INTERNATIONAL STANDARD

ISO 10119

Third edition
2020-06

Carbon fibre — Determination of density

Fibre de carbone — Détermination de la masse volumique

Reference number
ISO 10119:2020(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

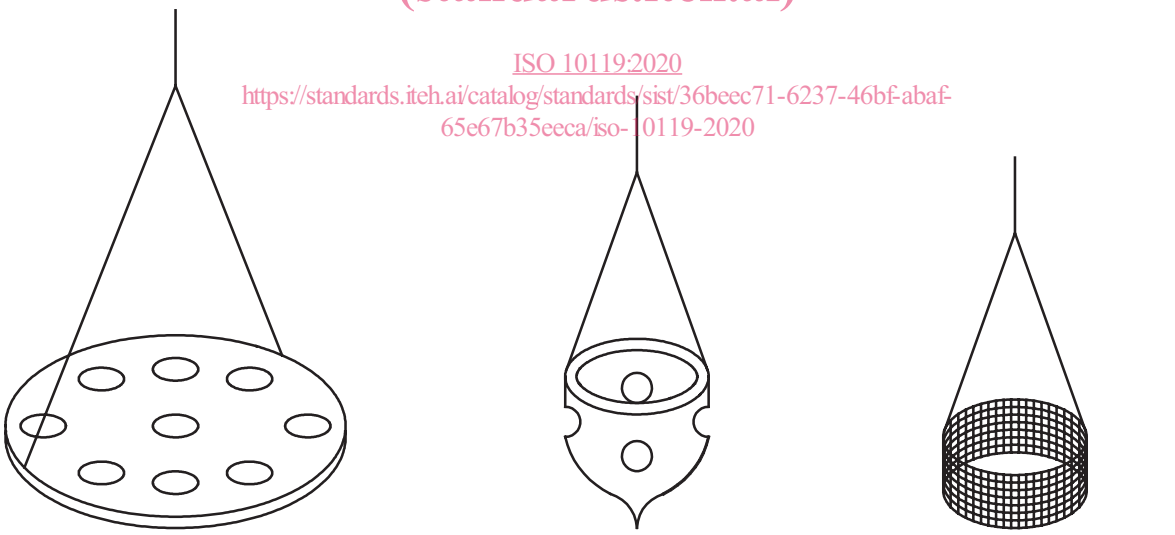
6.1.1 Principle

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:

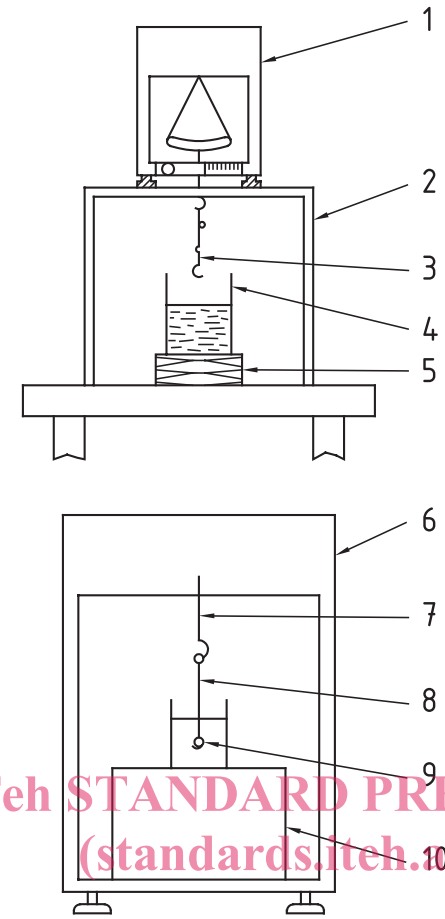
a) air bubbles adhering to the surfaces of the specimen when weighing in the immersion liquid;

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).