

Certified Reference Material

BAM-P110

BET Specific Surface Area of Titanium Dioxide (Anatase)
calculated from the nitrogen adsorption isotherm at 77.3 K

Certified Value

Specific Surface Area ^a A_{BET} in m ² /g	Uncertainty ^b U in m ² /g
107.8	1.6

^a Specific surface area calculated in a relative adsorption pressure range $0.05 \leq p/p_0 < 0.3$ as multi point BET model with minimum of five points described in ISO 9277

^b Uncertainty $U = k \cdot u_c$ calculated according to ISO Guide 35 and ISO/IEC Guide 98-3 with the coverage factor $k = 2$ (giving a level of confidence of approximately 95 %). The value of the combined standard uncertainty u_c of the certified property includes both an uncertainty contribution resulting from the inter-laboratory characterization, the study of inhomogeneities, stability of the material and the uncertainty contribution due to the measurement result variations of the single instruments (mean data set precision).

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:

Material description

A unit of the CRM BAM-P110 consists of a single glass bottle containing approximately 10 g of crystalline, pure anatase (99.5 %) titanium dioxide powder with a mean diameter of about 10 to 25 nm of pore width.

Recommended use

The reference material is intended for performance testing of instruments used for the determination of multi point BET specific surface area from the adsorption in a relative pressure range $0.05 \leq p/p_0 < 0.3$ of the nitrogen isotherm determined by the static volumetric method.

Handling

Prior to the measurement, outgassing of the sample is necessary. Outgassing has to be carried out in a vacuum. Heat the sample for degassing in a vacuum with a rate of about 5 K/min to 180 °C, then hold temperature at 180 °C for at least 3 hours. Afterwards, allow the sample to cool slowly back to ambient temperature. The final pressure should be < 0.1 Pa. The adsorption and the desorption branch of the N₂ isotherm has to be measured at 77.3 K. Perform the analysis following the instrument manufacturer's instructions. The recommended minimum sample intake is 0.8 g.

Determine the BET specific surface area in accordance with the international standard ISO 9277 using at least 5 adsorption isotherm data points in the relative pressure range $0.05 \leq p/p_0 < 0.3$ (BET evaluation range of the reference material). Use the value 0.162 nm² for the cross sectional area of nitrogen.

Transport and storage

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

Metrological traceability

The certified value is a method-defined (model dependent) parameter. The value is determined on the basis of the BET model described in ISO 9277. Under the condition that the model is applied as an integral 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.

Participating laboratories

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Bayerisches Zentrum für Angewandte Energieforschung e.V., Würzburg (Germany)
BEL Japan Inc., Osaka (Japan)
Centro Ricerche Fiat, Torino (Italy)
Delft Solids Solutions, Delft (The Netherlands)
ECSIN-Veneto Nanotech, Padova (Italy)
Fraunhofer-Institut für Keramische Technologien und Systeme, Dresden (Germany)
Instituto Nacional de Metrologia Brasil, Duque de Caxias (Brasil)
Micromeritics Instrument Corp., Norcross, GA (USA)
Micromeritics GmbH, Mönchengladbach (Germany)
NanoLogica AB, Södertälje (Sweden)
Oerlikon Metco WOKA GmbH, Barchfeld-Immelborn (Germany)
POROTEC GmbH, Hofheim (Germany)
PTT Global Chemical Public Company Limited, Rayong (Thailand)
Quantachrome GmbH, Odelzhausen (Germany)
Quantachrome Instruments, Boynton Beach (USA)
ThermoFisher Scientific, Milan (Italy)
University of Alicante, Alicante (Spain)
UNIZAR, Zaragoza (Spain)

Data set means of the participants in the inter-laboratory certification study

Data set no.	A_{BET} (m^2/g)
01	107.77
02	109.45
03	103.57
04	109.19
05	110.34
06	109.26
07	110.73
08	105.39
09	104.53
10	106.85
11	109.98
12	109.16
13	104.53
14	108.23
15	107.70
16	105.41
17	108.17
18	107.88
19	106.72
20	110.62
21	96.98 ^a
22	108.13
23	108.81
24	106.91
25	106.99 ^b

^a data set mean for the particular property detected as statistical outlier

^b one outlier of 6 replicate measurements detected

References

F. Emmerling, W. Unger, S. Rades, W. Bremser, A. Zimathies, C. Prinz
Certification Report of BAM-P110
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin 2016

Guidelines for the Development and Production of BAM Reference Materials.
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin 2016

ISO Guide 35

Reference materials - General and statistical principles for certifications.
International Organization for Standardization, Geneva (2006)

ISO/IEC Guide 98-3: Uncertainty of Measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM: 1995).

International Organization for Standardization, Geneva (2008)

ISO 9277

Determination of the specific surface area of solids by gas adsorption – BET method.
International Organization for Standardization, Geneva (2010)

Brunauer, S., Emmet, P. H., Teller, E.
Adsorption of Gases in Multimolecular Layers.
J. Amer. Chem. Soc., 60 (1938) 309 – 319

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