



Determination of the specific surface of powdered solids by BET

Basic principle

The determination of the specific surface of powdered substances is based on a low-temperature gas adsorption process introduced by Brunauer, Emmett and Teller (BET).

A defined amount of the sample is weighed into a tubular glass cuvette. Using a closed apparatus, the sample is then continuously gassed with a mixture of 30 % nitrogen and 70 % helium. After that, the sample is immersed into a dewar jar containing liquid nitrogen and cooled down to $-196\text{ }^{\circ}\text{C}$. This process is accompanied by the formation of a monomolecular nitrogen layer on the surface of the analyte. Once the equilibrium state is reached, complete desorption is induced by heating the sample. The amount of nitrogen set free is quantitatively determined with the aid of a thermal-conductivity detector and converted into a surface on the basis of the BET equation. Under the conditions applied by Chemetall, 1 cm^3 desorbed nitrogen equals a surface of 2.84 m^2 .

Procedure

The measurement is preceded by a calibration of the apparatus.

Sample preparation

The powdered sample is inserted into a U-shaped tubular cuvette of known weight ($\pm 0.1\text{ mg}$) until its horizontal part is completely filled. The cuvette is then fixed to the outgas position of the BET apparatus and gassed for 15 minutes with a mixture of 30 % nitrogen and 70 % helium (probe gas). The sample is heated for one hour at $150\text{ }^{\circ}\text{C}$ under probe gas flow and then cooled down to room temperature.

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Measurement

The cold cuvette is transferred to the sample position of the apparatus. Ambient air penetrated into the cuvette during transfer leads to a short increase in counts of the detector signal, the measurement is started as soon as the counts are back at a constant level close to zero and a fine adjustment of the apparatus to the zero value has taken place. A dewar flask filled with liquid nitrogen is used to cool the cuvette. The immersion of the sample into the liquid nitrogen triggers the automatic start of the measurement. Due to the adsorption of nitrogen on the particle surface, the flowmeter shows a decreasing flow rate of the probe gas. Once the detector signal display reaches 0, the dewar flask is automatically driven down and a heater is switched on. In the course of the desorption process which then takes place, previously bound nitrogen is set free leading to an increase in the detector signal to a maximum value and a subsequent asymptotic decrease towards zero. The measurement is stopped as the signal is zero. The resulting data output of the apparatus corresponds to the absolute surface of the weighed amount of sample in m².

Calculation

The calculation is based on the following equation:

$$\frac{a}{b-c} = \text{specific surface} \quad (\text{m}^2/\text{g})$$

with:

- a: absolute surface acc. to measurement (m²)
- b: weight of the filled tubular cuvette (g)
- c: weight of the empty tubular cuvette (g)

The precision of the BET method for analytical measurements is ± 0.05 m² absolute.

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Chemetall GmbH

Special Metals Division

Trakehner Straße 3
D-60487 Frankfurt am Main
P.O. Box 90 01 70
D-60441 Frankfurt a.M.
Phone +49 69 7165-3911
Fax +49 69 7165-2523
Mail specialmetals@chemetall.com

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