



Specific surface area by Brunauer-Emmett-Teller (BET) theory

Date

30.05.2016

Version

1.0 English

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1 Scope

This Standard Operating Procedure (SOP) describes the experimental procedure and settings of the specific surface area analysis by Brunauer-Emmett-Teller (BET) theory.

2 Basics

Within the increased use of nanomaterials (NM) in the last years also the efforts to detect the specific surface area of NM increased. The specific surface area determines the contact area of the NM with other surfaces / substances in the environment. NM show a larger specific surface area compared to their microscale pendants, with the consequence that it has been suggested that NM are more “reactive”. Thus, it is not surprising that the specific surface area is suggested to be a relevant dose metric e. g. in nanotoxicology. The surface area has been defined by ISO (2012) as “the quantity of accessible surface of a sample when exposed to either gaseous or liquid adsorbate phase.” The specific surface area is defined as the surface area of a material divided by its mass or its volume. In this SOP the specific surface area per mass is used (m²/g) and its determination based on the BET theory is described. Briefly, this theory/method is based on the physical multi layer adsorption of usually non-corrosive gases (like nitrogen, argon, carbon dioxide, etc.) on solid materials (e.g. nano-material powder) to determine the specific surface area data. The theory is the most common and standardized (ISO 9277:2010, DIN ISO 9277:2013) analysis technique for the measurement of the specific surface area published by Brunauer, Emmett and Teller (1938).

2.1 Background: BET theory

For detailed information on the theory refer to the literature (Brunauer et al. 1938, ISO 9277:2010, DIN ISO 9277:2013).

3 Materials & Instruments

3.1 Materials

The following materials and chemicals are required:

- (Nano-)material as powder
- Glas holder
- Microbalance
- Spatula
- Cleaning wipes
- Adsorption gas (e. g. nitrogen)

3.2 Instruments

The following instruments are required:

- BET surface area analyzer

Note: For the analysis a NOVA-2200 BET Surface Area Analyzer Quantachrome was used to record the UV/VIS spectra. The usage and maintenance of the instruments will be not described in detail in this SOP. Please refer to the manual.

4 Experimental procedure

The device has to be switched on / warm up for at least 30 min before starting an experiment. For an analysis the NM is filled in an instrument specific glass holder and weighted at least three times on a Microbalance. Afterwards the sample is placed in the instrument being evacuated, heated up for specific time and temperature. Afterwards the sample is cooled down and weighted again to determine possible mass losses. Now the sample/holder is placed in the BET measurement unit and the BET analysis starts by cooling down the sample to ~ 77 K, followed by nitrogen injection under various pressures to determine the N₂ displacement for specific surface area calculation.

4.1 Preparation and measurement

- Estimation/calculation of specific surface area of the NM
- Choose of adequate (regarding the estimated surface area and volume of the NM) glass holders for measurement
- Filling of sample into glass holder
- Weighing of sample material and estimation of total surface area inside the glass holder (min. of 5 m² is required)
- Placing the glass holders into BET heating unit
- Evacuating of holder using standard rotating pump unit
- Heating of sample for min. 18 h at 250°C (temperature can be varied/adapted to the NM)
- Cooling down of the sample to room temperature
- Weighing of sample after heat treatment (determination of sample mass)
- Transfer into BET measurement unit
- Programming of BET device
- Start of measurement program
 1. Cooling to ~77 K
 2. Injection of N₂
 3. Pressure variation (6 point measurement)
 4. Read out of results

4.2 Quantification

- Quantification is carried out as given by the instrument software as specific surface area (m²/g).

Note: Please refer to the instrument manual.

5 Safety precautions

For all working steps protective clothing, mask suitable for handling NM, safety goggles and (heat resistant) gloves have to be worn.

6 Waste disposal

NM have to be collected and disposed off separately.

7 Literature

ISO, ISO/TR 13014:2012: Nanotechnologies -- Guidance on physico-chemical characterization of engineered nanoscale materials for toxicologic assessment, 2012.

ISO 9277:2010 Determination of the specific surface area of solids by gas adsorption -- BET method

DIN ISO 9277:2013 Bestimmung der spezifischen Oberfläche von Feststoffen durch Gasadsorption nach dem BET -Verfahren

Brunauer, Stephen; Emmett, P. H.; Teller, Edward (1938). "Adsorption of Gases in Multimolecular Layers". *Journal of the American Chemical Society* 60 (2): 309–319. doi:10.1021/ja01269a023. ISSN 0002-7863.

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This SOP is valid from: 30.05.2016

This SOP replaces the SOP from: Primary version

Responsible for the implementation of this SOP: Measurement personal

Previous changes: Primary version (1.0)

Distribution: Management
Head of department
Measurement personal
Quality management

Signature, Date

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