Certified Reference Material

BAM-P108

BET Specific Surface Area of Activated Nanoporous Carbon

ausverkauft / out of stock

Certified Value

BET Specific Surface Area A_{BET} in m^2/g	**Uncertainty** U in m^2/g
550	**5**

This material was certified on the basis of the Guide for the Development 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].

Scope

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. A unit of BAM-P108 consists of a single bottle containing approximately 10 g of an activated nanoporous carbon material.

The certified value is only valid for static volumetric BET type instruments using nitrogen at 77.3 K. This CRM is not certified for adsorption/desorption isotherm form, pore volume, or pore size distribution by any other method.

Sample pretreatment

Outgassing of the sample has to be carried out in a vacuum. Starting at room temperature, the sample is to be heated up to 423 K (150 °C) in vacuum within 1 hour followed by degassing the sample at 150 °C for 1 hour. The final pressure should be < 0.1 Pa.

Measurement

The N_2 isotherm has to be measured at 77.3 K in the range of a relative pressure from 0.001 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 vapor pressure of the adsorptive 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 $0.001 \leq p / p_0 < 0.05$ (BET evaluation range of the reference material). 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 N_2 molecule in the monolayer. Use the value σ_{N_2} = 0.162 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 Pa, T_{STD} = 273.15 K, and R = 8.314 Pa m^3 K^{-1} mol^{-1} (molar gas constant).

Participating laboratories in the interlaboratory certification study

Adsor-Tech GmbH, Premnitz (Germany)
Aqura GmbH, Hanau (Germany)
BAM, Fachgruppe I.3, Berlin (Germany)
BAM, Fachgruppe V.4, Berlin (Germany)
BAM, Fachgruppe VII.1, Berlin (Germany)
Bayer Technology Services GmbH, Leverkusen (Germany)
Bayerisches Zentrum für Angewandte Energieforschung e. V., Würzburg (Germany)
BEL Japan Inc., Osaka (Japan)
Delft University of Technology (The Netherlands)
Hermsdorfer Institut für technische Keramik e. V. (HITK), Hermdorf (Germany)
Micromeritcs Instrument Corp., Norcross, GA (USA)
Micromeritics GmbH, Mönchengladbach (Germany)
NIST, Gaithersburg (USA)
POROTEC GmbH, Hofheim (Germany)
Quantachrome GmbH, Odelzhausen (Germany)
Quantachrome Instruments, Boynton Beach (USA)
Rubotherm - Präzisionsmesstechnik GmbH (Germany)
ThermoFisher Scientific, Milan (Italy)
University of Alicante (Spain)
University of Amsterdam (The Netherlands)
University of Kassel (Germany)
University of Provence, Marseille (France)
University of Siegen (Germany)
ZetA Partikelanalytik GmbH, Mainz (Germany)

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

Table 1 Results of the interlaboratory certification study

Instrument	Number of measurements	Instrument mean of BET surface area in m^2/g	Standard deviation in m^2/g
02	4	557.4172	3.1184
03	5	551.0915	2.9947
04	9	549.9794	7.1476
05	5	554.4014	0.7778
06	3	567.7787	13.1309
07	5	549.9947	5.0558
08	13	553.9951	3.1062
09	5	543.1369	8.3501
10	5	547.3748	2.1303
11	4	553.1877	7.5083
12	5	538.5192	0.3881
13	5	541.9908	2.4201
14	5	551.9457	2.3330
15	5	550.3417	2.1941
16	5	536.1633	1.2926
17	5	558.5714	4.5175
18	5	554.7706	5.6951
19	5	538.6001	1.8377
20	5	549.4533	1.4882
21	5	539.1813	5.9777
22	8	546.5963	3.9984
24	5	553.4188	5.5055
25	5	561.9434	6.0873
26	5	553.6956	2.6433
27	5	553.3998	5.9531

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

Storage of the material

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

Expiration of the certification

This certificate is valid for ten years after date of certification.

Certification report

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

References

[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/CD 9277
Determination of the specific surface area of solids by gas adsorption using the BET method.
ISO, Geneva 2007

[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 Siemieniewska, 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-P108
BAM Bundesanstalt für Materialforschung und -prüfung, Berlin 2007

BAM Bundesanstalt für Materialforschung und -prüfung

Berlin, Germany, December 10, 2007

Prof Dr U. Panne
Head of BAM Department I
Analytical Chemistry;
Reference Materials

Dr A. Thünemann
Head of BAM Division I.3
Structure Analysis;
Polymer Analysis

This reference material is provided by:

BAM Bundesanstalt für Materialforschung und -prüfung,
Richard-Willstätter-Straße 11, D-12489 Berlin, Germany

Phone: +49 30 8104 2061
Fax: +49 30 8104 1117

E-Mail: sales.crm@bam.de
Internet: www.webshop.bam.de