acid reacting with the carbonate fillers does not cause droplets to spit out of the beaker.

When the effervescence finished, three-quarters fill the boat or crucible with water and pour it all into the 250 ml beaker, repeating if necessary until all the residue has been transferred to the beaker.

Add an additional 50 ml of water to the beaker.

Place the filter, dried and weighed as described in 7.3.3.2, on the suction flask and apply the suction. Slowly pour the acid over the glass on to the filter.

Rinse the glass in the beaker with water and pour the water on to the filter; then rinse with denatured alcohol, agitating with a glass rod, and pour alcohol on to the filter.

Repeat this operation four or five times until the glass is thoroughly clean.

Transfer the glass on to the filter using the glass rod and a jet of denaturated ethanol. Rinse twice with denaturated ethanol.

Dry the filter to constant mass in the drying oven. Constant mass is reached when the difference in mass of two successive weighing is less than 0,5 % of the specimen mass.

Allow to cool in the desiccator to ambient temperature and weigh. Record the mass in grams as m_5.

Alternatively, if the fibre length is greater than 12 mm, the procedure described in Annex A may be used. The procedure given in 7.3.3.4 shall always be used as the reference method, however.

7.3.3.5 Separation of glass and filler when filler does not completely dissolve in hydrochloric acid

If filler insoluble in hydrochloric acid remains on the filler on filtering, determine the mass m_5 as described in 7.3.3.4.

Then, using tweezers (7.3.2.5), pick out all the glass from the filter. Reweigh the filter with the insoluble filler on it. Record this mass in grams as m_7.

NOTE If the glass filaments are very short, it might not be possible to separate them from the filler manually. In this case, the method given in this document is not capable of giving accurate values of the glass and filler contents separately, although the combined filler and glass content can be determined.

7.3.4 Expression of results

Calculate, for each specimen, the glass content M_{glass} and the filler content M_{filler}, expressed as a percentage of the initial mass, using Formulae (2) and (3), respectively:

$$M_{\text{glass}} = \frac{m_5 - m_4}{m_2 - m_1} \times 100 \tag{2}$$

$$M_{\text{filler}} = \left(\frac{m_3 - m_1}{m_2 - m_1} - \frac{m_5 - m_4}{m_2 - m_1} \right) \times 100 \tag{3}$$

where

m_1 is the initial mass, in grams, of the dry boat or crucible;

m_2 is the initial mass, in grams, of the dry boat or crucible plus the dried specimen;

m_3 is the final mass, in grams, of the boat or crucible plus residue after calcination;

m_4 is the mass, in grams, of the dry filter;

m_5 is the mass, in grams, of the filter plus contents after reacting the residue with acid.

In the case of moulding compounds containing volatile matter, when the drying stage was omitted, replace m_2 in the formula by m_6, where m_6 is the initial mass, in grams, of the dry boat or crucible plus undried test piece.

If filler insoluble in hydrochloric acid remained on the filter on filtering, replace m_4 in Formulae (2) and (3) by m_7, where m_7 is the mass, in grams, of the dry filter plus undissolved mineral filler.

If the results of the individual measurements differ by more than 5 % in relative value, carry out an additional determination on a third specimen taken from the same location in the elementary unit or laboratory sample. Express the result of the test as the average of the two (or three) individual measurements.