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Carbon fibre — Determination of size content

Fibres de carbone — Détermination du taux d'ensimage

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10548 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

This second edition cancels and replaces the first edition (ISO 10548:1994), which has been technically revised.

Carbon fibre — Determination of size content

1 Scope

This International Standard specifies test methods for the determination of the size content of carbon fibre yarn. It is applicable to continuous-filament yarns and staple-fibre yarns.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1886:1990, *Reinforcement fibres — Sampling plans applicable to received batches*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1

size

any material applied to fibres to facilitate the handling and use of the fibres

3.2

size content

mass of the size expressed as a percentage of the sized carbon fibre yarn

4 Principle

Test specimens are weighed before and after removal of the size by one of the following three methods:

- **method A**: size removal by Soxhlet extraction, to be used when the size is completely soluble in a suitable solvent;
- **method B**: size removal by chemical digestion using a mixture of sulfuric acid and hydrogen peroxide, used when the size is partially hardened and not completely soluble in solvents;
- **method C**: size decomposition by pyrolysis at high temperature in an atmosphere of nitrogen, used when the size is completely removed by pyrolysis.

Methods A and B require the last weighing to be made to constant mass by repetition of the final drying and weighing procedure. In those cases where known materials are being tested regularly, it is permitted to define, by experiment, a minimum time for drying which will ensure that constant mass has been reached. Particular care may be required if the carbon fibre or sizing is water-absorbent.

5 Test specimens

Two test specimens or more shall be taken from each lot or yarn sample. The size content is taken as the average of the two or more determinations.

If the determination is carried out for the purpose of lot acceptance, the lot received shall be sampled in accordance with ISO 1886.

When handling test specimens, gloves shall be worn or tweezers used to avoid damage to the specimens.

NOTE The specification or the person requiring the test may stipulate that additional determinations be carried out at different places within the elementary unit.

6 Conditioning

Elementary units and yarn samples shall be allowed to attain laboratory temperature before testing. Before weighing the yarn, it shall be dried if required.

7 Test methods

7.1 Method A: Solvent extraction

7.1.1 Apparatus and materials

7.1.1.1 Balance, accurate to 0,5 mg, readable to 0,1 mg.

7.1.1.2 Hot-air oven, capable of being controlled to ± 5 °C.

7.1.1.3 Desiccator, containing a suitable desiccant, for example silica gel, anhydrous calcium chloride or phosphorous pentoxide.

7.1.1.4 Soxhlet extractor, 200 ml capacity, with a 500 ml flask.

7.1.1.5 Soxhlet extraction thimbles, measuring 41 mm × 123 mm.

NOTE The sizes of the Soxhlet extractor, the flask and the Soxhlet extraction thimbles are given as an indication only and may be varied to suit individual requirements.

IMPORTANT — The extraction and all handling of organic solvents should be carried out in a fume cupboard.

7.1.1.6 Rubber gloves or **tweezers**.

7.1.1.7 Cutting blade.

7.1.1.8 Heating mantle, fitted with a power regulator capable of controlling the boiling rate of the solvent.

7.1.1.9 Distilled water, or an organic solvent such as butan-2-one (methyl ethyl ketone), tetrahydrofuran, dichloromethane (methylene chloride), acetone or dichloroethane.

NOTE The choice of solvent depends upon the type of size applied to the carbon fibre under test. This choice is best made by consultation between the customer and the supplier.

7.1.2 Procedure 1

7.1.2.1 Dry an extraction thimble (7.1.1.5) at 105 °C ± 5 °C in the oven (7.1.1.2) for 1 h and allow to cool to room temperature in a desiccator (7.1.1.3).

7.1.2.2 Weigh the thimble to the nearest 0,1 mg (m_1).

7.1.2.3 Place a test specimen in the thimble and weigh to the nearest 0,1 mg (m_2).

7.1.2.4 Place the thimble and its contents in the siphon chamber of the Soxhlet apparatus (7.1.1.4), and place a sufficient volume of solvent in the flask to ensure operation of the siphon during the reflux cycles.

7.1.2.5 Extract for 2 h, adjusting the heating mantle (7.1.1.8) so that at least four reflux cycles occur during the 2 h extraction period (a shorter extraction period may, however, be used if it can be demonstrated to give complete extraction).

7.1.2.6 Turn off the heating mantle. Wait 10 min for the apparatus to cool. Remove the thimble and contents. Allow to stand at ambient temperature for 10 min to allow excess solvent to evaporate.

7.1.2.7 Dry the thimble and contents for 1 h in the oven (7.1.1.2) at 110 °C ± 5 °C (or, if the solvent used has a boiling point higher than 100 °C, at a temperature 10 °C above the boiling point of the solvent) and allow to cool in a desiccator. Weigh to the nearest 0,1 mg.

7.1.2.8 Repeat the drying and weighing procedure until the difference in mass between two successive weighings (m_3) is less than ± 0,2 mg. If a minimum drying time known to give constant mass has been established (see clause 4), this requirement does not apply.

7.1.3 Procedure 2

7.1.3.1 If the specimen is free of loose fibre and can be coiled into a form which can be inserted into the Soxhlet extraction chamber without the danger of loss of fibre during extraction, an extraction thimble need not be used.

7.1.3.2 Weigh the test specimen to the nearest 0,1 mg (m_4).

7.1.3.3 Carry out the extraction as described in Procedure 1, but without the extraction thimble.

7.1.3.4 Dry the specimen for 1 h at 110 °C ± 5 °C [or a higher temperature (see 7.1.2.7)] and allow to cool in a desiccator. Weigh to the nearest 0,1 mg.

7.1.3.5 Repeat the drying and weighing procedure until the difference in mass between two successive weighings (m_5) is less than ± 0,2 mg. If a minimum drying time known to give constant mass has been established (see clause 4), this requirement does not apply.

7.1.4 Expression of results

7.1.4.1 Procedure 1

Calculate the size content SC, expressed as a percentage by mass, from the equation:

$$\mathrm{SC} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the initial mass, in grams, of the extraction thimble;

m_2 is the initial mass, in grams, of the thimble plus test specimen;

m_3 is the final mass, in grams, of the thimble plus test specimen after extraction.

7.1.4.2 Procedure 2

Calculate the size content SC, expressed as a percentage by mass, from the equation:

$$\mathrm{SC} = \frac{m_4 - m_5}{m_4} \times 100$$

where

m_4 is the initial mass, in grams, of the specimen;

m_5 is the final mass, in grams, of the specimen after extraction.

7.2 Method B: Digestion with sulfuric acid and hydrogen peroxide

7.2.1 Apparatus and reagents

7.2.1.1 Balance, accurate to 0,5 mg, readable to 0,1 mg.

7.2.1.2 Hot-air oven, capable of being controlled to $\pm$ 0,5 °C.

7.2.1.3 Desiccator, containing a suitable desiccant, for example silica gel, anhydrous calcium chloride or phosphorus pentoxide.

7.2.1.4 Fume cupboard.

7.2.1.5 Rubber gloves, **face shield** and **laboratory coat**.

7.2.1.6 Sintered-glass filters, porosity P 40, pore diameter 20 µm to 30 µm, capacity 30 ml to 70 ml.

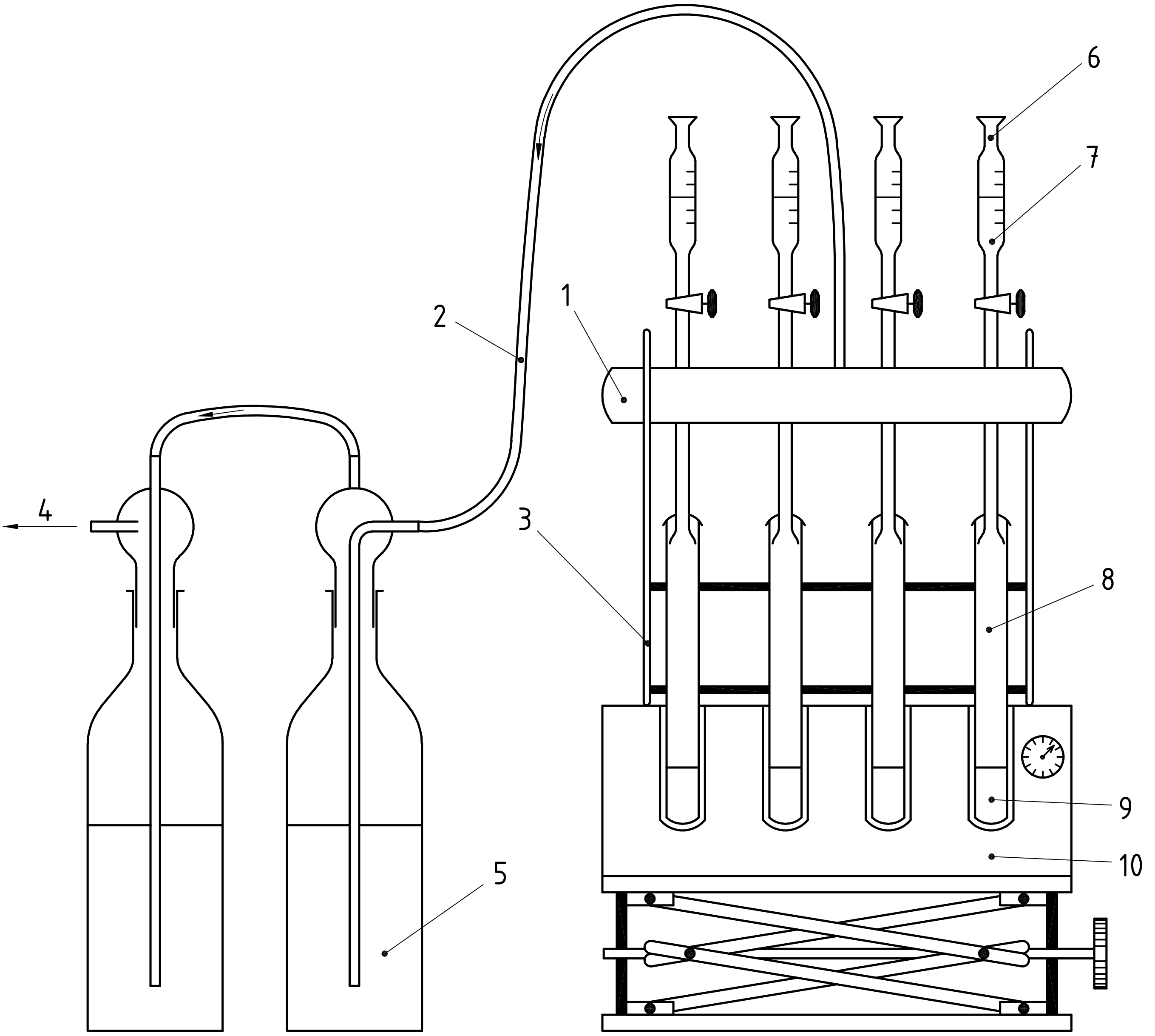
7.2.1.7 Cutting blade.

7.2.1.8 Dry-block heater (see Figure 1), fitted with an exhaust hood connected to a scrubbing system and suction pump, and also fitted with a fixed support rack designed to hold digestion tubes and graduated dropping funnels for the addition of reagents. The heating block shall be placed on a secure laboratory jack or similar item of equipment.

7.2.1.9 Concentrated sulfuric acid, at least 96 % (by mass) ($\rho =$ 1,84 g/ml).

7.2.1.10 Hydrogen peroxide solution, containing at least 30 % (by mass) H_2O_2. The volumes given in the procedure are for 30 % (by mass).

7.2.1.11 Sodium hydroxide solution, containing approximately 15 % (by mass) NaOH, for use in neutralizing acid fumes extracted by the exhaust hood.



Key

1	Exhaust hood
2	Extraction line for acid fumes
3	Support rack
4	To suction pump
5	Sodium hydroxide solution
6	Dropping funnel
7	Hydrogen peroxide
8	Digestion tube
9	Test specimen in concentrated sulfuric acid
10	Heating block

Figure 1 — Apparatus for method B: Chemical digestion

7.2.2 Procedure

IMPORTANT — This procedure must be carried out in a fume cupboard. During the oxidation operation, the fume cupboard door must be lowered. Face visor, gloves and laboratory coat must be worn.

7.2.2.1 Pre-heat the heating block (7.2.1.8) to 260 °C ± 5 °C for 20 min before use.

7.2.2.2 Fill each of the dropping funnels with approximately 25 ml of hydrogen peroxide solution (7.2.1.10).

7.2.2.3 Weigh a test specimen of carbon fibre yarn to the nearest 0,1 mg (m_1) and place it in a digestion tube. Add at least 50 ml of concentrated sulfuric acid (7.2.1.9). Swirl to ensure that the test specimen is immersed in the acid. Place the tube in the support rack. Repeat the procedure for other specimens if more than one determination is being carried out.

7.2.2.4 Place the exhaust hood (see 7.2.1.8) over the digestion tubes in the rack.

7.2.2.5 Start the suction pump (see Figure 1).

7.2.2.6 Slowly open the dropping funnels containing the hydrogen peroxide solution and allow about 20 ml of this solution to run into each digestion tube. The reaction mixture will fume as the reaction starts.

7.2.2.7 Use the laboratory jack to raise the heating block round the digestion tubes.

7.2.2.8 Allow the digestion tubes to remain in the heating block at 260 °C ± 5 °C for 5 min.

7.2.2.9 Lower the heating block away from the digestion tubes. If a solution is not clear, allow to cool for 5 min, add a further 10 ml of hydrogen peroxide solution and repeat the procedure from 7.2.2.7. If the solution is still not clear, repeat the digestion procedure on a fresh test specimen.

7.2.2.10 Allow the digested specimens to cool to room temperature.

7.2.2.11 Heat sintered-glass filters (7.2.1.6) (one for each digested specimen) in the oven (7.2.1.2) at 100 °C ± 5 °C for 1 h, allow to cool in a desiccator (7.2.1.3) and weigh to the nearest 0,1 mg (m_2).

7.2.2.12 Filter each solution slowly through a sintered-glass filter and wash each specimen with distilled water until the washings are neutral to indicator paper.

7.2.2.13 Dry the sintered-glass filters and specimens for 1 h in the oven at 110 °C ± 5 °C and allow to cool in a dessicator. Weigh to the nearest 0,1 mg.

7.2.2.14 Repeat the drying and weighing procedure until the difference in mass between two successive weighings (m_3) is less than ± 0,2 mg. If a minimum drying time known to give constant mass has been established (see clause 4), this requirement does not apply.

7.2.2.15 A correction factor will be required if the digestion causes a significant loss in mass of the carbon fibre. This can be determined by carrying out a blank test on unsized fibre.

7.2.3 Expression of results

Calculate the size content SC, expressed as a percentage by mass, from the equation:

$$\mathrm{SC} = \frac{m_1 - (m_3 - m_2)}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the specimen before digestion;

m_2 is the mass, in grams, of the sintered-glass filter;

m_3 is the mass, in grams, of the sintered-glass filter plus specimen after digestion.

7.3 Method C: Decomposition by pyrolysis

7.3.1 Principle

The size is destroyed by pyrolysis in an electric furnace in a stream of nitrogen at a temperature exceeding the decomposition temperature of the size.

NOTE The decomposition temperature is that temperature at which 100 % of the size is decomposed and removed from the fibres by the gas stream. Decomposition of the size is a function of temperature and time. The manufacturer of the yarn is obliged to specify the decomposition parameters of the size.

7.3.2 Apparatus

7.3.2.1 Balance, accurate to 0,5 mg, readable to 0,1 mg.

7.3.2.2 Electric furnace, capable of being controlled to $\pm$ 5 $^\circ$C.

7.3.2.3 Nitrogen supply unit, comprising a cylinder, reducing valve, flowmeter and connection tubing, and supplying nitrogen of purity greater than 99,996 %.

7.3.2.4 Specimen holder, such as a combustion boat or sagger made of stainless steel or ceramic material.

7.3.3 Procedure

7.3.3.1 Heat the specimen holder (7.3.2.4) for 15 min in the electric furnace (7.3.2.2) at a temperature exceeding the decomposition temperature of the size in a flow of nitrogen (see 7.3.2.3). Allow to cool in a desiccator. Weigh to the nearest 0,1 mg (m_2).

7.3.3.2 Weigh a test specimen to the nearest 0,1 mg (m_1).

7.3.3.3 Adjust the flow of nitrogen to the furnace so that the furnace will be swept out in 1 min.

7.3.3.4 Place the specimen in the weighed specimen holder and place in the electric furnace.

7.3.3.5 Decompose the size on the specimen for the specified time at the same temperature as in 7.3.3.1. During this time, gradually double the nitrogen flow.

7.3.3.6 Either switch off the furnace and allow the specimen to cool in the furnace at the increased nitrogen flow or transfer the specimen in its holder to a container filled with nitrogen at room temperature and allow the specimen to cool in the container.

When the specimen in the specimen holder has cooled sufficiently, transfer it to a desiccator. It is important to ensure that the specimen is cooled sufficiently before transferring it to the desiccator. If the specimen glows red in the air during the transfer, discard it and retest on a fresh specimen.

7.3.3.7 Allow the specimen plus specimen holder to cool to room temperature in the desiccator. Weigh the specimen plus specimen holder to the nearest 0,1 mg (m_3).

7.3.3.8 A correction factor will be required if the temperature and time of pyrolysis cause a significant loss in mass of the carbon fibre. This can be determined by carrying out a blank test on unsized fibre.

7.3.3.9 If the nature of the material under test permits, the procedure may be simplified by carrying it out without a specimen holder.

7.3.4 Expression of results

Calculate the size content SC, expressed as a percentage by mass, from the equation:

$$\mathrm{SC} = \frac{m_1 - (m_3 - m_2)}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the specimen before pyrolysis;

m_2 is the mass, in grams, of the specimen holder;

m_3 is the mass, in grams, of the specimen holder plus test specimen after pyrolysis.

8 Precision

The precision of these test methods is not known because interlaboratory data are not available. Interlaboratory data are being obtained and precision data will be added at the next revision.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the carbon fibre yarn tested;

c) the size content of the yarn;

d) the type of size;

e) the method and conditions of test used, including the following details:

  — method A: the solvent used,

  — method C: the temperature and time of pyrolysis;

f) the date of the test.

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