

**Federal Institute for Materials Research and Testing**

**CERTIFIED REFERENCE MATERIAL  
FOR THE GAS ADSORPTION**

***BAM-PM-102***  
***Material:  $\alpha$ -Alumina***

***with specific surface area (BET) of***

$$5.41 \text{ m}^2 \text{ g}^{-1} \pm 0.04 \text{ m}^2 \text{ g}^{-1}$$

<b>Mean of means <sup>1)</sup></b>	<b>5.41 m<sup>2</sup> g<sup>-1</sup></b>
<b>Uncertainty</b>	
<b>Standard deviation of the mean of means</b>	<b>0.04 m<sup>2</sup> g<sup>-1</sup></b>
<b>95% confidence interval</b>	<b>0.09 m<sup>2</sup> g<sup>-1</sup></b>
<b>Standard deviation of means</b>	<b>0.24 m<sup>2</sup> g<sup>-1</sup></b>

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).

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<sup>1)</sup> 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:  $a_{\text{nitrogen}} = 0.162 \text{ nm}^2$  (4)

### **2.4 Evaluation**

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

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

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 Werkstoffforschung 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  $\alpha$ -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 $S_{\text{BET}}$ $\text{m}^2 \text{g}^{-1}$	Standard deviation $\text{m}^2 \text{g}^{-1}$
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  $\alpha$ -alumina content of the sample ( $\text{Al}_2\text{O}_3$ ) is >99.76 % +/- 0.03.

#### **3.3 Thermal analysis**

When  $\alpha$ -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  $\alpha$ -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  $\mu\text{m}$  with an average particle size of circ. 150  $\mu\text{m}$ ; it was determined by laser diffraction analysis (see Figure 2).

#### **3.6 Density**

The density is 3.95  $\text{g/cm}^3$ , determined by applying helium at 293 K.

#### **3.7 Morphology**

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

#### **3.8 Recommendations**

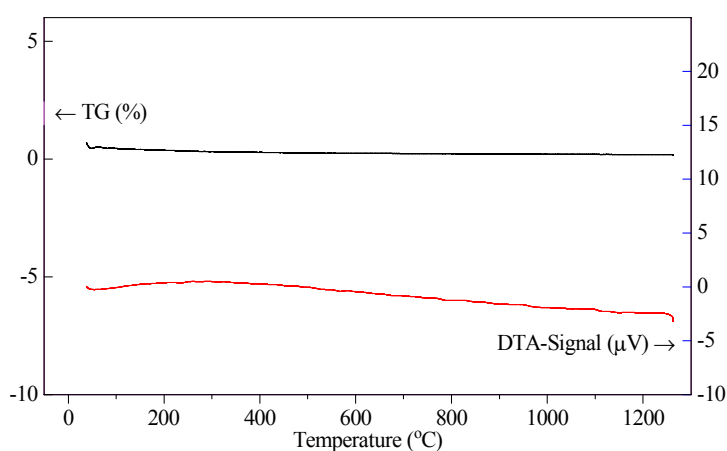
When the reference material will be used for calibrating measurement of instruments, it should be taken into account that the dead volume was measured by using helium.

#### **3.9 Durability**

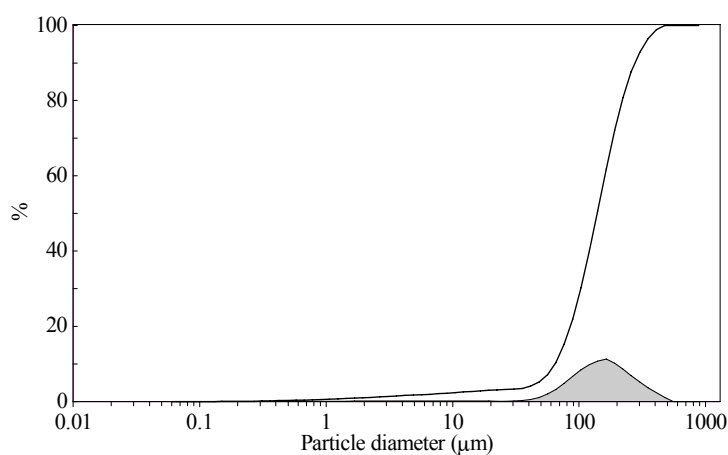
Durability of the reference material is guaranteed for two years from date of shipment provided the material is stored and handled appropriately.

#### 4. References

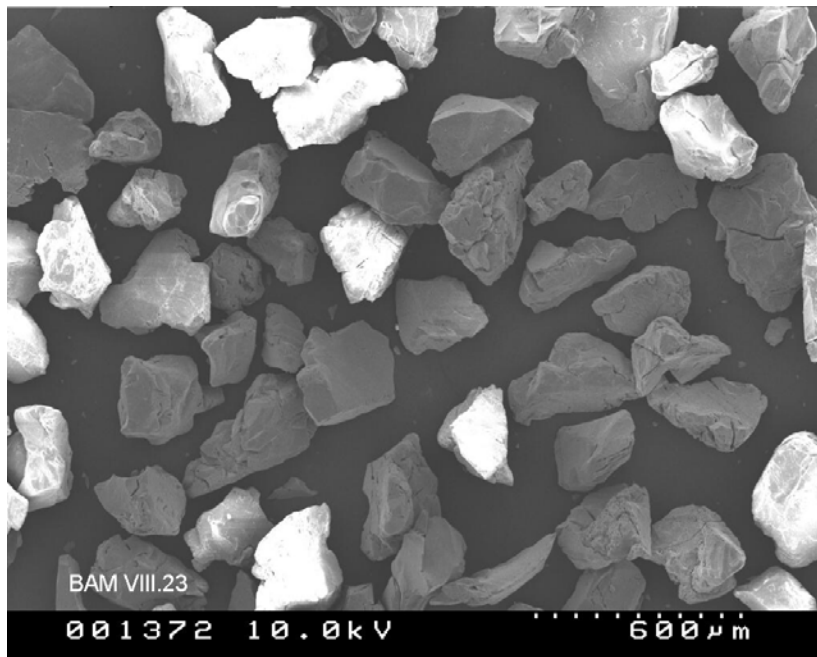
- (1) Guidelines for the production and certification of BCR reference materials, European Commission, Standards, Measurement & Testing Programme, 1994
- (2) DIN 66131: Determination of specific surface area of solids by means of gas adsorption after Brunauer, Emmett and Teller (BET), July 1993; Beuth 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  $\alpha$ -alumina



**Figure 2:** Particle size distribution of  $\alpha$ -alumina



**Figure 3:** Scanning electron micrograph of  $\alpha$ -alumina

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