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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

Reference number
ISO 14127:2008(E)

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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 (ρ_r) in accordance with ISO 1183-1, ISO 1183-2 or ISO 1183-3.

9.3 Determine the density of the carbon fibre (ρ_f) in accordance with ISO 10119.

9.4 Determine the density of the composite (ρ_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

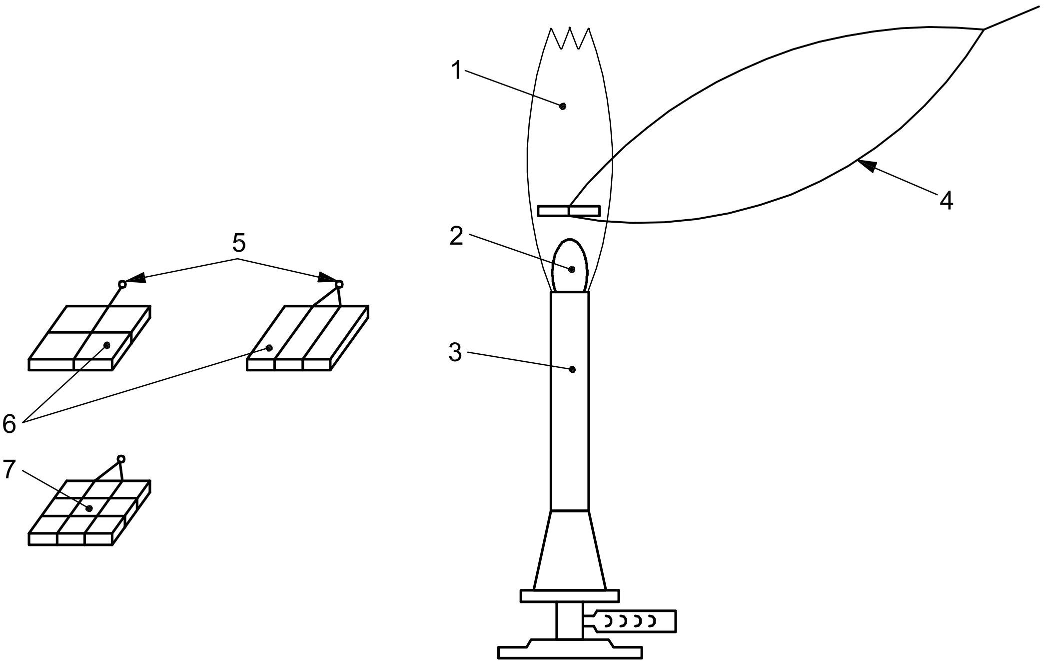
Measure the mass of the test specimen and that of the fibre by procedure A1 (combustion procedure), procedure A2 (nitric acid digestion procedure) or procedure A3 (digestion in sulfuric acid/hydrogen peroxide). When using method B, simply measure the thickness of the composite (see 10.3).

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)

10.2.1.3 Light the Bunsen burner with the air shut off. Adjust the height of the flame to between 15 cm and 20 cm. Gradually increase the supply of air to bring the height of the reducing flame (blue flame) to about 2 cm. Using tweezers to hold the nichrome wire binding the specimen, introduce the specimen horizontally into the flame a little above the top of the reducing zone of the flame, but without at any time touching the reducing zone, as shown in Figure 1.

10.2.1.4 Within five to twenty seconds, most of the resin in the specimen will be burnt off, producing a red flame and black soot. Continue to heat the specimen until its bottom surface becomes red hot. Then invert it until the other surface becomes red hot. When all the resin has been burnt off, the red flame will disappear. Continue heating until this happens. The total heating time will normally be 3 min or less.

10.2.1.5 Remove the specimen from the flame and place it on a metal plate to cool to room temperature. If the specimen is not weighed at once, place the plate plus specimen in a desiccator.

10.2.1.6 Weigh the specimen, including the nichrome wire, to the nearest 0,1 mg (m_2).

10.2.2 Procedure A2 (nitric acid digestion procedure)

10.2.2.1 Weigh a test specimen to the nearest 0,1 mg (m).

10.2.2.2 Using a measuring cylinder, measure 50 ml of concentrated nitric acid into a 200 ml conical flask.

10.2.2.3 Place the flask in an oil bath maintained at 120 °C ± 2 °C and place the test specimen in the flask.

10.2.2.4 Attach a reflux condenser to the flask and heat the flask for 90 min.

10.2.2.5 Dry a glass filter in an air-circulation oven at 105 °C ± 5 °C for 90 min, then cool it in a desiccator to room temperature and weigh it to the nearest 0,1 mg. Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Call this mass m_3.

10.2.2.6 After digestion, cool the solution and filter it through the glass filter, using vacuum suction, to separate off the fibre.

10.2.2.7 Wash out the flask with 20 ml to 30 ml of concentrated nitric acid, filtering the washings through the glass filter.

10.2.2.8 Wash the fibre retained on the filter with several approximately 100 ml portions of distilled water from a washing bottle and then with acetone.

10.2.2.9 Place the glass filter in an air-circulation oven and dry it at 105 °C ± 25 °C for 90 min.

10.2.2.10 After drying, remove the glass filter from the oven and place it in a desiccator for 30 min to cool to room temperature.

10.2.2.11 Weigh the glass filter plus fibre to the nearest 0,1 mg.

10.2.2.12 Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Call this mass m_4.

10.2.3 Procedure A3 (digestion in sulfuric acid/hydrogen peroxide mixture)

10.2.3.1 Weigh a test specimen to the nearest 0,1 mg (m).

10.2.3.2 Using a measuring cylinder, measure 30 ml of concentrated sulfuric acid into a 200 ml beaker.

10.2.3.3 Place the specimen in the concentrated sulfuric acid and cover the beaker with a watch-glass. To dissolve the resin, heat the beaker on a hot-plate (at 180 °C ± 10 °C) so that the sulfuric acid begins to fume.

10.2.3.4 When all the resin has dissolved and the sulfuric acid solution has become dark brown in colour, stop heating. Remove the beaker from the hot-plate.

10.2.3.5 Fill a syringe with about 20 ml of hydrogen peroxide solution. Slowly add small drops of hydrogen peroxide solution to the beaker. When all the fibres have floated up to the surface, leaving the sulfuric acid solution clear, add 2 ml more hydrogen peroxide solution and heat for 10 min prior to cooling to room temperature.

10.2.3.6 Dry a glass filter to constant mass (m_3), following the procedure described in 10.2.2.5.

10.2.3.7 Filter the contents of the beaker through the glass filter, using vacuum suction, to separate off the fibre.

10.2.3.8 Wash out the beaker with 20 ml to 30 ml of concentrated sulfuric acid, filtering the washings through the glass filter.

10.2.3.9 Wash the fibre retained on the filter with approximately 500 ml of distilled water from a washing bottle, ensuring that the washing water is close to pH 7. Then wash the fibre with acetone.

10.2.3.10 Place the glass filter in an air-circulation oven and dry it at 105 °C ± 5 °C for 90 min.

10.2.3.11 After drying, remove the glass filter from the oven and place it in a desiccator for 30 min to cool to room temperature.

10.2.3.12 Weigh the glass filter plus fibre to the nearest 0,1 mg.

10.2.3.13 Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Call this mass m_4.

10.3 Method B (thickness measurement method)

10.3.1 Measure the thickness at at least 10 points scattered uniformly across the surface of the composite.

NOTE It should be noted that the surface smoothness of the composite will affect the results of this measurement. The fibre content of a composite can be determined for the whole composite or for specific areas of the composite. Hence, a correction may need to be made for a non-uniform resin surface, such as that caused by the use of a peel ply during manufacture.

11 Calculation and expression of results

11.1 Method A (resin removal method)

11.1.1 In the case of procedure A1 (combustion procedure, see 5.1.1), calculate the fibre content by mass from Equation (1):

$$w_\mathrm{f} = \frac{m - m_1 + m_2}{m} \times 100 \qquad (1)$$

where

w_f is the fibre content (mass %);

m is the initial mass of the test specimen (g);

m_1 is the initial mass of the test specimen and nichrome wire (g);

m_2 is the mass of the test specimen and nichrome wire after combustion (g).

In the case of procedure A2 (nitric acid digestion procedure, see 5.1.2) and procedure A3 (digestion in sulfuric acid/hydrogen peroxide mixture, see 5.1.3), calculate the fibre content from Equation (2):

$$w_\mathrm{f} = \frac{m_4 - m_3}{m} \times 100 \tag{2}$$

where

m_3 is the mass of the glass filter (g);

m_4 is the mass of the test specimen and glass filter after digestion (g).

11.1.2 Calculate the fibre content by volume, the resin content by volume and the void content by volume from Equations (3), (4) and (5):

$$\varphi_\mathrm{f} = \frac{w_\mathrm{f} \times \rho_\mathrm{c}}{\rho_\mathrm{f}} \tag{3}$$

$$\varphi_\mathrm{r} = \frac{\left(100 - w_\mathrm{f}\right) \times \rho_\mathrm{c}}{\rho_\mathrm{r}} \tag{4}$$

$$\varphi_\mathrm{v} = 100 - (\varphi_\mathrm{f} + \varphi_\mathrm{r}) \tag{5}$$

where

φ_f is the fibre content (volume %);

φ_r is the resin content (volume %);

φ_v is the void content (volume %);

w_f is the fibre content (mass %);

ρ_c is the density of the test specimen (g/cm^3);

ρ_f is the density of the fibre (g/cm^3) (see Note);

ρ_r is the density of the resin (g/cm^3).

If the calculated value of the void content is negative, it is necessary to re-examine the densities of the resin, the fibre and the composite. In such cases, a solution shall be negotiated between the purchaser and supplier.

NOTE The density of the fibre can be obtained from the supplier's documentation.

11.2 Method B (thickness measurement method)

Calculate the fibre content by volume and the resin content by volume of the composite from the following equations:

$$\varphi_\mathrm{f} = \frac{N \times \rho_{A,\mathrm{p}}}{d \times \rho_\mathrm{f}} \times 10^{-1} \tag{6}$$

$$\varphi_\mathrm{r} = 100 - \varphi_\mathrm{f} \quad \text{(see Note 2)} \tag{7}$$

where

N is the number of plies in the composite;

$\rho_{A,\mathrm{p}}$ is the mass per unit area of the fibre in the plies of the prepreg (g/m^2) (see Note 1);

d is the thickness of the composite (mm);

ρ_f is the density of the fibre (g/cm^3) (see Note to 11.1.2).

NOTE 1 The mass per unit area of the fibre in prepregs can be obtained either from the supplier's documentation or by direct measurement using specimens taken from an area adjacent to the area of the prepreg used in the manufacture of the composite.

NOTE 2 The calculation of the resin content assumes a 0 % void content.

12 Precision

The precision data were determined from an interlaboratory trial organized in 1998 and involving four laboratories, the results of which were analysed in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3 (see Table 1).

The reproducibility is the precision under conditions in which test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment, and is expressed in terms of a reproducibility standard deviation or a reproducibility deviation.

The within-laboratory reproducibility is the precision under conditions in which test results are obtained with the same method on identical test material in the same laboratory, but with any operator, equipment and/or time of measurement.

Table 1 — Interlaboratory trial data

	Test procedure	Average result %	Within-laboratory reproducibility standard deviation $S_{R\mathrm{W}}$	Reproducibility standard deviation S_R
φ_f	Procedure A1	61,17	1,12	1,72
	Procedure A2	57,80	1,16	1,68
	Procedure A3	57,23	1,22	1,87
φ_r	Procedure A1	36,95	3,22	4,06
	Procedure A2	41,91	1,91	2,84
	Procedure A3	42,69	2,82	3,47

13 Test report

The test report shall include the following information:

a) a reference to this International Standard (ISO 14127:2008), indicating the procedure used (combustion procedure, nitric acid digestion procedure, digestion with sulfuric acid/hydrogen peroxide mixture or thickness measurement);

b) all details necessary for complete identification of the material tested, including type, source, manufacturer's code number and previous history, where these are known;

c) the method of preparation of the test specimens (moulding method used, moulding conditions, etc.);

d) the number of specimens tested;

e) the dimensions and/or mass of the specimens;

f) the temperature, humidity and time for conditioning of the specimens;

g) the test result for each individual specimen and, if specified by the party requesting the test, the mean value for all the specimens;

h) details of any deviations from this International Standard and any other information necessary;

i) the date of the test.

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