Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL
FOR THE GAS ADSORPTION

ausverkauft / out of stock

BAM-PM-103
Material: Alumina Type 60

with specific surface area (BET) of

156.0 ± 1.3 $m^2 g^{-1}$

Mean of means[1)]	**156.0 $m^2 g^{-1}$**
Uncertainty	
Standard deviation of the mean of means	**1.3 $m^2 g^{-1}$**
95%-confidence interval	**2.7 $m^2 g^{-1}$**
Standard deviation of means	**7.2 $m^2 g^{-1}$**

with specific pore volume
(adsorption, Gurvich) of

0.250 ± 0.002 $cm^3 g^{-1}$

Mean of means [1)]	**0.250 $cm^3 g^{-1}$**
Uncertainty	
Standard deviation of the mean of means	**0.002 $cm^3 g^{-1}$**
95%-confidence interval	**0.004 $cm^3 g^{-1}$**
Standard deviation of means	**0.008 $cm^3 g^{-1}$**

Certificate

[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.

with hydraulic pore radius of

3.18 ± 0.02 nm

Mean of means [2)]	**3.18 nm**
Uncertainty	
Standard deviation of the mean of means	**0.02 nm**
95%-confidence interval	**0.03 nm**
Standard deviation of means	**0.08 nm**

with most frequent pore radius of

1.93 ± 0.04 nm

Mean of means [2)]	**1.93 nm**
Uncertainty	
Standard deviation of the mean of means	**0.04 nm**
95%-confidence interval	**0.07 nm**
Standard deviation of means	**0.18 nm**

according to the 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) Pore volume, mean pore radius and most frequent pore radius according to DIN 66134 (3)

1. Scope

The reference material is intended for the calibration and checking of instruments, especially for determining the surface area, the specific pore volume, the mean pore radius, and the most frequent pore radius.
The parameters mentioned are material-specific quantities to characterize mesoporous solids by means of the gas adsorption method.
(Isotherm Type IV according to IUPAC classification (4)).

[2)] 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 5 hours at a specified vacuum, cooling slowly.

2.2 Measurement

The quantity of nitrogen adsorbed and desorbed was measured using 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)
- capillary condensation theory (5)

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.

The specific pore volume after Gurvich was determined from adsorption branch of the isotherm at p/p_0 = 0.99 according to DIN 66134.

The hydraulic pore radius was determined using the relation $2V/S_{BET}$ according to DIN 66134 with V being the pore volume calculated from the desorption branch of the isotherm by means of the model of Barrett, Joyner und Halenda using the Kelvin and Halsey equations.

The most frequent pore radius according to DIN 66134 was determined from the maximum of the pore size distribution dV/dr calculated from the desorption branch of the isotherm by means of the model of Barrett, Joyner und Halenda using the Kelvin and Halsey equations.

Participants in the interlaboratory study:

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Degussa AG, ZFE - OT, Hanau
FISONS Instruments S.p.A., Milano, Italy
Freiberger NE-Metall GmbH, Material- und Umwelttechnik, Freiberg/Sa.

Friedrich-Schiller-Universität, Institut für Physikalische Chemie, Jena
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Grace Davison, R & D, Silica/Adsorbents, Worms
HÜLS AG, Zentrale Analytik, Marl
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KGaA Merck, Darmstadt
Leuna-Katalysatoren GmbH, Leuna
Micromeritics GmbH, Neuss
Quantachrome, Eurasburg
Schaefer Kalk, Diez / Lahn
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Universität Leipzig, Institut für Physikalische und Theoretische Chemie, Leipzig
Universität des Saarlandes, Saarbrücken
Wissenschaftlich-technische Gesellschaft für Verfahrenstechnik, FIA e.V., Freiberg

Table 1

Evaluation of the interlaboratory study for determining the specific surface area of Alumina Type 60 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 $m^2\,g^{-1}$**
A01-01	2	161.2	0.1
A11-06	3	159.7	0.4
A60-40	9	157.7	1.8
D01-01	6	155.9	2.6
L03-02	9	130.9	8.9
L08-04	9	162.8	1.2
L09-37	8	155.5	1.1
L10-05	9	156.2	1.3
L13-34	7	136.8	9.6
L16-08	9	161.1	0.8
L23-36	9	155.2	2.2
L25-12	9	158.3	0.9
L26-35	3	160.6	2.4

Table 1 (cont.)

Laboratory	Number of measurements	Laboratory mean of S_{BET} $m^2\,g^{-1}$	Standard deviation $m^2\,g^{-1}$
L32-15	9	156.0	1.1
L34-17	8	156.6	1.0
L35-18	9	146.1	4.4
L39-21	8	156.7	1.0
L41-22	9	155.6	1.5
L45-24	6	153.8	1.7
L46-25	9	158.9	1.0
L49-26	8	157.2	0.6
L51-27	8	156.6	0.6
L54-30	9	158.3	0.8
L55-31	9	151.8	3.2
L56-32	9	162.1	1.2
L57-33	9	158.6	0.7
L58-38	9	153.6	1.4
L60-40	5	158.6	3.0
L61-41	6	162.9	1.0
S57-33	8	166.2	1.8

Table 2

Evaluation of the interlaboratory study for determining the specific pore volume of alumina type 60
Participating laboratories: 24
Parameter to be certified: *Gurvich specific pore volume (adsorption at $p/p_0 = 0.99$)*
Method: Gas adsorption at 77 K, adsorptive nitrogen

Laboratory	**Number of measurements**	**Laboratory mean of specific pore volume** $cm^3\,g^{-1}$	**Standard deviation** $cm^3\,g^{-1}$
A01-01	3	0.251	0.001
A11-06	3	0.252	0.002
A60-40	9	0.248	0.003
D01-01	6	0.246	0.003
L08-04	9	0.249	0.005
L09-37	9	0.256	0.022
L10-05	9	0.259	0.003
L13-34	7	0.228	0.009
L16-08	9	0.249	0.004
L23-36	9	0.244	0.004
L25-12	9	0.255	0.002
L26-35	2	0.259	0.001
L32-15	8	0.253	0.004
L35-18	9	0.235	0.008
L41-22	7	0.251	0.001
L45-24	7	0.247	0.004
L46-25	8	0.247	0.008
L54-30	9	0.248	0.004
L55-31	8	0.233	0.003
L56-32	9	0.261	0.003
L57-33	9	0.252	0.002
L60-40	5	0.249	0.004
L61-41	6	0.260	0.005
S57-33	7	0.259	0.004

Table 3

Evaluation of the interlaboratory study for determining the hydraulic pore radius of Alumina Type 60
Participating laboratories: 24
Parameter to be certified: *Hydraulic pore radius (2V/S_{BET})*
Method: Gas adsorption at 77 K, adsorptive nitrogen

Laboratory	**Number of measurements**	**Laboratory mean of hydraulic pore radius nm**	**Standard deviation nm**
A01-01	3	3.10	0.03
A11-06	3	3.16	0.02
A60-40	9	3.14	0.02
D01-01	6	3.16	0.02
L08-04	9	3.06	0.06
L09-37	9	3.31	0.27
L10-05	9	3.31	0.04
L13-34	7	3.34	0.18
L16-08	9	3.09	0.04
L23-36	9	3.15	0.03
L25-12	9	3.22	0.02
L26-35	2	3.20	0.06
L32-15	8	3.25	0.06
L35-18	9	3.21	0.05
L41-22	8	3.21	0.05
L45-24	7	3.24	0.04
L46-25	8	3.11	0.09
L54-30	9	3.14	0.04
L55-31	9	3.06	0.04
L56-32	9	3.22	0.03
L57-33	9	3.19	0.02
L60-40	4	3.16	0.02
L61-41	6	3.20	0.07
S57-33	7	3.11	0.05

Table 4

Evaluation of the interlaboratory study for determining the most frequent pore radius of Alumina Type 60
Participating laboratories: 26
Parameter to be certified: *Most frequent pore radius (BJH model, desorption branch)*
Method: Gas adsorption at 77 K, adsorptive nitrogen

Laboratory	**Number of measurements**	**Laboratory mean of most frequent pore radius** **nm**	**Standard deviation** **nm**
A01-01	3	1.97	0.004
A11-06	2	2.26	0.003
A60-40	8	1.81	0.007
D01-01	6	1.98	0.003
L03-02	9	1.70	0.138
L09-37	8	1.96	0.012
L10-05	9	1.86	0.067
L13-34	7	1.76	0.058
L16-08	8	1.77	0.039
L23-36	8	2.23	0.001
L25-12	9	1.88	0.078
L32-15	6	2.24	0.005
L35-18	8	1.75	0.013
L39-21	9	1.98	0.0001
L41-22	9	2.15	0.129
L45-24	7	1.70	0.283
L46-25	9	2.24	0.004
L49-26	8	1.99	0.006
L54-30	8	1.76	0.029
L55-31	9	1.79	0.467
L56-32	8	1.77	0.001
L57-33	8	2.23	0.006
L58-38	9	1.90	0.001
L60-40	4	1.81	0.002
L61-41	6	1.85	0.057
S57-33	9	1.87	0.107

3. Further information regarding the sample

3.1 Origin

The sample is a product of Merck KGaA, Darmstadt, Germany.

3.2 Isotherm and pore size distribution

See Figures 1 and 2.

3.3 Thermal analysis

When alumina type 60 is heated its endothermic mass losses are 3.3 %. This mass losses continue until 1273 K. At a temperature of 1443 K an exothermic effect begins which ends at 1533 K. Adsorbed water was removed. (see Figure 3).

3.4 Phase analysis (X-ray powder diffraction)

Alumina phase η with parts of γ.

3.5 Particle size distribution

The particle range of the material is between 30 and 250 µm. It was determined by laser diffraction analysis (see Figure 4).

3.6 Density

The density is 3.39 g cm^{-3}, determined by applying helium at 293 K.

3.7 Morphology

See Figure 5.

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 one year 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)

(5) L. H. Cohan, J. Am. Chem. Soc. **66**, 98 (1944)

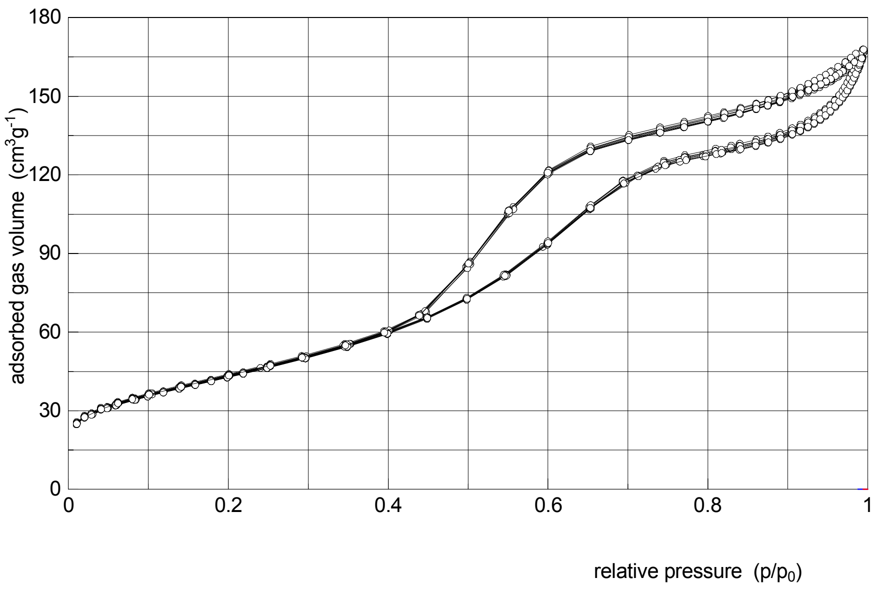

Figure 1: Adsorption isotherm for nitrogen at 77 K on alumina type 60 (overlay of 9 measurements)

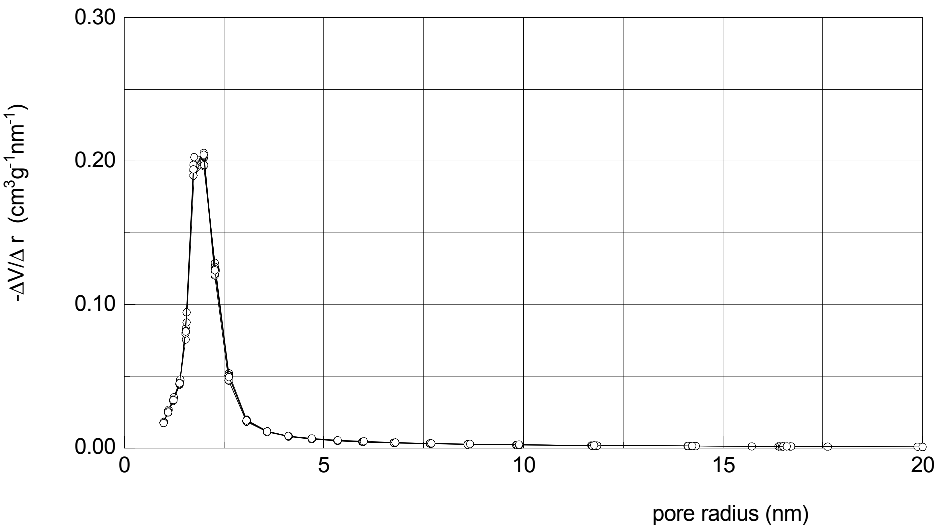

Figure 2: Pore size distribution of alumina type 60 (overlay of 9 measurements)

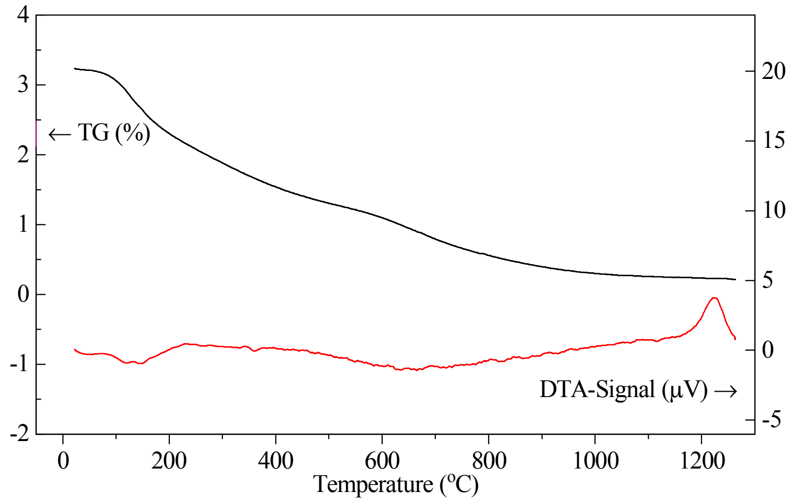

Figure 3: TG and DTA curves of alumina type 60

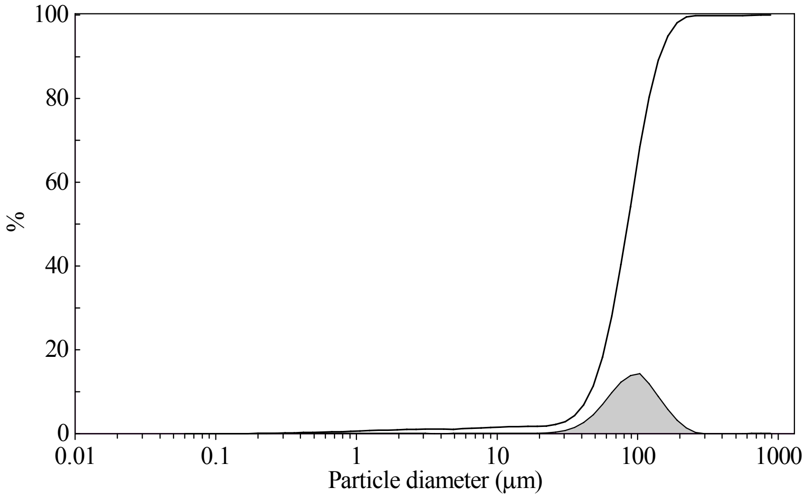

Figure 4: Particle size distribution of alumina type 60

Figure 5: Scanning electron micrograph of alumina type 60

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Date of shipment: ....

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