INTERNATIONAL STANDARD

**ISO 14127**

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# Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents

*Composites renforcés de fibres de carbone — Détermination des teneurs en résine, en fibre et en vide*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 14127 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

# Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents

## 1 Scope

This International Standard specifies methods for calculating the resin, fibre and void contents of a carbon-fibre-reinforced composite from the densities of the resin, the fibre and the composite and the mass of fibre in the composite (method A) and for calculating the fibre content from the thickness of the composite (method B).

Method A specifies three different resin removal procedures for the determination of the mass of fibre in the composite (viz a combustion procedure, a procedure by digestion in nitric acid and a procedure by digestion in a mixture of sulfuric acid and hydrogen peroxide). The selection of the procedure to be used is made by considering the combustibility of the resin used in the composite, its ability to decompose and the type of resin concerned. It should be noted that method A is only of limited applicability when filled resins are present that could prevent complete dissolution and/or combustibility of the resin.

Method B (thickness measurement method) is only applicable to composites moulded from prepregs of known fibre mass per unit area.



## 2 Normative references



The following referenced documents are indispensable for the application 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 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

ISO 1183-2, *Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method*

ISO 1183-3, *Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 10119, *Carbon fibre — Determination of density*

## 3 Health and safety

This International Standard limits itself to describing the determination of the resin, fibre and void contents of composites reinforced with carbon fibre. The conditions under which the test specimens, apparatus and reagents are handled shall comply with the national regulations in force in each country and the staff shall be informed of the hazards involved and appropriate precautions taken.

## 4 Terms and definitions

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

**4.1**
**fibre content by mass**
ratio of the mass of fibre in a composite to the total mass of the composite

NOTE It is expressed as a percentage.

**4.2**
**fibre content by volume**
ratio of the volume of fibre in a composite to the total volume of the composite

NOTE It is expressed as a percentage.

**4.3**
**void content**
ratio of the volume of the voids (hollow spaces) in a composite to the total volume of the composite



NOTE It is expressed as a percentage.



## 5 Principle

### 5.1 Method A (resin removal method)

#### 5.1.1 Procedure A1: combustion procedure

The mass of a test specimen is determined before and after combustion of the resin in the upper part of the reducing (non-oxygen) flame of a Bunsen burner.

NOTE The combustion procedure makes use of the relative ease of decomposition of resins, compared to carbon fibres, in inert gases. The procedure consists of heating a specimen of composite material with the reducing flame of a Bunsen burner so that only the resin is removed by combustion. However, its application is limited to resins that decompose completely by combustion. Therefore, this procedure is not applicable to resins that are not completely combustible, such as epoxy novolac and brominated systems. There are also drawbacks such as the fact that the accuracy of the combustion procedure is slightly inferior to that of the nitric acid digestion procedure and the sulfuric acid/hydrogen peroxide digestion procedure. Nonetheless, it is useful as a rapid test procedure which can be carried out safely and simply.

Because of the lack of reliability of the combustion procedure, its use shall be as agreed between the purchaser and supplier.

#### 5.1.2 Procedure A2: nitric acid digestion procedure

The mass of a test specimen is determined before and after digestion of the resin with concentrated nitric acid, which does not attack the carbon fibres excessively.

NOTE Both the nitric acid digestion procedure and the sulfuric acid/hydrogen peroxide digestion procedure make use of the fact that digestion of resins in a hot bath of nitric acid or sulfuric acid/hydrogen peroxide mixture is rapid compared

to carbon fibres (which resist digestion in such conditions). The procedure consists of soaking the composite in a hot bath of one of these reagents so that only the resins are removed by digestion. The nitric acid digestion procedure is applicable to all epoxy resins except acid anhydride curing substances. The sulfuric acid/hydrogen peroxide digestion procedure is applicable to all epoxy resins, phenolic resins and polyamide resins.

#### 5.1.3 Procedure A3: digestion in a sulfuric acid/hydrogen peroxide mixture

The mass of a test specimen is determined before and after digestion of the resin with an aqueous mixture of sulfuric acid and hydrogen peroxide, provided that the carbon fibres are not attacked.

See also the Note to 5.1.2.

### 5.2 Method B (thickness measurement method)

The thickness of the composite is measured over the entire surface of the composite. Using known values of the mass per unit area and the density of the reinforcement, the fibre content of the composite can be calculated.

## 6 Conditioning of test sample

A quantity of material sufficient to complete the tests is taken as the test sample and conditioned for a sufficient time to re-establish temperature equilibrium. This conditioning shall be carried out in one of the standard atmospheres specifed in ISO 291.



## 7 Apparatus and reagents



### 7.1 General

Normal laboratory equipment is required plus the following specific apparatus:

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

**7.1.2 Analytical balance**, accurate to 0,1 mg.

**7.1.3 Abrasive paper**, with grain size finer than 180 grit.

### 7.2 Method A

#### 7.2.1 Procedure A1 (combustion procedure)

**7.2.1.1 Bunsen burner**, compatible with the gas used.

**7.2.1.2 Nichrome wire**, about 0,2 mm in diameter.

#### 7.2.2 Procedure A2 (nitric acid digestion procedure)

**7.2.2.1 Borosilicate-glass vacuum filter**.

**7.2.2.2 200 ml conical flask**.

**7.2.2.3 100 ml measuring cylinder**.

**7.2.2.4 Water reflux condenser**, with a standard taper joint to fit the conical flask.

**7.2.2.5 Air-circulation drying oven**, capable of heating up to about 200 °C.

**7.2.2.6** **Acetone**, as specified in ISO 6353-2.

**7.2.2.7** **Concentrated nitric acid**, 62 % by mass.

**7.2.3 Procedure A3 (digestion in a mixture of sulfuric acid and hydrogen peroxide)**

**7.2.3.1** **Borosilicate-glass vacuum filter**.

**7.2.3.2** **Borosilicate-glass beaker**, minimum volume 200 ml.

**7.2.3.3** **100 ml measuring cylinder**.

**7.2.3.4** **Air-circulation drying oven**, capable of heating up to about 200 °C.

**7.2.3.5** **Acetone**, as specified in ISO 6353-2.

**7.2.3.6** **Concentrated sulfuric acid**, 96 % by mass.

**7.2.3.7** **30 % to 35 % hydrogen peroxide solution**.

**SAFETY PRECAUTIONS — When handling nitric acid, sulfuric acid or hydrogen peroxide, care shall be taken to prevent accidents, as follows. Wear rubber gloves, protective goggles, etc., in order to avoid direct contact of these reagents with the human body. If human skin does come into contact with one of these reagents, appropriate treatment shall be provided immediately. It is also necessary to ventilate the test area and/or room. Boiling 65 % hydrogen peroxide can decompose explosively; do not therefore allow it to concentrate by distillation. Waste chemicals should be kept separate and disposed of in a suitable way.**



### 7.3 Method B (thickness measurement method)



**7.3.1** **Ball micrometer** or **equivalent instrument**, reading to an accuracy of 0,01 mm, to measure the specimen thickness.

## 8 Test specimens

**8.1** The mass of the test specimens shall be 0,2 g to 0,5 g. They shall be less than 4 mm in thickness and shall be 6 mm to 10 mm in length and width.

**8.2** The locations from which the test specimens are taken shall be distributed randomly over the sample and be no nearer than 10 mm to any edge.

**8.3** The edges of the test specimens shall be ground square and smoothed with abrasive paper.

**8.4** At least three test specimens shall be taken unless otherwise specified by the party requesting the test.

## 9 Density measurements

**9.1** Determine the densities of the resin, the composite and the carbon fibre as follows.

**9.2** Determine the density of the resin ($\rho_r$) in accordance with ISO 1183-1, ISO 1183-2 or ISO 1183-3.

**9.3** Determine the density of the carbon fibre ($\rho_f$) in accordance with ISO 10119.

**9.4** Determine the density of the composite ($\rho_c$) by the immersion method specified in ISO 1183-1. Use water as the immersion liquid.

**9.5** When measuring resin and composite densities by the immersion method, it is essential that a correction be made for the mass of the wire used to suspend the specimen.

## 10 Procedures

### 10.1 General

Measure the mass of the test specimen and that of the fibre by procedure A1 (combustion procedure), procedure A2 (nitric acid digestion procedure) or procedure A3 (digestion in sulfuric acid/hydrogen peroxide). When using method B, simply measure the thickness of the composite (see 10.3).

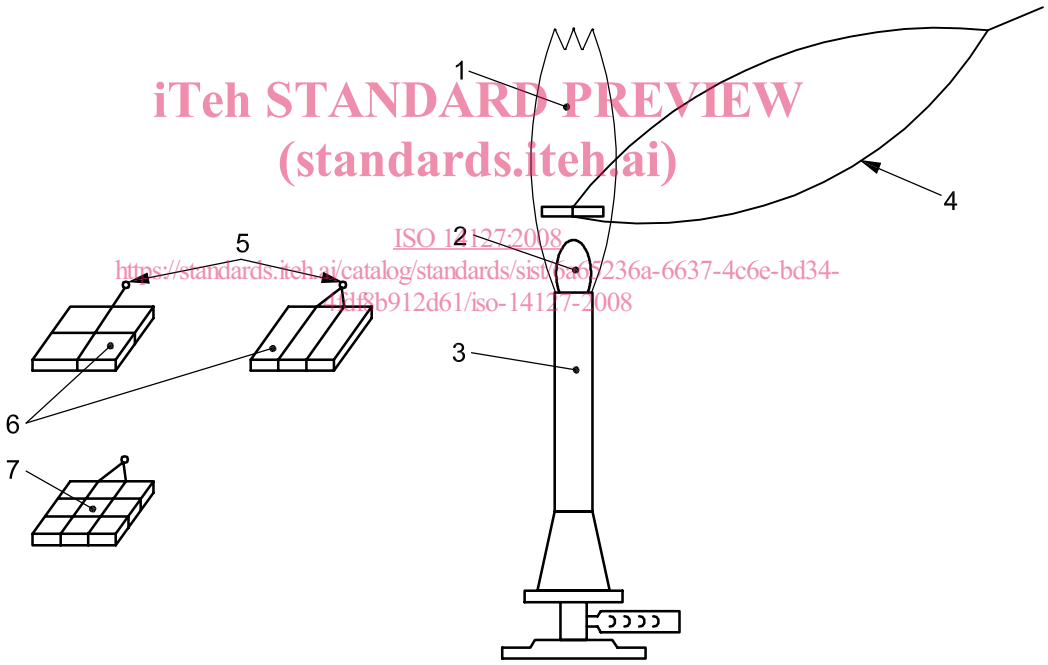
### 10.2 Method A

#### 10.2.1 Procedure A1 (combustion procedure)

**10.2.1.1** Weigh a test specimen to the nearest 0,1 mg ($m$).

**10.2.1.2** Bind the specimen with nichrome wire as shown in Figure 1. Weigh the specimen, including the nichrome wire, to the nearest 0,1 mg ($m_1$).






**Key**

1 flame
2 reducing zone of flame
3 Bunsen burner
4 tweezers
5 nichrome wire
6 specimen reinforced with unidirectional carbon fibres
7 specimen reinforced with woven carbon fibre fabric

**Figure 1 — Procedure A1 (combustion procedure)**