

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 1477 : 2007

(AFAC/JSA)

**Test methods for fibrous activated
carbon**

ICS 71.060.10

Reference number : **JIS K 1477 : 2007 (E)**

PROTECTED BY COPYRIGHT

 **Documents**
Document Sharing Hub

20 S

Date of Establishment: 1995-04-01

Date of Revision: 2007-11-20

Date of Public Notice in Official Gazette: 2007-11-20

Investigated by: Japanese Industrial Standards Committee
Standards Board
Technical Committee on Chemical Analysis

JIS K 1477:2007, First English edition published in 2008-04

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 2008

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

PROTECTED BY COPYRIGHT

Contents

	Page
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Sampling method	3
5 General matters	3
6 Test items	3
7 Test method	4
7.1 Specific surface area	4
7.2 Pore volume	7
7.3 Physical properties of fibre	8
7.4 Physical properties of sheet	12
7.5 Drying loss	13
7.6 pH value	14
7.7 Ignition residue	15
7.8 Toluene adsorption performance	16
7.9 Methylene blue adsorption performance	23
7.10 Iodine adsorption performance	29

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Association of Fibrous Activated Carbon (AFAC)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 1477 : 1995** is replaced with this Standard.

This **JIS** document is protected by the Copyright Law.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Test methods for fibrous activated carbon

1 Scope

This Japanese Industrial Standard specifies the test methods for fibrous activated carbon.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

- JIS B 7721 *Verification and calibration of the force measuring system of the tension/compression testing machines*
- JIS B 7956 *Continuous analyzers for hydrocarbons in ambient air*
- JIS K 0050 *General rules for chemical analysis*
- JIS K 0114 *General rules for gas chromatographic analysis*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 0804 *Gas detector tube measurement system (Length-of-stain type)*
- JIS K 1107 *Nitrogen*
- JIS K 1150 *Test method for silica gel*
- JIS K 1474 *Test methods for activated carbon*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8625 *Sodium carbonate (Reagent)*
- JIS K 8637 *Sodium thiosulfate pentahydrate (Reagent)*
- JIS K 8659 *Starch, soluble (Reagent)*
- JIS K 8680 *Toluene (Reagent)*
- JIS K 8897 *Methylene blue (Reagent)*
- JIS K 8913 *Potassium iodide (Reagent)*
- JIS K 8920 *Iodine (Reagent)*
- JIS K 8951 *Sulfuric acid (Reagent)*
- JIS K 9007 *Potassium dihydrogenphosphate (Reagent)*
- JIS K 9019 *Disodium hydrogenphosphate 12-water (Reagent)*
- JIS L 1906 *Test methods for non-woven fabrics made of filament yarn*
- JIS L 1913 *Test methods for nonwovens made of staple fibers*
- JIS P 3801 *Filter paper (for chemical analysis)*
- JIS R 1301 *Porcelain crucibles for chemical analysis*

- JIS R 3503 *Glass apparatus for chemical analysis*
JIS Z 0701 *Silicagel desiccants for packaging*
JIS Z 8401 *Guide to the rounding of numbers*
JIS Z 8703 *Standard atmospheric conditions for testing*
JIS Z 8802 *Methods for determination of pH of aqueous solutions*
JIS Z 8830 *Determination of the specific surface area of powders (solids) by gas adsorption method*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions in **JIS K 0211** and the following apply.

3.1 fibrous activated carbon

porous fibrous activated carbon having the higher ability of adsorption to the organic matters and the like in gas or aqueous solution

NOTE : It is also called “activated carbon fibre”.

It is obtained by carbonizing the fibre which is made of rayon, acrylic fibre, phenol resin or pitch and further reacting (activating) it with steam, carbon dioxide and the like at high temperature.

3.2 specific surface area

surface area per unit mass of fibrous activated carbon

3.3 pore volume

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

3.4 sheet

sheet shaped product such as non-woven fabric (felt) and fabric (cloth) composed of 100 % fibrous activated carbon or of fibrous activated carbon compounded with other fibre and the like

3.5 breakthrough curve

a curve obtained from the temporal change of the outlet concentration (C) of the fluid which flows out from a fixed bed when the adsorbable substance is fed to the fixed bed of fibrous activated carbon at a certain height (Z) under the constant condition of inlet concentration (C_0), temperature, flow rate of feeding (V) and the like

It means the process where this outlet concentration gradually rises from the value near to zero at the beginning, that is, the curve plotted based on the variation of the outlet concentration with time.

NOTE : Usually it is expressed by the ratio to the inlet concentration (C/C_0).

3.6 breakthrough

phenomenon in which the concentration of adsorbed substance at the outlet exceeds a certain allowable concentration

3.7 breakthrough time

time point in a breakthrough curve at which, for example, $C/C_0 = 0.1$ (C : outlet concentration, C_0 : inlet concentration) is obtained, and which can be the index of the adsorption rate

3.8 equilibrium adsorption

condition in which the adsorption and desorption are balanced, i.e. state occurring when the fibrous activated carbon, immersed in an aqueous solution in which adsorbed substance is dissolved or placed in the air containing adsorbable substance under a definite condition of temperature, adsorbs the adsorbed substance in the aqueous solution or in the air so that the concentration of the adsorbed substance is reduced gradually, after a certain period or longer until it becomes constant and can not be reduced any more

3.9 adsorption amount

the amount of adsorbable substance in aqueous solution or in air per unit mass of fibrous activated carbon

3.10 equilibrium adsorption amount

the amount of adsorbed substance in aqueous solution or in air which is adsorbed per unit mass by fibrous activated carbon under equilibrium adsorption condition

3.11 residual concentration (equilibrium concentration)

the concentration of substance remained in aqueous solution or in air under equilibrium adsorption condition

3.12 adsorption isotherm

a line which indicates the relation between the residual concentration (also called an equilibrium concentration) and the equilibrium adsorption amount at a definite temperature

4 Sampling method

Take the sample so as to represent a lot and cut the fibre in 10 mm or shorter in length, if necessary.

The size of the lot shall be decided between the parties concerned with delivery.

5 General matters

The general matters common to the test shall be in accordance with **JIS K 0050** and **JIS K 0115**.

6 Test items

The test items shall be as follows.

The test items shall be selected between the parties concerned with delivery according to the purpose of use.

- a) **Specific surface area**
- b) **Pore volume**

- c) **Physical properties of fibre** The physical properties of fibre shall be as follows.
 - 1) Fibre diameter
 - 2) Tensile strength
- d) **Physical properties of sheet** The physical properties of sheet shall be as follows.
 - 1) Mass per unit area
 - 2) Tensile strength and ratio of elongation
- e) **Drying loss**
- f) **pH value**
- g) **Ignition residue**
- h) **Toluene adsorption performance** The toluene adsorption performance shall be as follows.
 - 1) Breakthrough adsorption test
 - 2) Equilibrium adsorption amount
- i) **Methylene blue adsorption performance** The methylene blue adsorption performance shall be as follows.
 - 1) Breakthrough adsorption test
 - 2) Equilibrium adsorption amount
- j) **Iodine adsorption performance**
 - 1) Equilibrium adsorption amount

7 Test method

7.1 Specific surface area

7.1.1 Principle

The measurement of specific surface area of fibrous activated carbon shall be in accordance with BET method.

7.1.2 Apparatus and implement

The apparatus and implement shall be in accordance with **JIS K 1150**.

An example of nitrogen adsorption apparatus is shown in figure 1.

7.1.3 Operation

The operation shall be in accordance with **JIS K 1150** and the following.

- a) **Pretreatment of sample** Dry approximately 0.1 g to 0.2 g of sample in a measuring apparatus for 15 min or longer under the conditions of 120 °C or higher in temperature and 13 Pa or under in pressure.

- b) **Measurement of mass of sample** Remove the electric furnace for heating sample while exhausting the inside of system and returning to room temperature, and then close cock C_0 . Detach the sample tube to wipe off the grease, and weigh the mass. Calculate the difference between this mass and the mass of sample tube to obtain the mass of sample after dried.

The mass shall be weighed in the range of the specified amount ± 0.1 mg.

- c) **Measurement of dead volume** The measurement of dead volume shall be as follows.

- 1) After leaving it to cool down to the room temperature, exhaust it.
- 2) Immerse the sample tube in liquefied nitrogen up to its marked line. While it is being measured, maintain the liquid surface of liquefied nitrogen of this time at a definite level, especially in 4) and d) 3). At this time, immerse the sample tube in liquefied nitrogen to a depth 50 mm or greater as shown in figure 1, and maintain the variation in liquid surface of liquefied nitrogen within 2 mm. Supplement liquefied nitrogen by automatic control to fill a Dewar vessel with liquefied nitrogen and stopper it. In consideration of the variation in saturated vapour pressure of liquefied nitrogen, measure the temperature of liquefied nitrogen with the precision of ± 0.1 °C and carry out the correction of saturated vapour pressure. Use new liquefied nitrogen each time.

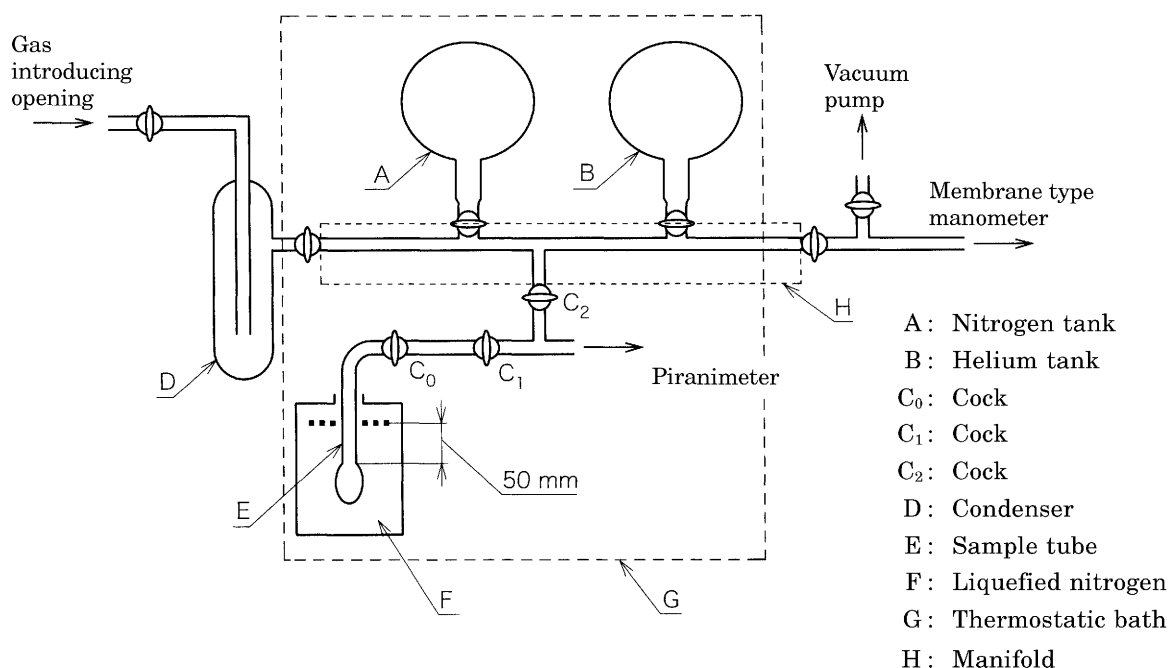


Figure 1 Example of nitrogen adsorption apparatus

- 3) Fill helium at approximately 50 Pa in the manifold, and record the pressure.
- 4) Introduce helium in the sample tube, and after reaching equilibrium, record the pressure.

- 5) Obtain the dead volume of the sample tube immersed in liquefied nitrogen from the pressure before and after introduction of helium and the volume of manifold according to the ideal gas law. Measure it before the measurement of adsorption or after the measurement by using helium at the same temperature as that of the measurement for the nitrogen adsorption amount.

At this time, in consideration of the variation in temperature before and after introducing helium, maintain the temperature of the whole apparatus within ± 0.1 °C or carry out the correction of dead volume by measuring the room temperature before and after introduction.

- d) **Measurement for nitrogen adsorption amount** The measurement for nitrogen adsorption amount shall be as follows.

- 1) Exhaust the inside of the system to 0.14 Pa or lower.
- 2) Fill the manifold with the nitrogen of Class 2 specified in **JIS K 1107**, and after reaching equilibrium, record the pressure.
- 3) Open the cocks of C_0 , C_1 and C_2 and introduce nitrogen in the sample tube. After reaching equilibrium, record the equilibrium pressure.

In such a case, the time required up to the measurement of equilibrium pressure is too short, which often becomes the cause of error. Therefore confirm that the change of pressure is not found within 10 min.

NOTE : Attention on the liquid surface of liquefied nitrogen and room temperature described in c) 5) shall also be paid in the operations of 2) and 3).

- 4) Obtain the adsorption amount from the pressure before and after introducing nitrogen, the volume of manifold and the dead volume.
- 5) Repeat the operations of 2), 3) and 4) until the relative pressure (P/P_0) of nitrogen becomes unity. Herein, P_0 means the saturated vapour pressure (Pa) of adsorbate and P means the equilibrium pressure (Pa).

7.1.4 Calculation

Specific surface area shall be calculated according to the following formula.

- a) **Calculation based on multipoint method** Calculate by using the data of nitrogen adsorption amount obtained in 7.1.3 d) according to formula (1).

$$\frac{P}{V_1(P_0 - P)} = \frac{1}{V_m C} + \frac{C - 1}{V_m C} \times \frac{P}{P_0} \dots\dots\dots (1)$$

where, P_0 : saturated vapour pressure of adsorbate (Pa)
 P : equilibrium pressure (Pa)
 V_1 : whole adsorption amount (cm³/g)
 V_m : adsorption amount of monomolecular layer (ml/g)
 C : BET constant

The measurement under relative pressure 0.10 or lower is recommended. When $\frac{P}{V_1(P_0 - P)}$ is plotted on the ordinate and $\frac{P}{P_0}$ 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 straight line, $\frac{C-1}{V_m C}$ and the intercept on the ordinate, $\frac{1}{V_m C}$.

Herein, the nitrogen adsorption isotherm of fibrous activated carbon indicates the type I classified by **IUPAC** (International Union of Pure and Applied Chemistry). **JIS Z 8830**, puts such a case outside its scope of application because BET plot does not become a straight line with the relative pressure in the range between 0.05 and 0.3. However, in this Standard, BET plot is made to be a straight line because the relative pressure is made 0.1 or lower, then the measurement of specific surface area according to BET method can be applied.

Calculate the specific surface area according to formula (2).

$$S_{BET} = s \times V_m \times \frac{N}{V_0} = 4.35 V_m \dots\dots\dots (2)$$

where, S_{BET} : BET specific surface area (m²/g)

s : area occupied by a nitrogen molecule (0.162 nm²)

N : Avogadro number

V_0 : gas volume under standard condition (22 414 cm³)

V_m : adsorption amount of monolayer (ml/g)

It is recommended that the calculation is processed according to the normal computer program.

- b) **Calculation based on one point method** For the fibrous activated carbon, the measurement under relative pressure 0.1 or under 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 being in the above-mentioned range of relative pressure, $C \gg 1$ is true and therefore the intercept on the ordinate, $\frac{1}{V_m C}$, of BET plot is small and can be simplified as shown in formula (3).

$$V_m = V_1 \left(1 - \frac{P}{P_0} \right) \dots\dots\dots (3)$$

where, P_0 : saturated vapour pressure of adsorbate (Pa)

P : equilibrium pressure (Pa)

V_1 : whole amount of adsorption (cm³/g)

V_m : adsorption amount of monomolecular layer (ml/g)

It is recommended that the calculation is processed according to the normal computer program.

7.2 Pore volume

7.2.1 Principle

The pore volume shall be the adsorption volume when the relative pressure is 0.995 from the nitrogen adsorption isotherm obtained in **7.1**.

7.2.2 Apparatus and operation

The apparatus and operation shall be in accordance with 7.1.2 and 7.1.3.

Since the measurement of nitrogen adsorption isotherm under 1.000 relative pressure is usually difficult, the correction of atmospheric pressure is carried out at the time of measurement, and the pore volume is obtained when the relative pressure as close to the saturated vapour pressure as possible is 0.995.

It is recommended that the calculation is processed according to the normal computer program.

7.3 Physical properties of fibre

7.3.1 Fibre diameter

The measurement of fibre diameter shall be conducted by means of either the laser oscillator or reflecting microscope.

a) **Laser oscillator** The measurement by laser oscillator shall be as follows.

1) **Apparatus** The apparatus shall be as follows.

1.1) **Laser oscillator** Helium-neon oscillator of 633 nm laser wavelength.

1.2) **Goniometer**

1.3) **Screen** capable of screening the diffraction pattern of laser beam.

2) **Operation** The operation shall be as follows.

2.1) After taking a bundle of fibre from the sample and fibrillating it, pull out mono-filament and take it as a test specimen.

2.2) Irradiate the laser beam oscillated from a laser oscillator to the test specimen and read the distance of the position where the diffraction intensity of diffraction pattern observed on the surface of screen at a sufficient distance becomes minimal.

3) **Number of times of test** It shall be decided between the parties concerned with delivery so as to take 30 times as a guide.

4) **Calculation** For the average fibre diameter, calculate the fibre diameter of mono-filament according to formula (4) and formula (5), average the results and then round off the resultant average to one decimal place according to JIS Z 8401.

$$\bar{D} = \frac{\sum D_i}{n} \dots\dots\dots (4)$$

$$D_i = \frac{2t \times \lambda}{L} \times 10^{-3} \dots\dots\dots (5)$$

where, $\bar{D}$: average value (μm)

D_i : fibre diameter of mono-filament (μm)

n : number of times of measurement

t : distance between test specimen and screen (mm)