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Carbon fibre — Determination of density

Fibre de carbone — Détermination de la masse volumique

Reference number
ISO 10119:2002(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

ISO 10119 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 10119:1992), which has been technically revised.

Annex A forms a normative part of this International Standard.

INTERNATIONAL STANDARD **ISO 10119:2002(E)**

Carbon fibre — Determination of density

1 Scope

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

- method A: liquid-displacement method;
- method B: sink/float method;
- method C: density-gradient column method.

Method C is the reference method.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents 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 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 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
density

the mass per unit volume of a substance at a specified temperature

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

6 Test methods

6.1 Method A: Liquid-displacement method

6.1.1 Principle

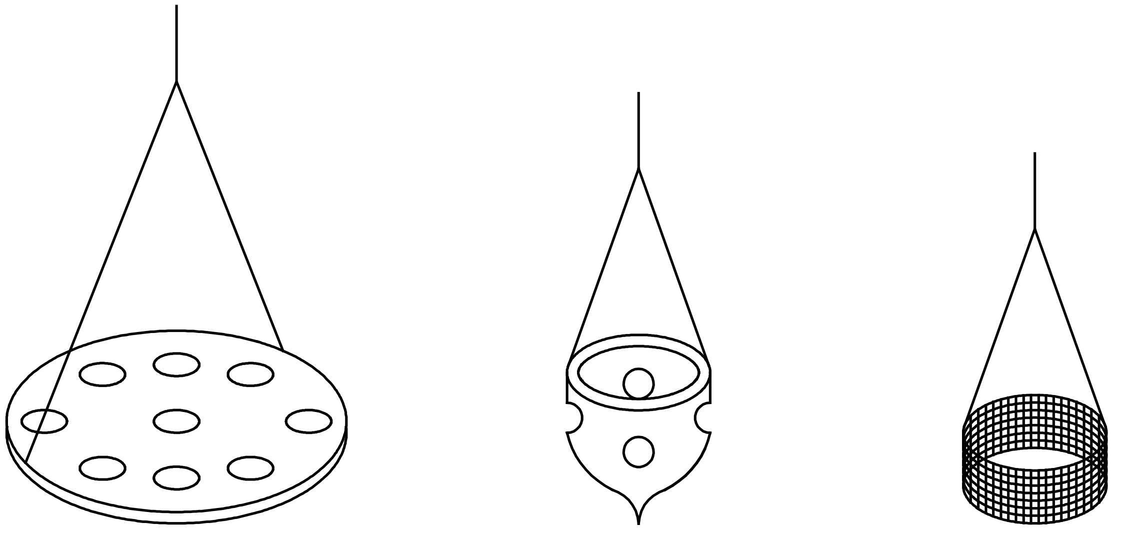
A specimen is weighed in air and then in a liquid which completely wets out the specimen and which has a known density at least 0,2 g/cm^3 less than that of the specimen. The difference in weight of the specimen in the two media is due to the Archimedean upthrust.

6.1.2 Apparatus and materials

Standard laboratory apparatus and the following:

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).



Perforated stainless-steel support **Perforated glass support** **Stainless-steel gauze support**

Figure 1 — Examples of test specimen supports

6.1.2.3 **Pyknometer** 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	$\rho_{23} = 0{,}79\ \text{g/cm}^3$;
acetone	$\rho_{23} = 0{,}79\ \text{g/cm}^3$;
methanol	$\rho_{23} = 0{,}80\ \text{g/cm}^3$;
dichloroethane	$\rho_{23} = 1{,}25\ \text{g/cm}^3$;
o-dichlorobenzene	$\rho_{23} = 1{,}31\ \text{g/cm}^3$;
trichloroethane	$\rho_{23} = 1{,}35\ \text{g/cm}^3$;
trichloromethane	$\rho_{23} = 1{,}48\ \text{g/cm}^3$;
carbon tetrachloride	$\rho_{23} = 1{,}59\ \text{g/cm}^3$.

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 pyknometer (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.

NOTE 1 The main sources of error are:

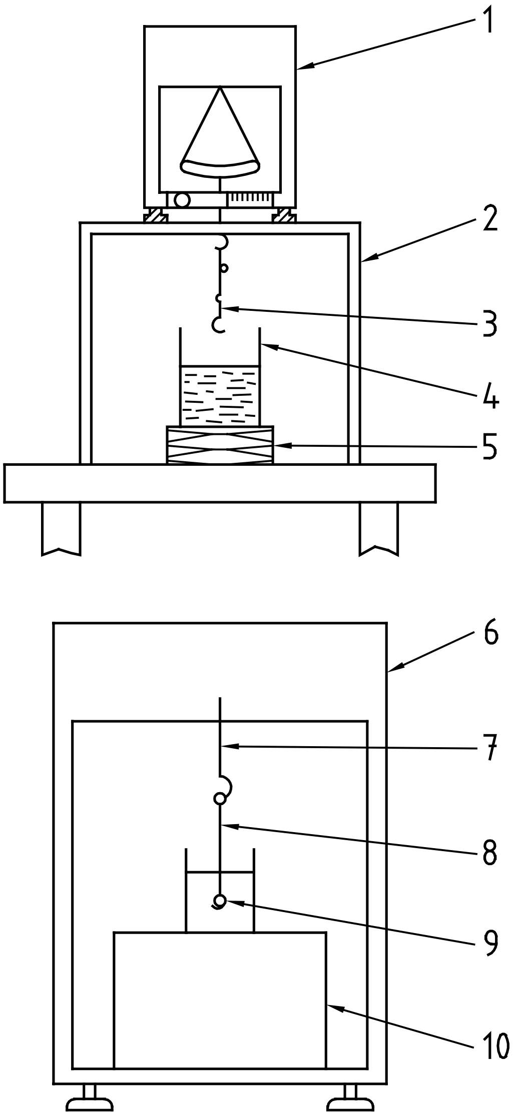
a) air bubbles adhering to the surfaces of the specimen when weighing in the immersion liquid;

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.

NOTE 2 A vacuum pump (6.1.2.5) or ultrasonic device (6.1.2.6) may be used to eliminate air bubbles.

NOTE 3 In order to minimize the adherence of air bubbles to the test specimen, it is recommended that one of the immersion liquids listed in 6.1.2.7 is 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 the equation:

$$\rho_\theta = \frac{w_1}{w_1 - w_2} \times \rho_\mathrm{L}$$

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

6.2.2.9 Immersion liquids: Two liquids which, when mixed, will cover the range of densities required (examples):

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.

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 flask (6.2.2.8) 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.

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

6.2.4.1.3 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 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 pyknometer in accordance with ISO 1675, or hydrometer, 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 ten thousand and covering the desired density range.

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

6.3.2.4 **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.

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 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.

6.3.4 Procedure

6.3.4.1 Set up the density-gradient column 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 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.

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.

7 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.

8 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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

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

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), 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.

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)], the column is filled from the bottom with a liquid of progressively increasing density, this liquid displacing the less dense liquid already in place towards the top.

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 6.3.2.1) containing the calibrated floats (6.3.2.2) (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 density range has to be.

The usual density range for a column of length 70 cm is 0,05 g/cm^3.

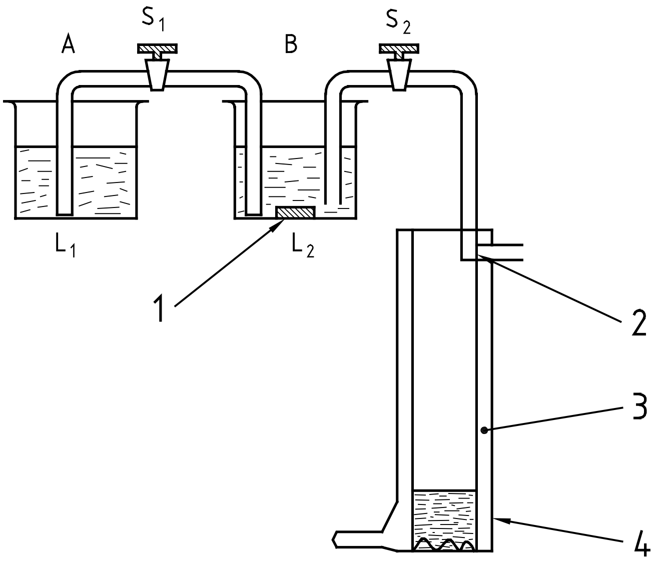
A.2.3 Fill vessels A and B with liquid L_1 or liquid L_2 as indicated in Figure A.1 a) or A.1 b). Each vessel shall contain a volume of liquid equal to or greater than half the volume of the column.

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

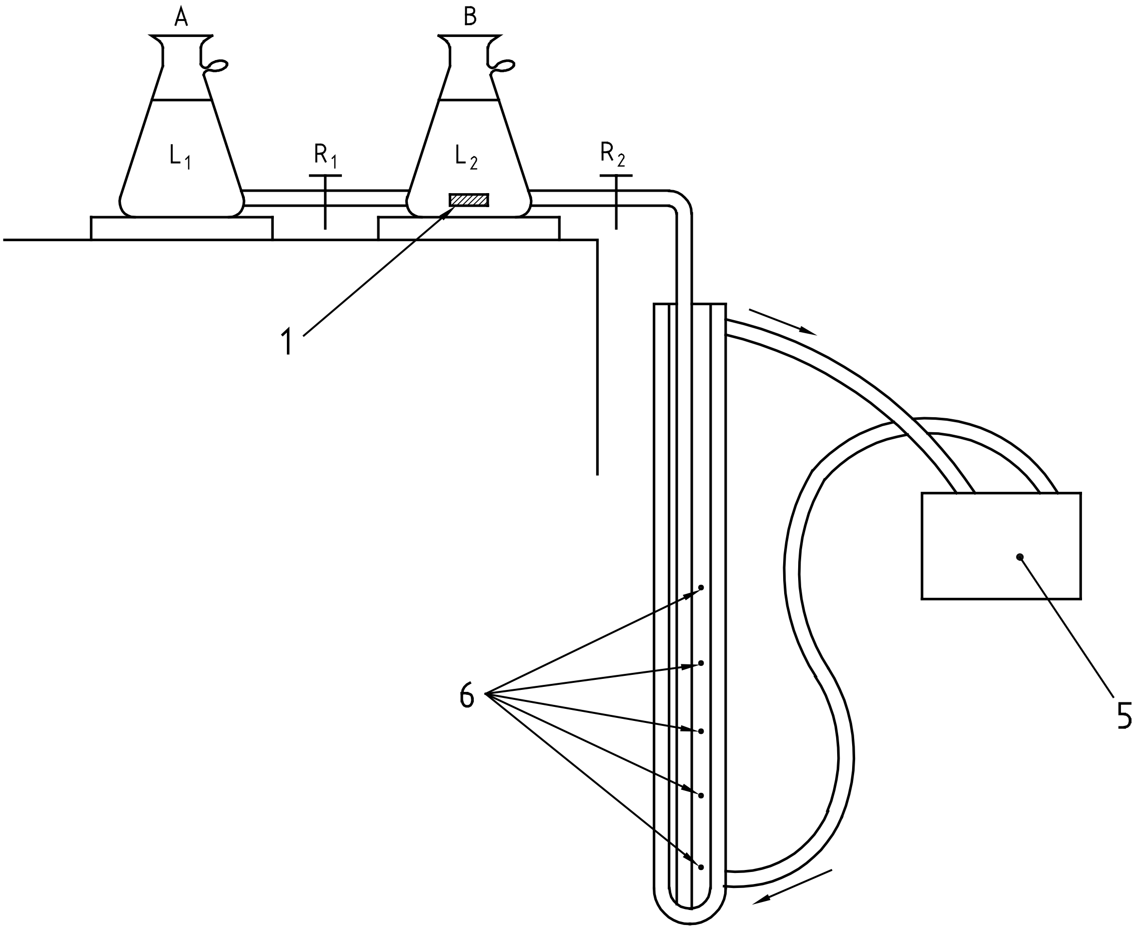
In the case of the method illustrated in Figure A.1 a), the spheres commence to rise with the open surface of the liquid and then separate from each other in order of increasing density.

In the case of the method 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.



a) Filling from the top



b) Filling from the bottom

Key

1	Magnetic stirrer
2	Capillary in contact with inside surface of column
3	Column
4	Thermostatted water jacket
5	Thermostat
6	Calibrated spherical floats

Vessel A: higher-density liquid L_1

Vessel B: lower-density liquid L_2

Figure A.1 — Schematic illustration of density-gradient column preparation by the continuous-filling method

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