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Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of mass per unit area and fibre mass per unit area

Plastiques renforcés de fibres — Mélanges à mouler et préimprégnés — Détermination de la masse surfacique et de la masse des fibres par unité de surface

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 documents 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 fourth edition cancels and replaces the third edition (ISO 10352:2010), which has been technically revised.

The main changes compared to the previous edition are as follows:

— determination of fibre mass per unit area by Method A, Method B, Method C, Method D and Method E 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.

Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of mass per unit area and fibre mass per unit area

1 Scope

This document specifies a method for the determination of the mass per unit area. It also specifies five methods (Method A to Method E) for the determination of the fibre mass per unit area of moulding compounds and prepregs. The five methods are as follows:

— Method A: Extraction by Soxhlet;

— Method B: Extraction by immersion in solvent in a beaker;

— Method C: Decomposition by loss ignition;

— Method D: Extraction by wet combustion;

— Method E: Method by calculation.

This document is applicable to the following types of materials:

— moulding compound and preimpregnated unidirectional sheet, tape, fabric and mats;

— prepregs in which any type of reinforcement (aramid, carbon, glass, etc.) and any type of matrix (thermosetting or thermoplastic) has been used.

Typically, reinforcement fibres are coated with sizing or finishes. These normally dissolve with the resin and are, therefore, included in the resin content.

This document is not applicable to the following types of prepregs:

— those containing reinforcements which are soluble (or partly soluble) in the solvents used to dissolve the resin.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 1889, *Reinforcement yarns — Determination of linear density*

ISO 4602, *Reinforcements — Woven fabrics — Determination of number of yarns per unit length of warp and weft*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
elementary unit
individual sample roll, sheet or pack which is intended for the measurement of the mass per unit area using this document

Note 1 to entry: Prepregs are usually supplied in rolls or in packs of sheets. In this context, an individual roll or pack of sheets is an elementary unit.

3.2
laboratory sample
sample taken from an *elementary unit* (3.1)

3.3
test specimen
specimen cut from a *laboratory sample* (3.2)

4 Principle

4.1 Determination of mass per unit area

The mass of a test specimen of known area is determined. Two different specimen sizes are specified, depending on the type of material. If the material has been manufactured using a solvent or if the volatile-matter content of the material is not negligible, the sample is conditioned in a specified atmosphere before test specimens are taken. The result is expressed as the mass per unit area.

4.2 Determination of fibre mass per unit area

4.2.1 Method A: Extraction in Soxhlet-type apparatus

Determination of mass by means of weighing to constant mass after extraction of the resin with suitable solvent in a Soxhlet-type apparatus. Solvent to use shall be able to extract all the resin component completely from the specimen.

4.2.2 Method B: Extraction by immersion in solvent in a beaker

Method B is similar to Method A (see 4.2.1) but faster. In case of dispute, Method A shall be applied.

4.2.3 Method C: Decomposition by loss on ignition

Determination of mass by means of weighing to constant mass after decomposition of the resin by ignition.

4.2.4 Method D: Extraction by wet combustion

Determination of mass by means of weighing to constant mass after extraction of the resin by acid digestion. Use a solution of concentrated sulphuric acid and hydrogen peroxide.

4.2.5 Method E: Method by calculation

If the linear density (in accordance with ISO 1889) and the number of yarns per unit (in accordance with ISO 4602) in use is known, the fibre mass per unit area can be simply calculated.

5 Apparatus and reagents

5.1 General

Normal laboratory equipment is required plus the following specific apparatus.

5.1.1 Balance, graduated in tenths of 0,1 mg and an accuracy of 0,5 mg.

5.1.2 Square template, with the dimensions given in Table 1 for the material being tested, with an accuracy of 0,1 mm on the length of each side.

5.1.3 Ancillary items, such as a sharp cutting tool and tweezers.

5.1.4 Desiccator, with containing a suitable drying agent (for example silica gel, calcium chloride or phosphorus pentoxide).

5.2 For Method A

5.2.1 Single thickness extraction thimble, nominal diameter 20 mm to 22 mm, nominal length 60 mm to 80 mm.

5.2.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.2.3 Extraction apparatus of the Soxhlet type, comprising a condenser, siphon tube and flask and provided with an electric heating mantle.

5.2.4 Suitable solvent for extraction, (analytical grade), solvent to use shall be able to extract all the resin component completely from the specimen.

NOTE 1 General solvent is methyl-ethyl-ketone (MEK), tetra-hydro-furan (THF), acetone, N-methyl-pyrrolidone (NMP), denatured ethanol and others.

NOTE 2 Other solvents or the combination of more than one solvent can be used in order to extract the full all of the resin component from the specimen.

5.3 For Method B

5.3.1 Container 400 ml, Erlenmeyer flask or beaker.

5.3.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.3.3 Suitable solvent for extraction, (analytical grade) solvent to use shall be able to extract all the resin component completely from the specimen.

NOTE 1 General solvent is methyl-ethyl-ketone (MEK), tetra-hydro-furan (THF), acetone, N-methyl-pyrrolidone (NMP), denatured ethanol and others.

NOTE 2 Other solvents or the combination of more than one solvent can be used in order to extract the full all of the resin component from the specimen.

5.3.4 Acetone (propanone)

5.4 For Method C

5.4.1 Crucible from porcelain or equivalent properties, with a capacity sufficient to completely contain the specimen.

5.4.2 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.4.3 Muffle designed electric furnace, capable of maintaining a temperature of 565 °C with an accuracy of 30 °C.

5.4.4 Bunsen flame.

5.4.5 Heatproof gloves and safety screen for eye protection.

5.4.6 Laboratory exhausts fan and/or ventilation system.

5.5 For Method D

5.5.1 Erlenmeyer 250 ml double necked, pear-shaped flask, with 50 ml dropping funnel equipped with air inlet and a water pump.

5.5.2 Heat source, with suitable temperature control.

5.5.3 Container 400 ml, Erlenmeyer flask or beaker.

5.5.4 20 ml sintered glass crucible and suitable filtration assembly.

5.5.5 Electric oven, capable of maintaining a temperature of 105 °C with an accuracy of 5 °C.

5.5.6 Protective equipment, with protective clothing and rubber gloves resistant to hydrogen peroxide and sulphuric acid solutions, safety screen for eye protection.

5.5.7 Concentrated sulphuric acid, with specific gravity: 1,84 to 1,89.

5.5.8 Hydrogen peroxide solution, with concentration: 300 g/l to 500 g/l.

5.5.9 Acetone (propanone).

5.5.10 Distilled water.

6 Conditioning and testing

6.1 Conditioning

6.1.1 Materials for which no conditioning is required

Conditioning is not required for the following materials:

— those which are known to have been manufactured without using a solvent and to have been stored under conditions close to standard atmospheric conditions;

