6 Test methods

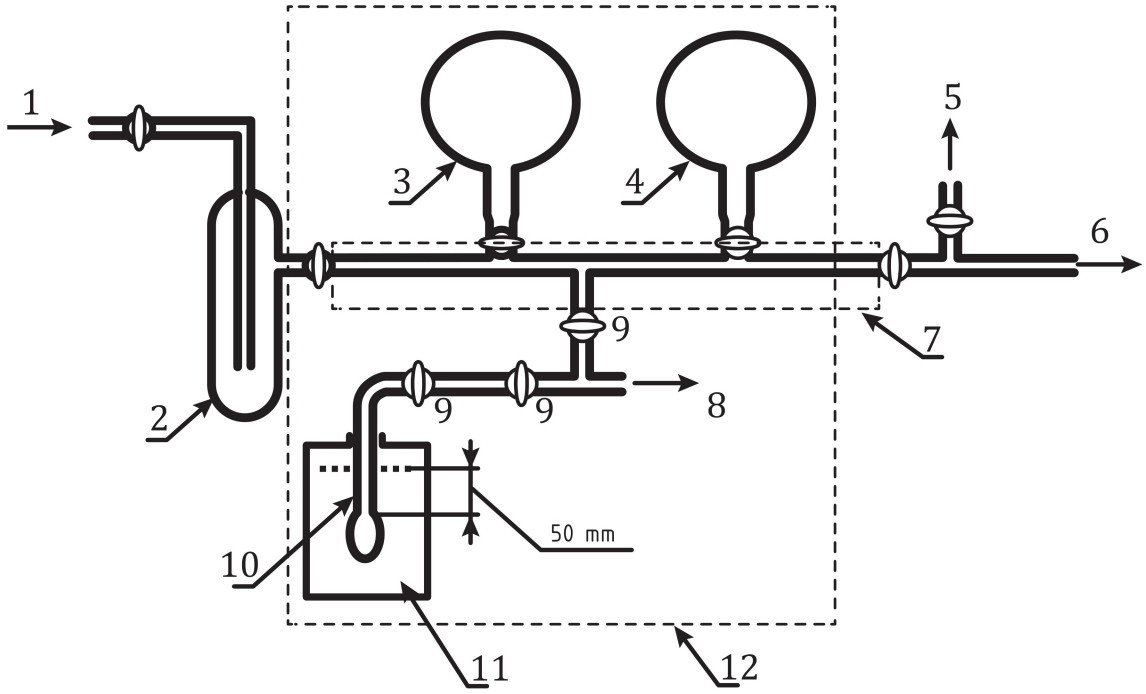
6.1 Specific surface area

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(C_0) stopcock
9(C_1) stopcock
9(C_2) 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 ±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 ±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 ±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 ≤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 ≤0,10 is recommended.

When $p/\left[V_1\left(p_0-p\right)\right]$ 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 ≤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).

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

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 nm^2;

N is Avogadro's number;

V_0 is the gas volume under standard conditions (22 414 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 ≤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;