Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR THE GAS ADSORPTION

Certificate

BAM-PM-101
Material: SiO_2

with specific surface area (BET) of

0.177 ± 0.004 $m^2 g^{-1}$

Mean of means[1)]	**0.177**	**$m^2 g^{-1}$**
Uncertainty		
Standard deviation of the mean of means	**0.004**	**$m^2 g^{-1}$**
95% confidence interval	**0.008**	**$m^2 g^{-1}$**
Standard deviation of means	**0.014**	**$m^2 g^{-1}$**

according to interlaboratory study carried out in accordance with the "Guidelines for the Production and Certification of BCR Reference Materials" (1)

Method	Gas adsorption at 77 K
Adsorptive	Krypton
Evaluation	BET method according to DIN 66131 (2)

1. Scope

The reference material is intended for the calibration and checking of instruments, especially for determining very small surface areas.
The parameters mentioned are material-specific quantities to characterize non-porous and macroporous solids by means of the gas adsorption method.

2. Measurement and evaluation

2.1 Pretreatment of the sample

Heating the specimen for one hour at 623 K at 0.1 Pascal
Keeping this temperature for 3 hours at a specified vacuum, cooling slowly

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[1)] The results were rounded off according to DIN 1333. Outliers determined by the Grubbs test (95 % significance level) were not included in the calculation of the mean value.

2.2 Measurement

The quantity of krypton adsorbed was measured by the static volumetric method.
BET range: p/p_0 from 0.05 to 0.3

2.3 Assumptions

- BET theory (3)
- molecular cross-sectional area of krypton: $a_{krypton}$ = 0.21 nm² (4)

2.4 Evaluation

The specific surface area in $m^2 g^{-1}$ was determined in accordance with DIN 66131 using the following equation:

$$S_{BET} = n_m \cdot a_{krypton} \cdot N_A$$

The monolayer capacity n_m was calculated by linear regression analysis from the slope and the intercept on the y-axis, $n_m = 1/(a+b)$, a = slope, b = intercept (BET-equation).
N_A is the Avogadro's constant.

Participants in the interlaboratory study:

BASF Aktiengesellschaft, Ammoniaklaboratorium, Ludwigshafen
Bundesanstalt für Materialforschung und -prüfung (BAM), Laboratorium Biowerkstoffe, Berlin
Bundesanstalt für Materialforschung und -prüfung (BAM), Laboratorium Sekundäreigenschaften von Referenzmaterialien, Berlin
Degussa AG, Hanau
FISONS Instruments S.p.A., Milano, Italy
Fraunhofer-Institut für Keramische Technologien und Sinterwerkstoffe, Dresden
Friedrich-Schiller-Universität Jena, Institut für Physikalische Chemie, Jena
GSF-Forschungszentrum für Umwelt und Gesundheit GmbH, Institut für Hydrologie, Oberschleißheim
Institut für Technologie und Umweltschutz e.V., Berlin
Leuna-Katalysatoren GmbH, Forschung/Texturlabor, Leuna
Micromeritics GmbH, Neuss
Universität des Saarlandes, Lehrstuhl für Werkstofftechnologie, Saarbrücken
Universität Erlangen-Nürnberg, Lehrstuhl für Technische Chemie, Erlangen
Universität Leipzig, Institut für Technische Chemie, Leipzig

Table 1

Evaluation of the interlaboratory study for determining the specific surface area of silica using the BET method.
Parameter to be certified: *BET specific surface area in $m^2 g^{-1}$*
Participating laboratories: 14
Method: gas adsorption at 77 K, adsorptive krypton

Laboratory	Number of measurements	Laboratory mean of S_{BET} $m^2 g^{-1}$	Standard deviation $m^2 g^{-1}$
L01-01	9	0.173	0.003
L03-02	9	0.184	0.006
L11-06	3	0.149	0.008
L22-11	9	0.174	0.006
L25-12	9	0.166	0.004
L26-35	2	0.161	0.005
L31-14	9	0.172	0.005
L37-19	6	0.169	0.003
L41-22	9	0.176	0.004
S52-28	7	0.188	0.055
L54-30	9	0.208	0.004
L56-32	8	0.189	0.005
L57-33	9	0.181	0.005
L60-40	9	0.183	0.004

3. Further information regarding the reference material

3.1 Origin

The material was prepared from ground pegmatite granite which was mechanically prepared and chemically purified.

3.2 Chemical analysis

The silica content of the material (SiO_2) is >99.99 %.

3.3 Thermal analysis

When the silica material is heated its mass losses are extremely low, i.e. ca. 0.1%, occurring mainly in the temperature range of up to 673 K (see Figure 1). They are essentially attributable to the desorption of adsorbed water. The reversible DTA effect with a peak temperature of 850 K has a characteristic shape with a shifted base line and a somewhat asymmetric peak, as is characteristic for modification change of quartz (low and high modification).

3.4 Phase analysis by X-ray powder diffraction

The material consists of crystalline quartz. No other crystalline SiO_2 modifications or phases can be identified. The detection limit under the test conditions is better than 0.5 mass %.

3.5 Particle size distribution

The particle range of the material is quite narrow, i.e. between 100 and 600 µm, with an average particle size of circ. 260 µm; it was determined by laser diffraction analysis (see Figure 2).

3.6 Density

The density is 2.65 g/cm³, determined by applying helium at 293 K.

3.7 Morphology

The particles have sharp and irregular surfaces (see Figure 3).

3.8 Recommendations

When the reference material will be used for calibrating measurement of instruments, it should be taken into account that the dead volume was measured by using helium.

3.9 Durability

Durability of the reference material is guaranteed for two years from date of shipment provided the material is stored and handled appropriately.

4. References

(1) Guidelines for the production and certification of BCR reference materials, European Commission, Standards, Measurement & Testing Programme, 1994

(2) DIN 66131: Determination of specific surface area of solids by means of gas adsorption after Brunauer, Emmett and Teller (BET), July 1993; Beuth Verlag GmbH, Berlin

(3) S. Brunauer, P.H. Emmett u. E. Teller, J. Amer. Chem. Soc. **60**, 309 (1938)

(4) K.S.W. Sing, D.H. Everett, R.A.W. Haul, L. Moscou, R. A. Pierotti, J. Rouquerol, T. Siemieniewska, Pure & Appl. Chem. **57** (1985) 603 (IUPAC Recommendations 1984)

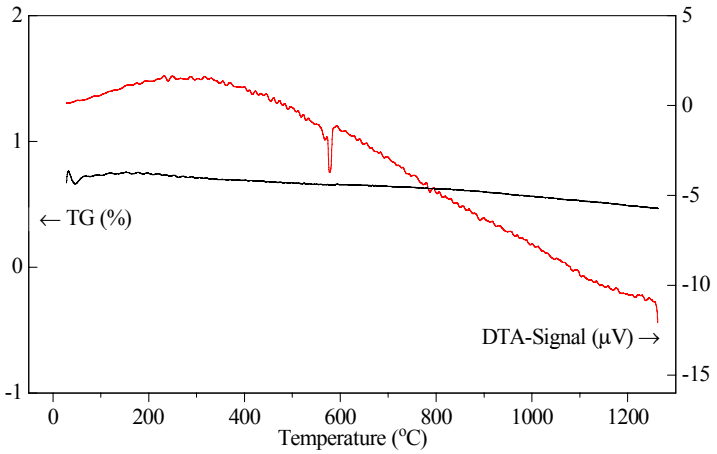

Figure 1: TG and DTA curves of silica

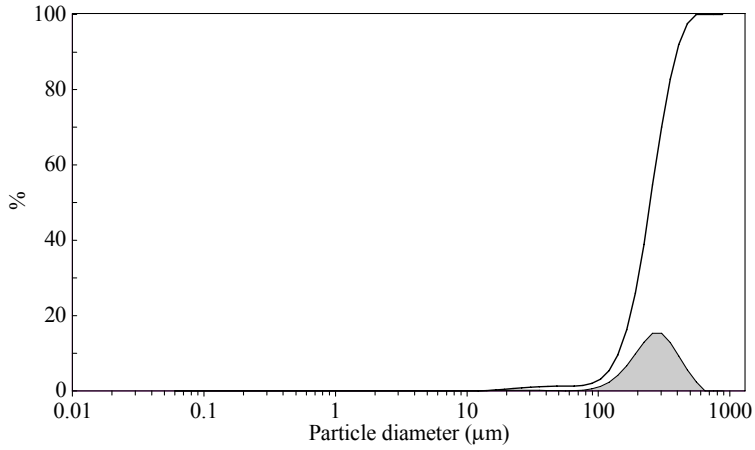

Figure 2: Particle size distribution of silica

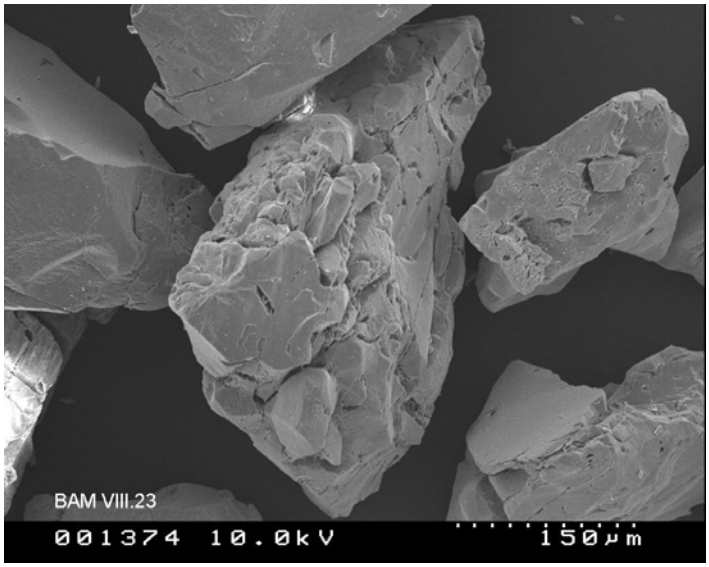

Figure 3: Scanning electron micrograph of silica

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Prof Dr A. Zschunke
Head of Department
Analytical Chemistry,
Reference Materials

Prof Dr K. Meyer
Head of Division
Inorganic Chemical Analysis,
Reference Materials

Bundesanstalt für Materialforschung und -prüfung (BAM)
Division I.1 Inorganic Chemical Analysis, Reference Materials
Branch Adlershof, Richard-Willstätter-Straße 11, D-12489 Berlin

phone: ++ 49-30-8104-1119/2061/5825
fax: ++ 49-30-8104-1117

e-mail: Angelika.Selmke@bam.de
e-mail: Peter.Klobes@bam.de

www.webshop.bam.de