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ISO 10119

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Carbon fibre — Determination of density

Fibres de carbone — Détermination de la masse volumique

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Reference number
ISO 10119:1992(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.

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International Standard ISO 10119 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 13, *Composites and reinforcement fibres*.

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Annex A forms an integral part of this International Standard.

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Carbon fibre — Determination of density

1 Scope

This International Standard specifies three methods for the determination of the density of desized carbon fibre yarn:

Method A:	Liquid-displacement method
Method B:	Sink/float method
Method C:	Density-gradient column method

The determination of density may also be carried out on sized fibre by agreement between customer and supplier. At low levels of size (less than 1%), the density obtained with sized fibre may be taken to be identical to that of unsized fibre.

Method C is the reference method.

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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 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 1675:1985, *Plastics — Liquid resins — Determination of density by the pyknometer method.*

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

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 density: The mass per unit volume of a substance at a specified temperature. This property is expressed in kilograms per cubic metre or in grams per cubic centimetre at the specified temperature. The recommended temperature is 23 °C.

4 Sampling

Sampling shall be carried out as specified by ISO 1886 in order to determine the number of elementary units (spools, etc.) to be sampled.

5 Conditioning and test conditions

Before testing, test specimens shall be conditioned in a standard test atmosphere as specified in ISO 291. During the test, the test apparatus and specimens shall be maintained at the same conditions as used for conditioning. The preferred conditions are 23 °C ± 2 °C and (50 ± 5) % RH.

6 Number of test specimens

Take at least three test specimens from each elementary unit.

7 Test methods

7.1 Method A: Liquid-displacement method

7.1.1 Principle

A specimen is weighed in air and then in a liquid which completely wets out the specimen and which has a known density at least 0,2 g/cm^3 less than that of the specimen. The difference in mass of the specimen in the two media is due to the Archimedean upthrust. This difference in mass, divided by the density of the liquid, gives the volume of the specimen. The mass of the specimen in air, divided by its volume, gives the density of the specimen.

7.1.2 Apparatus and materials

Standard laboratory apparatus and the following:

7.1.2.1 Analytical balance, accurate to 0,1 mg.

7.1.2.2 Suspension wire, made of stainless steel, of diameter 0,4 mm or less, or a **specimen support** made of glass or stainless steel, with perforations so that it can be immersed easily in the immersion liquid (see figure 1).

7.1.2.3 Pyknometer or **hydrometer**, accurate to 0,001 g/cm^3.

7.1.2.4 Beaker, made of borosilicate glass.

7.1.2.5 Support framework, suitable for use with the balance (7.1.2.1) (see figure 2).

7.1.2.6 Vacuum pump (optional).

7.1.2.7 Ultrasonic device (optional).

7.1.2.8 Desiccator.

7.1.2.9 Immersion liquids (examples):

Ethanol	$\rho_{23} = 0{,}79$ g/cm^3
Acetone	$\rho_{23} = 0{,}79$ g/cm^3
Methanol	$\rho_{23} = 0{,}80$ g/cm^3
Dichloroethane	$\rho_{23} = 1{,}25$ g/cm^3
o-Dichlorobenzene	$\rho_{23} = 1{,}31$ g/cm^3
Trichloroethane	$\rho_{23} = 1{,}35$ g/cm^3
Trichloromethane	$\rho_{23} = 1{,}48$ g/cm^3
Carbon tetrachloride	$\rho_{23} = 1{,}59$ g/cm^3

WARNING — Take the necessary safety precautions when handling these liquids.

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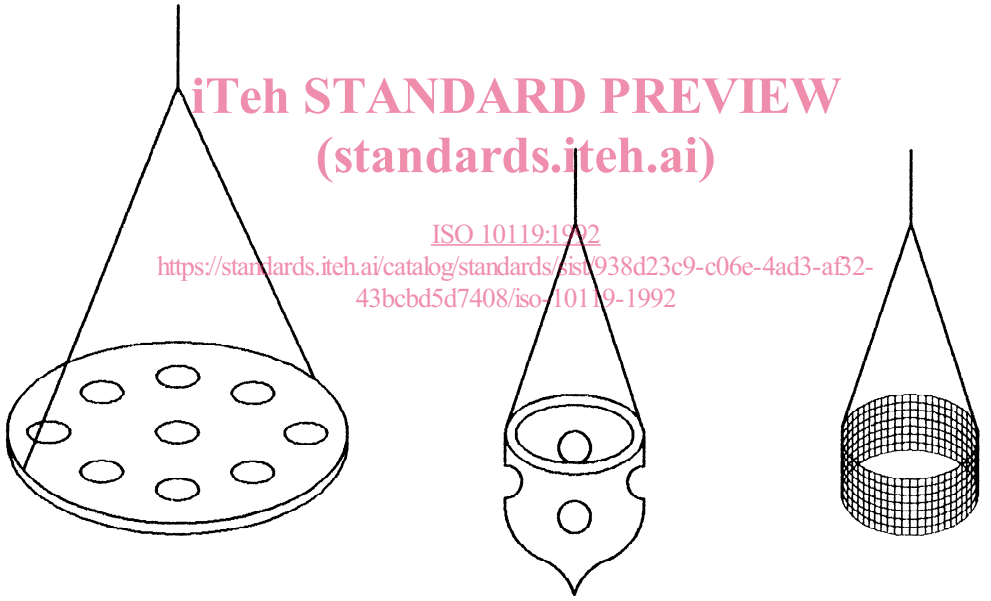

Figure 1 — Examples of test specimen supports

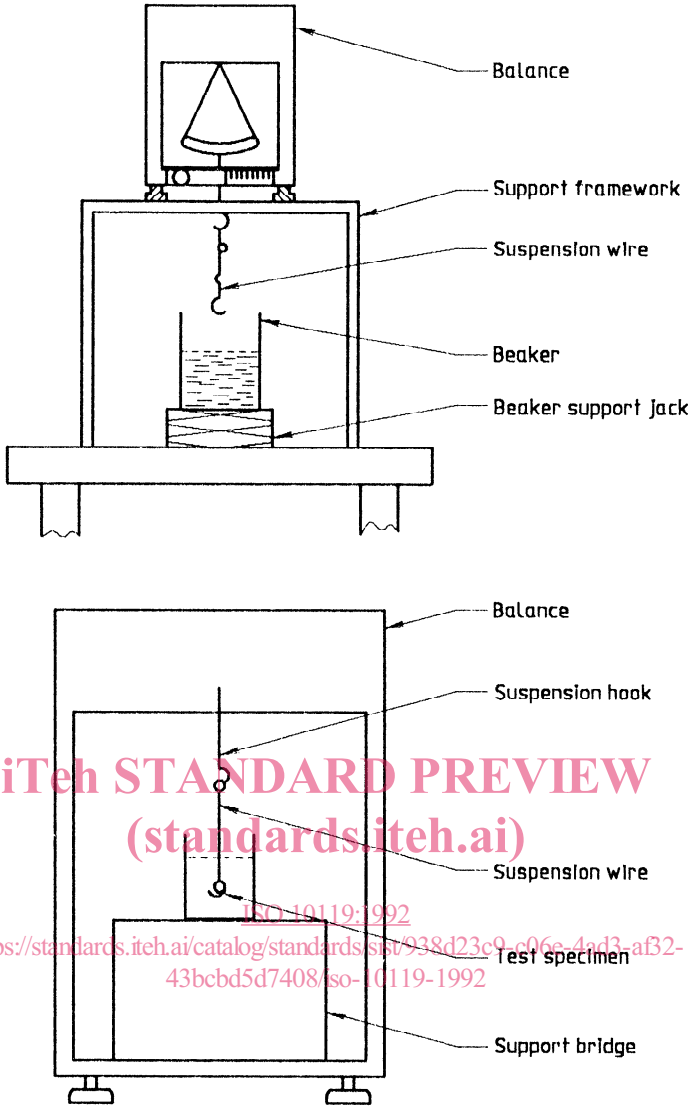

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Figure 2 — Examples of apparatus for determining density by the liquid-displacement method

7.1.3 Test specimens

For each test specimen, take a continuous length of yarn, desized in accordance with a method agreed on between the interested parties[1], unless otherwise stated, and having a mass of at least 0,2 g, and form it into a convenient shape, for example a bow or knot.

7.1.4 Procedure

Determine all masses by means of the analytical balance (7.1.2.1).

7.1.4.1 Determine the exact density of the immersion liquid (7.1.2.9) at the temperature of the test, using the pyknometer (see 7.1.2.3) in accordance with ISO 1675, or the hydrometer (see 7.1.2.3).

7.1.4.2 Weigh the suspension wire or the specimen support (7.1.2.2) in air to the nearest 0,1 mg (m_1).

7.1.4.3 Immerse the specimen support or suspension wire in the selected immersion liquid contained in the beaker (7.1.2.4). Adjust the level of the liquid in the beaker to a mark on the support or suspension wire chosen to give a specimen immersion of 10 mm when the specimen is subsequently weighed on the suspension wire or specimen support (see 7.1.4.6). Weigh the specimen support or suspension

1) An International Standard describing the method to be used to determine the size content will be published at a later date.

wire in the immersion liquid to the nearest 0,1 mg (m_2).

7.1.4.4 Attach a knotted test specimen to the suspension wire or place it on the specimen support, and weigh the test specimen and suspension wire or support in air to the nearest 0,1 mg (m_3).

7.1.4.5 Immerse the knotted test specimen, on the suspension wire or support, in the beaker containing the immersion liquid, remove any air bubbles by pressing the specimen against the sides of the beaker with a glass rod, or by using the vacuum pump (7.1.2.6) or the ultrasonic device (7.1.2.7).

7.1.4.6 Weigh the specimen plus suspension wire or support in the immersion liquid to the nearest 0,1 mg, taking care that the depth of immersion is the same as in 7.1.4.3 (m_4).

7.1.5 Expression of results

The density, in grams per cubic centimetre, of the test specimen at a temperature θ is given by the equation:

$$\rho_\theta = \frac{(m_3 - m_1)}{(m_3 - m_1) - (m_4 - m_2)} \times \rho_L$$

where

m_1 is the mass, in grams, of the suspension wire or specimen support in air;

m_2 is the mass, in grams, of the suspension wire or specimen support in the immersion liquid;

m_3 is the mass, in grams, of the suspension wire or specimen support and specimen in air;

m_4 is the mass, in grams, of the suspension wire or specimen support and specimen in the immersion liquid;

ρ_L is the density, in grams per cubic centimetre, of the immersion liquid.

7.2 Method B: Sink/float method

7.2.1 Principle

This method is based on the observation of the state of equilibrium of the carbon fibre in a liquid mixture that has the same density as the fibre.

Two versions of this method are specified:

Method B1: A dynamic method in which the mixture of liquids required to hold the test specimen in uniform suspension is made progressively.

Method B2: Test portions of finely chopped yarn are placed in a series of liquid mixtures of different known densities.

7.2.2 Apparatus and materials

7.2.2.1 Vacuum desiccator.

7.2.2.2 Thermometer.

7.2.2.3 Pyknometer or **hydrometer**, accurate to 0,001 g/cm^3.

7.2.2.4 Test tubes or **sample tubes**, of 5 cm^3 capacity, fitted with stoppers resistant to the liquid employed.

7.2.2.5 Measuring cylinder, of 250 cm^3 capacity.

7.2.2.6 Thermostat bath, capable of maintaining the temperature of the solution in the tubes at 23 °C ± 0,1 °C.

7.2.2.7 Tweezers.

7.2.2.8 Razor blades.

7.2.2.9 Liquid-storage flask, of 250 cm^3 capacity.

7.2.2.10 Immersion liquids: two liquids which, when mixed, will cover the range of densities required.

Acetone, methanol, ethanol, petroleum spirit	$\rho_{23} = 0{,}8$ g/cm^3
Trichloroethane	$\rho_{23} = 1{,}35$ g/cm^3
Carbon tetrachloride	$\rho_{23} = 1{,}59$ g/cm^3
Dibromoethane	$\rho_{23} = 2{,}17$ g/cm^3
Bromoform	$\rho_{23} = 2{,}89$ g/cm^3

WARNING — Take the necessary safety precautions when handling these liquids.

7.2.3 Test specimens

Take lengths of yarn with a mass of approximately 10 mg to 20 mg (method B1) or appproximately 100 µg portions of finely chopped fibre (method B2).

7.2.4 Procedure

7.2.4.1 Method B1

7.2.4.1.1 Prepare a mixture of the two selected immersion liquids (7.2.2.10) in the flask (7.2.2.9) to obtain a mixture whose density is less than that of the specimens. Mix the liquids thoroughly, bring the mixture to 23 °C ± 0,1 °C and maintain it at this temperature.

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7.2.4.1.2 Form a test specimen into a knot, place it in the liquid mixture then de-aerate under a vacuum of 10 mmHg, maintaining the vacuum for at least 2 min.

7.2.4.1.3 Add progressively several drops of the denser liquid, stirring to ensure thorough mixing. Continue the addition until the specimen remains in suspension in the middle of the flask. Wait 5 min. If the specimen sinks add several drops of the denser liquid, if it floats add several drops of the less dense liquid until the specimen remains stationary. Filter the liquid mixture and determine its density using the pyknometer (see 7.2.2.3) in accordance with ISO 1675, or the hydrometer (see 7.2.2.3).

7.2.4.2 Method B2

7.2.4.2.1 Prepare mixtures of immersion liquids (7.2.2.10) covering the required density range at increments of 0,02 g/cm^3. Determine the density of each mixture using the pyknometer in accordance with ISO 1675, or the hydrometer, noting the temperature at which the determinations were carried out. A small quantity of wetting agent may be added if necessary.

7.2.4.2.2 Fill six 5-cm^3 test tubes (7.2.2.4) with 2,5 cm^3 of the liquid mixture. Introduce into each test tube a quantity of finely chopped carbon fibres sufficient to cover a pin head (about 100 µg). Stopper and shake the tubes well, and allow the tubes to stand at the same temperature as that at which the determinations of the densities of the solutions were carried out.

7.2.4.2.3 After 60 min, observe the position of the fibres in the tubes against a white background.

7.2.4.2.4 The density of the yarn is given by the density of the mixture in which the majority of the fibres are held in suspension.

7.2.5 Expression of results

Express the density of the carbon fibre yarn in grams per cubic centimetre.

7.3 Method C: Density-gradient column

7.3.1 Principle

This method is based on the observation of the equilibrium position of a test specimen in a column of liquid having a linear density gradient.

Density-gradient columns are columns of liquid whose density increases uniformly from the top to the bottom of the column.

7.3.2 Apparatus and materials

7.3.2.1 Density-gradient column, consisting of a vertical graduated tube, open at the top, length approximately 1 m, diameter 40 mm to 50 mm, surrounded by a water jacket maintained at a temperature of 23 °C ± 0,1 °C. A stainless-steel basket, which can be raised and lowered by means of a wire not attacked by the liquids used, is situated at the base of the column.

7.3.2.2 A series of calibrated reference floats, approximately 5 mm to 6 mm in diameter, of different densities measured at 23 °C to an accuracy of one part in ten thousand and covering the desired density range.

7.3.2.3 Apparatus for filling the column, comprising a siphon, stopcock, glass tube, 2-litre vessel and magnetic stirrer.

7.3.2.4 Immersion liquids: two liquids which, when mixed, will cover the density range required. Typical mixtures are:

Ethanol, bromoform (density range 0,81 g/cm^3 to 2,89 g/cm^3)

Zinc chloride, water (density range 1,00 g/cm^3 to 2,00 g/cm^3)

Trichloroethane, ethylene dibromide (density range 1,35 g/cm^3 to 2,18 g/cm^3)

Carbon tetrachloride, ethylene dibromide (density range 1,59 g/cm^3 to 2,18 g/cm^3)

Carbon tetrachloride, bromoform (density range 1,59 g/cm^3 to 2,89 g/cm^3)

WARNING — Take the necessary safety precautions when handling these liquids.

7.3.3 Test specimens

Take test specimens of mass between 1 mg and 10 mg depending on the mass per unit length, and immerse them in the less dense of the two liquids for at least 10 min, taking care to eliminate all air bubbles.

Form each specimen into a suitable shape for insertion into the column. The form chosen shall be suited to the type of carbon fibre under test. The most suitable form for filament fibre is a knot or bow.

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7.3.4 Procedure

7.3.4.1 Set up the density-gradient column as described in annex A .

7.3.4.2 Carefully immerse a test specimen at the top of the column and wait until it has descended to an equilibrium position. Take care that no filaments rise to the surface and that no air bubbles are trapped inside the specimen.

7.3.4.3 When equilibrium has been reached, record the column graduation corresponding to the equilibrium position of the specimen and determine the corresponding density value from the column calibration curve.

NOTE 1 The time required to attain equilibrium can vary from several minutes to several hours. It will depend on the shape of the specimen, the density gradient in the column and the precision required.

Avoid contact with the sides of the column, and with specimens remaining in the column from previous tests, which may lead to a reduction in the rate of free fall of the specimen.

7.3.4.4 Remove specimens which have disintegrated by means of the "basket" designed to remove debris from the column. Carry out this procedure slowly in order to avoid disturbing the liquid in the column.

8 Precision

The precision of these test methods is not known because inter-laboratory data are not available. Inter-laboratory data are being obtained and a precision statement will be added at the next revision.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary to identify the fibre sample tested;

c) the method used (A, B1, B2 or C);

d) whether or not the fibre was size-free (method A only);

e) the pair of liquids used (methods B1, B2 and C), or the immersion liquid and its density (method A);

f) the number of specimens tested;

g) the mean value of the density, rounded to the nearest 0,01 g/cm^3;

h) details of any operation not included in this International Standard, and any incident noted during the test which may have influenced the results.

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Annex A
(normative)

Preparation of the density-gradient column

A.1 Principle

Two methods may be employed to prepare the density-gradient column used in method C.

In the first method [see figure A.1 a)], the column is filled from the top with liquids of progressively decreasing density, each liquid being allowed to run down the inside surface of the tube so that it settles above the more dense liquid already in place.

In the second method [see figure A.1 b)], filling is carried out from the bottom of the column with a liquid of progressively increasing density, which displaces upwards the lower-density liquid already in place.

A.2 Procedure

A.2.1 Set up the apparatus as shown in figure A.1 a) or figure A.1 b). Adjust the thermostat temperature to 23 °C ± 0,1 °C.

Place the basket (see 7.3.2.1) containing the calibrated floats (7.3.2.2) (preferably eight) at the base of the column.

A.2.2 Prepare the master liquids L_1 (higher density) and L_2 (lower density). Depending on the precision desired, these master liquids may be either the original liquids or mixtures whose densities span those of the fibres being tested. The higher the precision required, the narrower the range of densities chosen. A typical density range for a column of length 70 cm is 0,05 g/cm^3.

A.2.3 Fill vessels A and B with liquid L_1 or liquid L_2 as indicated in figure A.1 a) or A.1 b). Each vessel shall contain a volume of liquid equal to or greater than half the volume of the column.

Commence stirring the liquid nearest to the column. Prime the siphons S_1 and S_2 [see figure A.1 a)] or open the taps R_1 and R_2 [see figure A.1 b)] to give a filling time of the order of two hours.

In the case of the method illustrated in figure A.1 a), the spheres commence to rise with the free surface of the liquid and then separate from each other in order of decreasing density.

In the case of the system illustrated in figure A.1 b), the spheres rise from the base of the column successively in order of increasing density and then follow the rise of the liquid level.

A.2.4 Stopper the column and keep it at the thermostat temperature of 23 °C ± 0,1 °C for at least 24 h. At the end of this period, measure to the nearest millimetre the distance of each float from the base of the column and plot a curve relating equilibrium height to density. Repeat the filling procedure if a straight-line curve is not obtained. The life of a column is approximately one month, after which the column loses linearity.

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