



SLOVENSKI STANDARD SIST EN ISO 11667:2000

01-maj-2000

Nj`U_b]c`U Yb]dc`ja Yfb]a UHf]U]!`Na Yg]nUcV`_cj Ub`Y]b`dfYdfY[Y!
8c`c Yj Ub`Y`ga c`Yzj`U_Yb`nUc`U Yb`Y]b`a]bYfUb]`dc`b]!`A YrcXUn
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Fibre-reinforced plastics - Moulding compounds and prepregs - Determination of resin, reinforced-fibre and mineral-filler content - Dissolution methods (ISO 11667:1997)

Faserverstärkte Kunststoffe - Formmassen und Prepregs - Bestimmung des Gehaltes an Harz, Verstärkungsfaser und Mineralfüllstoff - Auflösungsverfahren (ISO 11667:1997)

Plastiques renforcés de fibre - Préimprégnés et compositions de moulage - Détermination des taux de résine, de fibre de renfort et de charge minérale - Méthodes par dissolution (ISO 11667:1997)

Ta slovenski standard je istoveten z: EN ISO 11667:1999

ICS:

83.120 Ube a] [|ã ^iã Reinforced plastics

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 11667

June 1999

ICS 83.120

English version

Fibre-reinforced plastics - Moulding compounds and prepregs -
Determination of resin, reinforced-fibre and mineral-filler content
- Dissolution methods (ISO 11667:1997)

Plastiques renforcés de fibre - Préimprégnés et
compositions de moulage - Détermination des taux de
résine, de fibre de renfort et de charge minérale - Méthodes
par dissolution (ISO 11667:1997)

Faserverstärkte Kunststoffe - Formmassen und Prepregs -
Bestimmung des Gehaltes an Harz, Verstärkungsfaser und
Mineralfüllstoff - Auflösungsverfahren (ISO 11667:1997)

This European Standard was approved by CEN on 6 May 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 1999, and conflicting national standards shall be withdrawn at the latest by December 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 11667:1997 has been approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

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REPUBLIKA SLOVENIJA
 MINISTRSTVO ŠOLSTVA, VEŠTINE IN ŠPORTA
 REPUBLIC OF SLOVENIA
 MINISTRY OF EDUCATION, SCIENCE AND SPORTS
 Ljubljana, 2000

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Annex ZA (normative)**Normative references to international publications
with their relevant European publications**

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

| <u>Publication</u> | <u>Year</u> | <u>Title</u> | <u>EN</u> | <u>Year</u> |
|--------------------|-------------|--|------------|-------------|
| ISO 291 | 1997 | Plastics - Standard atmospheres for conditioning and testing | EN ISO 291 | 1997 |

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INTERNATIONAL STANDARD

ISO
11667

First edition
1997-12-15

Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of resin, reinforced-fibre and mineral-filler content — Dissolution methods

*Plastiques renforcés de fibre — Préimprégnés et compositions
de moulage — Détermination des taux de résine, de fibre de renfort et
de charge minérale — Méthodes par dissolution*

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Reference number
ISO 11667:1997(E)

ISO 11667:1997(E)**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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11667 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Annex A of this International Standard is for information only.

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Fibre-reinforced plastics — Moulding compounds and prepregs — Determination of resin, reinforced-fibre and mineral-filler content — Dissolution methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies two dissolution methods for the determination of the resin, reinforcement-fibre and mineral-filler contents of moulding compounds and prepregs.

Method A: Extraction by Soxhlet. In cases of dispute, this method is the reference method.

Method B: Extraction by immersion in solvent in a beaker. This method uses simpler equipment, making it suitable for quality assurance testing.

This International Standard is applicable to the following types of material:

- prepregs made from yarns, rovings, tapes and fabrics;
- SMC, BMC and DMC moulding compounds.

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

This International Standard is not applicable to the following types of reinforced plastic:

- those containing reinforcements which are soluble (or partly soluble) in the solvents used to dissolve the resin;
- those in which the resin is partly or fully cured and so not fully soluble in organic solvents.

NOTE — ISO 1172:1996, *Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content — Calcination methods*, may be used where the resin is cured.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*.

ISO 472:1988, *Plastics — Vocabulary*.

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*.

3 Definitions

For the purposes of this International Standard, the relevant definitions given in ISO 472 apply, with the following addition:

3.1 elementary unit

Smallest normally commercially available entity of a given product.

4 Principle

The non-soluble material content (reinforcement fibre + filler) is obtained by the measurement of the difference in the mass of a test specimen before and after extraction by solvent under defined conditions.

The soluble part (resin) is extracted either in a Soxhlet apparatus (method A) or by immersion in a beaker (method B).

When the material under test contains fillers, the separation between fibres and fillers is obtained either by reacting the mineral filler with hydrochloric acid (method A) or by successive filtration without the use of acid (method B).

The masses before dissolution, after dissolution and after reaction with acid or filtration enable the reinforcement, resin and filler contents to be calculated.

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5 Sampling

5.1 The test shall be carried out on two specimens which are as identical as possible. On prepreg fabrics, the specimens shall be taken at least 50 mm from the edge.

Two test specimens are sufficient providing that the difference between the values obtained is 5 % or less. If this is not the case, a third specimen shall be tested which is as identical to the other two as possible.

5.2 In order to obtain an evaluation result that is as representative as possible of the resin, reinforcement-fibre and mineral-filler content of the elementary unit examined, this test procedure may need to be repeated a certain number of times, at specific places in the elementary unit. The number and location of the tests required should be defined either in the product specification or by the person requesting the analysis. Alternatively, the number and location may be decided by prior experience.

5.3 For all tests which are not carried out on elementary units, take test specimens that are as representative of the material under test as circumstances allow.

6 Preparation of test specimens

Unless otherwise specified, it is recommended that the test specimens be cut in a shape which allows them to fit into the thimble of the Soxhlet apparatus or the beaker used for the extraction.

The mass should preferably be in the range 2 g to 20 g.

NOTE — For SMC, BMC and DMC tested by method B, a larger test specimen may be used if this is more representative of the material.

In the case of preregs and moulding compounds which contain solvents or free monomer, care shall be taken to avoid loss of volatile matter. It is recommended that materials containing free monomer should not be left unprotected for more than 5 min before commencing the test procedure. For SMC, the protective release film shall not be removed from the laboratory sample or 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 is taken.

7 Test methods — General

The method used to dissolve the resin depends on the accuracy of the result required by the person ordering the test. The procedures for separating the reinforcement fibre from the mineral filler are different in the two methods.

The method requires the masses of all specimens to be brought to a constant value by repetition of the drying and dissolution stages and by reweighing until the difference between two consecutive weighings is less than 1 mg. In those cases where known materials are being tested regularly, however, it is permitted to define, by experimentation, minimum times for dissolution and drying, to be certain that constant mass has been reached.

8 Method A: Extraction by Soxhlet

8.1 Reagents

The solvent used shall be capable of completely extracting all the resin from the test specimen.

Solvents suitable for the more common resins include:

dichloromethane;

acetone;

methyl ethyl ketone (butan-2-one);

denatured ethanol.

Other solvents may be used as necessary to achieve complete extraction.

8.2 Apparatus

Normal laboratory equipment, plus the following:

8.2.1 Balance, accurate to 0,5 mg and graduated to 0,1 mg.

8.2.2 Soxhlet extraction apparatus with a **thimble** large enough to contain the whole of the test specimen.

8.2.3 Cutting tool.

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

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

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