

Certified Reference Material

BAM-P109

BET Specific Surface Area of Activated Nanoporous Carbon

Certified Value

BET Specific Surface Area A_{BET} in m ² /g	Uncertainty U in m ² /g
1396	24

This material was certified on the basis of the Guidelines for the Production of BAM Reference Materials [1]. The measurements and data evaluation were carried out according to ISO 9277 [2].

The certified value is expressed as the mean of the instrument averages of 25 instruments which participated in the interlaboratory comparison measurements. The uncertainty was calculated as $U = k \cdot u_c$ where u_c is the combined standard uncertainty for the average and $k=2$ is the coverage factor. The value of u_c includes both a combined estimate of the variation of the averages and the variation due to material inhomogeneity according to ISO Guide 35 [3].

Material Description

A unit of BAM-P109 consists of a single bottle containing approximately 10 g of an activated nanoporous carbon material (micro beads, diameters about 250 ... 500 μm).

Intended Use

The reference material is intended for the calibration and checking of instruments used for the determination of the BET specific surface area by the static volumetric method.

Transport and Storage

The material should be stored in the closed bottle at ambient temperature (20 °C to 25 °C) in a dry atmosphere.

Metrological Traceability

The certified value was determined in accordance with the Brunauer, Emmett, and Teller (BET) model. Under the conditions of this model as part of the traceability statement, the certified value is traceable to the base units of the SI via calibrated measurements of the quantities pressure, volume, and mass.

Expiration of the Certificate

This certificate is valid for two years from the date of shipment provided the reference material is stored under the recommended conditions.

Date of Shipment from BAM: _____

Handling

Outgassing of the sample has to be carried out in a vacuum. Starting at room temperature, the sample is to be heated up to 200 °C (473 K) in vacuum within 1 hour followed by degassing the sample at 200 °C for 3 hours. The final pressure should be < 0.1 Pa.

The N₂ isotherm at 77.3 K has to be measured at least in the range of a relative pressure from 0.01 to 0.1. Choose sample size and perform analysis following the instrument manufacturer's instructions.

Multipoint BET Evaluation

The monolayer capacity per unit mass of adsorbed nitrogen $n_{a,m}$ needs to be calculated from the isotherm data in accordance with the international standard ISO 9277 [2] as follows. Plot the BET function $y_{BET} = (p/p_0)/[n_a(1-p/p_0)]$ against relative pressure p/p_0 , where n_a is the amount of adsorbate per unit mass at the equilibrium pressure p and p_0 is the saturated vapour pressure of the adsorbate at the temperature of adsorption. Determine the best fit straight line by linear regression using at least 5 adsorption isotherm data points in the relative pressure range $0.012 \leq p/p_0 < 0.055$ (BET evaluation range of the reference material BAM-P109). From the gradient k_{BET} and the intercept on the y-axis, i_{BET} , calculate the monolayer capacity per unit mass of the adsorbent using $n_{a,m} = 1/(k_{BET} + i_{BET})$.

The BET specific surface area, A_{BET} , is then given by

$$A_{BET} = n_{a,m} \cdot N_A \cdot \sigma_{N_2}$$

where N_A is the Avogadro constant and σ_{N_2} the cross sectional area for the N₂ molecule in the monolayer.

Use the value $\sigma_{N_2} = 0.162 \text{ nm}^2$ [2], [4] for the calculation.

If your instrument records the adsorption isotherm in the form of the adsorbed specific gas volume V_a at standard temperature and pressure (STP) as a function of relative pressure, V_a must be converted into n_a using

$$n_a = \frac{p_{STD}}{R \cdot T_{STD}} \cdot V_a$$

with $p_{STD} = 101325 \text{ Pa}$, $T_{STD} = 273.15 \text{ K}$, and $R = 8.314 \text{ Pa m}^3 \text{ K}^{-1} \text{ mol}^{-1}$ (molar gas constant).

Participating Laboratories

Adsor-Tech GmbH, Premnitz (Germany)

Aqura GmbH, Hanau (Germany)

BAM, Fachgruppe I.3, Berlin (Germany)

BAM, Fachgruppe V.4, Berlin (Germany)

Bayer Technology Services GmbH, Leverkusen (Germany)

Bayerisches Zentrum für Angewandte Energieforschung e. V., Würzburg (Germany)

BEL Japan Inc., Osaka (Japan)

Evonik Degussa GmbH, Inorganic Materials, Köln (Germany)

Delft Solids Solutions, Delft (The Netherlands)

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin (Germany)

Hermsdorfer Institut für technische Keramik e. V. (HITK), Hermsdorf (Germany)

Micromeritics Instrument Corp., Norcross, GA (USA)

Micromeritics GmbH, Aachen (Germany)

POROTEC GmbH, Hofheim (Germany)

Quantachrome GmbH, Odelzhausen (Germany)

Quantachrome Instruments, Boynton Beach (USA)

Rubotherm - Präzisionsmesstechnik GmbH, Bochum (Germany)

Sachtleben Chemie GmbH, Duisburg (Germany)

ThermoFisher Scientific, Milan (Italy)

University of Alicante, Alicante (Spain)

University of Amsterdam, Amsterdam (The Netherlands)

ZetA Partikelanalytik GmbH, Mainz (Germany)

Three of the laboratories participated in the interlaboratory study with two BET instruments each.

Accepted Instrument Mean Values

Instrument	Number of measurements	Instrument mean of BET surface area in m ² /g	Standard deviation in m ² /g
01	5	1394	4
02	5	1394	11
03	5	1369	5
04	5	1380	4
05	5	1438	39
06	5	1419	18
08	5	1402	18
09	5	1401	45
10	5	1417	38
11	5	1395	14
12	5	1373	17
13	5	1354	9
14	5	1380	24
15	5	1403	14
16	5	1377	6
17	5	1381	6
18	5	1400	10
19	5	1445	27
20	5	1397	20
22	5	1406	9
23	5	1401	14
24	5	1403	5
25	5	1376	14

The results of two instruments were insufficient and could not be included in the evaluation.

Certification Report

An accompanying report in German [5] presenting the certification procedure and the statistical evaluation is available from BAM on request.

Literature

- [1] Guidelines for the Production of BAM Reference Materials.
BAM Bundesanstalt für Materialforschung und -prüfung, Berlin 2006
Web-Link: www.bam.de/pdf/service/referenzmaterialien/bam_rm_guidelines.pdf
- [2] ISO 9277
Determination of the specific surface area of solids by gas adsorption using the BET method.
ISO, Geneva, First Edition 1995 & FDIS of Second Edition 2010
- [3] ISO Guide 35
Certification of reference materials - General and statistical principles.
ISO, Geneva, 2006
- [4] Sing, K. S. W., Everett, D. H., Haul, R. A. W., Moscou, L., Pierotti, R. A., Rouquerol, J. and Siemieniowska, T.
Reporting physical adsorption data for gas/solid systems with special reference to the determination of surface area and porosity. (IUPAC Recommendations 1984)
Pure Appl. Chem., 57 (1985) 603 - 619
- [5] Klobes, P., Bremser, W., Zimathies, A. and Prinz, C.
Bericht zur Zertifizierung des Referenzmaterials BAM-P109
BAM Bundesanstalt für Materialforschung und -prüfung, Berlin 2010

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