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

气体吸附 BET 法测定固态物质比表面积

(English Translation)

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1 Scope

This Standard specifies the determination of the overall (see Note) specific external and internal surface area of disperse (e.g., nano-powders) or porous solids by measuring the amount of physically adsorbed gas according to the Brunauer, Emmett and Teller (BET) method

[1]

Note: For solids exhibiting a chemically heterogeneous surface, e.g., metal-carrying catalysts, the BET method gives the overall surface area, whereas the metallic portion of the surface area can be measured by chemisorption methods.

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 ~ 50 nm). Inaccessible pores are not detected. The BET method cannot reliably be applied to solids which absorb the measuring gas.

A strategy for specific surface area determination of microporous materials (type I isotherms) is described in Annex C.

2 Normative references

The following referenced documents are indispensable for the application 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 8213, Chemical products for industrial use - Sampling techniques - Solid chemical products in the form of particles varying from powders to coarse lumps

ISO 14488, Particulate materials - Sampling and sample splitting for the determination of particulate properties

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

adsorption

Adsorption isotherms may be obtained by volumetric, gravimetric, calorimetric or spectroscopic measurement or by the carrier gas method using continuous or discontinuous operation (see 6.3).

6.1 Sample preparation

Sampling shall be carried out in accordance with ISO 8213 and ISO 14488. 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), by spectroscopic methods, 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 an inert gas (e.g., helium). Degassing is complete when a steady value of the residual gas pressure p , of its composition or of the sample mass is reached.

Using the vacuum technique, isolate the heated sample container from the pump and trap (at time t_i in Figure 4). If the pressure is nearly constant over a period of 15 min ~

30 min, degassing is complete. Almost invariant pressure also confirms 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 conductivity within the sample cell.

For sensitive samples, a pressure-controlled heating (see Figure 5) is recommended.

This procedure consists in varying the heating rate in relationship to the gas pressure evolved from a porous material during the degassing under vacuum conditions. When a fixed pressure limit, p_L (usually around 7 Pa ~ 10 Pa), is surpassed due to the desorbed material from the sample surface, the temperature increase is stopped and the temperature is kept constant until the pressure falls below the limit. At that point, the system continues the temperature ramp. This procedure is particularly suitable for avoiding structural changes in microporous materials, when fast heating rates can damage fragile structures due to a vigorous vapour release. In addition, the method is very safe in preventing sample elutriation when water or other vapours are released from the pores in very fine powder materials.