Certification Report

Certified Reference Material

BAM-P115

Pore size parameters of nanoporous Titanium dioxide calculated from the nitrogen sorption isotherm at 77.3 K

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Summary

This report describes the certification of the porous reference material BAM-P115. The certified values determined by nitrogen adsorption and desorption at 77.3 K according to the international standards ISO 9277 [1] and ISO 15901-2 [2] are summarized in the table below.

Property		Value	U [a]	$2 \cdot s_x$ [b]	Unit
Specific Surface Area [c]	A_{BET}	147.3	2.8	7.3	m^2/g
Specific pore volume [d]	$V_{p,0.99}$	0.214	0.004	0.007	cm^3/g
Hydraulic pore diameter	$4V_{p,0.99}/A_{BET}$	5.79	0.07	0.07	nm
Modal pore diameter	$D_{BJH,des}$ [e]	4.75	0.21	0.46	nm
Modal pore diameter	$D_{BJH,ads}$ [f]	5.40	0.24	0.61	nm

[a] Uncertainty $U = k \cdot u_c$ calculated according to ISO Guide 35 [3] and ISO/IEC Guide 98-3 [4] with the coverage factor $k = 2$ (giving a level of confidence of approximately 95%). The value of the combined standard uncertainty u_c of the certified property includes both an uncertainty contribution resulting from the inter-laboratory characterization, the study of inhomogeneities, stability of the material and the uncertainty contribution due to variation in the measurement results from individual instruments (mean data set).

[b] Standard deviation of accepted laboratory mean values (reported as twice the standard deviation).

[c] Specific surface area calculated in a relative adsorption pressure range of $0.05 \le p/p_0 < 0.3$ as multi point BET model [5] with minimum of five points as described in ISO 9277.

[d] Single point total pore volume according to the Gurvich rule [6] determined from the adsorption branch of the isotherm at relative pressure $p/p_0 = 0.990$

[e] Pore size at maximum of the differential pore size distribution calculated from the desorption branch of the isotherm applying the BJH model [7] as described in ISO 15901-2

[f] Pore size at maximum of the differential pore size distribution calculated from the adsorption branch of the isotherm applying the BJH model [7] as described in ISO 15901-2

This report contains detailed information on the preparation of the CRM as well as on homogeneity investigation and on the analytical methods used for certification analysis.
The certified values are based on the results of 13 laboratories which participated in the certification inter-laboratory comparison.

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List of abbreviations

(if not explained elsewhere)

ANOVA	analysis of variance
BET	Brunauer, Emmett, Teller (method)
BJH	Barrett, Joyner, Halenda (method)
CI	confidence interval
CRM	certified reference material
GUM	ISO guide to the expression of uncertainty in measurement
ILC	inter-laboratory comparison (certification round robin)
RM	reference material

List of Symbols

df	degrees of freedom
k	coverage factor
l	number of accepted data sets in the inter-laboratory comparison
M	mean square sum
n	number of observations
p	pressure of the adsorptive in equilibrium with the adsorbate
p_0	saturation vapor pressure of the adsorptive
s_{bb}	standard uncertainty due to between-bottle (in)homogeneity
s_i	standard deviation of single data set
s_x	ILC standard deviation of a property value
U	expanded standard uncertainty of a property value
u'_{bb}	estimated standard uncertainty due to within bottle (in)homogeneity
u_{char}	standard uncertainty due to characterization
u_c	combined standard uncertainty of a property value
u_{hom}	standard uncertainty due to (in) homogeneity
u_{lts}	standard uncertainty due to long-term (in)stability
x	property value of a candidate material
x_{cert}	certified property value of a CRM

1. Introduction

The Certified Reference Material BAM-P115 is a nanoporous titanium dioxide (anatase) and intended for use in the calibration and performance testing of gas sorption instruments to check the values of the BET surface area, pore volume and pore size.

BAM-P 115 has been developed as an appropriate successor material for the ZRM BAM-P106, which is sold out due to a high number of inquiries.

With this material it is possible to validate gas sorption instruments in test laboratories and to carry out monitoring that is required according to ISO/IEC 17025 [8].

The certification of this CRM has been carried out based on BAM Management Manual; MH - 3.5 Production of reference materials [9], ISO 17034 [10] and relevant ISO Guides [3], [11].

2. Material

2.1 Selection and characterization of the candidate material

A nanoporous titanium dioxide (TiO_2, also called titania) in the modification anatase was selected from several tested candidate materials.
The titania candidate material TP Hombikat (Lot 10588097-5) was delivered by Venator Germany GmbH and consists of pure anatase according to the manufacturer's specifications.
Measurements with mercury porosimetry (Fig. 1) indicated two main pore sizes. In the macro pore range with 32 µm and in meso pore range with 7 nm where the mesopores are the focus of the certification. As additional properties a pore volume of 535 mm^3/g and a porosity of approximately 66% were measured.
As a result of XRD measurements at BAM, Division 6.3, it could be confirmed that the crystal modification of TP Hombikat (Lot 10588097-6) is pure anatase (see Fig. 2). The particle size was determined to be 30 µm, which is in accordance with the SEM data (Fig. 3).
Although anatase is metastable and thermodynamically less stable than rutile (the main modification of TiO_2), the phase conversion rate of anatase into rutile is virtually zero at temperatures up to about 873 K. Therefore, the long-term stability of anatase modification is not affected in the temperature range between room temperature and the recommended degassing temperature of 453.15 K (see Chapter 4. Stability).

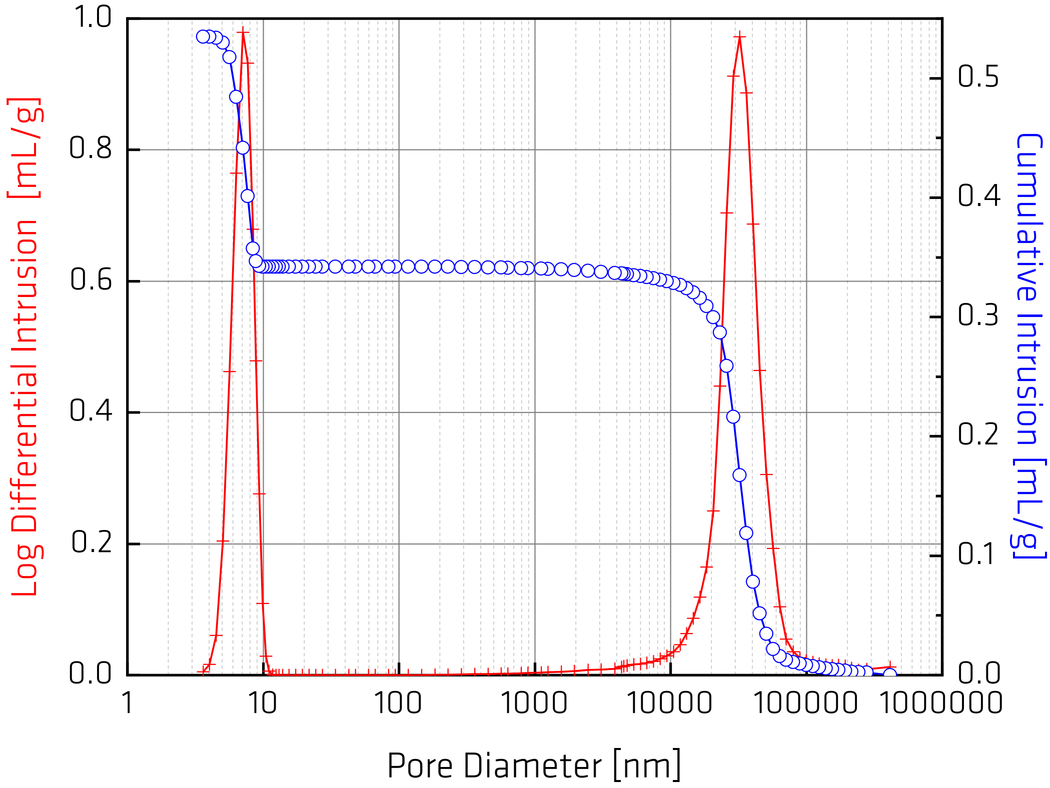

Fig. 1: Determination of pore size, pore volume and porosity by mercury porosimetry of the candidate material.

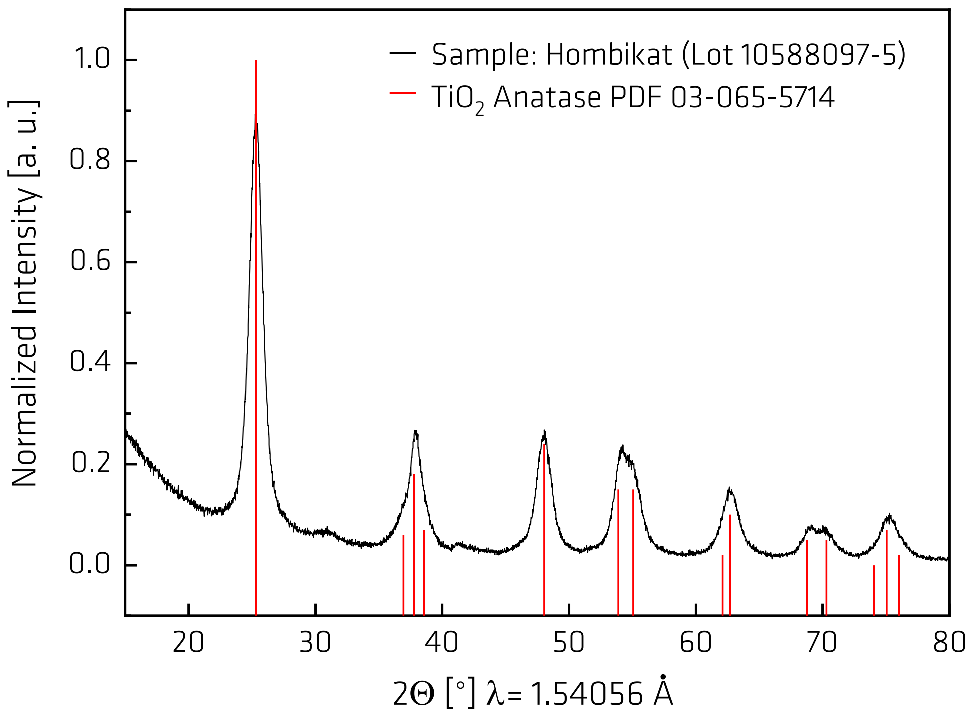

Fig. 2: X-ray powder diffraction pattern of the candidate material and comparison with the database entry powder diffraction profile [12] of titanium dioxide (anatase).

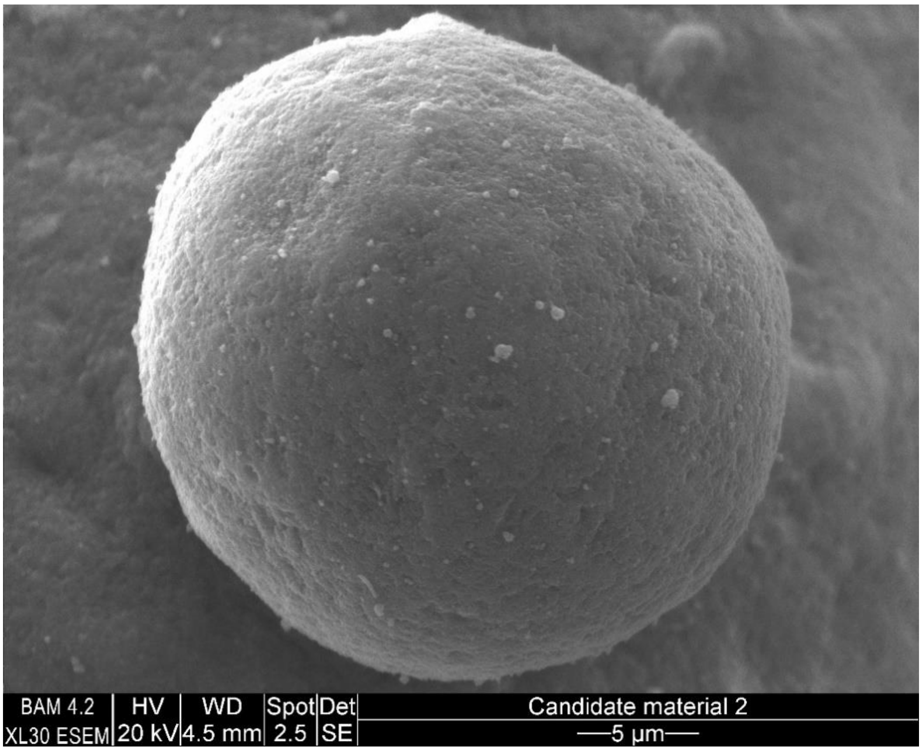

Fig. 3: SEM image of a particle of the candidate material (CRM BAM-P115) with a particle size of 30 µm.

2.2 Porosity characterization

Prior to the measurement, outgassing of the sample is necessary. Outgassing must be carried out in vacuum with a final pressure of < 10 Pa. For degassing in vacuum, the sample is to be heated with a rate of about 5 K/min to 453.15 K, then this temperature must be maintained for at least three hours. Afterwards, the sample to be allowed to slowly cool down to ambient temperature.

After sample preparation the pore size analysis is carried out with the gas adsorption method. This includes measurement of a low temperature physisorption isotherm (see Fig. 4) with a starting pressure of < 0.1 Pa. By means of appropriate models, a number of porosity parameters can be calculated from the isotherm data such as:

- the specific surface area A_{BET} in m^2/g according to the BET model of Brunauer, Emmett, and Teller [5] (see Fig. 5),
- the total mesopore volume $V_{\mathrm{p,0.99}}$ according to the Gurvich rule [6],
- the hydraulic pore diameter $4V_{\mathrm{p,0.99}}/A_{\mathrm{BET}}$,
- the pore diameter $D_{\mathrm{BJH,des}}$ according to the BJH method [7] of Barrett, Joyner and Halenda from the desorption branch of the isotherm (see Fig. 4 and 6),
- the pore diameter $D_{\mathrm{BJH,ads}}$ from the adsorption branch of the isotherm (see Fig. 4 and 7).

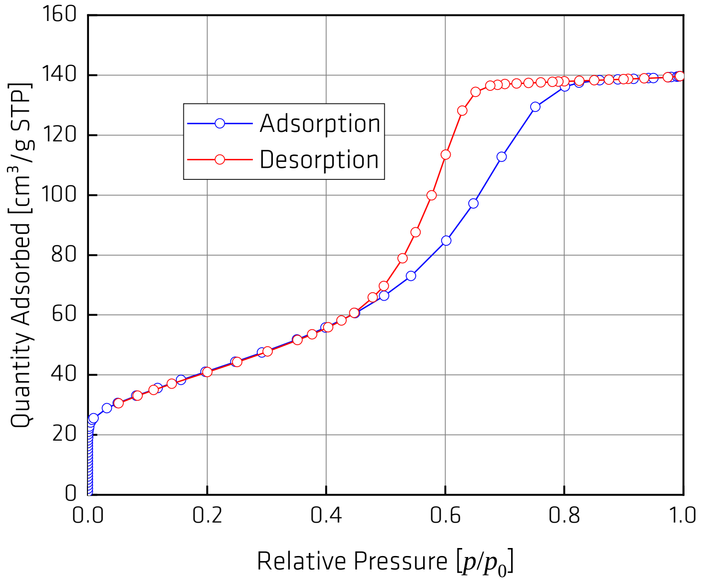

Fig. 4:

N_2 isotherm of BAM-P115 at 77.3 K

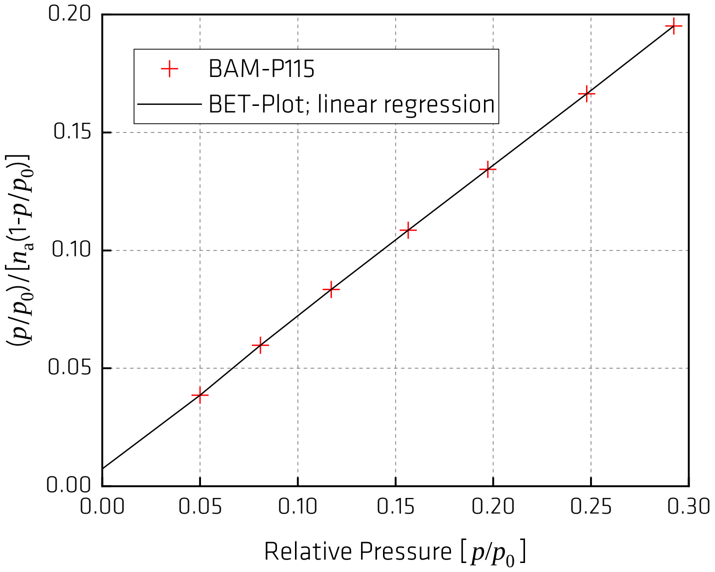

Fig. 5:

BET – Plot of BAM-P115

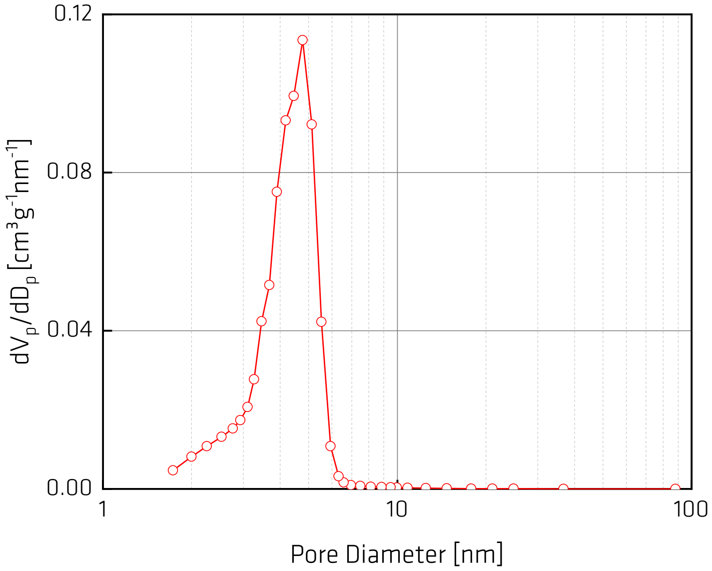

Fig. 6:

BJH pore volume distribution from the desorption branch of the N_2 isotherm

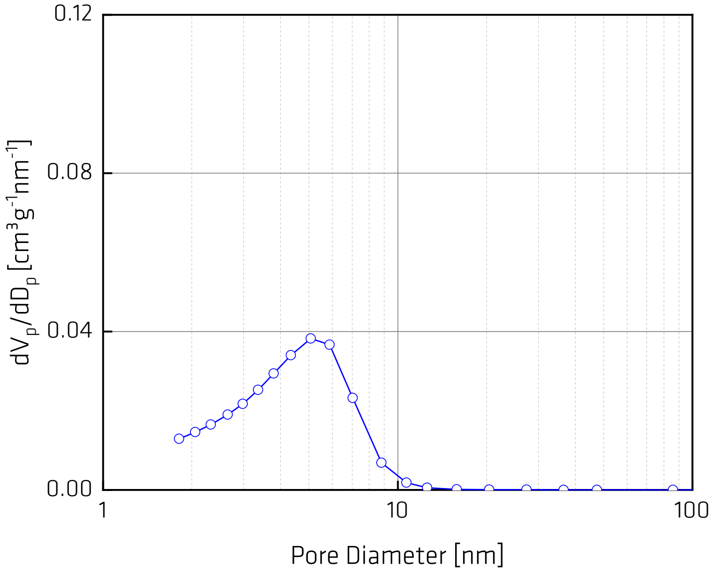

Fig. 7:

BJH pore volume distribution from the adsorption branch of the N_2 isotherm

2.3 Homogenization and bottling of the candidate material

Homogenization and subdividing of the sample material were carried out by means of an eight-port rotary sample divider PT 100 (Retsch, Germany) using the cross-riffling scheme [13] shown in Fig. 8.

Fig. 8: Cross riffling scheme used for subdividing the samples.

1	2	3	4	5	6	7	8		
⇓	⇓	⇓	⇓	⇓	⇓	⇓	⇓		
1-8	2-7	3-6	4-5	5-4	6-3	7-2	8-1	⇒	H
1-7	2-6	3-5	4-4	5-3	6-2	7-1	8-8	⇒	G
1-6	2-5	3-4	4-3	5-2	6-1	7-8	8-7	⇒	F
1-5	2-4	3-3	4-2	5-1	6-8	7-7	8-6	⇒	E
1-4	2-3	3-2	4-1	5-8	6-7	7-6	8-5	⇒	D
1-3	2-2	3-1	4-8	5-7	6-6	7-5	8-4	⇒	C
1-2	2-1	3-8	4-7	5-6	6-5	7-4	8-3	⇒	B
1-1	2-8	3-7	4-6	5-5	6-4	7-3	8-2	⇒	A

The initial amount of titanium dioxide powder (in its anatase polymorphic form) was subdivided into single units of at least 12 g and packaged in glass bottles of 30 mL volume. A total of 384 units were prepared.
24 out of these 384 units were used for testing the homogeneity and stability as well as for the interlaboratory comparison.

3. Homogeneity

For testing the homogeneity, 10 individual units of BAM-P115 were randomly selected. Three replicate measurements per bottle were carried out under identical conditions with the automated surface area and porosity analyzer ASAP 2020 (Micromeritics, Norcross USA) to detect the standard deviation between and within the bottles. The test results are summarized in Table 1 and the homogeneity contribution to the total uncertainty was calculated using a 1-way-ANOVA (see Annex 1).
To estimate the analyte-specific inhomogeneity contribution, u_{hom}, to the total uncertainty, two approaches were compared, Equations (1) and (2), according to ISO Guide 35 [3]. The approach which yielded the highest uncertainty value was included in the final calculation of u_{hom}.

$$s_{\text{bb}} = \sqrt{\frac{M_{between} - M_{within}}{n}} \quad (1)$$

$$u'_{bb} = \sqrt{\frac{M_{within}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \quad (2)$$

with

$M_{between}$	mean of squared deviations between bottles (from 1-way ANOVA)
M_{within}	mean of squared deviations within one bottle (from 1-way ANOVA)
n	number of replicate measurements per bottle
N	number of bottles selected for homogeneity study

In calculations of s_{bb} where $M_{within} > M_{between}$ the inhomogeneity contribution to the combined uncertainty was set to zero.

Table 1: Results of three replicate measurements on samples from a single bottle

Bottle	Data file	A_{BET}	$V_{p,0.99}$	$4V_p/A_{BET}$	$D_{BJH(des)}$	$D_{BJH(ads)}$
		m^2/g	cm^3/g	nm	nm	nm
A2(01) - 009	Hom-1547.smp	149.46	0.2162	5.79	4.60	5.22
	Hom-1548.smp	148.92	0.2155	5.79	4.61	5.23
	Hom-1549.smp	149.80	0.2175	5.81	4.55	5.30
B3(05) - 069	Hom-1557.smp	149.22	0.2170	5.82	4.60	5.31
	Hom-1558.smp	148.91	0.2149	5.77	4.53	5.22
	Hom-1559.smp	148.41	0.2149	5.79	4.58	5.26
H2(02) - 346	Hom-1560.smp	147.76	0.2147	5.81	4.60	5.29
	Hom-1561.smp	148.61	0.2148	5.78	4.59	5.33
	Hom-1562.smp	148.53	0.2168	5.84	4.58	5.22
D5(08) - 184	Hom-1563.smp	148.64	0.2164	5.82	4.58	5.27
	Hom-1564.smp	148.40	0.2155	5.81	4.60	5.23
	Hom-1565.smp	148.88	0.2164	5.81	4.59	5.32
E6(04) - 236	Hom-1566.smp	148.22	0.2145	5.79	4.57	5.25
	Hom-1567.smp	148.06	0.2144	5.79	4.61	5.29
	Hom-1568.smp	148.57	0.2160	5.82	4.61	5.32
E2(06) - 206	Hom-1570.smp	148.73	0.2162	5.81	4.58	5.25
	Hom-1572.smp	148.75	0.2160	5.81	4.61	5.24
	Hom-1573.smp	148.54	0.2150	5.79	4.58	5.25
C1(03) - 099	Hom-1574.smp	148.69	0.2157	5.79	4.57	5.26
	Hom-1575.smp	147.89	0.2143	5.80	4.53	5.24
	Hom-1576.smp	148.31	0.2166	5.84	4.61	5.24
G4(01) - 313	Hom-1577.smp	149.58	0.2172	5.81	4.61	5.25
	Hom-1579.smp	149.24	0.2158	5.78	4.59	5.26
	Hom-1581.smp	148.73	0.2153	5.79	4.57	5.28
A3(04) - 020	Hom-1582.smp	148.82	0.2154	5.79	4.53	5.28
	Hom-1583.smp	148.71	0.2153	5.79	4.53	5.23
	Hom-1584.smp	148.54	0.2159	5.81	4.61	5.25
C5(08) - 136	Hom-1585.smp	148.67	0.2159	5.81	4.54	5.23
	Hom-1586.smp	149.02	0.2172	5.82	4.61	5.23
	Hom-1587.smp	149.30	0.2174	5.82	4.60	5.28

Table 2: Analysis of variances calculated for specific surface area A_{BET}

Property	Source of variation	Square sum	Degrees of freedom (df)	Mean square sum (M)	F	F-crit. 95%
A_{BET}	Between Units	3.9613	9	0.4401	3.7056	2.3928
	Within Units	2.3756	20	0.1188		
$V_{p,0.99}$	Between Units	7.7849E-06	9	8.6499E-07	1.0266	2.3928
	Within Units	1.6852E-05	20	8.4261E-07		
$4V_p/A_{BET}$	Between Units	2.3734E-03	9	2.6371E-04	0.8759	2.3928
	Within Units	6.0216E-03	20	3.0108E-04		
$D_{BJH(des)}$	Between Units	4.2700E-03	9	4.7444E-04	0.5311	2.3928
	Within Units	1.7867E-02	20	8.9333E-04		
$D_{BJH(ads)}$	Between Units	5.9367E-03	9	6.5963E-04	0.5422	2.3928
	Within Units	2.4333E-02	20	1.2167E-03		

Table 3: Determination of the maximum inhomogeneity contribution u_{hom} of BAM-P115

Property	Inhomogeneity contribution s_{bb}	Inhomogeneity contribution u'_{bb}
A_{BET}	0.327295	0.111894
$V_{p,0.99}$	8.63729E-05	0.000298
$4V_p/A_{BET}$	0	0.005634
$D_{BJH(des)}$	0	0.009704
$D_{BJH(ads)}$	0	0.011325

Table 4: Inhomogeneity contribution u_{hom}

Property	u_{hom}	$u_{hom\ rel.}$	Unit
A_{BET}	0.327295	0.002201	m^2/g
$V_{p,0.99}$	0.000298	0.001381	cm^3/g
$4V_p/A_{BET}$	0.005634	0.000971	nm
$D_{BJH(des)}$	0.009704	0.002118	nm
$D_{BJH(ads)}$	0.011325	0.002153	nm

The statistical evaluation of the homogeneity testing results indicated that no significant inhomogeneity for the porosity parameters of the titania candidate material TP Hombikat could be determined.

4. Stability

Numerical results of the measurements used to monitor the stability of CRM BAM-P115 for the period between November 2018 and June 2020 are listed in Table 5. The stability measurements were carried out using the same sample preparation procedure and instrument as used for homogeneity testing. The respective diagrams for each porosity property are displayed in Figs. 9 – 13.

Table 5: Numerical results of stability monitoring

Data file	Test date	A_{BET}	$V_{p,0.99}$	$4V_p/A_{BET}$	$D_{BJH(des)}$	$D_{BJH(ads)}$
		m^2/g	cm^3/g	nm	nm	nm
Hom-1507.smp	22.11.2018	148.90	0.2152	5.79	4.76	5.33
Hom-1536.smp	18.12.2018	148.80	0.2152	5.78	4.55	5.25
Hom-1547.smp	17.01.2018	149.46	0.2162	5.79	4.60	5.22
Hom-1568.smp	15.02.2019	148.57	0.2160	5.82	4.61	5.32
Hom-1587.smp	15.03.2019	149.30	0.2174	5.82	4.60	5.28
Hom-1599.smp	05.04.2019	148.67	0.2146	5.77	4.58	5.23
Hom-1615.smp	22.05.2019	148.97	0.2160	5.80	4.54	5.30
Hom-1625.smp	07.06.2019	149.30	0.2142	5.74	4.60	5.25
CM2-1635.smp	25.06.2019	147.79	0.2143	5.80	4.60	5.28
Hom-1682.smp	02.08.2019	150.38	0.2143	5.70	4.79	5.32
Hom-1720.smp	15.10.2019	150.16	0.2147	5.72	4.83	5.34
115-1731.smp	22.11.2019	148.31	0.2124	5.73	4.79	5.30
115-1754.smp	13.01.2020	149.12	0.2142	5.75	4.80	5.30
115-1783.smp	04.02.2020	149.82	0.2152	5.75	4.75	5.22
115-1823.smp	12.05.2020	148.05	0.2139	5.78	4.80	5.30
115-1835.smp	04.06.2020	150.01	0.2159	5.76	4.77	5.07
	$\bar{x}_{Stab}$ [a]	149.10	0.2150	5.77	4.69	5.27
	$\bar{x}_{ILC}$ [b]	147.26	0.2142	5.79	4.75	5.40
	$s_{x,ILC}$	3.63	0.0035	0.03	0.31	0.23
	$\bar{x}_{Stab} + 2 \cdot s_{x,ILC}$	156.36	0.2220	5.83	5.31	5.73
	$\bar{x}_{Stab} - 2 \cdot s_{x,ILC}$	141.84	0.2080	5.71	4.07	4.81

[a] Stab = stability monitoring
[b] ILC = inter-laboratory comparison (certification study)

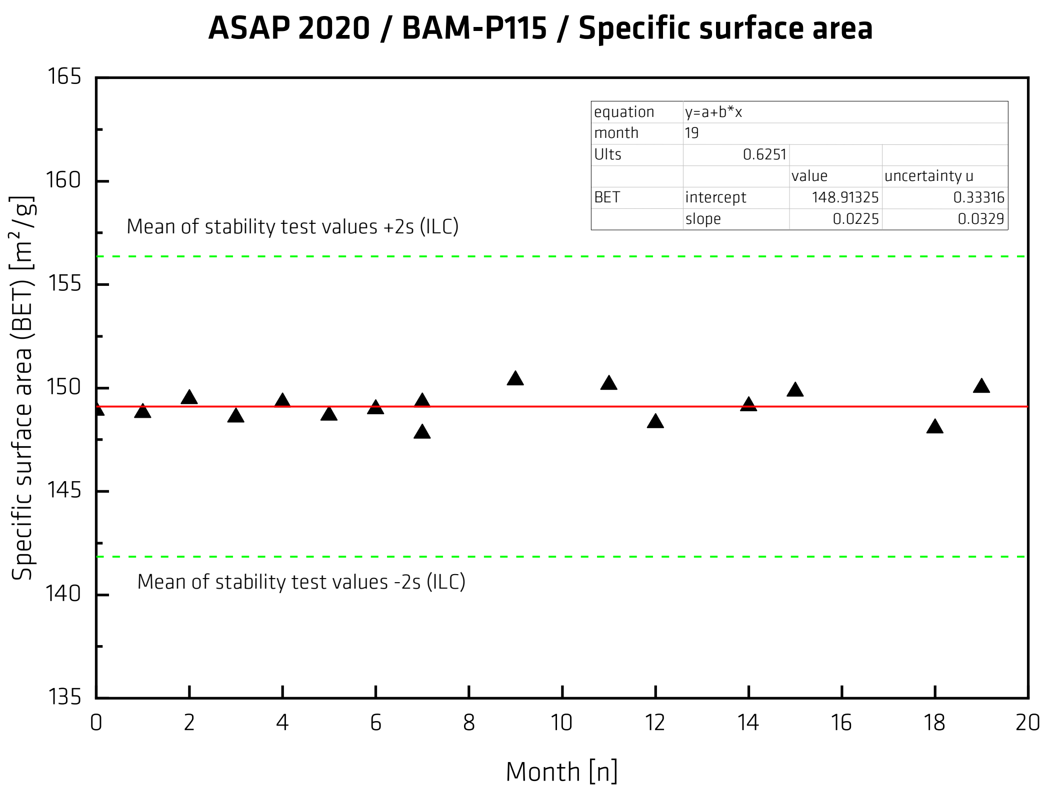

Fig. 9: Stability monitoring for the specific surface area A_{BET}

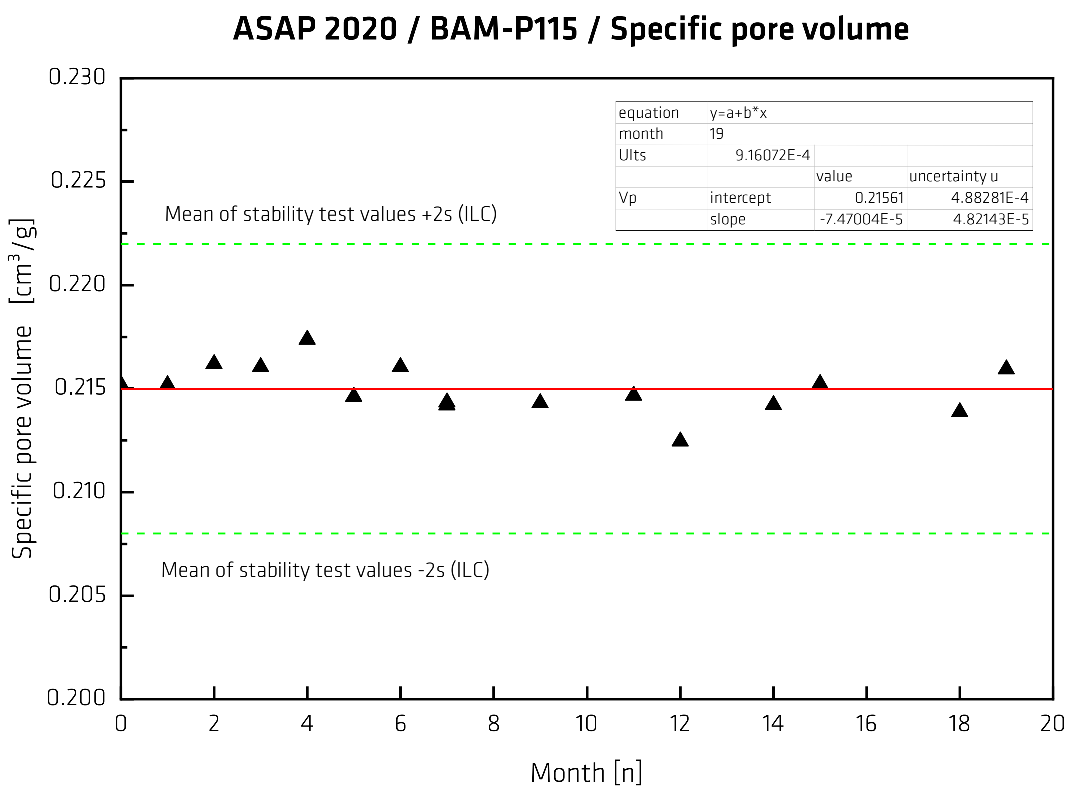

Fig. 10: Stability monitoring for the porosity property $V_{p,0.99}$ (specific pore volume)

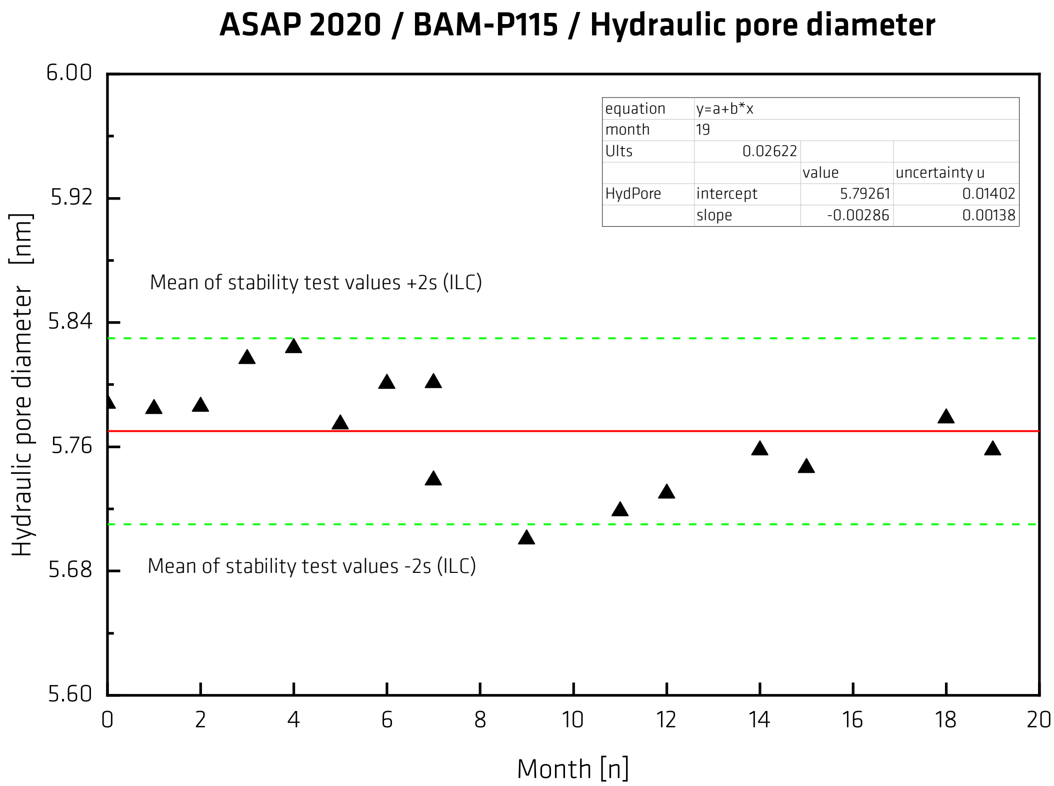

Fig. 11: Stability monitoring for the porosity property $4V_{p,0.99}/A_{BET}$ (hydraulic pore diameter)

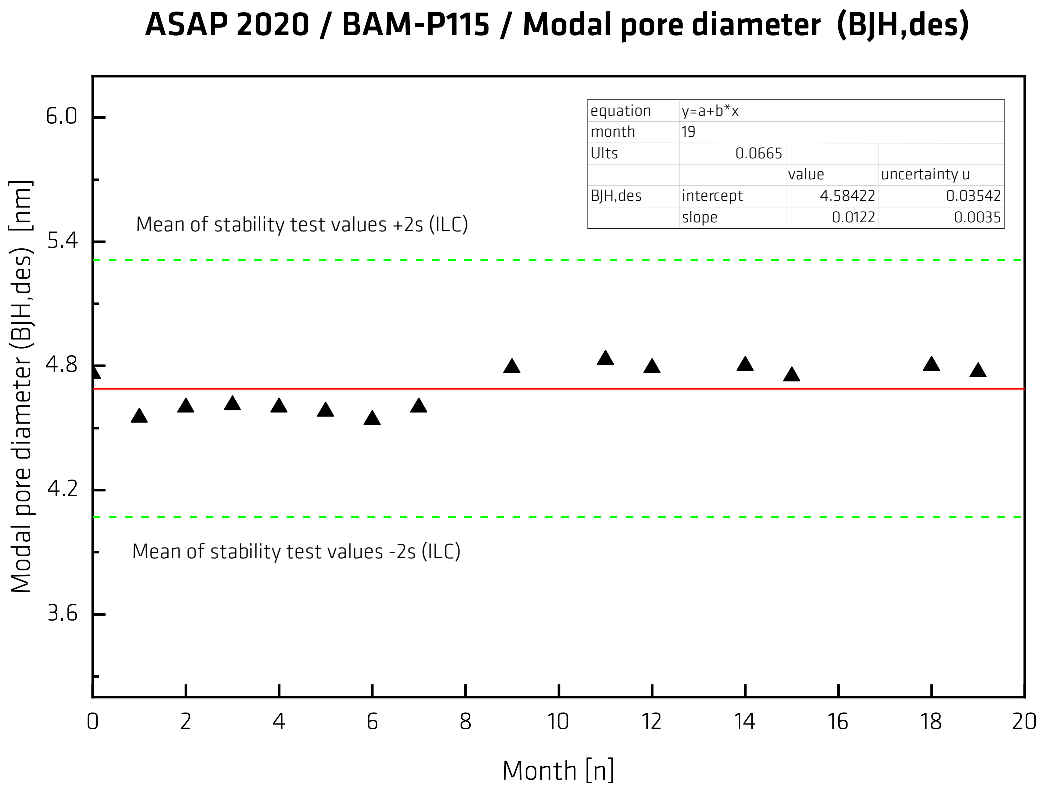

Fig. 12: Stability monitoring for the porosity property $D_{BJH(des)}$ (most frequent BJH pore diameter calculated from the desorption branch of the N_2 isotherm)

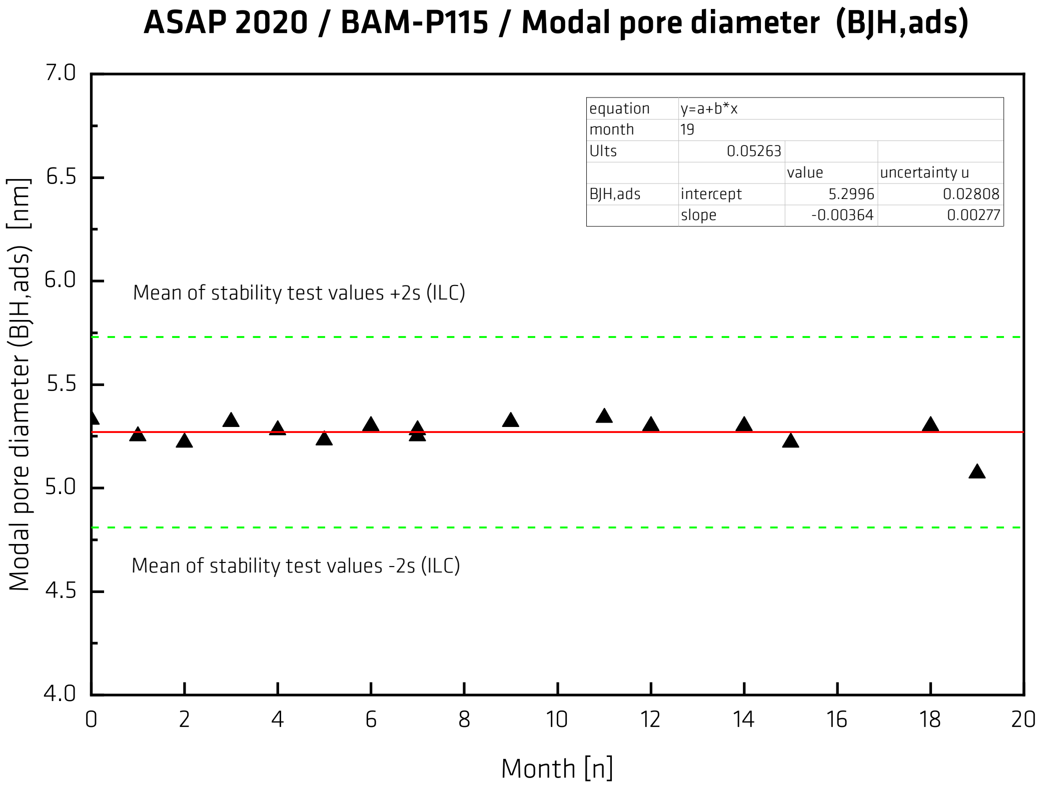

Fig. 13: Stability monitoring for the porosity property $D_{BJH(ads)}$ (most frequent BJH pore diameter calculated from the adsorption branch of the N_2 isotherm)

The results of the statistical evaluation of the stability data (see Table 6) indicate that no instability could be detected. However, the contribution of u_{lts} to the uncertainty of the certified value is not negligible and was therefore included in the final calculation of the combined uncertainty.

Table 6: Results of stability data evaluation according to ISO Guide 35

Property	Intercept	Slope	u(slope)	u_{lts}	$u_{lts\ rel}$	Instability
A_{BET}	148.91325	0.02250	0.03290	0.62510	0.00419	no
$V_{p,0.99}$	0.21561	-0.000075	0.000048	0.000916	0.00426	no
$4V_p/A_{BET}$	5.79261	-0.00286	0.00138	0.02622	0.00454	no
$D_{BJH(des)}$	4.58422	0.01220	0.00350	0.06650	0.01418	no
$D_{BJH(ads)}$	5.29960	-0.00364	0.00277	0.05263	0.00999	no

The shelf life of CRM BAM-P115 as estimated based on the stability monitoring data is at least two years for a carefully closed bottle stored at temperatures below 30°C under dry conditions.

5. List of participating laboratories

3P INSTRUMENTS GmbH & Co. KG, Odelzhausen (Germany)

Anton Paar QuantaTec Inc., Boynton Beach (USA)

Bundesanstalt für Materialforschung und –prüfung (BAM), Div. 6.3, Berlin (Germany)

Bundesanstalt für Materialforschung und –prüfung (BAM), Div. 5.4, Berlin (Germany)

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin (Germany)

Institute of Chemical and Engineering Sciences (ICES), Jurong Island (Singapore)

Instituto Nacional de Técnica Aeroespacial, Madrid (Spain)

Instituto Pedro Nunes, Coimbra (Portugal)

Particle Testing Authority - Micromeritics GmbH, Unterschleißheim (Germany)

Particle Testing Authority - Micromeritics Instrument Corp., Norcross, GA (USA)

University Industry Research Laboratory (UIRL), Johor Bahru (Malaysia)

Ural Scientific Research Institute for Metrology (UNIIM), Yekaterinburg (Russian Federation)

28 laboratories were invited to participate in the inter-laboratory comparison. 12 laboratories agreed to participate and submitted results. One laboratory participated with two instruments.

Most of the participating laboratories had already participated in previous inter-laboratory comparisons in the field of gas adsorption measurements. In addition, the laboratories confirmed that their quality management systems were in accordance with ISO/IEC 17025 or ISO 17034. The types of instruments used by the participants are listed in Table 7.

Table 7: Types of instruments used by the participants

Type of instrument	Number	Manufacturer
3P-Meso222	1	3P INSTRUMENTS GmbH & Co. KG
ASAP 2000	1	Micromeritics Instrument Corporation, Norcross, USA
ASAP 2010	1	
ASAP 2020	2	
ASAP 2420	1	
TriStar II Plus 3030	1	
TriStar II Plus	1	
Autosorb-iQ	1	Anton Paar QuantaTec Inc., Boynton Beach, USA
Autosorb-iQ 2	2	
NOVA 2000e	1	
Surfer	1	Thermo Fisher Scientific S.p.A. Milan, Italy
Total	13	

6. Results of the interlaboratory comparison and uncertainty estimation

6.1 Experimental results

The inter-laboratory comparison for the certification of BAM-P115 was performed according to the BAM Management Manual; MH - 3.5 Production of reference materials [9]. Data evaluation and statistical tests were carried out using the software package SoftCRM [14].

The candidate material for the participants was prepared from a mixture of 14 randomly selected bottles from the entire charge (see section 2.3). These bottles were merged and divided by means of an eight-port micro rotary riffler (Quantachrome, Germany) into units of approximately 8 g. The reduction of the original sample quantity was carried out in order to provide the participants with an optimal sample quantity for the analyses. A sample quantity of 0.8 g was planned for each analysis.

Each participating laboratory received one bottle together with the instructions for sample preparation and instrumental analysis. The data were evaluated according to ISO 9277 and ISO 15901-2. 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 8 and displayed graphically in Figs. 14 - 19. The error bars for the data set means represent the standard deviation of the measurements per data set.

Table 8: Data set means of the participants in the inter-laboratory comparison (ILC)

Property (x)→	A_{BET}	$V_{p,0.99}$	$4V_p/A_{BET}$	$D_{BJH(des)}$	$D_{BJH(ads)}$
Data set no.↓	m^2/g	cm^3/g	nm	nm	nm
C01	143.77	0.2093	5.827	4.414	4.913
C04	150.17	0.2146	5.735	4.548	4.936
C06	148.62	0.2152	5.791	4.560	5.100
C07	143.75	0.2085	5.802	4.578	5.292
C08	151.05	0.2169	5.744	4.585	5.304
C10	148.67	0.2160	5.808	4.619	5.324
C11	144.73	0.2108	5.826	4.805	5.328
C13	139.20	0.2008 [a]	5.766	4.805	5.417
C15	145.59	0.2116	5.812	4.809	5.523
C17	149.83	0.2190	5.847	4.884	5.625
C19	151.46	0.2181	5.760	4.901	5.719
C21	147.36	0.2134	5.793	5.011	5.781
C27	150.25	0.2172	5.782	5.240	5.884
l	13	12	13	13	13
$\bar{x}$ [b]	147.26	0.2142	5.792	4.751	5.396
s_x [c]	3.63	0.0035	0.034	0.228	0.306
$\frac{s_x}{\sqrt{l}}$	1.01	0.0010	0.009	0.063	0.085
$\bar{x}+1\cdot s_x$	150.90	0.2177	5.825	4.979	5.702
$\bar{x}-1\cdot s_x$	143.63	0.2107	5.758	4.523	5.090
$\bar{x}+2\cdot s_x$	154.53	0.2212	5.859	5.206	6.007
$\bar{x}-2\cdot s_x$	140.00	0.2072	5.725	4.295	4.785

[a] Insufficient data set mean for the particular property statistically detected as outlier and are therefore not included in the calculation

[b] Average of the accepted data set means

[c] Standard deviation of the accepted data set mean value.

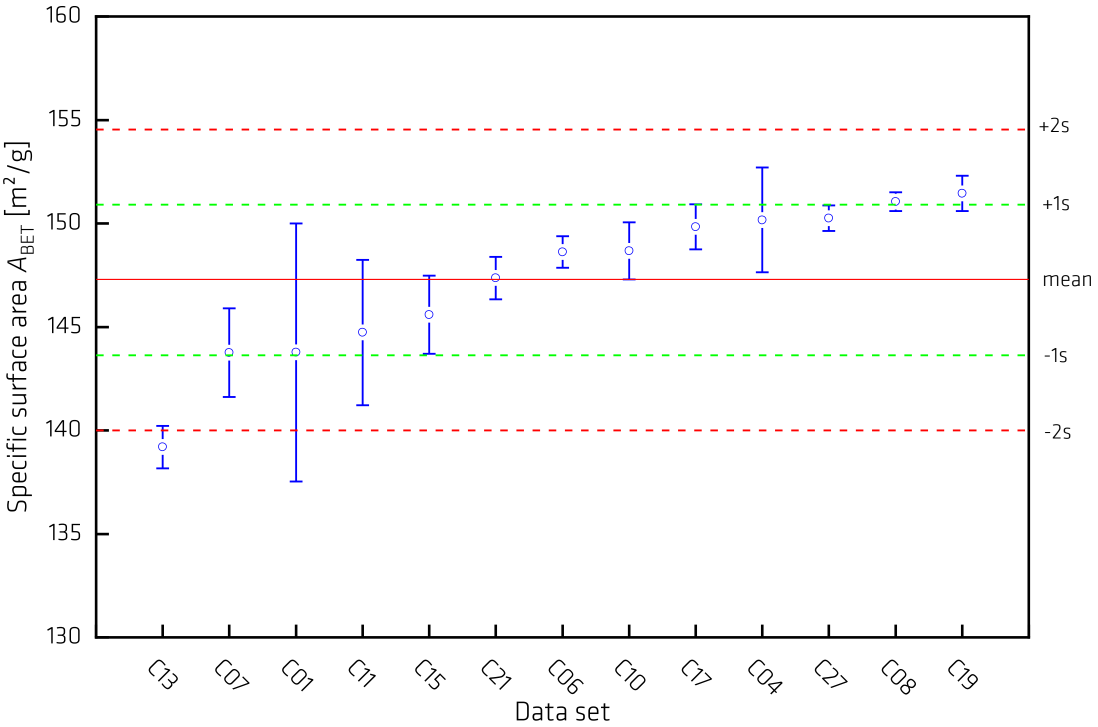

Fig. 14: Submitted and accepted calculated results for A_{BET} with calculated data set means and standard deviations (l = 13)

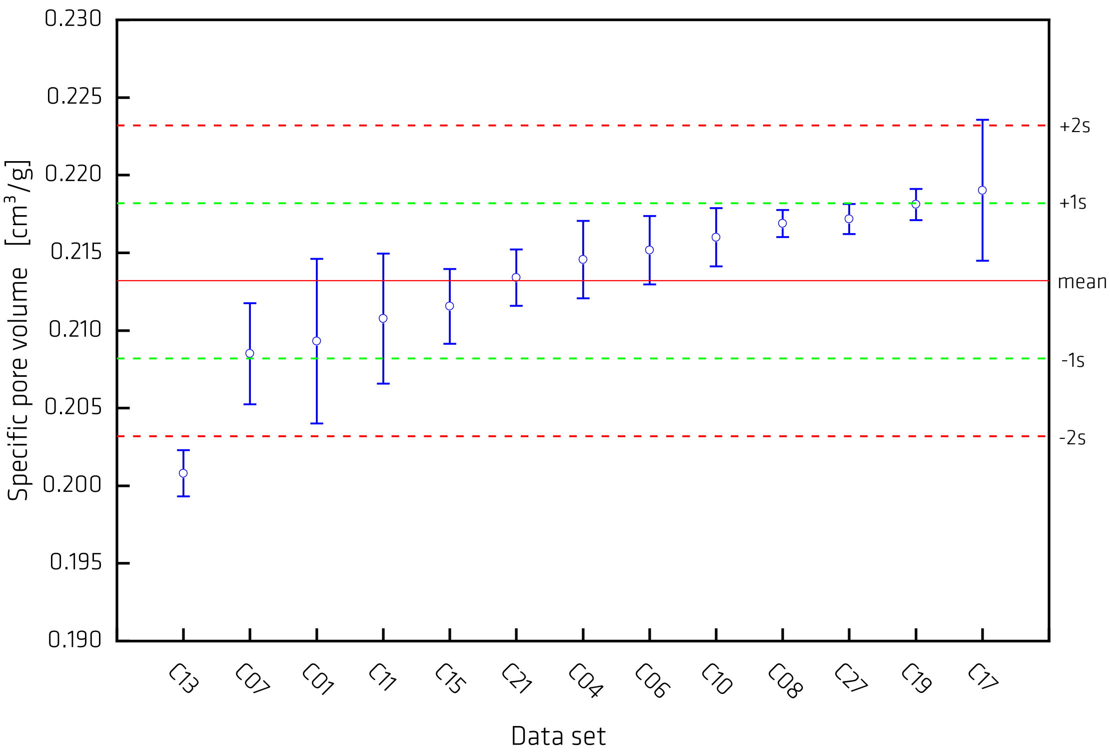

Fig. 15: Submitted results for $V_{p,0.99}$ with calculated data set means and standard deviations (n = 13)

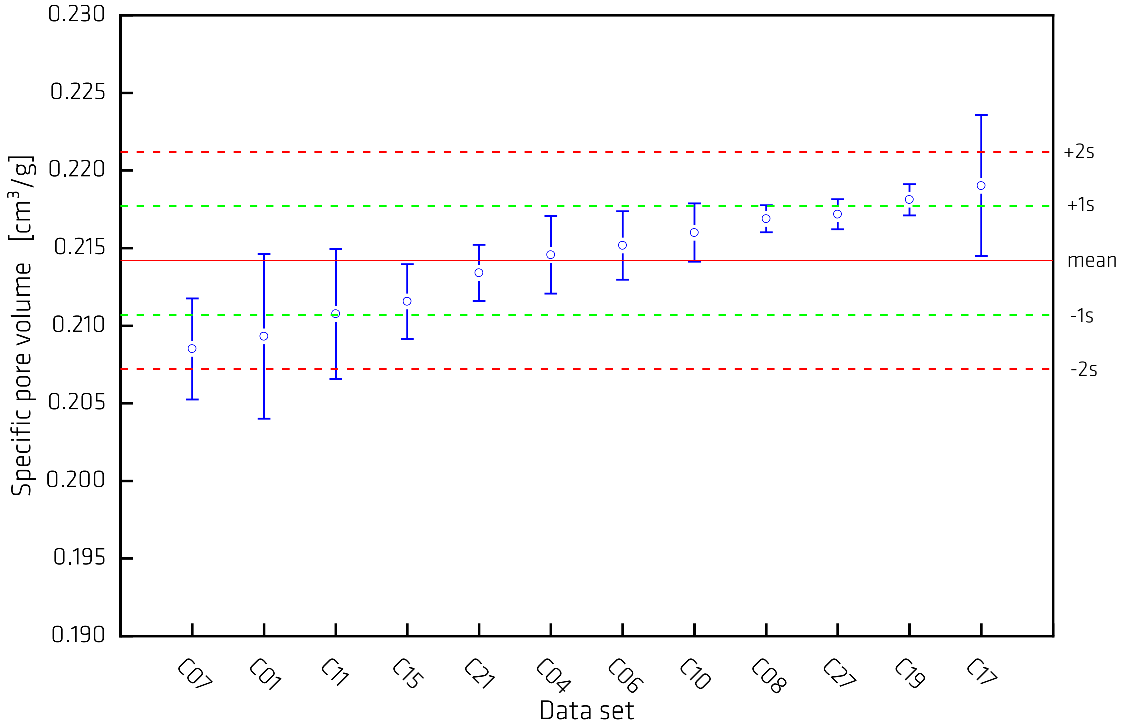

Fig. 16: Confirmed calculated results without outlier for $V_{p,0.99}$ with calculated data set means and standard deviations (l = 12)

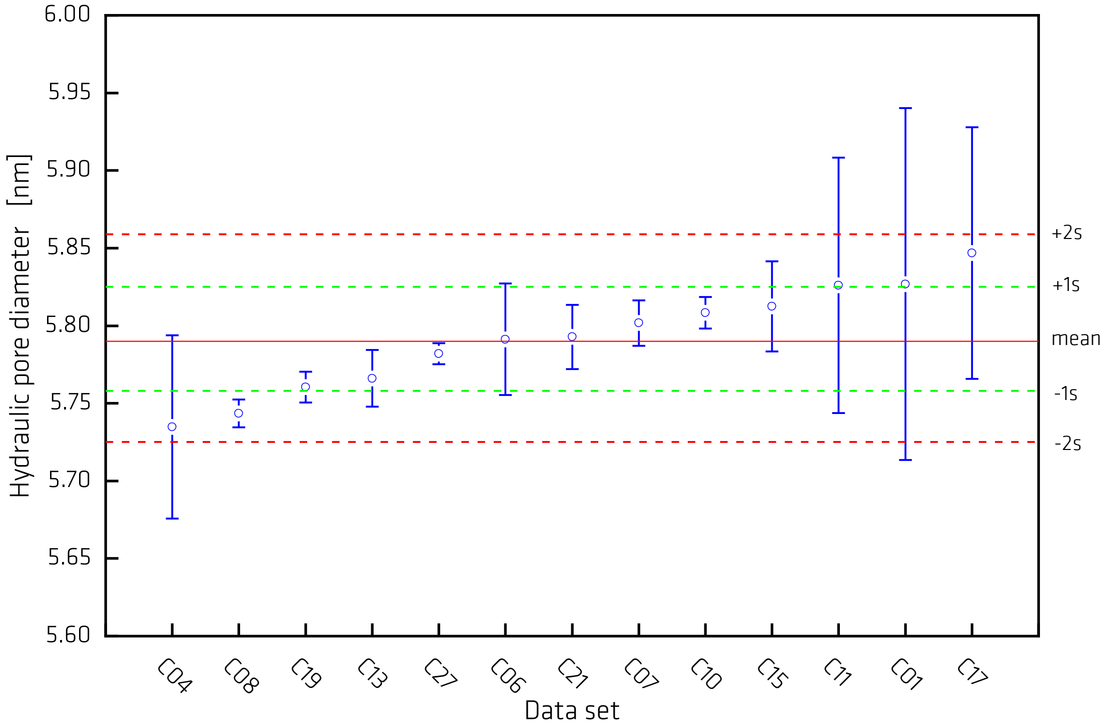

Fig. 17: Submitted and accepted calculated results for $4V_{p,0.99}/A_{BET}$ with calculated data set means and standard deviations (l = 13)

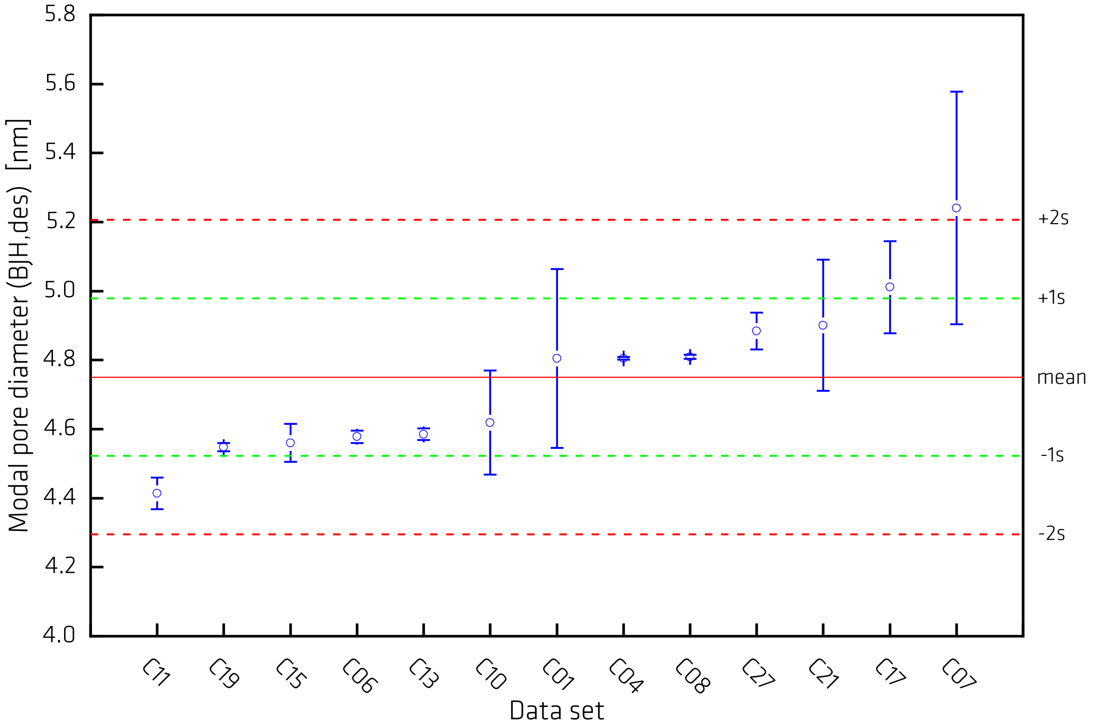

Fig. 18: Submitted and accepted calculated results for $D_{\mathrm{BJH(des)}}$ with calculated data set means and standard deviations (l = 13)

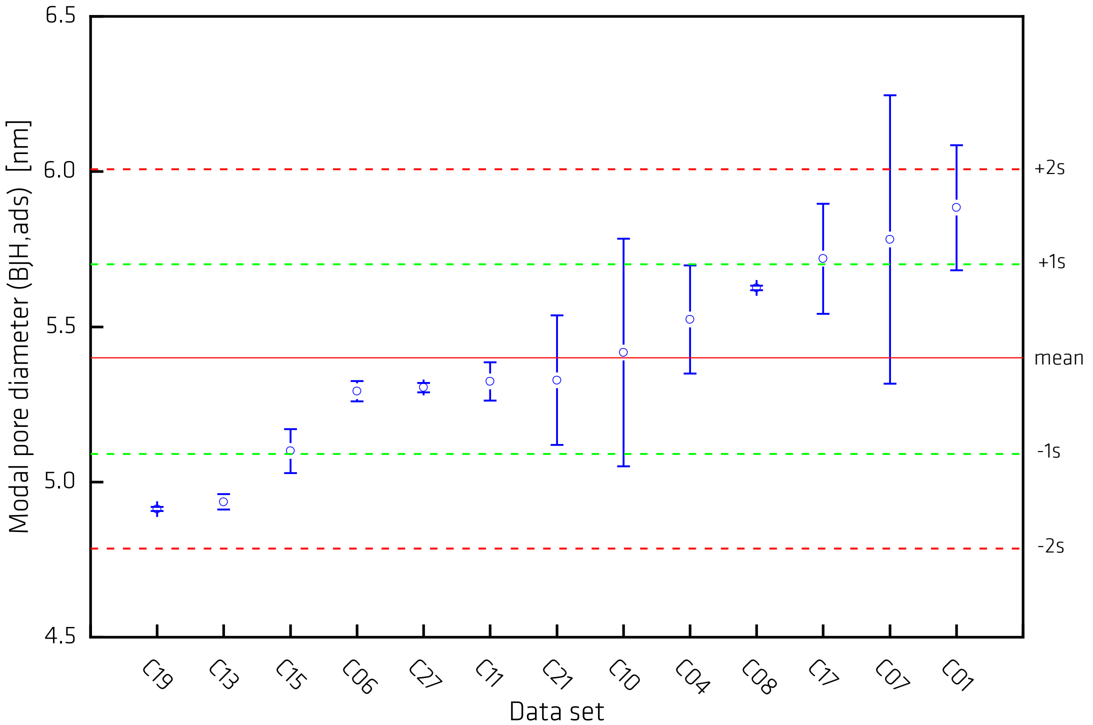

Fig. 19: Submitted and accepted calculated results for $D_{\mathrm{BJH(ads)}}$ with calculated data set means and standard deviations (l = 13)

6.2 Statistical evaluation

The statistical evaluation included all measurement data submitted by the participants of the inter-laboratory comparison and the same standardized procedure was used for sample measurement for all participants.
However, it was not to be assumed that the individual measured values in the inter-laboratory comparison would come from a common population, since the participants used different instruments (see Table 7). This was confirmed by observing different laboratory mean values and heterogeneous standard deviations (see Table 8).

The following statistical parameters were calculated:

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

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
- Test for skewness and kurtosis

As a result of the statistical analysis one outlier was detected, corresponding to the total mesopore volume $V_{p,0.99}$. This outlier (data set C13) was excluded from the calculations of the certified value and its uncertainty. The results of the calculations with the evaluation software SoftCRM [13] are presented in Table 9.

Table 9: Evaluation of the ILC data using the software program SoftCRM

Property (x)	$\bar{x}$	s_x	u_{char}	CI	Unit	Pooling	l
A_{BET}	147.26	3.63	1.01	2.20	m^2/g	no	13
$V_{p,0.99}$	0.2142	0.0035	0.0010	0.0022	cm^3/g	No	12
$4V_{p,0.99}/A_{BET}$	5.792	0.034	0.009	0.020	nm	No	13
$D_{BJH,des}$	4.751	0.228	0.063	0.138	nm	No	13
$D_{BJH,ads}$	5.396	0.306	0.085	0.185	nm	no	13

The relative uncertainties of u_{hom} and u_{lts} were used to adjust the uncertainty in the homogeneity $u_{hom}(x)$ and stability $u_{lts}(x)$ values for the accepted data sets of the inter-laboratory comparison (ILC) participants. This adjustment was required before the combined uncertainties could be calculated.

Table 10: Adaptation of u_{hom} and u_{lts} to the ILC data sets

Property (x)	$\bar{x}$	$u_{hom\ rel.}$	$u_{hom}(x)$	$u_{lts\ rel.}$	$u_{lts}(x)$
A_{BET}	147.26	0.002201	0.32407	0.00419	0.61704
$V_{p,0.99}$	0.2142	0.001381	0.00030	0.00426	0.00091
$4V_{p,0.99}/A_{BET}$	5.792	0.000971	0.00562	0.00454	0.02629
$D_{BJH,des}$	4.751	0.002118	0.01006	0.01418	0.06736
$D_{BJH,ads}$	5.396	0.002153	0.01161	0.00999	0.05390

The combined uncertainty $u_c(x)$ was calculated according to Equation (4) using the numerical values summarized in Table 11. This includes the standard uncertainty of the mean of the accepted means of the inter-laboratory comparison , the contribution of the homogeneity, the stability and the uncertainty contribution due to the measurement results variations of the single instruments (mean data set precision).

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

Table 11: Values of the uncertainty components for the specific surface area of BAM-P115

Property (x)	$\bar{x}$	u_{char}	$u_{hom}(x)$	$u_{lts}(x)$	$\frac{1}{l}\sqrt{\sum_{i=1}^{l} s_i^2}$	$u_c(x)$	U	l	Unit
A_{BET}	147.26	1.01	0.32407	0.61704	0.65662	1.39032	2.78064	13	m²/g
$V_{p,0.99}$	0.2142	0.0010	0.00030	0.00091	0.00085	0.00163	0.00326	12	cm³/g
$4V_{p,0.99}/A_{BET}$	5.792	0.009	0.00562	0.02629	0.01400	0.03172	0.06343	13	nm
$D_{BJH,des}$	4.751	0.063	0.01006	0.06736	0.03966	0.10102	0.20205	13	nm
$D_{BJH,ads}$	5.396	0.085	0.01161	0.05390	0.05478	0.11502	0.23004	13	nm

The certified values of each porosity parameter with a reasonable number of digits and the respective expanded uncertainty (rounded according to DIN 1333 [15]) are summarized in Table 12.

Table 12: Final values for the certified specific surface area of BAM-P115

Property	Certified value x_{cert}	Expanded uncertainty $U = k \cdot u_c$ (k = 2)	Unit
A_{BET}	147.3	2.8	m^2/g
$V_{\mathrm{p,0.99}}$	0.214	0.004	cm^3/g
$4V_{\mathrm{p,0.99}}/A_{\mathrm{BET}}$	5.79	0.07	nm
$D_{\mathrm{BJH,des}}$	4.75	0.21	nm
$D_{\mathrm{BJH,ads}}$	5.40	0.24	nm

7. Metrological traceability

The certified values of specific surface area and porosity parameters are determined according to ISO 9277 and ISO 15901-2 and are traceable to the base units of the SI via calibrated measurements of the quantities pressure, volume and mass.

8. Information on the proper use of the CRM

8.1 Recommended use

Prior to the measurement, outgassing of the sample is necessary. Outgassing must be carried out in vacuum with a final pressure of < 10 Pa. For degassing in vacuum, the sample is to be heated with a rate of about 5 K/min to 453.15 K, then this temperature must be maintained for at least three hours. Afterwards, the sample to be allowed to slowly cool down to ambient temperature.

The adsorption and desorption branch of the N_2 isotherm must be measured at 77.3 K. The analysis should be performed following the instrument manufacturer's instructions. The recommended minimum sample intake is 0.8 g.

The sample preparation station should have a separate vacuum circuit in addition to the analysis station, or the preparation should be carried out in a separate heating station. For instruments with a combined vacuum system, measurements and sample preparation should not be performed together, as condensation in glass vessels can occur during sample preparation.

8.2 Transport, storage and handling

CRM BAM-P115 can be shipped at ambient temperature. Upon receipt the material should be stored at a temperature below 30 °C in its original tightly closed bottle. When handling the sample, the bottle should be left open as briefly as possible. Care should be taken to avoid moisture once the bottle is opened. BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened bottles. The material should be used as is from the bottle. However, before taking a sub-sample re-homogenisation by manual shaking of the closed bottle is strongly recommended.

8.3 Shelf life

The initial stability study after storage of selected units at different temperatures did not reveal any statistically significant deterioration of the certified properties. However, starting with dispatch of the material from BAM the validity of the certificate expires after 24 months. Post-certification monitoring measurements will be conducted in appropriate periods to keep this information up to date.

8.4 Safety information

The usual laboratory safety precautions must be applied. No hazardous effects are to be expected when the material is used under conditions commonly adopted for the analysis of samples. It is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

8.5 Legal notice

Neither BAM, its contractors nor any person acting on their behalf:

(a) make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document does not infringe any privately-owned intellectual property rights; or

(b) assume any liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this document.

9. Information on and purchase of the RM

Certified reference material CRM BAM-P115 is supplied by

Bundesanstalt für Materialforschung und -prüfung (BAM)
Fachbereich 1.6: Anorganische Referenzmaterialien
Richard-Willstätter-Str. 11, D-12489 Berlin, Germany
Phone +49 (0)30 - 8104 2061
Fax: +49 (0)30 – 8104 72061
E-Mail: sales.crm@bam.de

Each bottle of CRM BAM-P115 will be distributed together with a detailed certificate containing the certified values and their uncertainties, the mean values and standard deviations of all accepted data sets and information on the analytical methods used and the names of the participating laboratories.
Information on certified reference materials can be obtained from BAM,
https://www.bam.de.
www.webshop.bam.de

10. References

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11. Annexes

Annex 1: Calculation of uncertainty contribution of inhomogeneity

TiO_2 - HOMBIKAT-5

Value: **A_BET**

$0.05 \le p/p_0 \le 0.3$

Homogeneity: **10 samples x 3 replicates**

	Replicates		
Bottle-No.	**1**	**2**	**3**
	m^2/g	**m^2/g**	**m^2/g**
A2(01) - 009	149.46	148.92	149.80
B3(05) - 069	149.22	148.91	148.41
H2(02) - 346	147.76	148.61	148.53
D5(08) - 184	148.64	148.40	148.88
E6(04) - 236	148.22	148.06	148.57
E2(06) - 206	148.73	148.75	148.54
C1(03) - 099	148.69	147.89	148.31
G4(01) - 313	149.58	149.24	148.73
A3(04) - 020	148.82	148.71	148.54
C5(08) - 136	148.67	149.02	149.30

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	3.961298	9	0.440144	3.705604654	0.0070454	2.392814
Within Groups	2.375559	20	0.118778			
Total	6.336857	29				

s_bb	0.327295	mean_total	148.730887
u'_bb	0.111894	s_method	0.344642
u_hom	0.327295	s_method_r	0.002317
u_hom_r	0.002201		

u_hom is the maximum uncertainty of s_bb and u'_bb

TiO_2 - HOMBIKAT-5

Value: **PV_GUR**

Homogeneity: **10 samples x 3 replicates**

Bottle-No.	**Replicates 1**	**2**	**3**
	cm^3/g	**cm^3/g**	**cm^3/g**
A2(01) - 009	0.2162	0.2155	0.2175
B3(05) - 069	0.2170	0.2149	0.2149
H2(02) - 346	0.2147	0.2148	0.2168
D5(08) - 184	0.2164	0.2155	0.2164
E6(04) - 236	0.2145	0.2144	0.2160
E2(06) - 206	0.2162	0.2160	0.2150
C1(03) - 099	0.2157	0.2143	0.2166
G4(01) - 313	0.2172	0.2158	0.2153
A3(04) - 020	0.2154	0.2153	0.2159
C5(08) - 136	0.2159	0.2172	0.2174

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	7.7849E-06	9	8.64989E-07	1.026561404	0.45306704	2.392814
Within Groups	1.68522E-05	20	8.42608E-07			
Total	2.46371E-05	29				

s_bb	8.63729E-05	mean_total	0.215831
u'_bb	0.000298	s_method	0.000918
u_hom	0.000298	s_method_r	0.004253
u_hom_r	0.001381		

u_hom is the maximum uncertainty of s_bb and u'_bb

TiO_2 - HOMBIKAT-5

Value: **Hydraulic pore diameter $4V_{p,0.99}/A_{BET}$**

Homogeneity: **10 samples x 3 replicates**

	Replicates		
Bottle-No.	**1**	**2**	**3**
	[nm]	**[nm]**	**[nm]**
A2(01) - 009	5.79	5.79	5.81
B3(05) - 069	5.82	5.77	5.79
H2(02) - 346	5.81	5.78	5.84
D5(08) - 184	5.82	5.81	5.81
E6(04) - 236	5.79	5.79	5.82
E2(06) - 206	5.81	5.81	5.79
C1(03) - 099	5.79	5.80	5.84
G4(01) - 313	5.81	5.78	5.79
A3(04) - 020	5.79	5.79	5.81
C5(08) - 136	5.81	5.82	5.82

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.002373434	9	0.000263715	0.875892785	0.56147645	2.392814
Within Groups	0.006021625	20	0.000301081			
Total	0.008395059	29				

s_bb	0	mean_total	5.804127
u'_bb	0.005634	s_method	0.017352
u_hom	0.005634	s_method_r	0.002990
u_hom_r	0.000971		

u_hom is the maximum uncertainty of s_bb and u'_bb

TiO_2 - HOMBIKAT-5

Value:	**BJH (des) pore diameter ∆V/∆Dp max [nm]**		
Homogeneity:	**10 samples x 3 replicates**		

Bottle-No.	**Replicates 1 [nm]**	**2 [nm]**	**3 [nm]**
A2(01) - 009	4.60	4.61	4.55
B3(05) - 069	4.60	4.53	4.58
H2(02) - 346	4.60	4.59	4.58
D5(08) - 184	4.58	4.60	4.59
E6(04) - 236	4.57	4.61	4.61
E2(06) - 206	4.58	4.61	4.58
C1(03) - 099	4.57	4.53	4.61
G4(01) - 313	4.61	4.59	4.57
A3(04) - 020	4.53	4.53	4.61
C5(08) - 136	4.54	4.61	4.60

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.0042700	9	0.000474444	0.531094527	0.8349	2.3928
Within Groups	0.0178667	20	0.000893333			
Total	0.022136667	29				

s_bb	0	mean_total	4.582333
u'_bb	0.009704	s_method	0.029889
u_hom	0.009704	s_method_r	0.006523
u_hom_r	0.002118		

u_hom is the maximum uncertainty of s_bb and u'_bb

TiO_2 - HOMBIKAT-5

Value:	**BJH (ads) pore diameter ΔV/ΔDp max [nm]**		
Homogeneity:	**10 samples x 3 replicates**		

	Replicates		
Bottle-No.	**1**	**2**	**3**
	[nm]	**[nm]**	**[nm]**
A2(01) - 009	5.22	5.23	5.30
B3(05) - 069	5.31	5.22	5.26
H2(02) - 346	5.29	5.33	5.22
D5(08) - 184	5.27	5.23	5.32
E6(04) - 236	5.25	5.29	5.32
E2(06) - 206	5.25	5.24	5.25
C1(03) - 099	5.26	5.24	5.24
G4(01) - 313	5.25	5.26	5.28
A3(04) - 020	5.28	5.23	5.25
C5(08) - 136	5.23	5.23	5.28

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.005936667	9	0.00065963	0.542161339	0.82671417	2.392814
Within Groups	0.024333333	20	0.001216667			
Total	0.03027	29				

s_bb	0	mean_total	5.261000
u'_bb	0.011325	s_method	0.034881
u_hom	0.011325	s_method_r	0.006630
u_hom_r	0.002153		

u_hom is the maximum uncertainty of s_bb and u'_bb

Annex 2: Measurement results of ILC-participants

Inter-laboratory comparison for the certification of BAM-P115

Data set no.	Specific surface area A_{BET} [m^2/g]					Mean value	Standard deviation
	Replicate						
	1	**2**	**3**	**4**	**5**		
C01	136.2070	137.8020	147.5210	148.0700	149.2320	143.7664	6.2292
C04	153.2390	147.8720	151.6050	150.8640	147.2600	150.1680	2.5352
C06	149.6546	148.6418	149.0217	147.9686	147.7901	148.6154	0.7657
C07	145.7610	145.3370	141.3460	142.5530	-	143.7493	2.1428
C08	150.4011	151.0773	151.1878	150.9109	151.6634	151.0481	0.4573
C10	149.0330	149.8520	147.1280	147.3150	150.0430	148.6742	1.3809
C11	140.0600	145.5000	147.8000	148.0900	142.2000	144.7300	3.5184
C13	140.3300	139.2700	139.9400	137.7300	138.7200	139.1980	1.0272
C15	143.0112	148.0423	146.4283	145.8106	144.6654	145.5916	1.8888
C17	148.5580	148.7200	150.5180	150.7190	150.6390	149.8308	1.0918
C19	150.3047	150.9050	151.8306	151.7794	152.4563	151.4552	0.8477
C21	147.0320	147.8810	145.8070	148.5300	147.5500	147.3600	1.0242
C27	149.3683	149.8585	150.8202	150.4973	150.7012	150.2491	0.6165

Inter-laboratory comparison for the certification of BAM-P115

Data set no.	**Gurvich Vp (cm^3/g)**					Mean value	Standard deviation
	p/p0 = 0,990						
	1	**2**	**3**	**4**	**5**		
C01	0.2025	0.2048	0.2139	0.2116	0.2137	0.2093	0.005299
C04	0.2175	0.2150	0.2155	0.2141	0.2107	0.2146	0.002492
C06	0.2185	0.2138	0.2163	0.2130	0.2143	0.2152	0.002202
C07	0.2117	0.2109	0.2054	0.2060	-	0.2085	0.003259
C08	0.2158	0.2173	0.2172	0.2162	0.2179	0.2169	0.000870
C10	0.2170	0.2170	0.2140	0.2140	0.2180	0.2160	0.001871
C11	0.2090	0.2111	0.2142	0.2149	0.2046	0.2108	0.004187
C13	0.2010	0.2010	0.2030	0.1990	0.2000	0.2008	0.001483
C15	0.2076	0.2141	0.2117	0.2126	0.2116	0.2116	0.002411
C17	0.2139	0.2145	0.2217	0.2210	0.2240	0.2190	0.004543
C19	0.2165	0.2178	0.2188	0.2185	0.2190	0.2181	0.001005
C21	0.2125	0.2132	0.2110	0.2157	0.2146	0.2134	0.001826
C27	0.2159	0.2165	0.2178	0.2174	0.2183	0.2172	0.000970

Inter-laboratory comparison for the certification of BAM-P115

Data set no.	Hydraulic pore diameter					Mean value	Standard deviation
	$4V_{p,0.99}/A_{BET}$						
	1	**2**	**3**	**4**	**5**		
C01	5.9470	5.9449	5.7999	5.7152	5.7272	5.8268	0.113445
C04	5.7743	5.8148	5.6853	5.6767	5.7229	5.7348	0.059024
C06	5.8389	5.7525	5.8055	5.7581	5.8011	5.7912	0.035943
C07	5.8095	5.8044	5.8127	5.7803	-	5.8017	0.014687
C08	5.7387	5.7548	5.7452	5.7313	5.7475	5.7435	0.008926
C10	5.8120	5.7910	5.8160	5.8150	5.8080	5.8084	0.010213
C11	5.9700	5.8000	5.8000	5.8000	5.7600	5.8260	0.082341
C13	5.7417	5.7593	5.7912	5.7741	5.7642	5.7661	0.018298
C15	5.8078	5.7862	5.7844	5.8334	5.8504	5.8124	0.029054
C17	5.7599	5.7694	5.8907	5.8652	5.9489	5.8468	0.080984
C19	5.7621	5.7723	5.7648	5.7577	5.7454	5.7605	0.009952
C21	5.7810	5.7670	5.7890	5.8090	5.8180	5.7928	0.020717
C27	5.7812	5.7796	5.7766	5.7784	5.7937	5.7819	0.006824

Inter-laboratory comparison for the certification of BAM-P115

Data set no.	BJH (des)					Mean value	Standard deviation
	ΔV/Δd max [nm]						
	1	**2**	**3**	**4**	**5**		
C01	4.7330	4.7340	5.0589	4.4429	5.0542	4.8046	0.2588
C04	4.8040	4.8010	4.8070	4.8040	4.8100	4.8052	0.0034
C06	4.5600	4.5600	4.5800	4.5900	4.6000	4.5780	0.0179
C07	5.5492	5.5148	4.9488	4.9488	-	5.2404	0.3370
C08	4.8010	4.8080	4.8170	4.8070	4.8130	4.8092	0.0061
C10	4.5760	4.8800	4.5690	4.5790	4.4890	4.6186	0.1508
C11	4.3500	4.3900	4.4300	4.4300	4.4700	4.4140	0.0456
C13	4.5740	4.5846	4.5907	4.6099	4.5665	4.5851	0.0167
C15	4.6000	4.6000	4.6000	4.5000	4.5000	4.5600	0.0548
C17	4.7726	5.0770	5.0616	5.0716	5.0738	5.0113	0.1336
C19	4.5307	4.5404	4.5565	4.5569	4.5545	4.5478	0.0117
C21	5.1840	4.6970	4.7950	4.9880	4.8400	4.9008	0.1899
C27	4.7900	4.9100	4.9000	4.9000	4.9200	4.8840	0.0532

Inter-laboratory comparison for the certification of BAM-P115

Data set no.	BJH (ads)						Mean value	Standard deviation
	ΔV/Δd max [nm]							
	1	**2**	**3**	**4**	**5**			
C01	5.7941	5.7939	5.7914	6.2442	5.7954		5.8838	0.2015
C04	5.4400	5.4500	5.8350	5.4480	5.4440		5.5234	0.1742
C06	5.3100	5.3200	5.3100	5.2400	5.2800		5.2920	0.0327
C07	5.5294	5.5400	6.4774	5.5784	-		5.7813	0.4645
C08	5.6160	5.6180	5.6270	5.6310	5.6320		5.6248	0.0074
C10	4.9080	5.8370	5.6970	5.2710	5.3700		5.4166	0.3665
C11	5.2400	5.2800	5.3500	5.3600	5.3900		5.3240	0.0619
C13	4.9157	4.9253	4.9357	4.9778	4.9253		4.9360	0.0244
C15	5.1000	5.0000	5.1000	5.1000	5.2000		5.1000	0.0707
C17	5.7972	5.7942	5.4014	5.8028	5.8000		5.7191	0.1776
C19	4.9084	4.9046	4.9161	4.9206	4.9162		4.9132	0.0065
C21	5.2660	5.2940	5.6930	5.1800	5.2070		5.3280	0.2090
C27	5.3200	5.3100	5.3100	5.2800	5.3000		5.3040	0.0152