
Test methods for fibrous activated carbon

Méthodes d'essai pour les charbons actifs fibreux

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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.

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 on 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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

This document is based on the Japanese Industrial Standard JIS K 1477.

Introduction

Fibrous Activated Carbon (FAC) was developed and has been produced commercially in Japan since the 1980s. The demand for FAC products first increased in the northeast Asian region and has expanded globally since 2010. FAC contributes to conservation of the environment and improves the quality of life and health all over the world.

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Test methods for fibrous activated carbon

1 Scope

This document specifies test methods for determining the properties of fibrous activated carbon, including specific surface area, pore volume, fibre and sheet properties, mass loss on drying, pH value, total ash content, and performance for toluene adsorption, methylene blue adsorption and iodine adsorption.

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 472, *Plastics — Vocabulary*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 9073-1, *Textiles — Test methods for nonwovens — Part 1: Determination of mass per unit area*

ISO 9073-3, *Textiles — Test methods for nonwovens — Part 3: Determination of tensile strength and elongation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1

fibrous activated carbon

activated carbon fibre

FAC

fibre, made from fibrous rayon, acrylic, phenol resin or pitch, which has been carbonized and then activated by reacting with steam, carbon dioxide or chemical methods at high temperature to give it higher ability to adsorb organic compounds from gas or aqueous solution

3.2

specific surface area

surface area of fibrous activated carbon per unit of mass

3.3

pore volume

total sum of the pore volume of fibrous activated carbon per unit of mass

3.4 sheet

sheet-shaped product composed of 100 % fibrous activated carbon or of fibrous activated carbon compounded with other fibre

EXAMPLE Non-woven fabric (felt) and fabric (cloth).

3.5 breakthrough curve

curve illustrating a temporal change in the outlet concentration (c) of the fluid from a fixed bed of fibrous activated carbon when the substance to be adsorbed is fed onto this fixed bed at a defined height under constant parameters such as inlet concentration (c_0), temperature, feed flow rate, etc.

Note 1 to entry: When obtaining a breakthrough curve, the outlet concentration gradually rises from a value near zero at the beginning, i.e. the curve plotted shows the variation in outlet concentration with time. Usually it is plotted as the ratio of outlet concentration to inlet concentration c/c_0 vs time.

3.6 breakthrough

conditions at which the concentration of adsorbed substance at the outlet exceeds a defined allowable concentration

3.7 breakthrough time

point in time on a breakthrough curve at which a defined ratio c/c_0 is reached, where c is the outlet concentration and c_0 is the inlet concentration

EXAMPLE $c/c_0 = 0,1$ is obtained, and this can be used as an index of the adsorption rate.

3.8 equilibrium adsorption

condition in an adsorption test in which the adsorption and desorption of a specified substance on fibrous activated carbon are balanced, so that the concentration of the adsorbed substance is reduced gradually until it becomes constant and cannot be reduced any further

Note 1 to entry: The fibrous activated carbon is either immersed in an aqueous solution in which the substance to be adsorbed is dissolved or placed in air containing substance to be adsorbed, under defined temperature, and the rate of adsorption is measured.

3.9 adsorption amount

amount of substance adsorbed from aqueous solution or from air per unit mass of fibrous activated carbon

3.10 equilibrium adsorption amount

amount of substance adsorbed from aqueous solution or from air per unit mass of fibrous activated carbon under equilibrium adsorption conditions

3.11 residual concentration equilibrium concentration

concentration of substance to be adsorbed which remains in aqueous solution or in air under equilibrium adsorption conditions

3.12 adsorption isotherm

line indicating the relation between the residual concentration and the equilibrium adsorption amount at a defined temperature

4 Sampling method

Take the test samples so as to represent the lot to be tested. Cut the test fibres in lengths of 10 mm, or shorter if necessary.

The size of the set of test samples shall be decided between the parties concerned with delivery.

5 Properties to be tested

5.1 General

The test method shall be selected after sufficiently understanding the purpose of the test, the analysis method and the nature of reagents. The properties to be tested shall be selected from the list in [5.2](#) by the parties concerned with delivery, in accordance with the destined end use of the FAC.

Water equivalent to Grade 1 as specified in ISO 3696 shall be used for the tests.

5.2 Specific tests

Tests for determining the physical properties of fibrous activated carbon are listed below:

- a) specific surface area;
- b) pore volume;
- c) physical properties of fibre;

The following physical properties of fibre shall be determined:

- 1) fibre diameter;
- 2) tensile strength;
- d) physical properties of sheet;

The following physical properties of sheet shall be determined:

- 1) mass per unit area;
- 2) tensile strength and elongation ratio;
- e) drying loss;
- f) pH value;
- g) total ash content;
- h) toluene adsorption performance, including:
 - 1) breakthrough adsorption;
 - 2) equilibrium adsorption amount;
- i) methylene blue adsorption performance, including:
 - 1) breakthrough adsorption;
 - 2) equilibrium adsorption amount;
- j) iodine adsorption performance, including the equilibrium adsorption amount.

6 Test methods

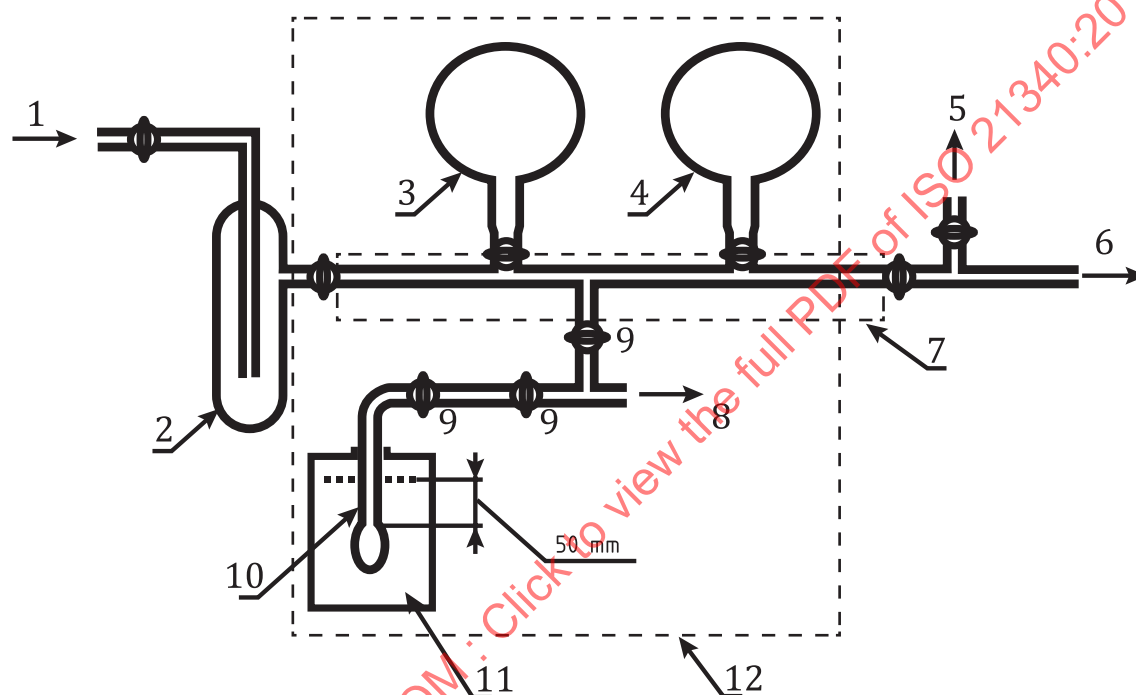
6.1 Specific surface area

6.1.1 Principle

Measurement of the specific surface area of fibrous activated carbon shall be carried out using the Brunauer-Emmett-Teller (BET) method.

6.1.2 Apparatus

An example of suitable nitrogen adsorption apparatus is shown in [Figure 1](#).



Key

- 1 gas introducing opening
- 2 condenser
- 3 nitrogen tank
- 4 helium tank
- 5 vacuum pump
- 6 membrane type manometer
- 7 manifold
- 8 Pirani gauge
- 9(C₀) stopcock
- 9(C₁) stopcock
- 9(C₂) stopcock
- 10 sample tube
- 11 liquid nitrogen
- 12 thermostatic bath

Figure 1 — Example of nitrogen adsorption apparatus

6.1.3 Test procedure

The test shall be carried out in the following steps:

a) Sample pretreatment

Place approximately 0,1 g to 0,2 g of sample in a sample tube of known mass in the measuring apparatus. Dry for ≥ 15 min at a temperature of ≥ 120 °C and at a pressure of ≤ 13 Pa.

b) Measurement of sample mass

Remove the electric furnace for drying the sample while evacuating the inside of the system, and allow the system to return to room temperature, and close stopcock C_0 . Detach the sample tube, wipe off any grease and weigh the total mass. Calculate the difference between this mass and the mass of sample tube in a) to obtain the mass of the sample after drying, to the nearest $\pm 0,1$ mg.

c) Measurement of dead volume

- 1) Evacuate the apparatus after cooling down to room temperature.
- 2) Immerse the sample tube in liquid nitrogen up to the marked line. During measurement, maintain the surface of the liquid nitrogen at this defined level, especially in steps c) 4) and d).
- 3) At this time, immerse the sample tube in liquid nitrogen to a depth of ≥ 50 mm, as shown in [Figure 1](#), and maintain the surface level of the liquid nitrogen within ± 2 mm. Supplement liquid nitrogen by automatic control to fill the Dewar vessel with liquid nitrogen. To take into account the variation in saturated vapour pressure of liquid nitrogen, measure the temperature of the liquid nitrogen with a precision of $\pm 0,1$ °C and carry out the correction of saturated vapour pressure. Use fresh liquid nitrogen for each measurement.
- 4) Fill the manifold with helium to approximately 50 Pa, and record the pressure.
- 5) Introduce helium into the sample tube and, after reaching equilibrium, record the pressure.
- 6) Obtain the dead volume of the sample tube immersed in liquid nitrogen using the pressure before and after introduction of helium and the volume of the manifold, according to the ideal gas law. Measure the dead volume either before or after the measurement of nitrogen adsorption, using helium at the same temperature used for the measurement of the amount of nitrogen adsorption [see d) below].

At this time, taking into account the variation in tube temperature before and after introduction of helium, maintain the temperature of the entire apparatus within $\pm 0,1$ °C, or carry out the correction of dead volume by measuring the room temperature before and after helium introduction.

d) Measurement of the amount of nitrogen adsorption

- 1) Evacuate the system to $\leq 0,14$ Pa.
- 2) Fill the manifold with nitrogen of $>99,995$ % purity by volume and after reaching equilibrium, record the pressure.
- 3) Open stopcocks C_0 , C_1 and C_2 and introduce nitrogen into the sample tube. After reaching equilibrium, record the pressure. Confirm that no change of pressure occurs within 10 min.

NOTE In some cases, the time required to reach equilibrium pressure is too short, which can become a source of error.

Attention shall also be paid to the surface of liquid nitrogen and the room temperature, as described in c) 5), when carrying out steps 2) and 3) above.

- 4) Obtain the adsorption amount from the pressure before and after introducing nitrogen, the volume of the manifold and the dead volume.

- 5) Repeat steps 2), 3) and 4) above until the relative pressure (p/p_0) of nitrogen becomes unity, where p_0 is the saturated vapour pressure, expressed in pascals, of adsorbate and p is the equilibrium pressure, expressed in pascals.

6.1.4 Calculation of specific surface area

6.1.4.1 Specific surface area shall be calculated using one of the following methods.

6.1.4.2 Multipoint method

First, using [Formula \(1\)](#), calculate the specific surface area using the data on the amount of nitrogen adsorption obtained in [6.1.3 d\)](#):

$$\frac{p}{V_1(p_0 - p)} = \frac{1}{V_m \cdot C} + \left(\frac{C-1}{V_m \cdot C} \right) \frac{p}{p_0} \quad (1)$$

where

p_0 is the saturated vapor pressure of adsorbed nitrogen, expressed in pascals;

p is the equilibrium pressure, expressed in pascals;

V_1 is the total volume of nitrogen adsorbed, expressed in cubic centimetres per gram;

V_m is the adsorbed volume of the monomolecular layer, expressed in millilitres per gram;

C is the BET constant.

Measurement under a relative pressure of $\leq 0,10$ is recommended.

When $p/[V_1(p_0 - p)]$ is plotted on the ordinate and p/p_0 is plotted on the abscissa, a straight line can be obtained within this range, and V_m and C can be obtained from the slope of the straight line and the intercept on the ordinate using [Formula \(1\)](#).

NOTE In this case, the nitrogen adsorption isotherm of fibrous activated carbon indicates a type I classified by International Union of Pure and Applied Chemistry (IUPAC). ISO 9277 puts such a case outside its scope of application because the BET plot is not a straight line with a relative pressure between 0,05 and 0,3. However, in this document, the BET plot is made to be a straight line because the relative pressure is made $\leq 0,10$, and the measurement of specific surface area according to the BET method can be applied.

Next, calculate the specific surface area using [Formula \(2\)](#).

$$S_{\text{BET}} = s \cdot V_m \cdot \frac{N}{V_0} = 4,35 V_m \quad (2)$$

where

S_{BET} is the BET specific surface area, expressed in square metres per gram;

s is the area occupied by a nitrogen molecule, i.e. $0,162 \text{ nm}^2$;

N is Avogadro's number;

V_0 is the gas volume under standard conditions ($22\,414 \text{ cm}^3$);

V_m is the volume of the adsorbed monomolecular layer, expressed in cubic centimetres per gram.

It is recommended that the calculation be carried out using the normal computer programme.

6.1.4.3 One-point method

For fibrous activated carbon, measurement under a relative pressure of $\leq 0,10$ is recommended, and the specific surface area can be obtained by measuring the adsorption amount at only one point in the range of relative pressure. When in the above-mentioned range of relative pressure, $C > 1$ is true and therefore the intercept on the ordinate, $1/(V_m C)$ of the BET plot is small and can be simplified as shown in [Formula \(3\)](#):

$$V_m = V_1 \left[1 - \left(\frac{p}{p_0} \right) \right] \quad (3)$$

where

p_0 is the saturated vapor pressure of adsorbed nitrogen, expressed in pascals;

P is the equilibrium pressure, expressed in pascals;

V_1 is the total volume of nitrogen adsorbed, expressed in cubic centimetres per gram;

V_m is the volume of the adsorbed monomolecular layer, expressed in cubic centimetres per gram.

It is recommended that the calculation be carried out using the normal computer programme.

6.2 Pore volume

6.2.1 Principle

The pore volume shall be the adsorption volume when the relative pressure is 0,995 from the nitrogen adsorption isotherm obtained in [6.1](#).

6.2.2 Apparatus and procedure

The apparatus and procedure shall be in accordance with [6.1.2](#) and [6.1.3](#).

Since measurement of the nitrogen adsorption isotherm under a relative pressure of 1,000 is usually difficult, correction of atmospheric pressure is carried out at the time of measurement, and the pore volume is obtained when a relative pressure of 0,995 is as close as possible to the saturated vapour pressure.

It is recommended that the calculation be carried out using the normal computer programme.

6.3 Physical properties of fibrous activated carbon

6.3.1 Fibre diameter

The measurement of fibre diameter shall be carried out using either a laser oscillator or a reflecting microscope.

a) Laser oscillator

1) apparatus

- i) laser oscillator, a helium-neon oscillator with laser wavelength of 633 nm;
- ii) goniometer;

iii) screen, to visualize the diffraction pattern of the laser beam;

2) procedure

- i) Fibrillate a bundle of fibre from the test sample, pull out a single monofilament and use it as the test specimen.
- ii) Irradiate the monofilament test specimen with the oscillating laser beam and record the distance from the screen at which the intensity of the diffraction pattern observed becomes minimal;
- iii) The number of measurements shall be decided between the parties concerned with delivery; 30 measurements are recommended.

3) calculation

To obtain the average fibre diameter D , calculate the diameter of the monofilament according to [Formula \(4\)](#) and average the results [[Formula \(5\)](#)], rounding off to one decimal place.

$$D_i = \frac{2l \cdot \lambda}{L} \times 10^{-3} \quad (4)$$

where

D_i is the diameter of monofilament, expressed in micrometres;

l is the distance between test specimen and screen, expressed in millimetres;

λ is the wavelength of helium-neon laser, expressed in nanometres;

L is the minimal distance between a pair of diffraction intensities nearest to the centre of the screen surface, expressed in millimetres.

$$D = \frac{\sum D_i}{n} \quad (5)$$

where

D is the average diameter of monofilament, expressed in micrometres;

D_i is the diameter of monofilament calculated with [Formula \(4\)](#), expressed in micrometres;

n is the number of measurements.

b) Reflecting microscope

1) apparatus

- i) reflecting microscope;
- ii) planimeter;
- iii) image-analysing apparatus;
- iv) dryer;

- v) polishing machine;
- 2) procedure
 - i) Take a bundle of fibre approximately 100 mm in length, bundle the end of it and insert the end into a tube of polytetrafluoroethylene of approximately 6 mm inside diameter.
 - ii) With embedding resin, uniformly impregnate the fibre protruding from the tube and draw it through the tube to align the fibres. Put the fibre bundle in a dryer, arranging the fibres parallel to each other, to harden the resin.

NOTE For the embedding resin, epoxy-based or acrylic-based thermosetting polymers such as methacrylate esters or acrylate esters are used.

- iii) Cut the bundle of resin-hardened fibre to approximately 25 mm in length, and fix it so that its longitudinal direction is perpendicular to the silicone rubber mold. Embed it by pouring in resin, and harden it in a dryer.
- iv) Polish the hardened bundle of fibre along the section perpendicular to the direction of fibre, using a polishing machine to finish it to a specular surface, and use this as the test specimen.
- iv) Fix the test specimen so that the specular surface is perpendicular to the optical axis of the reflecting microscope, and photograph a section. Calculate the fibre diameter based on the cross-sectional area of a single filament obtained either from the enlarged photo, using a planimeter, or from the microscope image using an image-analysing apparatus.
- v) The number of tests shall be in accordance with 6.3.1 a) 2) iii).

3) Calculation

To obtain the average fibre diameter D , calculate the diameter of the monofilament using [Formula \(6\)](#) from the cross-sectional area measured, and average the results [[Formula \(7\)](#)], rounding off to one decimal place.

$$D = \frac{\sum D_i}{n} \quad (6)$$

$$D_i = \sqrt{\frac{4A}{\pi}} \times 10^3 \quad (7)$$

where

D is the average value of the fibre diameter, expressed in micrometres;

D_i is the fibre diameter of monofilament, expressed in micrometres;

A is the cross-sectional area of monofilament, expressed in square millimetres;

n is the number of measurements.

6.3.2 Tensile strength

6.3.2.1 Principle

The test piece, which is a single filament fixed to a mount, is fixed to a tensile tester. Force is gradually applied to the test piece, and the force at which the test piece is broken is recorded as the tensile strength of a single filament.

6.3.2.2 Apparatus

A **tensile tester**, capable of maintaining a constant cross-head velocity and automatically recording the relation of load to elongation, shall be used.

6.3.2.3 Procedure

The tensile test shall be carried out as follows.

a) Preparation of the test piece

A bundle of fibre is taken from the sample and fibrillated sufficiently. A monofilament ≥ 15 mm in length is pulled out individually, and stretched along the central line of the test mount previously verified to be a straight line, and is fixed at two positions with adhesive to give the specified monofilament length.

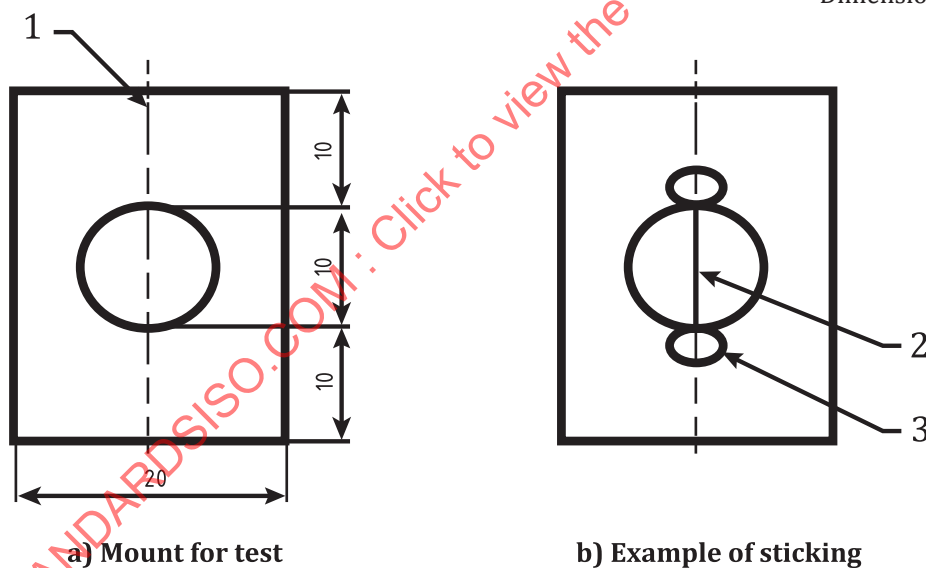
Examples of a test mount and attachment of the single filament are shown in [Figure 2](#).

The adhesive used shall be either rosin-based or epoxy resin-based.

Due to the adhesion procedure for the monofilament and possible cutting of the monofilament ends by the chuck of the tester, the recommended thickness of test mount is 0,2 mm to 0,4 mm.

When the sample is fibrillated and the monofilament fibres are glued on the mount for test, the fibres shall not be damaged by applying excess force.

Dimensions in millimetres



Key

- 1 central line
- 2 mono-filament
- 3 adhesive

Figure 2 — Examples of mount for tensile test and sticking

b) Test procedure

For the measurement of tensile strength, fix the test piece to the grips of the tensile tester and pull both ends at the central part of the test mount under the following conditions:

- length of test piece: 10,0 mm $\pm$ 0,2 mm;

— rate of elongation: 0,5 mm/min to 10,0 mm/min.

Measure the force required for rupture of the test piece.

The number of tests carried out shall be in accordance with 6.3.1 a) 2).

6.3.2.4 Calculation

Calculate the tensile strength for each test according to [Formula \(8\)](#) and obtain the average value to two significant figures.

$$\sigma = F / A \quad (8)$$

where

σ is the tensile strength of the monofilament, expressed in newtons per square millimetre;

F is the force applied to the monofilament at rupture, expressed in newtons;

A is the cross-sectional area of the monofilament, expressed in square millimetres.

Calculate the cross-sectional area, A , of the monofilament according to [Formula \(9\)](#).

$$A = (\pi / 4) D_i^2 \times 10^{-6} \quad (9)$$

where

D_i is the monofilament fibre diameter obtained according to [Formulae \(6\)](#) and [\(7\)](#) in 6.3.1 b) 3), expressed in micrometres.

6.3.2.5 Number of times of test

The number of times of test shall be in accordance with 6.3.1 a) 3).

6.4 Physical properties of fibrous activated carbon sheet

6.4.1 Principle

The physical properties of sheet to be measured are: the mass per unit area, tensile strength and elongation at break. The mass per unit area shall be obtained in accordance with ISO 9073-1; the tensile strength and elongation shall be obtained in accordance with ISO 9073-3.

6.4.2 Mass per unit area

a) Apparatus

Any of the following implements shall be used:

- 1) **punch cutter**, capable of cutting a test specimen measuring $\geq 62\,500\text{ mm}^2$;
- 2) **template**, having an area of shape measuring $\geq 62\,500\text{ mm}^2$;
- 3) **razor blade**;
- 4) **steel ruler**, with 1 mm graduations, or smaller;

5) **balance**, capable of weighing the mass of test specimen to a precision of 0,1 %;

b) Measurement atmosphere

Adjust the atmosphere to standard conditions of 20 °C ± 2 °C and (65 ± 2) % relative humidity.

c) Test procedure

1) Take three or more sheets of test specimen of ≥ 62 500 mm² (e.g. 250 mm × 250 mm) from the sample by using a punch cutter or template and a razor blade. When sheets of the required dimensions cannot be taken from the sample, cut it into rectangles of the closest possible size, measure the dimensions using a steel ruler, and calculate the area.

2) After conditioning the test specimens in the standard atmosphere specified in b) for ≥ 12 h, weigh the mass of the test specimens under the standard conditions.

d) Calculation

Calculate the mass per unit area according to [Formula \(10\)](#), obtain the average value and round off this value to three significant figures.

$$m_A = m/A \quad (10)$$

where

m_A is the mass per unit area, expressed in grams per square metre;

m is the average value of mass of the test piece, expressed in grams;

A is the area of the test piece, expressed in square metres.

6.4.3 Tensile strength and elongation

a) Apparatus

The tensile testing machine shall have a constant elongation rate and be capable of automatically recording the load and the free length of test pieces between grips.

b) Test atmosphere

The test atmosphere shall be in accordance with [6.4.2 b\)](#).

c) Test procedure

1) Take both lengthwise and crosswise respectively, seven sheets of test pieces of 50 mm ± 0,5 mm width, and of a length allowing 200 mm between the grips, e.g. 300 mm, and a uniform distance of ≥ 100 mm from each end of the sample. Condition the samples as mentioned above for 12 h or longer, and then measure the dimensions.

When measuring, the samples may be mounted with ≤ 50 mm in width and ≤ 200 mm in length between grips, in accordance with the agreement between the parties concerned with delivery. Such agreement shall be mentioned in the record.

2) Mount the test piece for the first test load with the length between grips of tensile testing machine of 200 mm ± 1 mm.

The first test shall be the degree of load where the test piece is held taut. The measurement may be carried out by inserting a rubber sheet, pasteboard or the like at the place of contact with the grip, so that the test piece does not slip while it is being measured.

- 3) Apply load at an elongation rate of 100 mm/min $\pm$ 10 mm/min until rupture of the test piece.
An elongation rate other than 100 mm/min may be used, according to agreement between the parties concerned with delivery. Such agreement shall be mentioned in the record.
- 4) Record the strength of test pieces under the maximum load to the nearest 0,1 N and record the elongation under the maximum load to the nearest 1 mm. Calculate the rate of test piece elongation from this elongation.
- 5) Obtain the average value of tensile strength and the average value of elongation rate of five points, excluding the maximum and minimum values of tensile strength in the lengthwise direction and crosswise direction respectively. Record the average tensile strength value to one decimal place, and the average elongation rate value to two significant figures.

6.5 Drying loss

6.5.1 Principle

The sample is dried in a thermostatic dryer and the loss in mass is measured.

6.5.2 Apparatus

6.5.2.1 Cylindrical glass weighing bottle, 45 mm in diameter and 60 mm in length, with lid.

6.5.2.2 Desiccator, with Type A silica gel desiccant.

6.5.2.3 Chemical or electronic force balance, capable of weighing to the nearest 0,1 mg.

6.5.2.4 Thermostatic dryer, capable of regulating the temperature to 115 °C $\pm$ 5 °C.

6.5.3 Procedure

- a) Weigh sufficient sample to the nearest 1 mg, to give approximately 1 g in mass after drying, in a cylindrical weighing bottle ([6.5.2.1](#)) of known mass. Spread the sample in uniform thickness on the bottom surface of the bottle.
- b) Heat the weighing bottle and its lid in a thermostatic dryer ([6.5.2.4](#)) maintained at 115 °C $\pm$ 5 °C for approximately 3 h or longer. At this time, a temporary permeable cover such as wire net may be used on the weighing bottle to prevent the sample from scattering.
- c) After cooling the sample in a desiccator ([6.5.2.2](#)), put on the lid and weigh the bottle to the nearest 1 mg.

6.5.4 Calculation

Calculate the loss of mass on drying according to [Formula \(11\)](#), and round off the result to one decimal place.

$$w_B = \frac{m_1 - m_2}{m_1 - m_3} \times 100 \quad (11)$$

where

w_B is the loss of mass on drying, expressed as a percent (mass fraction);

m_1 is the mass of sample and weighing bottle before drying, expressed in grams;

m_2 is the mass of sample and weighing bottle after drying, expressed in grams;

m_3 is the mass of the weighing bottle, expressed in grams.

6.6 pH value

6.6.1 Principle

The sample is boiled in water and after cooling the pH value of the filtrate or suspended solution is measured using a pH-meter.

6.6.2 Reagents and materials

6.6.2.1 Water, equivalent to Grade 1 as specified in ISO 3696, with a conductivity of 0,2 mS/m to 1,0 mS/m (2 μ S/cm to 10 μ S/cm) at 25 °C.

6.6.2.2 pH meter.

6.6.2.3 Filter paper.

6.6.3 Apparatus

The usual laboratory apparatus and, in particular, the following.

6.6.4 Procedure

- Place approximately 0,5 g of sample (converted to dried mass), in a 200 ml beaker. Convert the dried mass of sample by using [Formula \(11\)](#) for the loss of mass on drying, as calculated in [6.5.4](#).
- Add 100 ml of water ([6.6.2.1](#)), heat gently and boil for 5 min.
- After cooling to room temperature, filter the sample through filter paper ([6.6.2.3](#)). Add water to the filtrate to give a volume of 100 ml, agitate it sufficiently, and measure the pH value using the pH-meter ([6.6.2.2](#)).

The purpose of filtration is to prevent the sample from floating to the liquid surface, entanglement with the pH meter, etc. The pH value may be measured under conditions of suspended sample.

6.7 Total ash content

6.7.1 Principle

The sample is incinerated in an electric furnace and the residue obtained is weighed.

6.7.2 Apparatus

6.7.2.1 Porcelain crucible, of 50 ml capacity.

When the sample is bulky, a crucible of 100 ml capacity may be used.

6.7.2.2 Desiccator, as specified in [6.5.2.2](#).

6.7.2.3 Balance, as specified in [6.5.2.3](#).

6.7.2.4 Thermostatic dryer, as specified in [6.5.2.4](#).

6.7.2.5 Electric furnace, capable of heating to a controlled temperature of 800 °C to 900 °C.

6.7.3 Procedure

- Heat a portion of the FAC in a thermostatic dryer ([6.7.2.4](#)) at $115\text{ °C} \pm 5\text{ °C}$ for approximately 3 h and cool in a desiccator. Weigh out approximately 1 g of the sample, to the nearest 1 mg, into a porcelain crucible ([6.7.2.1](#)) brought to constant mass beforehand. Spread the sample uniformly in the crucible and put on a lid.
- Put the sample and the crucible in an electric furnace ([6.7.2.5](#)). Heat furnace and sample gently at the start, then raise the temperature gradually to incinerate the sample. Continue heating at 800 °C to 900 °C for 1 h to incinerate the sample completely.
- Remove the crucible and contents from the furnace and cool in a desiccator ([6.7.2.2](#)) to room temperature. Determine the mass of incinerated sample (ash) to the nearest 1 mg.

6.7.4 Calculation

Calculate the total ash content of the fibrous activated carbon sample using [Formula \(12\)](#), and round off the result to one decimal place.

$$w_A = (m_S/m_A) \times 100 \quad (12)$$

where

w_A is the total ash content of the sample, expressed as a percent (mass fraction);

m_S is the mass of sample after drying, expressed in grams;

m_A is the mass of sample after incineration, expressed in grams.

6.8 Toluene adsorption performance

6.8.1 Breakthrough adsorption test

6.8.1.1 Principle

A defined amount of gas containing toluene vapour of specified concentration is passed through the sample. The toluene concentration in the gas exiting the sample is measured by a detection tube for gas measurement, by continuous hydrocarbon analysis or by gas chromatography, and the breakthrough time is obtained.

6.8.1.2 Reagent

Toluene, as specified in ISO 6353-2, shall be used.

In handling toluene, which is highly inflammable, exposure to flame shall be avoided. Also, since it is harmful, inhalation of the vapour or any contact with the mucous membranes or the skin shall be avoided.

6.8.1.3 Apparatus

6.8.1.3.1 **Thermostatic dryer**, as specified in [6.5.2.4](#).

6.8.1.3.2 **Desiccator**, as specified in [6.5.2.2](#).

6.8.1.3.3 **Balance**, as specified in [6.5.2.3](#).

6.8.1.3.4 **Column for breakthrough adsorption test** (see example in [Figure 3](#)).

6.8.1.3.5 **Toluene breakthrough adsorption test apparatus** (see example in [Figure 4](#)).

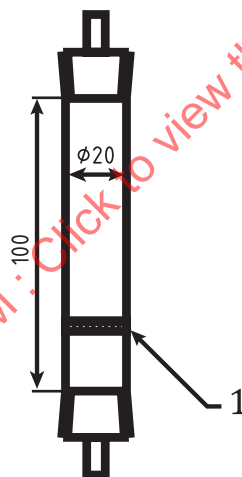
6.8.1.3.6 **Thermostatic bath**; i.e. a constant-temperature air vessel or a constant-temperature room, capable of maintaining the sample at the set temperature ± 1 °C.

6.8.1.3.7 **Gas chromatograph**.

6.8.1.3.8 **Detection tube for gas measurement**.

6.8.1.3.9 **Continuous hydrocarbon analyser**.

Dimensions in millimetres



Key

1 glass filter

Figure 3 — Example of column for toluene breakthrough adsorption test

6.8.1.4 Preparation of gas containing toluene vapour

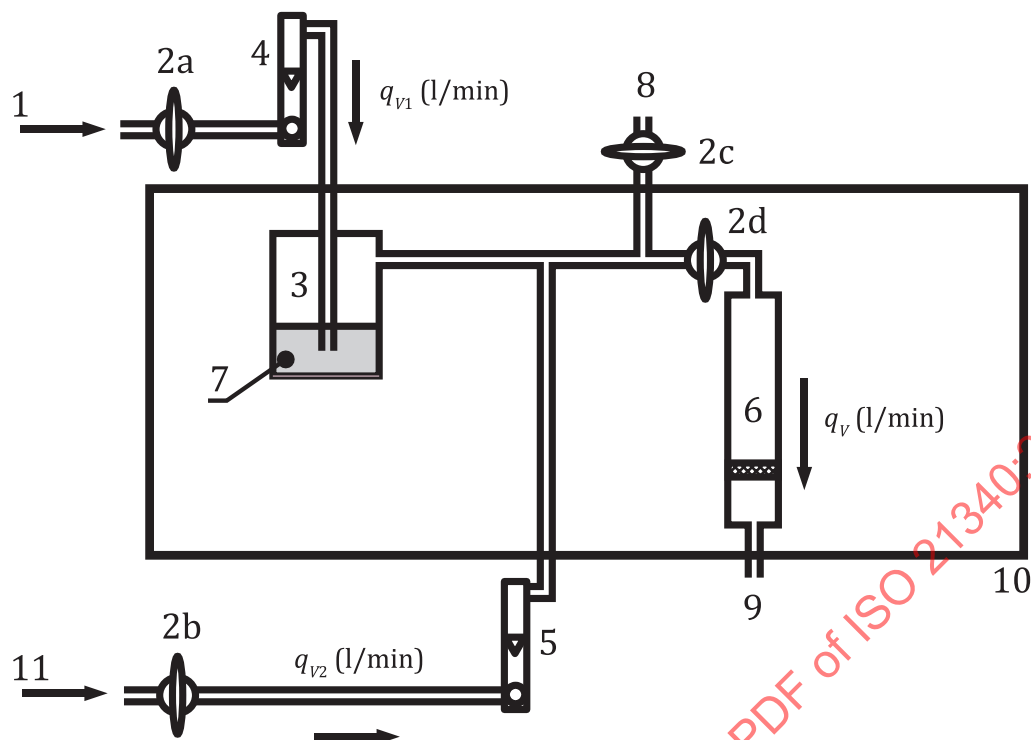
Pour toluene into the toluene vapour-generating bottle illustrated in [Figure 4](#), and adjust stopcock 2a to introduce a flow of dry air or nitrogen of the calculated volume q_{V1} (l/min). Adjust stopcock 2b to introduce a flow of dry air for dilution of the calculated amount q_{V2} (l/min). Calculate the flow rates of q_{V1} and q_{V2} of dry air according to [Formulae \(13\)](#) and [\(14\)](#).

$$q_{V1} = \left(\frac{q_V \cdot c_i}{10^6} \right) \left(\frac{101,325}{p} - 1 \right) \quad (13)$$

$$q_{V2} = q_V \left(1 - \frac{101,325 c_i}{p \times 10^6} \right) \quad (14)$$

where

- q_{V1} is the flow rate of dry air passed through the toluene vapour-generating bottle, expressed in litres per minute;
- q_{V2} is the flow rate of dry air for dilution, expressed in litres per minute;
- c_i is the concentration of toluene passed through the sample, expressed as a volume fraction (ppm);
- q_V is the flow rate of gas containing toluene vapour, expressed in litres per minute;
- p is the saturated vapour pressure of toluene at the measurement temperature (°C), expressed in kilopascals;
- 101,325 is the conversion factor (1 atm = 101,325 kPa).



Key

- 1 air for generation of toluene vapour
- 2a, 2b, 2c, 2d two way stopcocks
- 3 toluene vapour-generating bottle
- 4 flow meter for air containing toluene vapour
- 5 flow meter for air for dilution
- 6 column for breakthrough adsorption test
- 7 toluene
- 8 excess gas outlet
- 9 gas exhaust outlet (measurement of vapour concentration)
- 10 thermostatic bath
- 11 air for dilution
- q_v flow rate of gas containing toluene vapour (l/min)
- q_{v1} flow rate of dry air exiting toluene vapour-generating bottle (l/min)
- q_{v2} flow rate of dry air for dilution (l/min)

Figure 4 — Example of toluene breakthrough adsorption test apparatus

6.8.1.5 Breakthrough adsorption test conditions

In the case of cloth samples, the bulk density and height of the packed bed shall be measured under the conditions given in parentheses in e) and f) below, due to its higher density compared with felt and fibre.

- a) Measurement temperature: $25\text{ °C} \pm 5\text{ °C}$;
- b) Toluene concentration: $750\text{ ppm} \pm 250\text{ ppm}$ (volume fraction);
- c) Flow rate: $25\text{ cm/s} \pm 5\text{ cm/s}$;
- d) Inside diameter of column for breakthrough adsorption test: 20 mm;

- e) Bulk density: $0,075 \text{ g/ml} \pm 0,025 \text{ g/ml}$ ($0,225 \text{ g/ml} \pm 0,075 \text{ g/ml}$);
- f) Height of packed bed: $45 \text{ mm} \pm 5 \text{ mm}$ ($15 \text{ mm} \pm 5 \text{ mm}$);
- g) Mass of sample: $1 \text{ g} \pm 0,05 \text{ g}$.

6.8.1.6 Breakthrough adsorption test procedure

The test shall be carried out in the following steps.

- a) After heating the sample in a thermostatic drier at $115 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ for approximately 3 h, stopper it tightly and leave it to cool in a desiccator to room temperature.

Sheet-shaped samples such as felt and cloth are dried after punching them into a circle of diameter 20 mm.
- b) Weigh sufficient dried and cooled sample to the nearest 1 mg to obtain a test sample of $1 \text{ g} \pm 0,05 \text{ g}$ in mass; fill the adsorption test column with sample so that the height of the packed column is $45 \text{ mm} \pm 5 \text{ mm}$.
- c) Pass the specified air through the toluene breakthrough adsorption test apparatus at $25 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ and prepare the air mixed with toluene vapour so the concentration of toluene (c_i) is $750 \text{ ppm} \pm 250 \text{ ppm}$ (volume fraction). Adjust the temperature to $\pm 1 \text{ }^{\circ}\text{C}$ of the set temperature.
- d) Attach the column for breakthrough adsorption test in the specified position on the test apparatus. Control the flow rate by opening and closing stopcock 2c or 2d, and pass the air mixed with toluene vapour through the test column to adsorb toluene. At this time, regulate flow rates q_{V1} and q_{V2} so that the gas flow rate through the sample in the adsorption test column is 20 cm/s to 30 cm/s , adjusting for any variation in flow rate during the test.
- e) After introducing the gas mixed with toluene vapour, sample the gas exiting the column for the breakthrough adsorption test at adequate time intervals and continuously record the concentration of toluene vapour in this gas using a gas chromatograph, a detection tube for gas measurement or a continuous hydrocarbon analyser. The interval between gas samplings should be approximately 5 min.
- f) When the concentration of toluene vapour in the gas exiting the column reaches approximately 50 % of the concentration in the gas entering the column, terminate the gas feed operation.

NOTE In general, feeding of adsorbent is continued until its concentration reaches around 90 % or 100 % of the inlet concentration (the condition where the whole layer of adsorbent is considered to reach equilibrium). The length of layer in which adsorption progresses (length of the adsorption band is usually from 10 % breakthrough point to 90 % breakthrough point) is obtained and the adsorption rate is determined from it. The equilibrium adsorption amount can be investigated in this way.

- g) Plot the concentration of toluene vapour measured in e) against time, and note the time at which the concentration of toluene vapour in gas exiting the test column reaches 10 % of the concentration of toluene vapour in gas entering the test column, as the breakthrough time.

6.8.2 Equilibrium adsorption amount

6.8.2.1 Principle

Air containing a specified concentration of toluene vapour is passed through the sample at a defined rate, and the equilibrium adsorption amount is obtained from the increase in sample mass when the mass becomes constant.

6.8.2.2 Reagent

Toluene, as specified in ISO 6353-2, shall be used.

6.8.2.3 Apparatus

6.8.2.3.1 **Thermostatic dryer**, as specified in [6.5.2.4](#).

6.8.2.3.2 **Desiccator**, as specified in [6.5.2.2](#).

6.8.2.3.3 **Balance**, as specified in [6.5.2.3](#).

6.8.2.3.4 **Test tube** for the adsorption test, an example is shown in [Figure 5](#).

6.8.2.3.5 **Thermostatic bath**, i.e. a constant-temperature air bath or constant-temperature water bath capable of maintaining the set temperature ± 1 °C.

6.8.2.3.6 **Toluene vapour adsorption test apparatus**, an example is shown in [Figure 6](#).

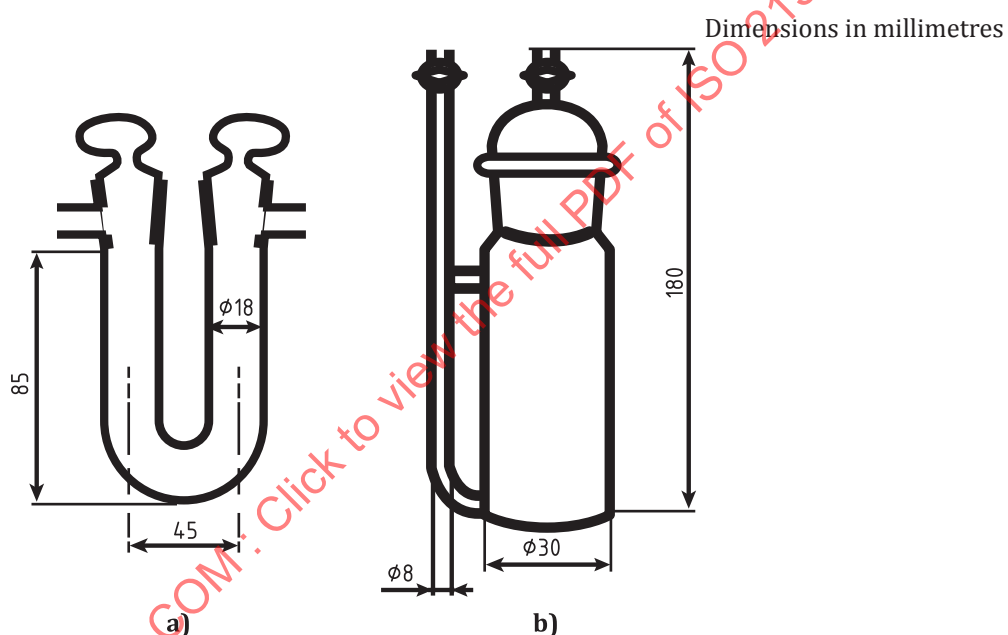
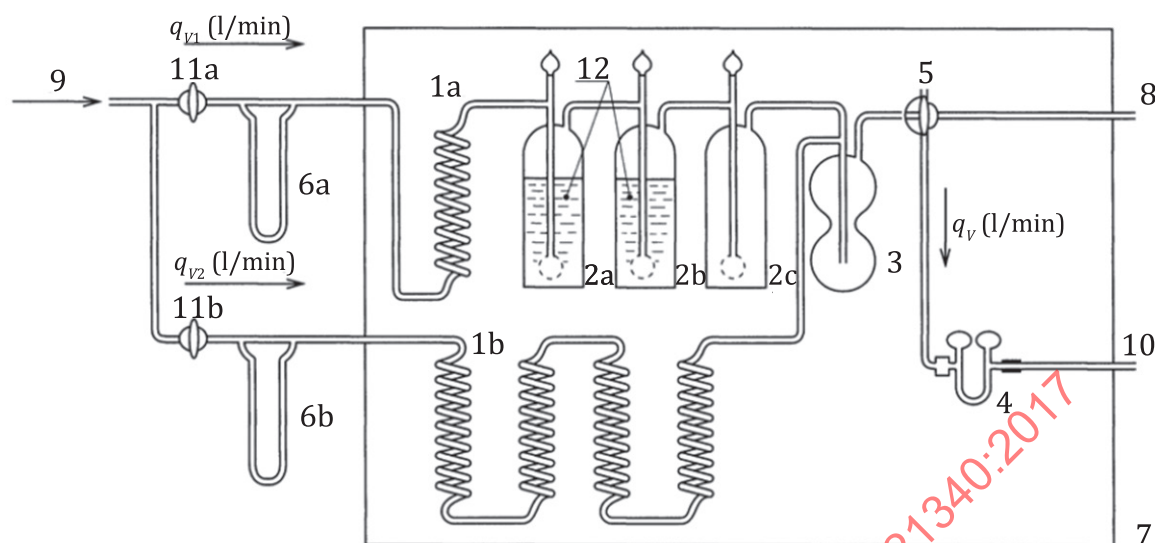


Figure 5 — Example of test tube for adsorption performance test



Key

- 1a, 1b coiled tubes for temperature control
- 2a, 2b, 2c gas wash-bottles with interchangeable ground joint filter plate (250 ml)
- 3 gas mixing bottle, bulb inside diameter 60 mm, double-sphere continuous type
- 4 test tube for adsorption test
- 5 three-way stopcock
- 6a flow meter for toluene vapour-generating air
- 6b flow meter for air for dilution
- 7 thermostatic bath
- 8 exhaust outlet for excess gas
- 9 dry air inlet
- 10 test gas outlet
- 11a, 11b stopcocks for adjusting gas flow rate
- 12 toluene
- q_v flow rate of gas containing toluene vapour (l/min)
- q_{v1} flow rate of dry air passed through toluene vapour-generating bottle (l/min)
- q_{v2} flow rate of dry air for dilution (l/min)

Figure 6 — Example of toluene vapour adsorption performance test apparatus

6.8.2.4 Preparation of gas containing toluene vapour

After adjusting the thermostat to the specified bath temperature, pour toluene in the toluene vapour-generating bottles 2a and 2b shown in Figure 6. Set stopcock 5 beforehand to allow flow in the direction of exhaust outlet for excess gas, 8. Introduce dry air of calculated volume q_{v1} by adjusting the flow rate (l/min) with stopcock 11a. Introduce dry air of calculated volume q_{v2} for dilution (l/min) by adjusting stopcock 11b.

Calculate the flow rate of dry air, q_{v1} and q_{v2} , according to Formulae (13) and (14) given in 6.8.1.4.

6.8.2.5 Procedure

The test procedure shall be carried out in the following steps.

- a) Weigh the mass of the adsorption test tube to the nearest 1 mg.

- b) Place $\geq 0,5$ g of the sample in the adsorption test tube as uniformly as possible. After heating in a thermostatic dryer at $115\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for approximately 3 h, stopper the sample hermetically and cool it to room temperature in a desiccator.
- c) Weigh the mass of the adsorption test tube containing sample to the nearest 1 mg. Determine the mass of dried sample, m_0 .
- d) To prepare the air mixed with the specified concentration of toluene vapour, pass the specified air through the toluene vapour adsorption performance test apparatus at a fixed temperature of $25\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. Maintain the temperature at $\pm 1\text{ }^{\circ}\text{C}$ of this temperature.
- e) Attach the adsorption test tube containing the sample at the specified position on the toluene vapour adsorption test apparatus, and switch stopcock 5 to allow flow in the direction of 10 (see [Figure 6](#)). Pass the air mixed with toluene vapour through the adsorption test tube to adsorb toluene on the sample. Maintain a constant flow rate during this procedure.
- f) After ≥ 30 min, remove the adsorption test tube, wipe off any moisture with dry paper or cloth and immediately weigh the mass.
- g) Again attach the adsorption test tube containing sample at the specified position on the toluene vapour adsorption test apparatus. After passing the air mixed with toluene vapor over the sample again for 15 min, remove the adsorption test tube, wipe it with dry paper or cloth, and immediately weigh the mass.
- h) Repeat procedure g) until the subsequent increase in mass of the adsorption test tube drops to ≤ 5 mg. Determine the total increase in mass of the sample, Δm .

6.8.2.6 Calculation

Calculate the equilibrium adsorption amount of toluene according to [Formula \(15\)](#) and round off the result to one decimal place.

$$w_q = (\Delta m / m_0) \times 100 \quad (15)$$

where

w_q is the equilibrium adsorption amount of toluene vapour, expressed as a percent (mass fraction);

Δm is the increase in mass of sample due to toluene adsorption, expressed in grams;

m_0 is the original mass of dried sample, expressed in grams.

6.9 Methylene blue adsorption performance

6.9.1 Breakthrough adsorption test

6.9.1.1 Principle

The sample is filled in a column, and methylene blue solution is passed through at a flow rate of 30 ml/min. The adsorption of the solution passed through is measured and a breakthrough curve is plotted from the change in concentration of residual methylene blue with time. The time at which the outlet concentration reaches 10 % of the inlet concentration is determined from the breakthrough curve, and is taken as the breakthrough time of methylene blue.

6.9.1.2 Reagents

6.9.1.2.1 Potassium dihydrogen phosphate (1/15 mol/l): Dissolve 9,08 g of potassium dihydrogen phosphate (KH_2PO_4) as specified in ISO 6353-3 in sufficient water to make 1 000 ml solution.

6.9.1.2.2 Disodium hydrogen phosphate (1/15 mol/l): Dissolve 23,88 g of disodium hydrogen phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) as specified in ISO 6353-2 in sufficient water to make 1 000 ml solution.

6.9.1.2.3 Phosphate buffer solution (pH 7): Mix potassium dihydrogen phosphate solution ([6.9.1.2.1](#)) and disodium hydrogen phosphate solution ([6.9.1.2.2](#)) at a volume ratio of 4:6.

6.9.1.2.4 Methylene blue solution: Weigh out 0,24 g (as dry mass) of methylene blue [purity >98,5 % mass fraction] in a 1 000 ml volumetric flask, and dissolve it by adding phosphate buffer solution ([6.9.1.2.3](#)). Add more buffer solution ([6.9.1.2.3](#)) up to the marked line.

The dry mass of methylene blue, whose characteristics change when dried, shall have been obtained previously, as follows: heat 1 g of methylene blue in a thermostatic dryer maintained at $105\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$ for 4 h; allow to cool in a desiccator using silica gel as desiccant; and obtain the loss in mass upon drying w (% mass fraction). Applying this value to [Formula \(16\)](#), convert the mass of undried methylene blue m_u to dried mass m_d .

$$m_d = [m_u / (100 - w)] \times 100 \quad (16)$$

where

m_d is the mass of methylene blue, converted to dry mass, expressed in grams;

m_u is the mass of undried methylene blue sample, expressed in grams;

w is the loss in mass upon drying, expressed as a percent (mass fraction).

6.9.1.3 Apparatus

6.9.1.3.1 Thermostatic dryer, as specified in [6.5.2.4](#).

6.9.1.3.2 Photometer, i.e. a photometric spectrophotometer.

6.9.1.3.3 Column for breakthrough adsorption test, having inside diameter 20 mm, height (trunk part) 100 mm to 150 mm and with an incorporated glass filter. An example is shown in [Figure 7](#).

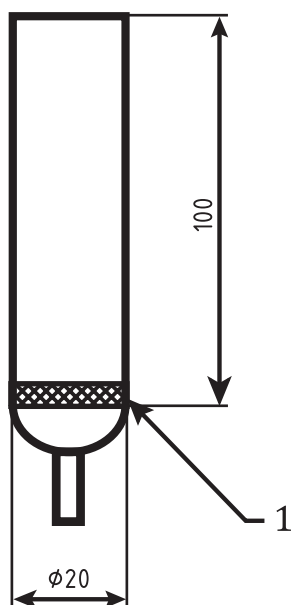
6.9.1.3.4 Constant flow-rate pump, capable of adjusting flow rate in the range 20 ml/min to 100 ml/min.

6.9.1.3.5 Methylene blue breakthrough adsorption test apparatus. An example is shown in [Figure 8](#).

6.9.1.4 Test conditions

- Measurement temperature: $25\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$;
- Concentration of methylene blue solution: 0,24 g/l;
- Flow rate: 30 ml/min;
- Bulk density: $0,100\text{ g/ml} \pm 0,050\text{ g/ml}$;
- Height of packed bed: $45\text{ mm} \pm 5\text{ mm}$;
- Mass of sample: $1,5\text{ g} \pm 0,05\text{ g}$.

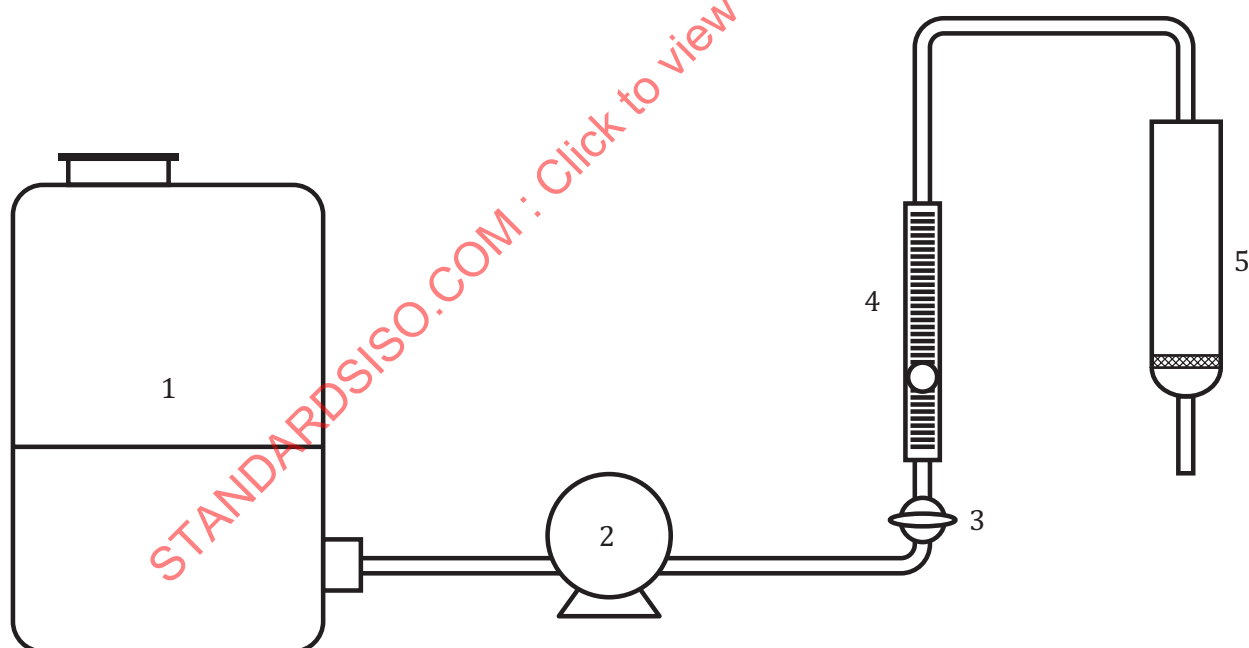
Dimensions in millimetres



Key

- 1 glass filter

Figure 7 — Example of column for breakthrough adsorption test



Key

- 1 storage bottle for methylene blue solution
 2 constant flow rate pump
 3 stopcock for flow rate adjustment
 4 flow meter for methylene blue solution
 5 column for breakthrough adsorption test

Figure 8 — Example of methylene blue breakthrough adsorption test apparatus

6.9.1.5 Procedure

The test procedure shall be carried out in the following steps.

- Heat about 3 g of FAC in a thermostatic dryer at $115\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for approximately 3 h, stopper it tightly and cool to room temperature in a desiccator.
- Accurately weigh out a sample from a) to the nearest 1 mg, to give $1,5\text{ g} \pm 0,05\text{ g}$ in mass. Prepare a slurry of the sample with water, mixing and deaerating sufficiently. Fill the column for the breakthrough adsorption test with this slurry to create a layer $40\text{ mm} \pm 5\text{ mm}$ in height, under suction to ensure no air bubbles are contained within the layer.
- Attach the test column filled with the sample to the methylene blue breakthrough adsorption test apparatus, and introduce the methylene blue solution at a flow rate of 30 ml/min. The constant flow rate pump should be adjusted beforehand to a flow rate of 30 ml/min.
- Sample the solution (eluant) at specified intervals. It is recommended to estimate the breakthrough time from previous test results of the equilibrium adsorption amount of methylene blue, and to take samples at shorter intervals in the neighbourhood of the estimated breakthrough time.
- Place the eluant in an absorption cell of 10 mm optical path length, and measure the absorbance of methylene blue at a wavelength of 665 nm using a photometer, with phosphate buffer solution (pH 7) as the control.

6.9.1.6 Preparation of the working curve

- Place 10 ml of methylene blue solution in a 100 ml flask using a volumetric pipette and add phosphate buffer solution (pH 7) up to the marked line. Take 5 ml, 10 ml, 25 ml and 50 ml from this solution and place in 500 ml volumetric flasks respectively. Add phosphate buffer solution (pH 7) up to the marked line.
- Based on these standard solutions, prepare the working curve by plotting the concentrations (0,24 mg/l to 2,40 mg/l) of methylene blue solution against the absorbance at 665 nm wavelength.

6.9.1.7 Preparation of methylene blue breakthrough curve

Obtain the concentrations of methylene blue in the eluant samples from the working curve (6.9.1.6). Plot the concentrations of methylene blue in the eluant samples thus obtained on the ordinate, and plot the sampling time on the abscissa to prepare the breakthrough curve.

6.9.1.8 Breakthrough time

From the breakthrough curve obtained in 6.9.1.7, take, as the breakthrough time of methylene blue, the time when the methylene blue eluant concentration at the test column outlet reaches 10 % (24 mg/l) of the concentration of methylene blue solution fed into the test column.

6.9.2 Equilibrium adsorption amount

6.9.2.1 Principle

The sample is added to a solution of known methylene blue concentration. After adsorption of methylene blue onto the sample reaches equilibrium, the solution is filtered, and the absorbance of the filtrate is measured. The amount of methylene blue adsorbed is determined from the filtrate concentration. The adsorption isotherm is prepared from this test result and the equilibrium adsorption amount of methylene blue, defined as when the filtrate concentration of methylene blue reaches 0,24 mg/l, is obtained from the adsorption isotherm.

6.9.2.2 Reagents

Methylene blue solution is prepared in accordance with [6.9.1.2](#), except that the mass of methylene blue (purity >98,5 % mass fraction) shall be 1,2 g (as dry mass).

6.9.2.3 Apparatus

6.9.2.3.1 Thermostatic dryer, as specified in [6.5.2.4](#).

6.9.2.3.2 Shaker, capable of reciprocating 200 times to 300 times per minute with a horizontal amplitude of 40 mm to 50 mm.

6.9.2.3.3 Photometer, i.e. photometric spectrophotometer.

6.9.2.3.4 Filter paper, of diameter 20 mm.

6.9.2.3.5 Funnel, with flat perforated plate of diameter 15 mm.

6.9.2.4 Procedure

The test procedure shall be carried out in the following steps.

- a) To prepare the sample, cut the FAC to lengths of ≤ 3 mm, heat in a thermostatic dryer ([6.9.2.3.1](#)) at $115\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for ≥ 3 h and then cool in a desiccator.
- b) Weigh out the specified amount of sample to the nearest 1 mg, transfer it into a 100 ml Erlenmeyer flask with interchangeable ground joint, and add 25 ml of methylene blue solution. When the sample is weighed out, weigh it by dividing into three stages or more so as not to exceed 0,02 g in the interval of mass corresponding to the adsorption performance of methylene blue expected in 0,1 g to 0,3 g of sample.
- c) Shake the flask containing the sample plus methylene blue solution at room temperature for 30 min.
- d) Place 10 ml of methylene blue solution in a 500 ml flask using a volumetric pipette, and add phosphate buffer solution (pH 7) up to the marked line. Place 5 ml of this solution in another 500 ml volumetric flask and add phosphate buffer solution (pH 7) up to the marked line. The concentration of methylene blue solution is now 0,24 mg/l.
- e) Filter, under reduced pressure, 20 ml of methylene blue solution d) which has passed through the sample, using the funnel ([6.9.2.3.5](#)) with flat perforated plate and the filter paper ([6.9.2.3.4](#)).
- f) Place a portion of the filtrate in an absorption cell of 10 mm optical path length and measure the absorbance at 665 nm wavelength using a photometer ([6.9.2.3.3](#)), taking the phosphate buffer solution (pH 7) as the control.

6.9.2.5 Preparation of working curve

Prepare the working curve as follows.

- a) Place 10 ml of methylene blue solution in a 100 ml flask by using a volumetric pipette and add phosphate buffer solution (pH 7) up to the marked line. Take 5 ml, 10 ml, 25 ml and 50 ml from this solution and place in 500 ml volumetric flasks respectively. Add phosphate buffer solution (pH 7) up to the marked line.
- b) Based on these standard solutions, prepare the working curve by plotting the concentrations (0,24 mg/l to 2,40 mg/l) of methylene blue solution against the absorbance at 665 nm wavelength obtained in [6.9.2.4 f\)](#), and from this curve obtain the residual concentration of methylene blue in the filtrate samples.

6.9.2.6 Calculation

Calculate the mass adsorption of methylene blue onto the sample, in milligrams per gram, according to [Formula \(17\)](#), using the residual concentration c_r of methylene blue in the filtrate obtained in [6.9.2.5](#).

$$Q = \frac{(1200 - c_r)(25 \times 10^{-3})}{m} \quad (17)$$

where

Q is the mass adsorption of methylene blue on the sample, expressed in milligrams per gram;

c_r is the residual concentration of methylene blue, expressed in milligrams per litre;

m is the mass of sample, expressed in grams;

1 200 is the initial concentration of methylene blue solution, expressed in milligrams per litre;

25×10^{-3} is a conversion factor.

6.9.2.7 Preparation of adsorption isotherm in methylene blue solution

Plot the residual concentration of methylene blue solution obtained in [6.9.2.5](#) on the abscissa and the adsorbed amount of methylene blue calculated in [6.9.2.6](#) on the ordinate of log-log graph paper, and prepare the adsorption isotherm.

6.9.2.8 Calculation of adsorption performance for methylene blue

Calculate the equilibrium mass adsorption, in milligrams of methylene blue per gram of sample, at which the residual concentration of methylene blue reaches 0,24 mg/l (obtained from the adsorption isotherm obtained in [6.9.2.7](#)).

Calculate the methylene blue adsorption performance according to [Formula \(18\)](#). Express the adsorption performance in units of 10 ml/g.

$$M = (Q/1\,200) \times 1\,000 \quad (18)$$

where

M is the adsorption performance of methylene blue, expressed in millilitres per gram;

Q is the mass adsorption of methylene blue, expressed in milligrams per gram;

1 200 is the initial concentration of methylene blue solution, expressed in milligrams per litre.

An example adsorption isotherm is illustrated in [Figure 9](#), and an example of adsorption test data is indicated in [Table 1](#).