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Carbon fibre — Determination of density

Fibre de carbone — Détermination de la masse volumique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 10119:2002), which has been technically revised.

The main changes compared to the previous edition are as follows:

— gas pycnometer method (method D) has been added;

— the calibration of the measurement cell and expansion cell have been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Density is a parameter that characterizes the basic physical properties of carbon fibre, and is also an important parameter for calculating the tensile strength and tensile modulus of carbon fibre.

ISO 10119:2002 describes three methods (A, B and C) of using liquid to determine the density of carbon fibre. In this edition, the gas pycnometer method is added as method D.

Gas pycnometer method uses inert gas instead of liquids to measure the volume of fibres, powders and cellular materials so as to obtain the density. The method give a much higher resolution (i.e. a factor of 100 times better).

With the development of electronic technology, fully automatic instruments are commercially available, which allow faster throughput testing which are suitable for large scale testing. In addition, there is no environmental pollution because no organic solvent is used.

Carbon fibre — Determination of density

1 Scope

This document specifies four methods for the determination of the density of carbon fibre tested as a yarn:

— method A: liquid-displacement method;

— method B: sink/float method;

— method C: density-gradient column method;

— method D: gas pycnometer method.

Method C is the reference method in cases of dispute, etc.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1675, *Plastics — Liquid resins — Determination of density by the pyknometer method*

ISO 10548, *Carbon fibre — Determination of size content*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
density
mass per unit volume of a substance at a specified temperature

Note 1 to entry: This property is expressed in grams per cubic centimetre or in kilograms per cubic metre at the specified temperature. The recommended temperature is 23 °C.

4 Test specimens — General requirements

Test specimens shall be taken from desized samples unless otherwise agreed between the supplier and the customer. To remove the size, use the solvent extraction, chemical digestion or pyrolysis method specified in ISO 10548. The determination of the density may also be carried out on sized fibre by agreement between customer and supplier. The density of sized fibre may be taken to be identical to that of unsized fibre when the size content is low.

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 ± 2) °C and (50 ± 10) % relative humidity.

6 Test methods

6.1 Method A: Liquid-displacement method

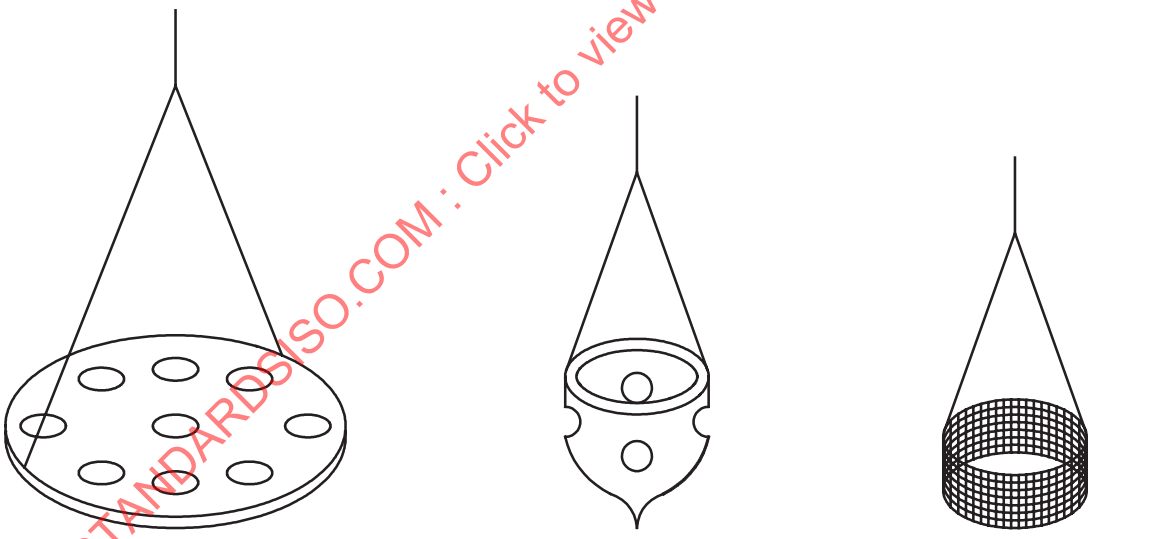
6.1.1 Principle

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 weight of the specimen in the two media is due to the Archimedean upthrust.

6.1.2 Apparatus and materials

6.1.2.1 Analytical balance, readable to 0,1 mg, with a maximum permissible error of 0,5 mg, and with a range from 0 g to 100 g.

6.1.2.2 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).



a) Perforated stainless-steel support | **b) Perforated glass support** | **c) Stainless-steel gauze support**

Figure 1 — Examples of test specimen supports

6.1.2.3 Pycnometer or **hydrometer,** maximum permissible error 0,001 g/cm^3.

6.1.2.4 Beaker, made of borosilicate glass.

6.1.2.5 Vacuum pump (optional).

6.1.2.6 Ultrasonic device (optional).

6.1.2.7 Immersion liquids (examples):

ethanol	ρ_{23} = 0,79 g/cm^3;
acetone	ρ_{23} = 0,79 g/cm^3;
methanol	ρ_{23} = 0,80 g/cm^3;
dichloroethane	ρ_{23} = 1,25 g/cm^3;
o-dichlorobenzene	ρ_{23} = 1,31 g/cm^3;
trichloroethane	ρ_{23} = 1,35 g/cm^3;
trichloromethane	ρ_{23} = 1,48 g/cm^3;
carbon tetrachloride	ρ_{23} = 1,59 g/cm^3.

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

6.1.3 Test specimen

Take a continuous length of yarn and form it into a convenient shape, for example a bow or knot.

6.1.4 Procedure

6.1.4.1 Carry out all weighings using the analytical balance (6.1.2.1).

6.1.4.2 Determine the exact density of the immersion liquid (6.1.2.7) at the temperature of the test, using the pycnometer (see 6.1.2.3) in accordance with ISO 1675, or the hydrometer (see 6.1.2.3).

6.1.4.3 Weigh the specimen in air to the nearest 0,1 mg (w_1). If the specimen is weighed using a suspension wire or specimen support (6.1.2.2), the wire or support shall be tared or weighed and, if weighed, its weight shall be deducted from subsequent weighings of the specimen.

6.1.4.4 Immerse the test specimen in the beaker (6.1.2.4) containing the immersion liquid (6.1.2.7) and remove any air bubbles by agitating the specimen or by pressing it. Weigh the specimen to the nearest 0,1 mg (w_2), watching the balance display for a few seconds to make sure that it does not drift as a result of convection currents.

The main sources of error are:

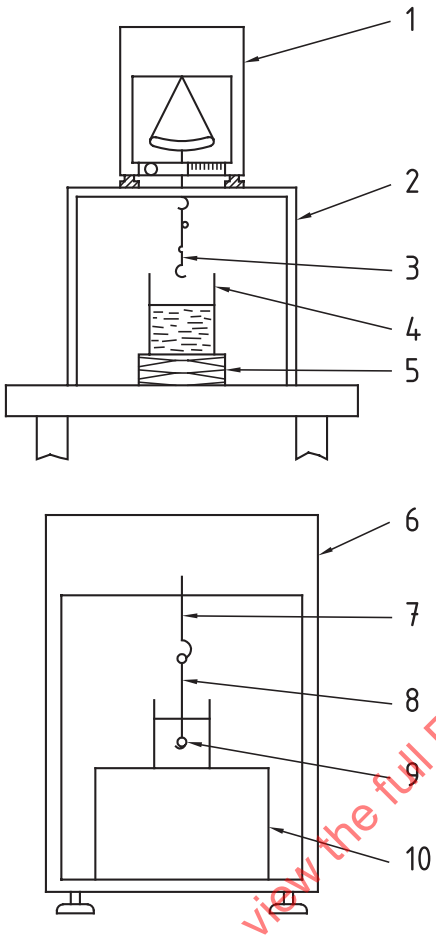
a) air bubbles adhering to the surfaces of the specimen when weighing in the immersion liquid;

b) surface tension effects on the specimen or suspension wire;

c) convection currents in the liquid in which the specimen is suspended, to minimize which the temperature of the liquid and of the air in the balance case should be the same.

A vacuum pump (6.1.2.5) or ultrasonic device (6.1.2.6) can be used to eliminate air bubbles.

In order to minimize the adherence of air bubbles to the test specimen, one of the immersion liquids listed in 6.1.2.7 should be used. If water is used, it is permissible to add a trace (say 1 part in 10 000) of surface-active material such as a detergent to the water.



Key

1	balance	6	balance
2	support framework	7	suspension hook
3	suspension wire	8	suspension wire
4	beaker	9	test specimen
5	beaker support jack	10	support bridge

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

6.1.5 Expression of results

The density, in grams per cubic centimetre, of the test specimen at a temperature θ is given by Formula (1):

$$\rho_\theta = \frac{w_1}{w_1 - w_2} \times \rho_L \tag{1}$$

where

w_1 is the weight, in grams, of the specimen in air;

w_2 is the weight, in grams, of the specimen in the immersion liquid;

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

6.2 Method B: Sink/float method

6.2.1 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.

6.2.2 Apparatus and materials

6.2.2.1 Thermometer.

6.2.2.2 Pycnometer or **hydrometer,** maximum permissible error 0,001 g/cm^3.

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

6.2.2.4 Measuring cylinder, of 250 cm^3 capacity.

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

6.2.2.6 Tweezers.

6.2.2.7 Razor blades.

6.2.2.8 Vacuum pump.

6.2.2.9 Immersion liquids, two liquids which, when mixed, covers the range of densities required (examples):

acetone, methanol, ethanol, petroleum spirit	ρ_{23} = 0,8 g/cm^3;
trichloroethane	ρ_{23} = 1,35 g/cm^3;
carbon tetrachloride	ρ_{23} = 1,59 g/cm^3;
dibromoethane	ρ_{23} = 2,17 g/cm^3;
bromoform	ρ_{23} = 2,89 g/cm^3.

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

6.2.3 Test specimens

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

6.2.4 Procedure

6.2.4.1 Method B1

6.2.4.1.1 Prepare a mixture of the two selected immersion liquids (6.2.2.9) in the measuring cylinder (6.2.2.4) to obtain a mixture whose density is less than that of the specimens. Mix the liquids thoroughly, place the measuring cylinder in the thermostatic bath (6.2.2.5) so that the mixture to 23 °C ± 0,1 °C and maintain it at this temperature.

6.2.4.1.2 Form a test specimen into a knot, place in the liquid mixture then use a vacuum pump (6.2.2.8) to de-aerate under a vacuum of 60 hPa, maintaining the vacuum for at least 2 min.

6.2.4.1.3 Progressively add 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 measuring cylinder. 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 suspended in the liquid. Filter the liquid mixture and determine its density using the pycnometer (see 6.2.2.2) in accordance with ISO 1675, or the hydrometer (see 6.2.2.2).

6.2.4.2 Method B2

6.2.4.2.1 Prepare mixtures of immersion liquids (6.2.2.9) covering the required density range at increments of 0,2 g/cm^3. Determine the density of each mixture using the pycnometer (6.2.2.2) in accordance with ISO 1675, or hydrometer (6.2.2.2), noting the temperature at which the determinations were carried out. A small quantity of wetting agent may be added if necessary.

6.2.4.2.2 Fill six 5 cm^3 test tubes (6.2.2.3) 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.

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

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

6.2.5 Expression of results

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

6.3 Method C: Density-gradient column

6.3.1 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.

6.3.2 Apparatus and materials

6.3.2.1 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.

6.3.2.2 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 10 thousand and covering the desired density range.

6.3.2.3 Apparatus for filling the column, comprising a siphon, stopcock, glass tube, 2 l vessel and magnetic stirrer.

6.3.2.4 Immersion liquids, two liquids which, when mixed, covers 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.

6.3.3 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 lower density of the two immersion fluids (see 6.3.2.4) 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.

6.3.4 Procedure

6.3.4.1 Set up the density-gradient column (6.3.2.1) as described in Annex A.

6.3.4.2 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.

6.3.4.3 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 The time required to attain equilibrium can vary from several minutes to several hours. It depends 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.

6.3.4.4 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.

6.4 Method D: Gas pycnometer method

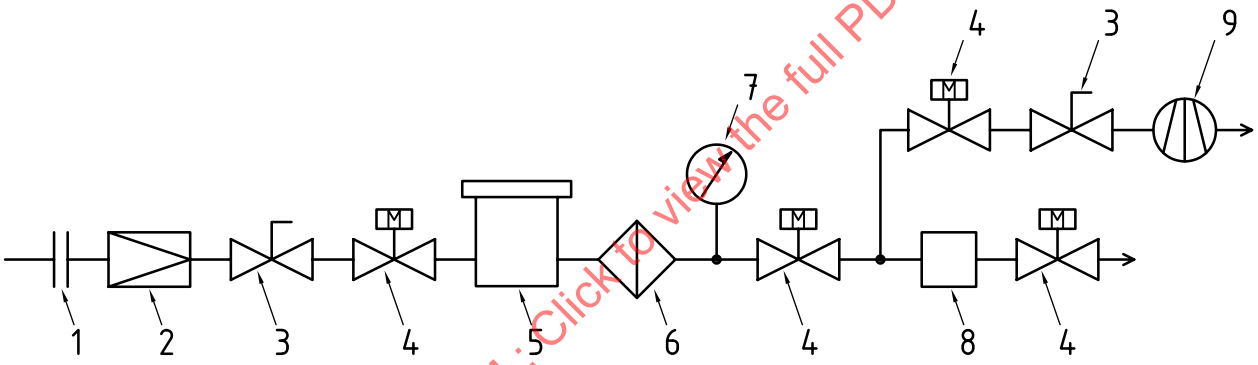
6.4.1 Principle

This method is based on Boyle's Law, which states that the decrease in volume of a confined gas results in a proportionate increase in pressure. Both helium and diatomic nitrogen are used because they behave as ideal gases at room temperature. A gas pycnometer makes use of this behaviour by making two precise pressure measurements on two known calibrated volumes. The density of specimen is obtained by its volume determined by measuring the pressure change of a confined amount of gas. Normally commercial instruments are used for these measurements.

The smaller the molecules of the gas the narrower the pores that can be penetrated will be. Hence, for high-accuracy measurements, helium is preferred.

6.4.2 Apparatus and materials

6.4.2.1 Gas pycnometer, consisting of measurement cell, expansion cell, valves, piping, circuit boards, pressure sensors and external interfaces (see schematic in Figure 3). Normally, a commercial version of this equipment is used, which does not show these working parts. The volume of measurement cell and expansion cell should be calibrated with a standard sample of known volume. The calibration method given in Annex B shall be applied.



Key

1	gas in	6	filter
2	relief valve	7	transducer
3	metering valve	8	expansion cell
4	solenoid valve	9	vacuum pump
5	measurement cell		

Figure 3 — Schematic examples of a gas pycnometer

6.4.2.2 Thermostatic bath, capable of maintaining the temperature of measurement cell at 23 °C ± 0,5 °C.

6.4.2.3 Analytical balance, readable to 0,1 mg, with a maximum permissible error of 0,5 mg, and with a range from 0 g to 200 g.

6.4.2.4 Measurement gas, preferably helium, or other non-corrosive and non-adsorbing gas, such as nitrogen. The purity of gas is 99,99 % or higher.

6.4.2.5 Suitable cutting tool, such as scissors or knife.

6.4.2.6 Vacuum pump (optional).

6.4.3 Test specimens

Take test specimens of appropriate mass depending on the volume of measurement cell and cut them with a suitable tool (6.4.2.5) to a length not exceeding the height of the measurement cell.

6.4.4 Procedure

6.4.4.1 Check the gas pycnometer (6.4.2.1) to ensure no leakage.

6.4.4.2 Weigh the measurement cell with the analytical balance (6.4.2.3) to the nearest 0,1 mg (m_1). Place the fibre vertically to the measurement cell and fill the cell volume to a minimum of 60 % of its full capacity. Then weigh the measurement cell and the specimen to the nearest 0,1 mg (m_2).

6.4.4.3 Put the weighed measurement cell and specimen into gas pycnometer. Seal the measurement cell and maintain the temperature at 23 °C ± 0,5 °C in general by thermostatic bath (6.4.2.2). Purge by multiple pulses with the measurement gas (6.4.2.4) to replace the air from measurement cell and expansion cell.

NOTE A vacuum pump (6.4.2.6) can be used to remove the air from measurement cell and expansion cell.

6.4.4.4 Open all the valves and leave the measurement cell and expansion cell full of measurement gas at atmospheric pressure. The readout of the pressure sensor should be zero.

6.4.4.5 By closing the valve of expansion cell, gas is allowed to flow into the measurement cell until the desired pressure is reached. The recommended pressure is 0,1 MPa. Close the intake valve and record the readout of the pressure sensor as p_1.

6.4.4.6 Open the valve of expansion cell so that the gas in the measurement cell flows into the expansion cell. Record the readout of the pressure sensor as p_2.

6.4.4.7 The density (ρ), in grams per cubic centimetre, of the test specimen is given by Formula (2):

$$\rho = \frac{m_2 - m_1}{V_{\text{meas}} + \dfrac{V_{\text{exp}}}{1-(p_1/p_2)}} \tag{2}$$

where

m_1 is the weight, in grams, of the measurement cell;

m_2 is the weight, in grams, of the measurement cell and the specimen;

V_{meas} is the volume, in cubic centimetre, of the measurement cell;

V_{exp} is the volume, in cubic centimetre, of the expansion cell;

p_1 is the pressure, in pascal, of the gas only in the measurement cell;

p_2 is the pressure, in pascal, of the gas in the measurement cell after expansion into the expansion cell.

6.4.4.8 Repeat procedure 6.4.4.4 to 6.4.4.7 until the standard deviation of five consecutive density single values is less than 0,000 5 g/cm^3. Take the arithmetic mean of the five single values as the density of the test specimen.

NOTE The currently commercialized gas pycnometer has been able to automate the procedure 6.4.4.4 to 6.4.4.7.

7 Precision

The precision of these test methods is not known at the time of publication.

8 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 10119:2020;

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

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

d) whether or not the fibre was size-free (if the sample was desized, give the method used);

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

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 document, and any incident noted during the test which may have influenced the results.