



BSI Standards Publication

Test methods for fibrous activated carbon

National foreword

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Test methods for fibrous activated carbon

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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.

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

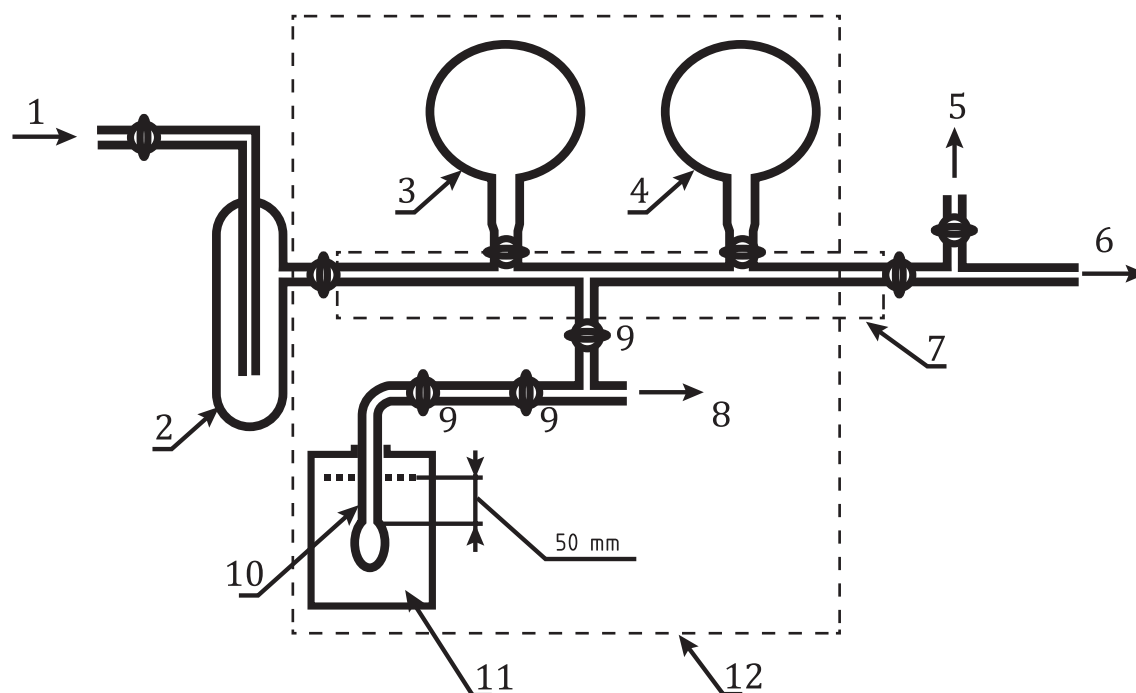
6.1 Specific surface area

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