Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR THE GAS ADSORPTION

Certificate

BAM-PM-102
Material: α-Alumina

with specific surface area (BET) of

5.41 $m^2 g^{-1}$ ± 0.04 $m^2 g^{-1}$

Mean of means [1)]	**5.41**	**$m^2 g^{-1}$**
Uncertainty		
Standard deviation of the mean of means	**0.04**	**$m^2 g^{-1}$**
95% confidence interval	**0.09**	**$m^2 g^{-1}$**
Standard deviation of means	**0.24**	**$m^2 g^{-1}$**

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	Nitrogen
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 of small surface areas.
The parameters mentioned are material-specific quantities to characterize non-porous and macroporous solids by means of the gas adsorption method (Isotherm Type II).

[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. Measurement and evaluation

2.1 Pretreatment of the sample

Heating the specimen for one hour at 523 K at 0.1 Pascal
Keeping this temperature for 3 hours at a specified vacuum, cooling slowly

2.2 Measurement

The quantity of nitrogen 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 nitrogen: $a_{nitrogen}$ = 0.162 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:

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

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 physikalische Kenngrößen; Porenstruktur, Berlin
Bundesanstalt für Materialforschung und -prüfung (BAM), Laboratorium Sekundäreigenschaften von Referenzmaterialien, Berlin
CONDEA Chemie GmbH, Anorganische Spezialchemikalien, Brunsbüttel
Degussa AG, ZFE - OT, Hanau
FISONS Instruments S.p.A., Milano, Italy
Forschungsinstitut für Leder- und Kunstledertechnologie gGmbH, Freiberg/Sa.
Forschungs- und Materialprüfungsanstalt Baden-Württemberg, Otto-Graf-Institut, Stuttgart
Freiberger NE-Metall GmbH, Material- und Umwelttechnik, Freiberg/Sa.
Friedrich-Schiller-Universität Jena, Institut für Physikalische Chemie, Jena
GSF-Forschungszentrum für Umwelt und Gesundheit, GmbH, Oberschleißheim
Hochschule für Architektur und Bauwesen, Forschungsbereich Baustoffe,Weimar
HÜLS AG, Zentrale Analytik, Marl
Institut für Angewandte Forschung, Reutlingen
Institut für Festkörper und Werkstofforschung Dresden e.V. , Dresden
Institut für Polymerforschung e. V., Dresden
Merck KGaA, Darmstadt
Leuna-Katalysatoren GmbH, Leuna
Micromeritics GmbH, Neuss
Philips GmbH, Aachen
Quantachrome, Eurasburg
Schaefer Kalk, Diez/Lahn

TU Bergakademie Freiberg, Institut Keramische Werkstoffe, Freiberg /Sa.
Universität Erlangen-Nürnberg, Lehrstuhl für Technische Chemie, Erlangen
Universität Gesamthochschule Essen, Fachbereich Bauphysik und Materialwirtschaft, Essen
Universität Kassel, Fachgruppe Thermodynamik, Kassel
Universität Leipzig, Institut für Physikalische und Theoretische Chemie, Leipzig
Universität des Saarlandes, Saarbrücken
Wissenschaftlich-technische Gesellschaft für Verfahrenstechnik, FIA e.V., Freiberg/Sa.

Table 1

Evaluation of the interlaboratory study for determining the specific surface area of α-alumina using the BET method.
Participating laboratories: 30
Parameter to be certified: *BET specific surface area*
Method: Gas adsorption at 77 K, adsorptive nitrogen

Laboratory	**Number of measurements**	**Laboratory mean of S_{BET} $m^2 g^{-1}$**	**Standard deviation $m^2 g^{-1}$**
L01-01	9	5.96	0.04
L03-02	8	5.21	0.08
L05-03	9	5.49	0.02
L08-04	8	5.63	0.43
L09-37	8	5.39	0.06
A11-06	3	5.46	0.09
L13-34	7	5.10	0.64
L15-07	8	5.91	0.12
L16-08	9	5.31	0.10
L21-40	9	5.52	0.12
L23-36	8	5.33	0.02
L25-12	8	5.40	0.02
L26-35	3	5.35	0.09
L30-13	9	5.32	0.08
L32-15	8	5.36	0.03
L34-17	7	5.40	0.06
L35-18	9	5.37	0.04
L38-20	8	5.51	0.13
L39-21	9	5.40	0.02
L41-22	9	5.45	0.02
L45-24	7	5.40	0.08
L46-25	9	5.52	0.03
L49-26	9	5.07	0.15
L52-28	9	4.82	0.12
L54-30	9	5.46	0.04
L55-31	9	5.84	0.24
L56-32	9	5.00	0.17
L57-33	9	5.53	0.07
S57-33	9	5.35	0.70
L61-41	6	5.52	0.03

3. Further information regarding the sample

3.1 Origin

The sample is a product of the Ceralox-Corp. Tucson, Arizona, USA.

3.2 Chemical analysis

The α-alumina content of the sample (Al_2O_3) is >99.76 % +/- 0.03.

3.3 Thermal analysis

When α-alumina is heated its mass losses are extremely low, 0.03 %. DTA-effects can be not identified. The failure of DTA-effects and the low mass losses confirm the presence of α-alumina (see Figure 1).

3.4 Phase analysis by X-ray powder diffraction

The material consists of corundum. No other alumina forms 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 between 40 and 500 μm with an average particle size of circ. 150 μm; it was determined by laser diffraction analysis (see Figure 2).

3.6 Density

The density is 3.97 g/cm^3, 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 three 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)

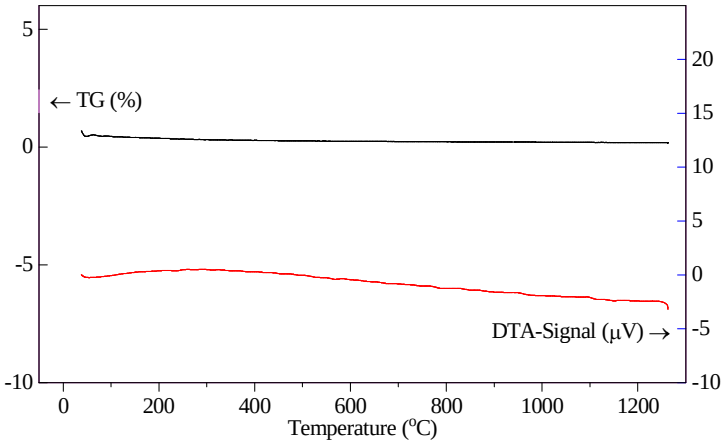

Figure 1: TG and DTA curves of α-alumina

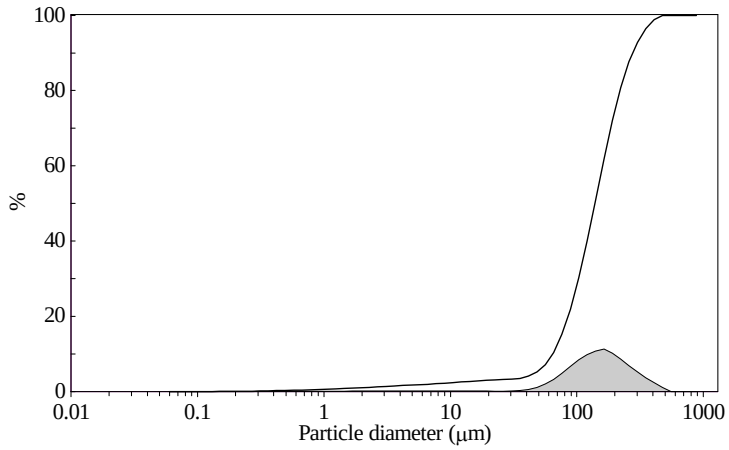

Figure 2: Particle size distribution of α-alumina

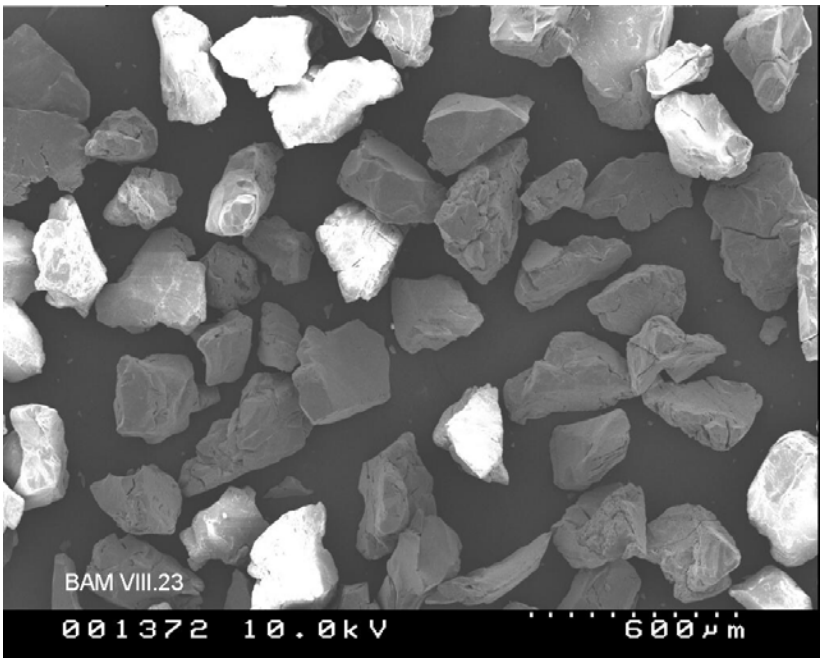

Figure 3: Scanning electron micrograph of α-alumina

Date of certification: 1996-08-30

Date of shipment: .....

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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

This reference material is supplied by:

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

Phone: +49 30 8104 2061
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