BAM

Certification Report

Certified Reference Material BAM-P128

Porosity Properties of Macroporous Alumina Ceramic

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Summary

This report describes the certification of the porous reference material BAM-P128 by mercury intrusion porosimetry in the low pressure range. The certified values determined by mercury intrusion porosimetry according to the international standards ISO 15901-1 [1] and DIN 66133 [2] are summarized in Table 1.

Table 1: Certified values determined by mercury intrusion porosimetry.

Property		Value [a]	Uncertainty [b]	Unit
Specific pore volume [c]	V_p	220	6	mm^3/g
Median pore diameter [d]	d_{50}	27.6	1.0	µm

[a] Mean value of the means of accepted data sets each derived from at least 19 single values.

[b] Uncertainty $U = k\ u_c$ calculated according to ISO Guide 35 [4] and ISO/IEC Guide 98-3 [5] with the coverage factor $k = 2$ (giving a level of confidence of approximately 95 %). The combined standard uncertainty u_c of each certified property includes an uncertainty contribution resulting from the interlaboratory testing, the study of inhomogeneities and stability of the material.

[c] Specific pore volume V_p calculated from the mercury intrusion with maximum pressure in the low-pressure device. Described in ISO 15901-1 [1] and DIN 66133 [2].

[d] Median pore diameter d_{50} calculated according to the Washburn equilibrium model [6] as described in ISO 15901-1 [1] and DIN 66133 [2].

Informative Value

Property		Value	Uncertainty	Unit
Density	ρ_s	3.6405	0.0019	g/cm^3

A unit of the CRM BAM-P128 consists of a package with six cylindrical porous burnt alumina ceramics (diameter: 5.5 mm, length: 20 mm).
The certified values are method-defined (model dependent) parameters. The used evaluation models are applied as an integral part of the traceability statement. The certified values are traceable to the base units of the SI via calibrated measurements of the quantities pressure, volume, and mass.

The reference material is intended for performance checking of instruments used for the determination of specific pore volume and median pore diameter from the low pressure branch of the mercury intrusion up to a maximum pressure between 0.2 and 0.4 MPa, depended on the specification of the used device.

The certificate of BAM-P128 is valid for three years from the date of shipment, providing that the reference material is stored under the recommended conditions.

List of abbreviations

(Unless not explained in particular sections of this report)

ANOVA	Analysis of Variance
CI	confidence interval
CRM	certified reference material
GUM	ISO guide to the expression of uncertainty in measurement
ILC	interlaboratory comparison
MU	measurement uncertainty
RM	reference material
TI	tolerance interval

List of symbols

k	coverage factor
l	number of accepted data sets in the interlaboratory comparison
n	number of replicate measurements
MS	mean squares sum
$\nu_{\mathrm{MS}_{\mathrm{within}}}$	degrees of freedom of $\mathrm{MS}_{\mathrm{within}}$
s_x	ILC standard deviation of a property value
U	expanded standard uncertainty of a property value
u_{bb}	standard uncertainty due to (in)homogeneity between units
u_{char}	standard uncertainty due to characterization
u_{c}	combined standard uncertainty of a property value
u_{lts}	standard uncertainty due to long-term (in)stability
x	property value of a candidate material
$\bar{x}$	mean value
x_{cert}	certified property value of a CRM
x_{i}	result of a single measurement in the experiment

Contents page

1.	Intention of the certification project	1
2.	Description of the material	2
2.1	Selection and source of the candidate material	2
2.2	Porosity characterization	4
3.	Homogenization and subdividing of the candidate material	6
4.	Homogeneity and stability testing	6
4.1	Homogeneity	6
4.2	Stability testing	9
5.	List of participating laboratories	13
6.	Results of the interlaboratory testing and statistical uncertainty estimation	14
6.1	Experimental results	14
6.2	Statistical evaluation	16
7.	References	18

1. Intention of the certification project

Today, pore size analysis by means of mercury intrusion porosimetry is one of the most frequently applied methods for the characterization of nano- and macroporous materials, together with gas adsorption. Only two national metrological institutes worldwide provide reference materials for mercury intrusion porosimetry. Among those, BAM provides a variety of reference materials for mercury intrusion porosimetry. NIST provides two reference materials for this method, one of those together with BAM.

The new reference material BAM-P128 represents an important addition to the reference materials already available, taking into account aspects of the measurement itself, which will be discussed in brief below.

A measurement is split into two phases. The first one corresponds to a maximum pressure slightly above the atmospheric pressure, whereas the second one corresponds to a maximum pressure given by the operator or instrument specification (typically up to a pressure of 400 MPa). The measurement in the two pressure ranges is technically implemented in two separate measuring stations.

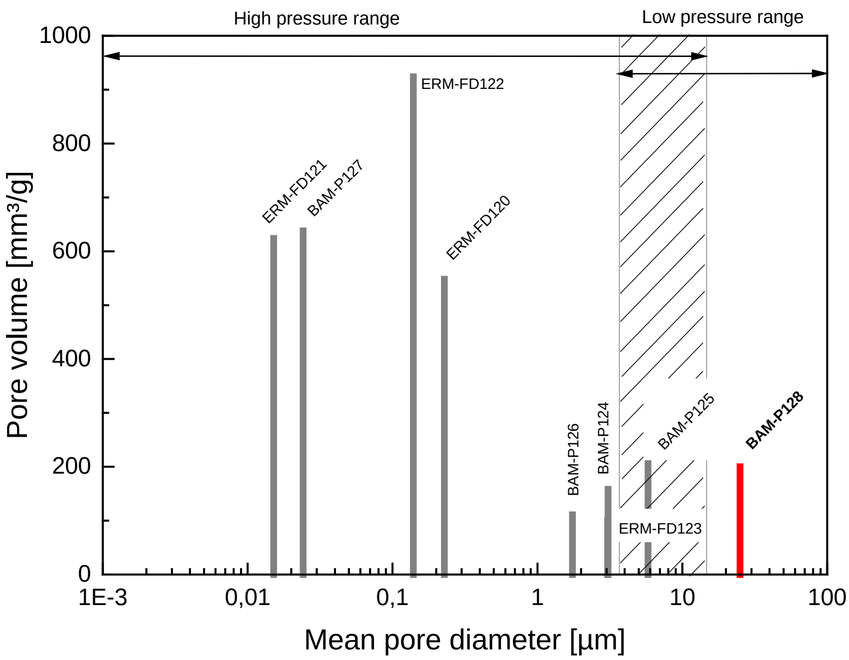


Fig. 1: Overview of current (grey bars) and new (red bar) BAM certified reference materials for mercury intrusion porosimetry.

For both measuring stations (low and high pressure) it is important that the pressure sensors and the volume calibration are tested using reference materials. All available reference materials for mercury intrusion porosimetry were developed for the high pressure range. The new reference material CRM BAM-P128 completes the range of currently available reference materials for mercury intrusion porosimetry (see Fig. 1). At the same time, it is the first reference material for testing the low pressure range worldwide.

The certification of this new CRM has been carried out on the basis of BAM Guidelines for the Development of Reference Materials [3] and relevant ISO Guides [4-5, 7-8].

2. Description of the material

2.1 Selection and source of the candidate material

CRM BAM-P128 consists of macroporous extruded and burnt alumina ceramic (Al_2O_3). The porous ceramic candidate material RAPOR® P20, manufactured and delivered by Rauschert Heinersdorf-Pressig GmbH (Pressig, Germany), was chosen from a number of tested candidate materials. The material is intended as the first mercury intrusion reference material for the low pressure devices of mercury intrusion porosimetry.

The candidate material RAPOR® P20 was characterized by X-ray powder diffraction. The material was homogenized in a ball mill (Pulverisette 23, Fritsch, Germany). The resulting fine powder was filled into a glass capillary (diameter: 0.5 mm). Data collection was performed using a Bruker D8 DISCOVER diffractometer using monochromatic Copper $K\alpha_1$-radiation and a Johansson monochromator in the incident beam. The diffracted intensities were collected using the following conditions: 2 θ range: 5°-70°, step size: 0.009°, step time: 1 s, 2 h per measurement. The resulting diffractogram is depicted in Fig 2. The strongest reflections coincide with the reflections for corundum (Al_2O_3, PDF 46-1212). Additional reflections of mullite ($Al_2(Al_{2.8}Si_{1.2})O_{9.6}$, PDF 79-1276) could also be detected [9].

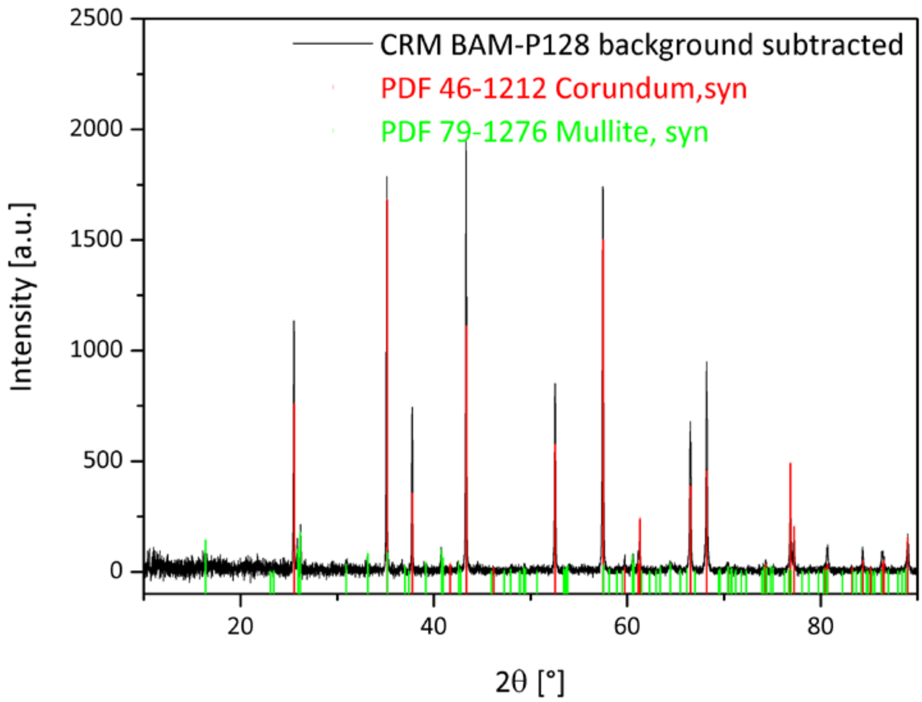


Fig. 2: Diffraction pattern of a CRM BAM-P128 sample (black line). The pattern shows sharp reflexes indicating a high degree of crystallinity of the sample. Red bars indicate the reflections of corundum, green bars indicate those of mullite.

The skeletal (apparent) density of CRM BAM-P128 was measured by He-Pycnometry [10].

Property	Value [a]	Uncertainty [b]	Unit
Density [c] ρ_s	3.6405	0.0019	g/cm³

[a] Mean value of 60 measurements at 20 °C.

[b] Uncertainty u_{comb} calculated by combined uncertainty of balance u_m, device u_{pyc} and replicate measurements u_v.

$$u_{\mathrm{comb}} = \sqrt{u_{\mathrm{m}}^2 + u_{\mathrm{pyc}}^2 + u_{\mathrm{v}}^2}$$

[c] Skeletal density, apparent density for porous and unporous solids described in DIN 66137-2 [10].

2.2 Porosity characterization

The sample material was tempered at 105 °C in a drying oven for three hours. The pore size analysis was carried out in the low pressure range between 0.2 and 0.4 MPa [11-12]. In the low pressure station the maximum available pressure range should be exploited. This pressure range correlates with the measurement of the macropore system with a pore width range of 0.36 µm to over 450 µm. Based on an appropriate mercury intrusion the following porosity parameters can be calculated for the certification of BAM-P128.

- Specific pore volume, V_p [1]
  The maximum value of intruded mercury in the pore system in the macropore range (see Fig. 3).

- Median pore diameter d_{50}
  The exerted pressure is inversely proportional to the clear width of the pore entrance. For pores of cylindrical shape, the Washburn equation [6] gives the relation between pressure and diameter (see Equation (1)).

$$d_\mathrm{p} = \frac{-4\gamma cos\theta}{p} \qquad (1)$$

The mercury surface tension, γ, with 0.480 N·m^{-1} (480 dynes/cm) and a contact angle, θ, of 140.0 degrees are used for the convert of pressure readings to the pore diameter d_{50} (see Fig. 4).

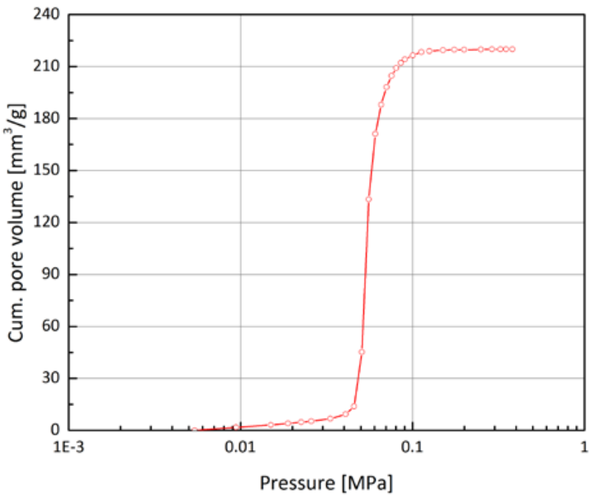


Fig. 3: Complete mercury intrusion in low pressure range up to 0.4 MPa.

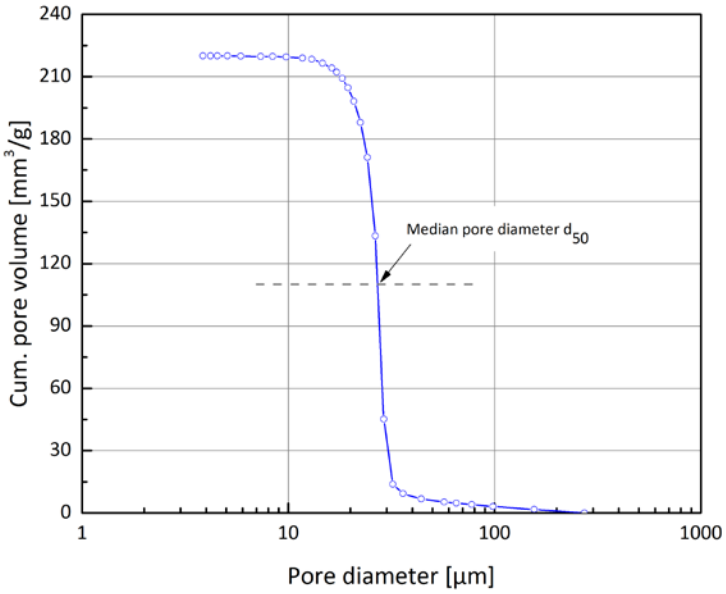


Fig. 4: Graphical representation of the median pore diameter d_{50} by calculation of the half height of the cumulative pore volume in the low pressure range.

3. Homogenization and subdividing of the candidate material

A total amount of 1500 cylinders was purchased from Rauschert Heinersdorf-Pressig GmbH (Pressig, Germany). 150 pieces were reserved for stability testing, further characterization, and retained sample. Homogenization and subdivision of the candidate material were carried out by random compilation. The initial amount of BAM-P128 was 1350 pieces. The distribution of these pieces for testing and sale was based on a matrix calculation from A1 to O15 with a total number of 225 units consisting of six pieces of BAM-P128 (see Fig. 5).

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1		o					x							x	
2					o						x				o
3												x	o		
4								x	o				x		
5		x				o									
6			o				x								
7											o				x
8					x			o							
9			x	o									x		
10						x						o			
11							o								
12				x		x				o					
13		o												x	
14										x		x		o	
15	o	x							x						

Fig. 5: Matrix scheme for the distribution of BAM-P128. Circles indicate samples for the homogeneity study. The crosses indicate samples distributed for interlaboratory testing.

4. Homogeneity and stability testing

4.1 Homogeneity

For homogeneity tests, 16 individual units of BAM-P128 were randomly selected. Two replicate measurements per unit were carried out under repeatability conditions to detect the within-unit and between unit standard deviation. The tests were carried out on an automated porosity analyzer AutoPore III (Micromeritics, Norcross USA). The test results are summarized in Table 2.

Table 2: Results of homogeneity testing between and within units (2 replicates for each unit).

Unit	Data file	V_p [mm³/g]	d_{50} [μm]
A15-1	A15-2284.smp	219.3	26.7677
A15-2	A15-2287.smp	221.5	26.4976
B1-1	B1-2298.smp	220.1	26.7285
B1-2	B1-2299.smp	218.2	26.7854
B13-1	B13-2304.smp	218.1	26.4327
B13-2	B13-2306.smp	221.4	26.7231
C6-1	C6-2277.smp	216.8	26.3786
C6-2	C6-2280.smp	219.2	26.0934
D9-1	D9-2245.smp	223.0	27.0675
D9-2	D9-2248.smp	216.9	26.4807
E2-1	E2-2257.smp	220.9	27.3038
E2-2	E2-2259.smp	220.1	27.1658
F5-1	F5-2268.smp	219.2	26.8717
F5-2	F5-2270.smp	224.1	26.8878
G11-1	G11-2295.smp	221.0	26.6998
G11-2	G11-2296.smp	221.5	26.4299
I4-1	I4-2301.smp	222.8	26.7154
I4-2	I4-2302.smp	219.6	26.3624
J12-1	J12-2292.smp	219.5	26.5824
J12-2	J12-2293.smp	219.0	26.0955
K7-1	K7-2281.smp	217.0	25.9871
K7-2	K7-2282.smp	223.0	26.5490
L10-1	L10-2263.smp	218.0	27.3176
L10-2	L10-2266.smp	221.6	26.9639
M3-1	M3-2253.smp	220.1	27.2774
M3-2	M3-2255.smp	219.6	26.7497
N14-1	N14-2272.smp	222.4	26.9279
N14-2	N14-2275.smp	222.9	27.1378
O2-1	O2-2289.smp	221.9	26.9164
O2-2	O2-2291.smp	224.1	26.8798
H8-1	H8-2238.smp	219.7	27.1682
H8-2	H8-2239.smp	223.6	27.2875
	$\overline{x}_{\mathrm{Hom}}$	220.503	26.75725

To obtain the inhomogeneity contribution u_{bb}, a 1-way Analysis of Variances (ANOVA) was carried out with the experimental homogeneity data. The inhomogeneity contribution is included in the total uncertainty budget of each porosity parameter [4] [13]. The u_{bb} values for BAM-P128 (see Table 5) were calculated in two different ways. For the specific pore volume V_p of the calculated uncertainty u_{bb} of the mean square between-unit is smaller than within-unit (see Table 3). Therefore, the u_{bb} value must be obtained from equation (2). In second case the uncertainty u_{bb} for pore diameter d_{50} is calculated with values of Table 4 and the equation (3).

Table 3: Analysis of Variances calculated for specific pore volume V_p

Source of Variation	Square sum	Degrees of freedom	Mean Square sum (MS)	StDev	F	F-crit. 95%
Between Units	52.4847	15	3.4990	MSB < MSW	0.6696	2.3522
Within Units	83.6050	16	5.2253	2.2859		

In case MSB < MSW use:

$$u_{bb} = \sqrt{\frac{MS_{within}}{n}} \cdot \sqrt[4]{\frac{2}{v_{MS_{within}}}} \qquad (2)$$

Table 4: Analysis of Variances calculated for median pore diameter d_{50}

Source of Variation	Square sum	Degrees of freedom	Mean Square sum (MS)	StDev	F	F-crit. 95%
Between Units	3.1835	15	0.2122	0.2787	3.7337	2.3522
Within Units	0.9095	16	0.0568	0.2384		

$$u_{bb} = \sqrt{\frac{MS_{\text{between}} - MS_{\text{within}}}{n}} \qquad (3)$$

Table 5: Inhomogeneity contributions u_{bb} of BAM-P128.

Property	u_{bb}	Unit
V_p	0.9611	mm^3/g
d_{50}	0.2787	µm

The statistical evaluation of the homogeneity testing results indicated that no significant inhomogeneity could be determined for the porosity parameters of BAM-P128. Therefore this candidate material has been suitable for the certification as BAM-P128.

4.2 Stability testing

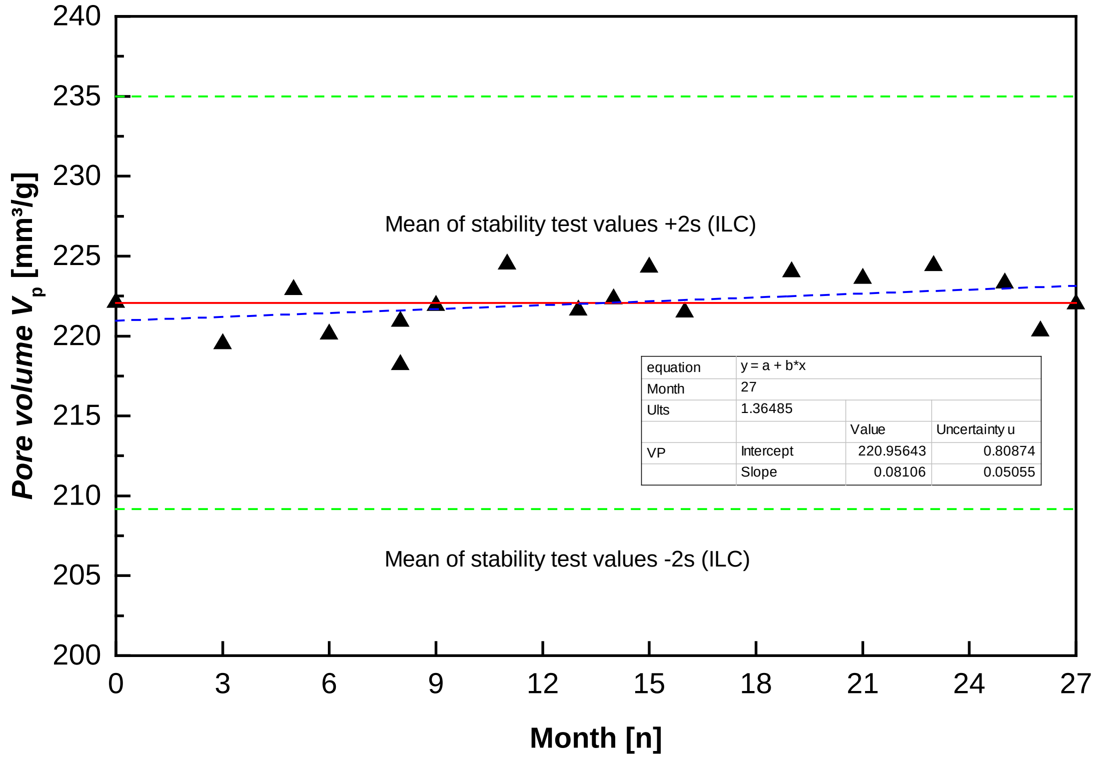
The results of the stability measurements of BAM-P128 are listed in Table 6 for the period between February 2013 and May 2015. The stability measurements were carried out with the same automated porosity analyzer AutoPore III (Micromeritics, Norcross USA) as for homogeneity testing. The respective diagrams for each porosity property are depicted in Fig. 6 and 7.

Table 6: Numerical results of stability monitoring.

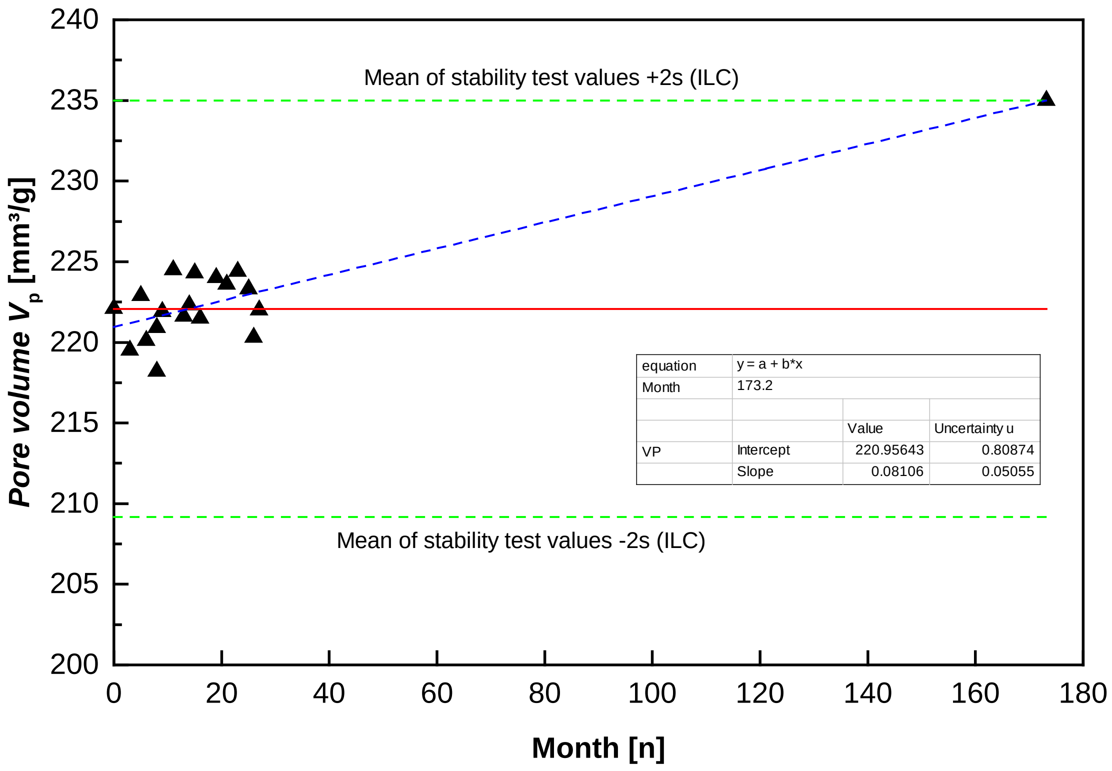
Data file	Test date	V_p	d_{50}
		mm³/g	µm
RP-2233.smp	13.02.2013	222.1	27.1415
RP-2322.smp	21.05.2013	219.5	26.3768
P20-2358.smp	15.07.2013	222.9	26.3359
P20-2372.smp	19.08.2013	220.1	26.1775
RP-2375.smp	09.10.2013	218.2	25.8603
RP-2382.smp	30.10.2013	220.9	25.8536
RP-2398.smp	25.11.2013	221.9	25.9901
RP-2406.smp	13.01.2014	224.5	26.0938
P20-2433.smp	05.03.2014	221.6	26.3097
RP-2465.smp	30.04.2014	222.3	25.8765
RP-2475.smp	12.05.2014	224.3	26.1185
RP-2523.smp	26.06.2014	221.5	26.1765
RP-2538.smp	03.09.2014	224.0	26.5285
RP-2558.smp	13.11.2014	223.6	26.3178
RP-2587.smp	06.01.2015	224.4	26.0726
RP-2593.smp	17.03.2015	223.3	26.1092
RP-2616.smp	07.04.2015	220.3	25.8575
RP-2637.smp	12.05.2015	222.0	26.1541
	$\bar{x}_{\text{Stab}}$ [a]	222.078	26.1861
	$\bar{x}_{\text{ILC}}$ [b]	219.865	27.6000
	$s_{x,\text{ILC}}$	6.459	1.3190
	$\bar{x}_{\text{Stab}} + 2 \cdot s_{x,\text{ILC}}$	234.996	28.8241
	$\bar{x}_{\text{Stab}} - 2 \cdot s_{x,\text{ILC}}$	209.160	23.5481

[a] Stab = stability monitoring

[b] ILC = interlaboratory comparison

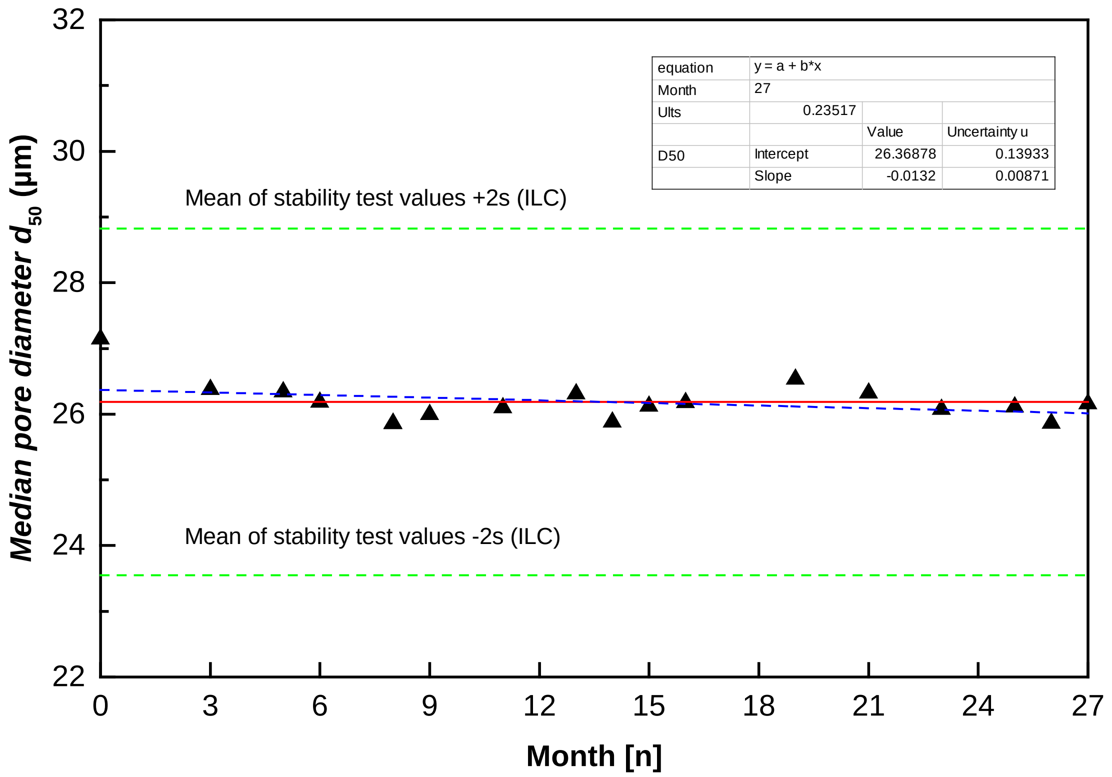


a)



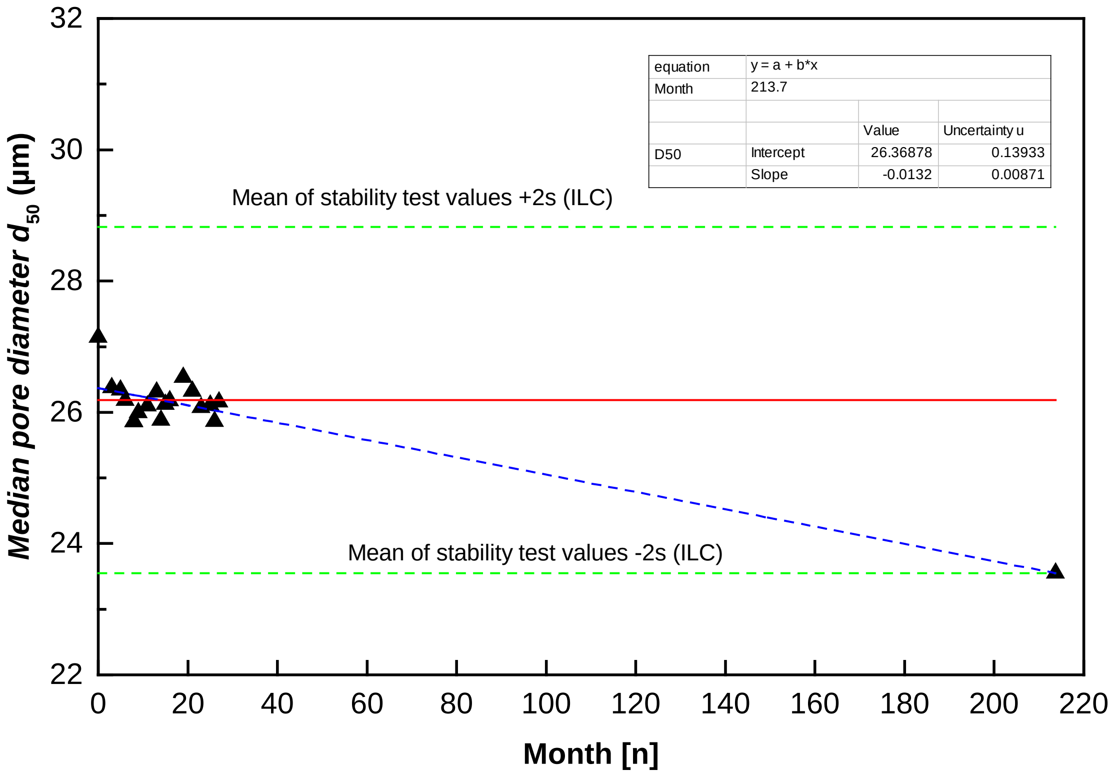
b)

Fig. 6: a) Stability monitoring for the porosity property V_p (specific pore volume). b) Estimation of long-term stability.



equation	y = a + b*x	
Month	27	
Ults	0.23517	
	Value	Uncertainty u
D50 Intercept	26.36878	0.13933
Slope	-0.0132	0.00871
Mean of stability test values +2s (ILC)
Mean of stability test values -2s (ILC)
Median pore diameter d_{50} (µm)
Month [n]

a)



equation	y = a + b*x	
Month	213.7	
	Value	Uncertainty u
D50 Intercept	26.36878	0.13933
Slope	-0.0132	0.00871
Mean of stability test values +2s (ILC)
Mean of stability test values -2s (ILC)
Median pore diameter d_{50} (µm)
Month [n]

b)

Fig. 7: a) Stability monitoring for the porosity property d_{50} (median pore diameter). b) Estimation of long-term stability.

The results of the statistical evaluation of the stability data (see Table 7) indicate that no instability could be detected for each porosity parameter. Nevertheless a contribution u_{lts} to the uncertainty of the certified values was included in the final calculation of the combined uncertainty on the basis of ISO Guide 35.

Table 7: Results of stability data evaluation according to ISO Guide 35.

Property	intercept	Slope	u(slope)	$u_{lts}(x)$	instability	Negligible uncertainty contribution
V_p	220.95643	0.08106	0.05055	1.36485	no	no
d_{50}	26.36878	-0.0132	0.00871	0.23517	no	no

The shelf life of BAM-P128, estimated on the basis of the stability monitoring data, is at least 27 months for storage of the carefully closed unit at temperatures below 25 °C under dry conditions.

Based on the present monitoring, a stability of at least 15 years is guaranteed based on the data summarized in Table 7. The stability will be monitored and documented periodically.

5. List of participating laboratories

AQura GmbH, Hanau (Germany)

AQura GmbH, Marl (Germany)

BAM Federal Institute for Materials Research and Testing, Div. 1.3, Berlin (Germany)

BAM Federal Institute for Materials Research and Testing, Div. 7.1, Berlin (Germany)

Forschungsinstitut für Anorganische Werkstoffe – Glas/Keramik- GmbH, Höhr-Grenzhausen (Germany)

Fraunhofer-Institut für Angewandte Polymerforschung IAP, Potsdam (Germany)

Fraunhofer-Institut für Solare Energiesysteme ISE, Freiburg (Germany)

Hoffmann & Co Elektrokohle AG, Bad Goisern (Austria)

Institut für Technische Chemie an der Universität Leipzig, Leipzig (Germany)

Leibniz-Institut für Oberflächenmodifizierung e.V., Leipzig (Germany)

Micromeritics GmbH, Aachen (Germany)

Micromeritics Instrument Corp., Norcross, GA (USA)

POROTEC GmbH, Hofheim (Germany)

Quantachrome GmbH, Odelzhausen (Germany)

Quantachrome Instruments, Boynton Beach (USA)

Rauschert Kloster Veilsdorf GmbH, Veilsdorf (Germany)

Technische Universität Dresden, Dresden (Germany)

Technische Universität München, München (Germany)

ThermoFisher Scientific, Milan (Italy)

Most of the laboratories had already participated in previous interlaboratory comparisons in the field of mercury intrusion porosimetry organized by BAM. Three laboratories took part for the first time. Therefore, these laboratories were asked to report their quality assurance of performed control measurements to check the instrument performance. The types of instruments used by the participants are listed in Table 8.

Table 8: Types of instruments used by the participants.

Type of instrument	Number	Manufacturer
Autopore III Autopore IV	1 4	Micromeritics Instrument Corporation, Norcross, GA (USA)
PASCAL 140	9	ThermoFisher Scientific, Milan (Italy)
Poremaster 33 Poremaster 60 Poremaster 60 GT	2 3 1	Quantachrome Instruments Corporation, Boynton Beach, FL (USA)
Total *	20	

* One laboratory participated with two instruments.

6. Results of the interlaboratory testing and uncertainty estimation

6.1 Experimental results

The interlaboratory comparison for the certification of BAM-P128 was performed on the basis of the Guidelines for the Production of BAM Reference Materials [3]. Data evaluation and statistical tests were carried out using the software package SoftCRM [14]. Each participating laboratory received closed units containing six pieces of the candidate material together with the instructions for running the tests and the data evaluation according to ISO 15901-1 [1] and DIN 66133 [2]. The laboratories had to perform five replicate measurements with each participating instrument. The mean values for the porosity parameters gained by each instrument are shown in Table 9 and displayed graphically in Fig. 8 and Fig. 9. The error bars of the data points for the data set mean represent the standard deviation of the two certification measurements per data set.

Table 9: Data set means as provided by the participants in the interlaboratory comparison (ILC).

Property $x \rightarrow$	V_p	d_{50}
Data set no. ↓	mm³/g	µm
01	236.60 [a]	26.7555
02	224.84	26.2267
03	213.41	28.342
04	220.58	29.511
05	217.44	26.28086
06	216.96	26.95
07	217.19	28.09307
08	223.08	26.07532
09	217.83	28.934
10	212.00	29.9764
11	223.36	26.21606
12	224.50	28.94824
13	215.23	28.28
14	222.86	26.91974
15	209.48	28.3424
16	237.44	26.086
17	223.44	27.148
18	212.08	26.32833
19	225.52	29.67402
20	220.20	26.912
l	19	20
$\bar{x}$ [b]	219.865	27.600
s_x [c]	6.459	1.319
$\frac{s_x}{\sqrt{l}}$	1.481	0.295
$\bar{x}+1\cdot s_x$	226.324	28.919
$\bar{x}-1\cdot s_x$	213.406	26.281
$\bar{x}+2\cdot s_x$	232.783	30.238
$\bar{x}-2\cdot s_x$	206.947	24.962

[a] value excluded for technical reasons., [b] average of the accepted data set means for the particular property on the basis of 5 single measurements each, [c] standard deviation of the data set means for the particular property.

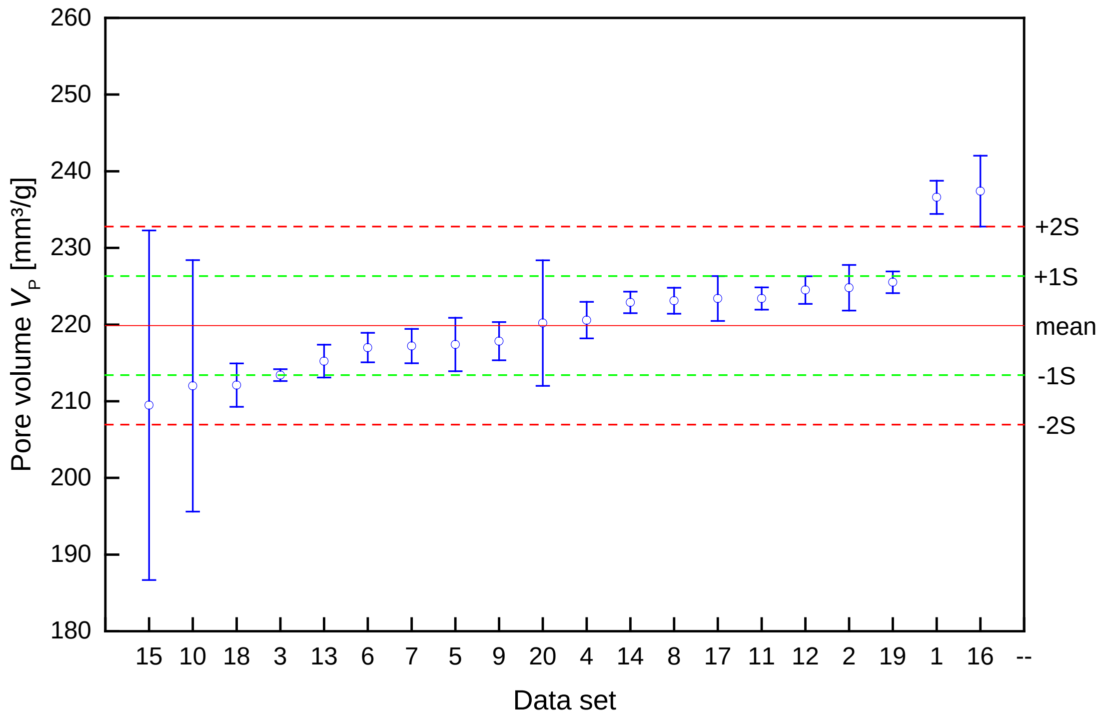


Fig. 8: Calculated means of data sets for V_p.

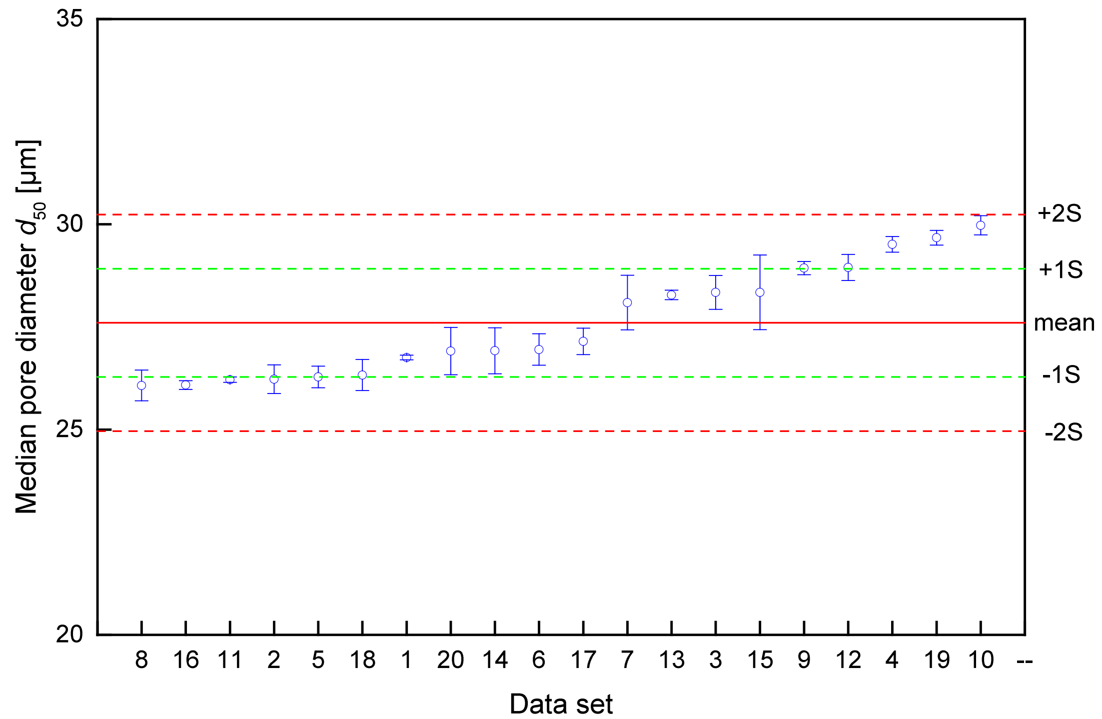


Fig. 9: Calculated means of data sets for d_{50}.

6.2 Statistical evaluation

According to ISO Guide 35 [4], an important aspect for the statistical treatment of the experimental data is to obtain the uncertainties of the certified values obtained with different instruments used by the different participating laboratories (see Table 8). Moreover, all participants in the interlaboratory study followed the same standardized procedure. Significant differences caused by different implementations in different laboratories were expected. This has been confirmed by the observation of very heterogeneous standard deviations indicating that the single experimental data did not belong to the same "mother distribution" and data pooling was not allowed. Therefore, the statistical treatment was performed using the laboratory mean values for each parameter to be certified.

The following statistical parameters were calculated:

- the mean of data set means
- the standard deviation of the distribution of laboratory means, and the standard deviation of the mean of laboratory means
- the confidence interval of the mean of laboratory means at the significance level of 0.05

The statistical tests carried out (at significance levels of 0.05 and 0.01) were:

- Cochran test for the identification of outliers with respect to laboratory variance
- Grubbs test for the identification of outliers with respect to the mean
- Dixon and Nalimov test for the verification of possible outlier indications
- Kolmogorov-Smirnov Test (Lilliefors version) for the normality test

As a result of the statistical analysis, two Nalimov stragglers were detected for the specific pore volume and Cochran outliers for both parameters under certification. Stragglers as well as Cochran outliers are retained in the data sets. For technical reason one Nalimov straggler was excluded. The results after deleting the outlying instrument mean for the accepted data are presented in Table 10.

Table 10: Statistical evaluation of the ILC data.

Property x	$\bar{x}$	s_x	u_{char}	CI	TI	Unit	Pooling	l
V_{p}	219.865	6.459	1.481	3.113	17.982	mm^3/g	no	19
d_{50}	27.600	1.319	0.295	0.617	3.630	µm	no	20

The plausibility of the obtained means of the instrument means has been checked by comparison between the results of the homogeneity measurements, the stability study, and the ILC measurements (see Table 11).

Table 11: Plausibility comparison of the mean values obtained from different tests.

Property x	Mean values from homogeneity test	Mean values from stability test	Mean values from ILC	Plausibility remark
V_p	220.503	222.078	219.865	compatible within the corresponding uncertainties
d_{50}	26.757	26.186	27.600	

The combined uncertainty $u_c(x)$ for each certified value was calculated according to Equation (4) using the numerical values summarized in Table 12 as the combination of the standard uncertainty of the mean of the instrument means, the contribution of the variation between the units, the long term stability contribution, and the uncertainty contribution due to the measurement result variations of the single instruments (mean data set precision).

$$u_c^2(x) = u_{char}^2 + u_{bb}^2 + u_{lts}^2 + \frac{1}{l^2}\sum_{i=1}^{l} s_i^2 \qquad \text{with} \qquad u_{char}^2 = \frac{s_x^2}{l} \tag{4}$$

Table 12: Values of the uncertainty components for the porosity parameters of BAM-P128.

Property	$\overline{x}$	$u_{char}(x)$	$u_{bb}(x)$	$u_{lts}(x)$	$\frac{1}{l}\sqrt{\sum_{i=1}^{l} s_i^2}$	$u_c(x)$	$U(x)$	l	Unit
V_p	219.865	1.481	0.9611	1.36485	1.62491	2.76048	5.52095	19	mm^3/g
d_{50}	27.600	0.295	0.2787	0.23517	0.08792	0.47721	0.95443	20	µm

The certified values of each porosity parameter with a reasonable number of digits and the respective expanded uncertainties (rounded according to DIN 1333 [15]) are summarized in Table 1.

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