SLOVENSKI STANDARD
SIST ISO 9277:2000

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Doloļ itev specifiļ ne povrġine trdnih snovi (v zrncih) z adsorpcijo plina s pomoļ jo metode BET

Determination of the specific surface area of solids by gas adsorption using the BET method

Détermination de l'aire massique (surface spécifique) des solides par adsorption de gaz à l'aide de la méthode BET

Ta slovenski standard je istoveten z: **ISO 9277:1995**

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Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9277 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

Annexes A and B of this International Standard are for information only.

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Determination of the specific surface area of solids by gas adsorption using the BET method

1 Scope

Ths International Standard specifies the determination of the total1) specific external and internal surface area of disperse or porous solids by measuring the amount of physically adsorbed gas according to the method of Brunauer, Emmett and Teller (BET method) [1], based on the 1984 IUPAC Recommendations [2].

The BET method is applicable only to adsorption isotherms of type II (disperse, nonporous or macroporous solids) and type IV (mesoporous solids, pore diameter between 2 nm and 50 nm) (see figure 1). Inaccessible pores are not detected. The BET method cannot reliably be applied to type I isotherms or to solids which absorb the measuring gas.

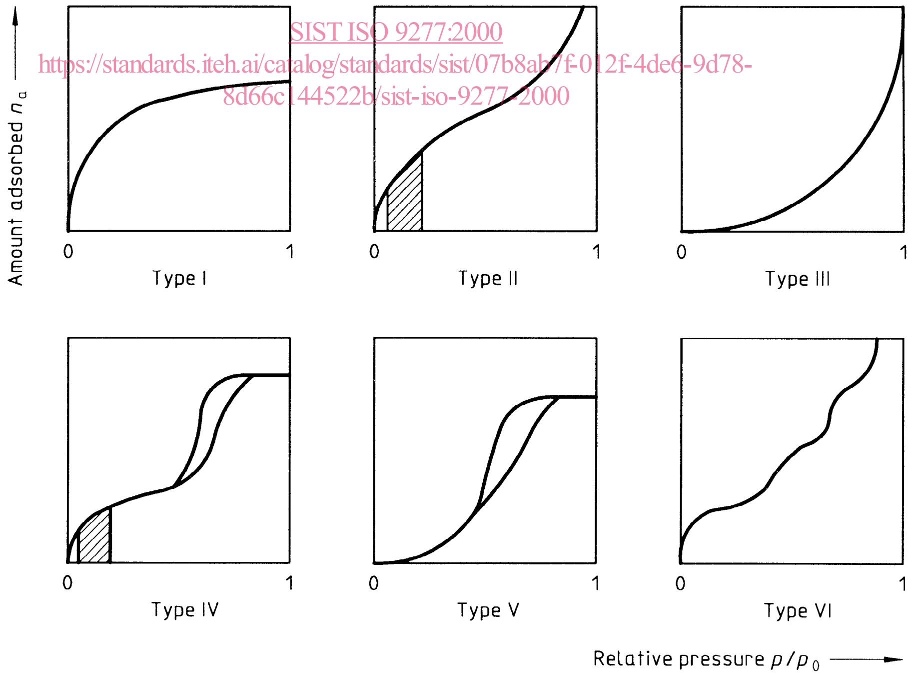

Figure 1 — IUPAC classification of adsorption isotherms (typical BET range is indicated in types II and IV by the hatched area)

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1) For solids exhibiting a chemically heterogeneous surface, e.g. metal-carrying catalysts, the BET method gives the total surface area, whereas the metallic portion of the surface area may be measured by chemisorption methods.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 8213:1986, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps.*

3 Definitions

For the purposes of this International Standard the following definitions apply.

3.1 adsorption: Enrichment of the adsorptive at the external and accessible internal surfaces of a solid.

3.2 physisorption: Weak bonding of the adsorbate, reversible by small changes in pressure or temperature.

3.3 adsorptive: Measuring gas to be adsorbed.

3.4 adsorbent: Solid which adsorbs the measuring gas.

3.5 adsorbate: Enriched adsorptive at the surface of the adsorbent.

3.6 surface area: Area of the external surface of a solid plus the internal surface of its accessible macro- and mesopores.

3.7 adsorption isotherm: Relation between the quantity of adsorbate and the equilibrium pressure of the adsorptive at constant temperature.

4 Symbols and units

Table 1 presents the symbols used in this International Standard, together with their SI units. For comparison purposes, the IUPAC [2] symbols are also given. All specific dimensions are related to sample mass, in grams.

Table 1 — Symbols

ISO symbol	Parameter	SI unit	IUPAC symbol
a_m	molecular cross-sectional area occupied by an adsorbed molecule in a complete monolayer	nm^2	a_m
a_s	specific surface area	$m^2\ g^{-1}$	a_s
C	BET parameter	1 1)	C
L	Avogadro constant (= 6,022 × 10^{23})	mol^{-1}	L
m	mass of the solid sample	g	m
m_a	specific mass adsorbed	1 1)	m^a
n_a	specific amount adsorbed	$mol \cdot g^{-1}$	n^a
n_m	specific monolayer capacity of adsorbate; amount of adsorbate needed to cover the surface with a complete monolayer of molecules	$mol \cdot g^{-1}$	n_m, n_m^a
$n_{m,mp}$	specific monolayer capacity derived from multipoint measurement	$mol \cdot g^{-1}$	—
$n_{m,sp}$	specific monolayer capacity derived from single-point measurement	$mol \cdot g^{-1}$	—
p	pressure of the adsorptive in equilibrium with the adsorbate	Pa	p
p_0	saturation vapour pressure of the adsorptive	Pa	p^0
p/p_0	relative pressure of the adsorptive	1 1)	p/p^0
R	molar gas constant (= 8,314)	$J \cdot mol^{-1} \cdot K^{-1}$	R
t	time	min	t

ISO symbol	Parameter	SI unit	IUPAC symbol
T	temperature	K	T
V_a	specific volume adsorbed	$mm^3 \cdot g^{-1}$	V^a

1) According to ISO 31-0, the coherent SI unit for any quantity of dimension one (at present commonly termed "dimensionless") is the unit one, symbol 1.

5 Principle

The method specified involves the determination of the amount of adsorbate or adsorptive gas required to cover the external and the accessible internal pore surfaces of a solid (see figure 2) with a complete monolayer of adsorbate. This monolayer capacity can be calculated from the adsorption isotherm using the BET equation (1) (see 6.1). Any gas may be used, provided it is physically adsorbed by weak bonds at the surface of the solid (van der Waals forces), and can be desorbed by a decrease in pressure at the same temperature.

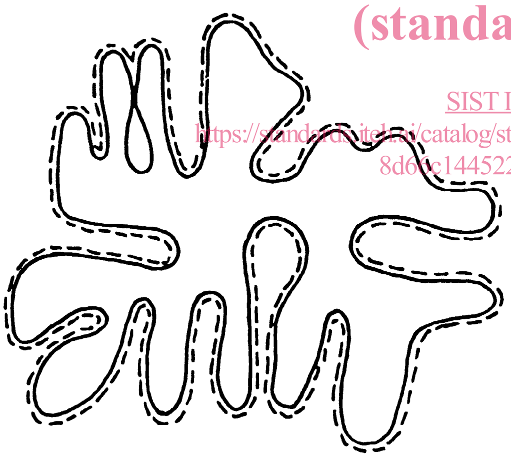

Figure 2 — Particle with surface detected by the adsorption method shown by dotted line

Nitrogen at its boiling point (about 77 K) is usually the most suitable adsorptive. If the sensitivity of the instrument when using nitrogen is insufficient for low surface areas, adsorptives of heavier molecules or of vapour pressure lower than nitrogen, e.g. krypton, may be used. The results of measurements with different adsorptives may deviate from each other because of different molecular areas, different accessibilities to pores and different measuring temperatures.

The adsorptive gas is admitted to the sample container which is held at a constant temperature. The amounts adsorbed are measured in equilibrium with the adsorptive gas pressure p and plotted against relative pressure, p/p_0, to give an adsorption isotherm. Adsorption isotherms may be obtained by volumetric, gravimetric, calorimetric or spectroscopic measurement or by the carrier gas method using continuous or discontinuous operation.

6 Procedure

6.1 Sample preparation

Sampling shall be carried out in accordance with ISO 8213. Prior to the determination of an adsorption isotherm, remove physically adsorbed material from the sample surface by degassing, while avoiding irreversible changes to the surface. Ascertain the maximum temperature at which the sample is not affected by thermogravimetric analysis (see figure 3) or by trial experiments using different degassing conditions of time and temperature. When vacuum conditions are used, degassing to a residual pressure of approximately 1 Pa or better is usually sufficient. Degassing of the sample can also be performed at elevated temperature by flushing with helium or with the adsorptive. Degassing is complete when a steady value of the residual gas pressure p, of its composition or of the sample mass m is reached.

Using the vacuum technique, isolate the heated sample container from the pump and trap (at time t_a in figure 4). If the pressure does not rise significantly over a period of 15 min to 30 min, degassing is complete. This procedure also establishes the absence of leaks. The specific surface area should be related to the mass of the degassed sample.

After degassing, the sample container is cooled to the measuring temperature. It should be noted that, at low gas pressures, the temperature of the sample needs some time to equilibrate due to the reduced thermal conduction of the cooling bath.