Certification Report

Certified Reference Material

BAM-P114

BET Specific Surface Area of
Titanium Dioxide (Anatase/Rutile)
calculated from the
nitrogen adsorption isotherm at 77.3 K

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Coordinator: Dr. F. Emmerling
Collaboration: C. Prinz, A. Zimathies
Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 6.3
Richard-Willstätter-Str. 11
12489 Berlin, Germany
Phone: +49 30 8104 1133
Fax: +49 30 8104 71133
E-mail: Franziska.Emmerling@bam.de

Summary

This report describes the certification of the porous reference material BAM-P114. The certified value determined by nitrogen adsorption at 77.3 K according to the international standard ISO 9277 [1] is given in the table below.

Property	Value	U [a]	$2 \cdot s_x$ [b]	Unit
Specific surface area [c] A_{BET}	24.48	0.30	0.4	m^2/g

[a] Expanded uncertainty $U = k \cdot u_c$ calculated according to ISO Guide 35 [2] and ISO/IEC Guide 98-3 [3] 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 interlaboratory characterization, the study of inhomogeneities, stability of the material, and the uncertainty contribution due to the measurement result variations of the single instruments (mean data set precision).

[b] Twofold standard deviation s_x of accepted laboratory means.

[c] Specific surface area A_{BET} calculated in a relative adsorption pressure range of $0.05 \leq p/p_0 < 0.3$ as multipoint BET model [4] with a minimum of five supporting points as described in ISO 9277.

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 value is based on the results of 13 laboratories which participated in the certification interlaboratory comparison.

Content

List of abbreviations

(if not explained elsewhere)

ANOVA	1-way analysis of variance
BET	Brunauer, Emmett, Teller (method)
CRM	certified reference material
GUM	ISO guide to the expression of uncertainty in measurement
ILC	interlaboratory comparison (certification study)
RM	reference material

List of Symbols

df	degrees of freedom
k	coverage factor
l	number of accepted data sets in the interlaboratory 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}	between-unit component of variance from a homogeneity study, expressed as a standard deviation
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_c	combined standard uncertainty of a property value
u_{char}	standard uncertainty due to characterization
u_{hom}	standard uncertainty associated with heterogeneity
u_{lts}	standard uncertainty associated with long term stability (adapted)
u_{Stab}	standard uncertainty due to long-term (in)stability
x	property value of a candidate material
$\overline{x}$	mean property value of a candidate material
x_{cert}	certified property value of a CRM

1. Introduction

The certified reference material BAM-P114 is a porous titanium dioxide. This CRM is intended for performance testing of gas sorption instruments used for determining the Brunauer-Emmet-Teller (BET) specific surface area of powders and porous solids.
The development of this CRM serves to extend the range of porous reference materials with a specific surface area above 20 m^2/g.
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 [5].
The certification of this new CRM has been carried out based on BAM Management Manual [6], ISO 17034 [7] and relevant ISO Guides [2], [8].

2. Material

2.1 Selection and characterization of the candidate material

A nanoscale titanium dioxide (TiO_2, also called titania) consisting of a mix of the modifications of anatase and rutile was selected from several tested candidate materials.
The titania candidate material TP Hombikat (Lot 10588097-4) was delivered by Venator Germany GmbH and consists of pure anatase according to the manufacturer's specifications.

Measurements with mercury porosimetry up to 400 MPa (Fig. 1) indicated a pore sizes of 30 nm, a pore volume of 159 mm^3/g and a porosity of approximately 64%.
As a result of XRD measurements at BAM, Division 6.3, it could be determined that the crystal modification of TP Hombikat (Lot 10588097-4) is a mix of anatase and rutile (see Fig. 2). The mixing ratio was determined using the Rietveld refinement for powder diffraction pattern analysis with Bruker TOPAS software. The analysis showed a ratio of 56% anatase and 44% rutile. The particle size was determined with main distribution from 10 to 100 µm, which agrees with the SEM data (Fig. 3).
Although anatase is metastable and thermodynamically less stable than rutile, the phase conversion rate of anatase into rutile is virtually zero at temperatures up to about 873 K. Therefore, the long-term stability of this mixture of modifications in BAM-P114 is not affected in the temperature range between room temperature and the recommended degassing temperature of 453.15 K (see chapter 4. Stability).

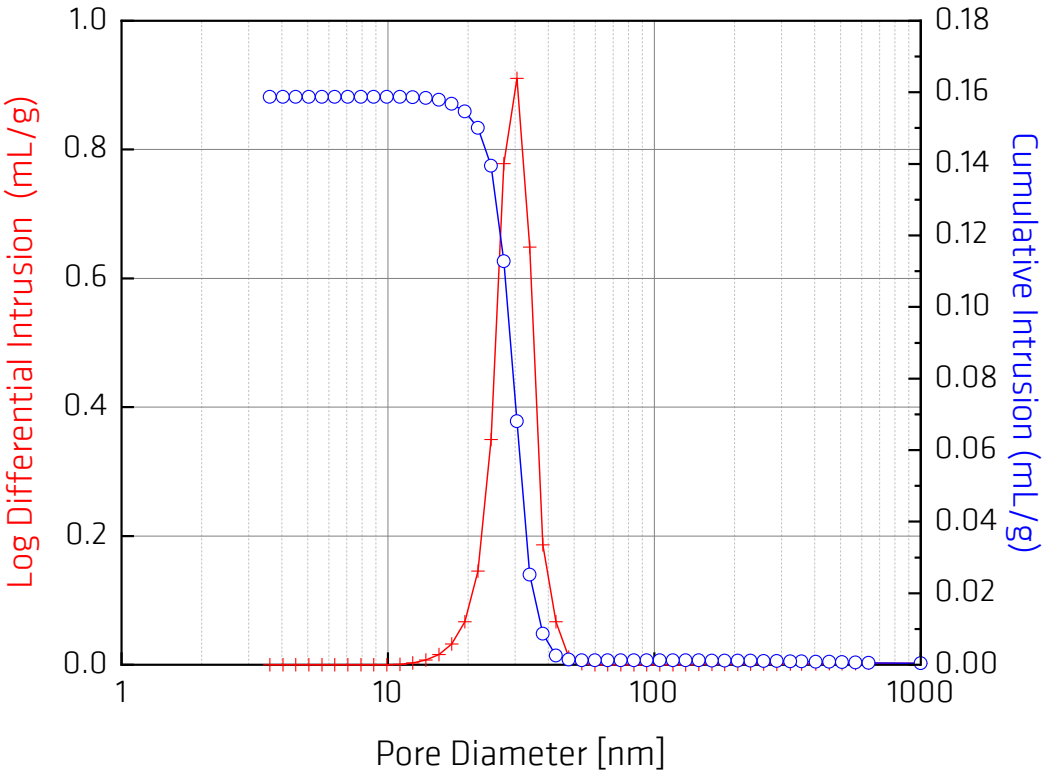

Fig. 1: Determination of pore size, pore volume and porosity of the candidate material by mercury porosimetry.

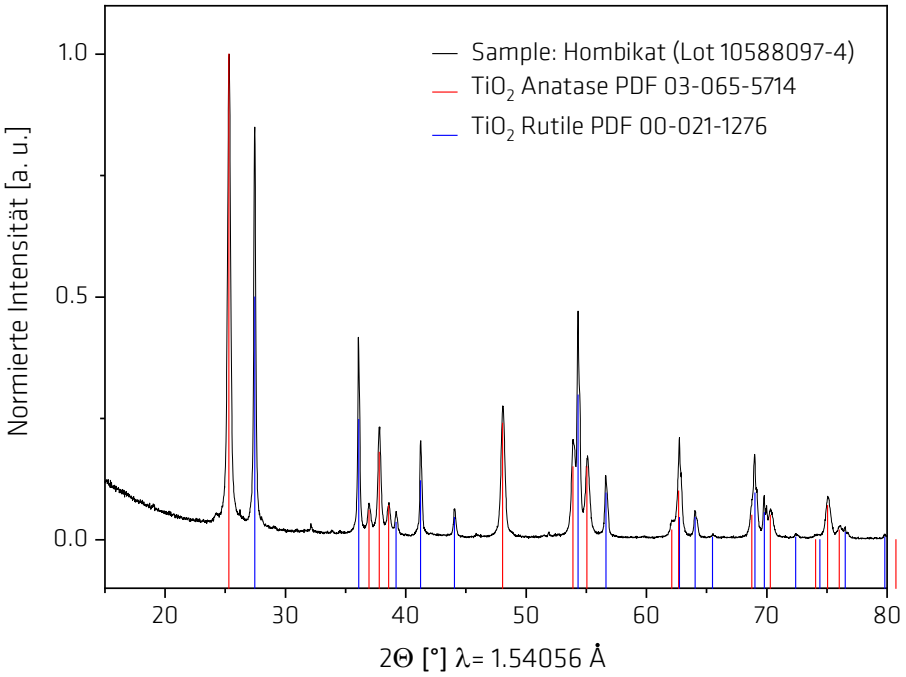

Fig. 2: X-ray powder diffraction pattern of the candidate material and comparison to the powder diffraction file (pdf) database entry [9] of titanium dioxide (mixture of anatase and rutile).

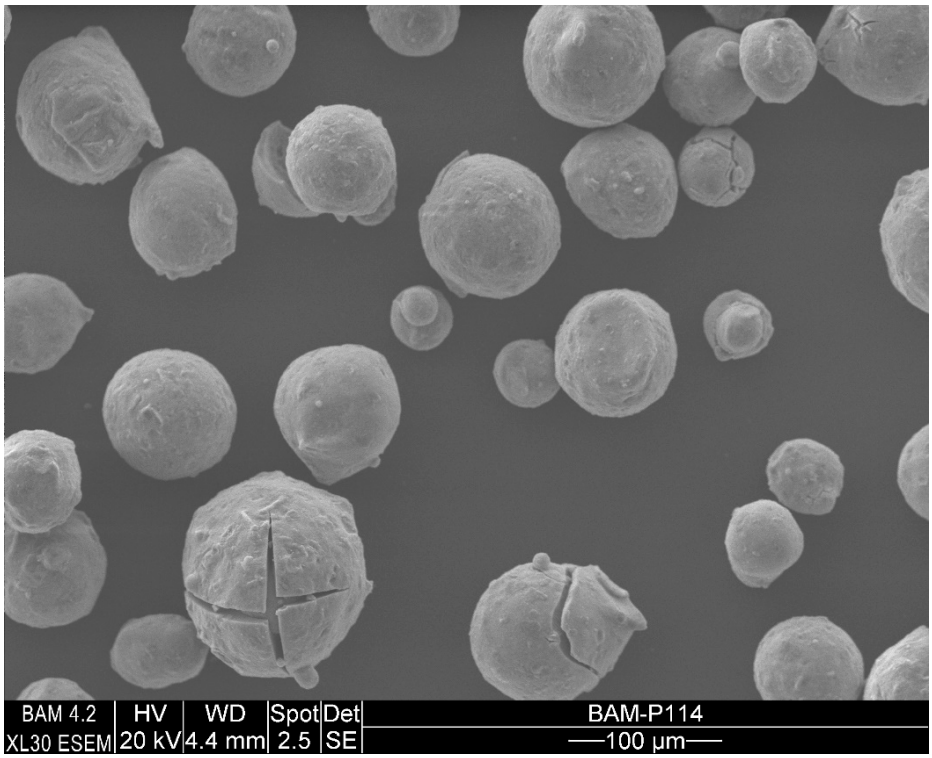

Fig. 3: SEM image of the candidate material (CRM BAM-P114) with a particle size distribution ranging from 10 to 100 µm.

2.2 Specific surface area characterization

Prior to measurement, sample preparation is necessary. Outgassing must be carried out in vacuum with a final pressure of < 10 Pa. The sample is then heated in a vacuum at a rate of about 5 K/min to 453.15 K, then held at 453.15 K for at least three hours. Afterwards, the sample is cooled slowly to ambient temperature.

After sample preparation, the first step of specific surface area determination is using the gas adsorption method for the measurement of a low temperature physisorption isotherm (see Fig. 4) or only the adsorption branch up to at least p/p_0 0.5 (see Fig. 5).

The certified value of specific surface area is calculated for multipoint BET data analysis by using the linear form (see Fig. 6) with a minimum of five adsorption points in a relative pressure range of $0.05 \leq p/p_0 < 0.3$ according to the BET model as described in ISO 9277. The value used for cross-sectional area of nitrogen was 0.162 nm^2.

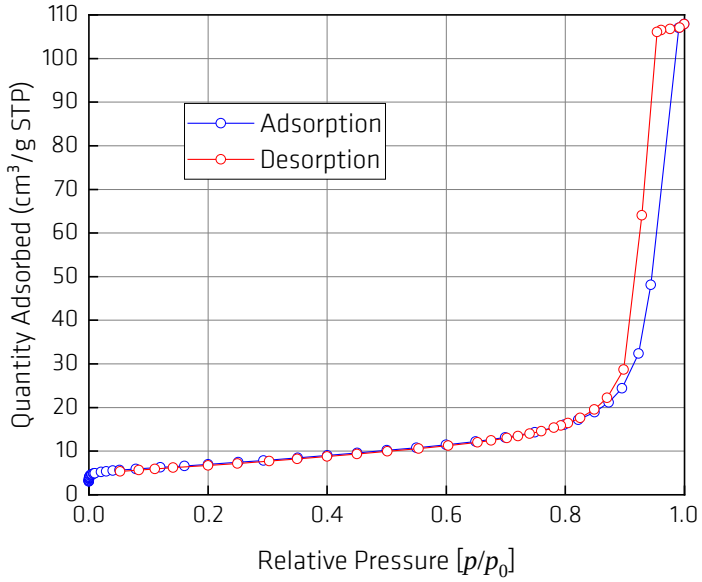

Fig. 4:

N_2 isotherm of BAM-P114 at 77.3 K

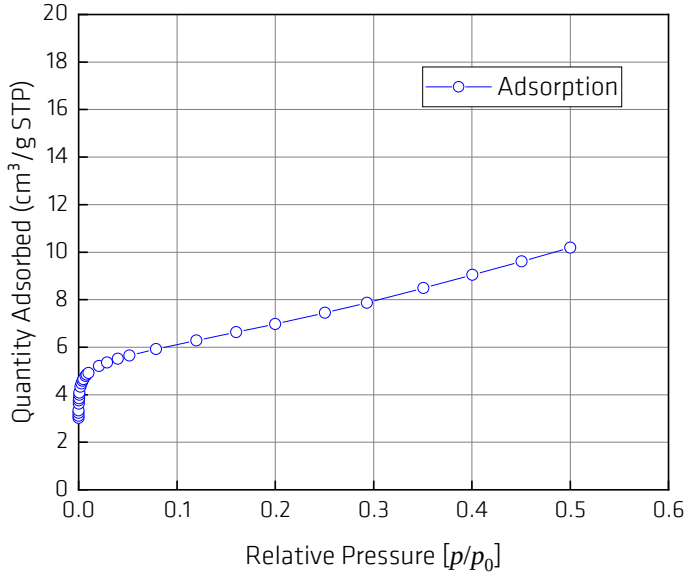

Fig. 5:

Adsorption branch

of BAM-P114 at 77.3 K

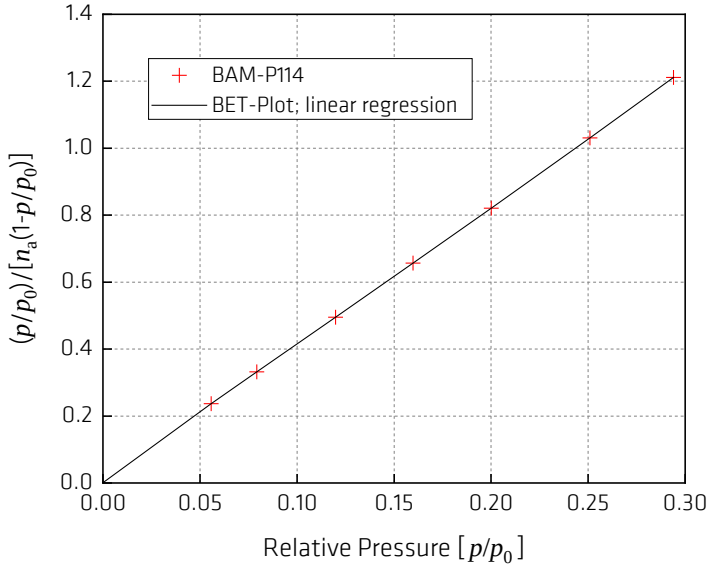

Fig. 6:

BET – Plot of BAM-P114

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 [10] shown in Fig. 7.

Fig. 7: 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 was subdivided into single units of at least 18 g filled in glass bottles of 30 mL volume. The total number of units was 256.
23 units thereof have been used for testing the homogeneity and stability as well as for the certification interlaboratory comparison.

3. Homogeneity

For testing the homogeneity, ten individual units of BAM-P114 were randomly selected. Three replicate measurements per bottle were carried out under repeatability 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 contribution of inhomogeneity to the total uncertainty was calculated using a 1-way ANOVA (see Annex 1).
The estimation of the inhomogeneity contribution u_{hom} to be included into the total uncertainty budget was identical with the greater value of the standard uncertainty between the bottles (eq.1) and the uncertainty potentially hidden under the within-bottle variability (eq. 2) according to ISO Guide 35 [2], and the BAM Management Manual [6]:

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

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

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

Table 1: Results of replicate measurements with samples from single bottles

Bottle	Data file	A_{BET} [m^2/g]	Data file	A_{BET} [m^2/g]	Data file	A_{BET} [m^2/g]
	Result no. 1		Result no.2		Result no. 3	
D4(08)	Homo-1644.smp	24.3638	Homo-1645.smp	24.3049	Homo-1646.smp	24.3060
C1(04)	Homo-1647.smp	24.4664	Homo-1648.smp	24.4819	Homo-1649.smp	24.4117
F3(03)	Homo-1650.smp	24.2290	Homo-1651.smp	24.2150	Homo-1652.smp	24.1854
H3(04)	Homo-1653.smp	24.2769	Homo-1654.smp	24.1937	Homo-1655.smp	24.2499
B2(06)	Homo-1656.smp	24.3168	Homo-1657.smp	24.2073	Homo-1658.smp	24.2849
E3(01)	Homo-1659.smp	24.4434	Homo-1660.smp	24.4010	Homo-1661.smp	24.4991
D2(06)	Homo-1662.smp	24.4157	Homo-1663.smp	24.4109	Homo-1664.smp	24.4166
A3(05)	Homo-1665.smp	24.5221	Homo-1666.smp	24.4390	Homo-1667.smp	24.4661
G2(07)	Homo-1668.smp	24.1506	Homo-1674.smp	24.3928	Homo-1676.smp	24.3564
G3(02)	Homo-1678.smp	24.3730	Homo-1679.smp	24.4041	Homo-1680.smp	24.3387
Mean total ($\overline{x}_{hom}$)		24.3508				

Table 2: 1-way ANOVA table of the homogeneity study on specific surface area A_{BET}

Source of variation	Square sum	Degrees of freedom (df)	Mean square sum (M)	F	F-crit. 95%
Between Units	0.24597	9	0.02733	9.01482	2.39281
Within Units	0.06063	20	0.00303		
s_bb	0.09000				
u'_bb	0.01788				

The tested bottles are significantly different (F > F-crit. 95%). This requires that the greatest inhomogeneity estimate (s_bb) is included in the uncertainty budget of the certified value.

Table 3: Inhomogeneity contribution

Property	u_{hom_max} (s_bb)	u_{hom_r}	Unit
A_{BET}	0.09000	0.00370	m^2/g

4. Stability

The stability measurements of BAM-P114 are listed in Table 4 for the period between July 2019 and June 2021. These results of the specific surface area A_{BET} are depicted in Figure 8 together with the trend of stability calculated by using the linear regression. All measurements were carried out with the same instrument ASAP 2020 (Micromeritics, Norcross, USA) used for the homogeneity study.

Table 4: Numerical results of stability monitoring

Data file	Test date	A_{BET}
		m^2/g
HOM01644.smp	04.07.2019	24.3638
HOM01680.smp	31.07.2019	24.3387
HOM-1699.smp	19.09.2019	24.5613
HOM-1719.smp	15.10.2019	24.4661
HOM-1736.smp	28.11.2019	24.5401
HOM-1748.smp	17.12.2019	24.4344
HOM-1804.smp	19.03.2020	24.5248
114-1850.smp	07.07.2020	24.6021
114-1894.smp	01.10.2020	24.6831
114-1909.smp	23.10.2020	24.6586
114-1956.smp	15.12.2020	24.5806
114-2010.smp	15.03.2021	24.5502
114-2052.smp	03.05.2021	24.3176
114-2079.smp	02.06.2021	24.4496
	$\bar{x}_{\mathrm{Stab}}$ [a]	24.5052
	$\bar{x}_{\mathrm{ILC}}$ [b]	24.4758
	$s_{x,\,\mathrm{ILC}}$	0.1938
	$\bar{x}_{\mathrm{Stab}} + 2 \cdot s_{x,\,\mathrm{ILC}}$	24.8928
	$\bar{x}_{\mathrm{Stab}} - 2 \cdot s_{x,\,\mathrm{ILC}}$	24.1176

[a] Stab = stability monitoring

[b] ILC = interlaboratory comparison (certification study)

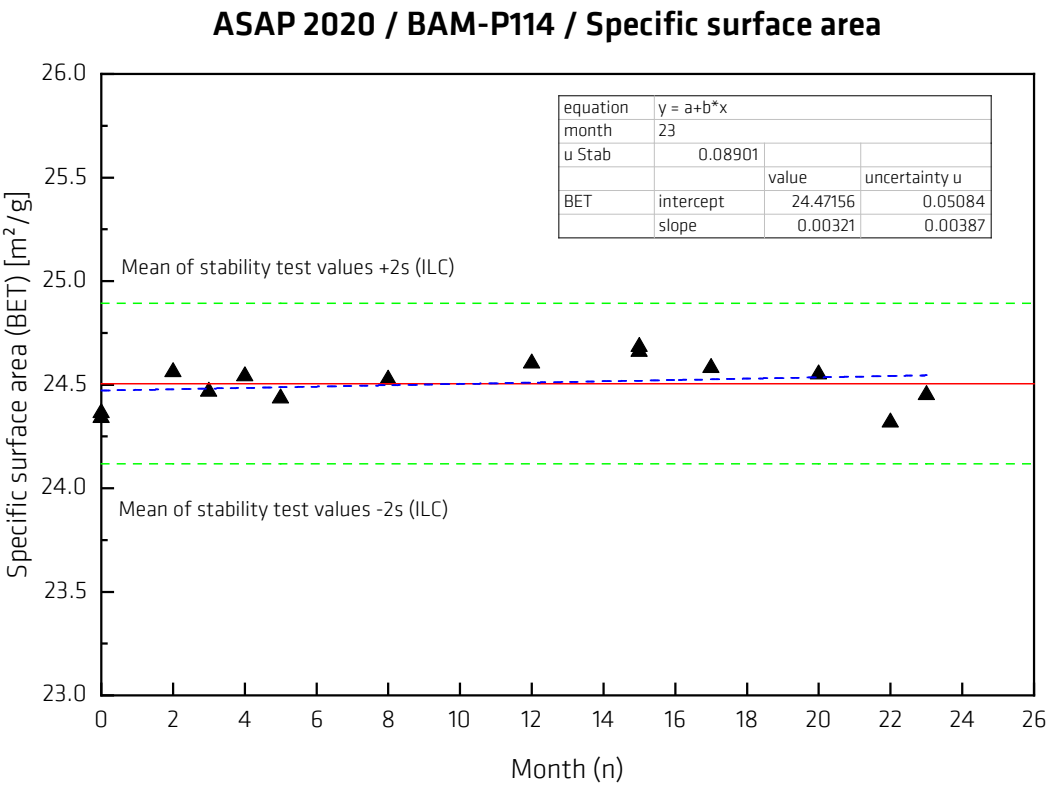

Fig. 8: Stability monitoring for the specific surface area A_{BET}

The linear regression calculation shows that the slope is not significantly different from zero. From this, the uncertainty budget $u_{Stab}(x)$ and the relative value $u_{Stab\_r}(x)$ is determined using Equation 3 and presented in Table 5. The calculated value of the uncertainty of the stability is included in the final calculation of the combined uncertainty.

$$u_{Stab}(x) = u_{slope} \cdot month \qquad (3)$$

Table 5: Results of stability data evaluation

Property	Intercept	Slope	u_{slope}	$u_{Stab}(x)$	$u_{Stab\_r}(x)$	Instability
A_{BET} in m²/g	24.47156	0.00321	0.00387	0.08901	0.00364	no

The shelf life of CRM BAM-P114 estimated based on the stability monitoring data is at least eight years for a carefully closed bottle stored at temperatures below 30°C under dry conditions.

5. List of participating laboratories

ALFATESTlab, Cinisello Balsamo (Italy)
Bundesanstalt für Materialforschung und –prüfung (BAM), Div. 6.3, Berlin (Germany)
ChemiLytics GmbH & Co. KG, Goslar (Germany)
Evonik Technology & Infrastructure GmbH, Hanau-Wolfgang (Germany)
Forschungsinstitut für Anorganische Werkstoffe -Glas/Keramik- GmbH, Höhr-Grenzhausen (Germany)
Institute of Chemical and Engineering Sciences (ICES), Jurong Island (Singapore)
Instituto Pedro Nunes, Coimbra (Portugal)
Oerlikon Metco WOKA GmbH, Barchfeld-Immelborn (Germany)
Particle Testing Authority - Micromeritics Instrument Corp., Norcross, GA (USA)
TUBITAK NIM, Gebze, Kocaeli (Turkey)
Universiti Teknologi Malaysia, Johor Bahru (Malaysia)
Ural Scientific Research Institute for Metrology (UNIIM), Yekaterinburg (Russian Federation)

Twelve laboratories were invited to the certification interlaboratory comparison. Twelve laboratories agreed to participate and submitted results. One laboratory participated with two instruments.

Most of the participating laboratories had already participated in previous interlaboratory comparisons in the field of gas adsorption measurements. In addition, the laboratories confirmed that their quality management was in accordance with ISO/IEC 17025 or ISO 17034. The types of instruments used by the participants are listed in Table 6.

Table 6: Types of instruments used by the participants

Type of instrument	Number	Manufacturer
NOVA 4000e	1	Anton Paar QuantaTec Inc., Boynton Beach, USA
ASAP 2000	1	Micromeritics Instrument Corporation, Norcross, USA
ASAP 2010	1	
ASAP 2020	2	
ASAP 2420	3	
TriFlex	2	
TriStar 3000	1	
TriStar II Plus 3030	1	
Surfer	1	Thermo Fisher Scientific S.p.A. Milan, Italy
Total *	13	

* One laboratory participated with two instruments

6. Results of the interlaboratory comparison and uncertainty estimation

6.1 Experimental results

The interlaboratory comparison for the certification of BAM-P114 was performed according to the BAM Management Manual [6]. Data evaluation and statistical tests were carried out using the software eCerto [11] and SoftCRM [12].

Each participating laboratory received one bottle together with the instructions for sample preparation and instrumental analysis. The data was evaluated according to ISO 9277 within a relative pressure range of $0.05 \leq p/p_0 < 0.3$. The value used for the cross-sectional area of nitrogen was 0.162 nm^2. The laboratories had to perform five replicate measurements with each participating instrument.

The mean values and standard deviations for the specific surface area gained by each instrument are shown in Table 7 and displayed graphically in Fig. 9.

Table 7: Data set means of the participants in the ILC

	A_{BET}
Data set no.	m^2/g
P01	24.4238
P02	24.4984
P03	24.8270
P04	24.4258
P05	24.1974
P06	24.3032
P07	24.5439
P08	24.3904
P09	24.4760
P10	24.8130
P11	24.4381
P12	24.6270
P13	24.2222
l	13
$\overline{x}$ [a]	24.4758
s_x [b]	0.1938
$\frac{s_x}{\sqrt{l}}$	0.0537
$\overline{x}+1\cdot s_x$	24.6696
$\overline{x}-1\cdot s_x$	24.2821
$\overline{x}+2\cdot s_x$	24.8634
$\overline{x}-2\cdot s_x$	24.0883

[a] Average of the accepted data set means

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

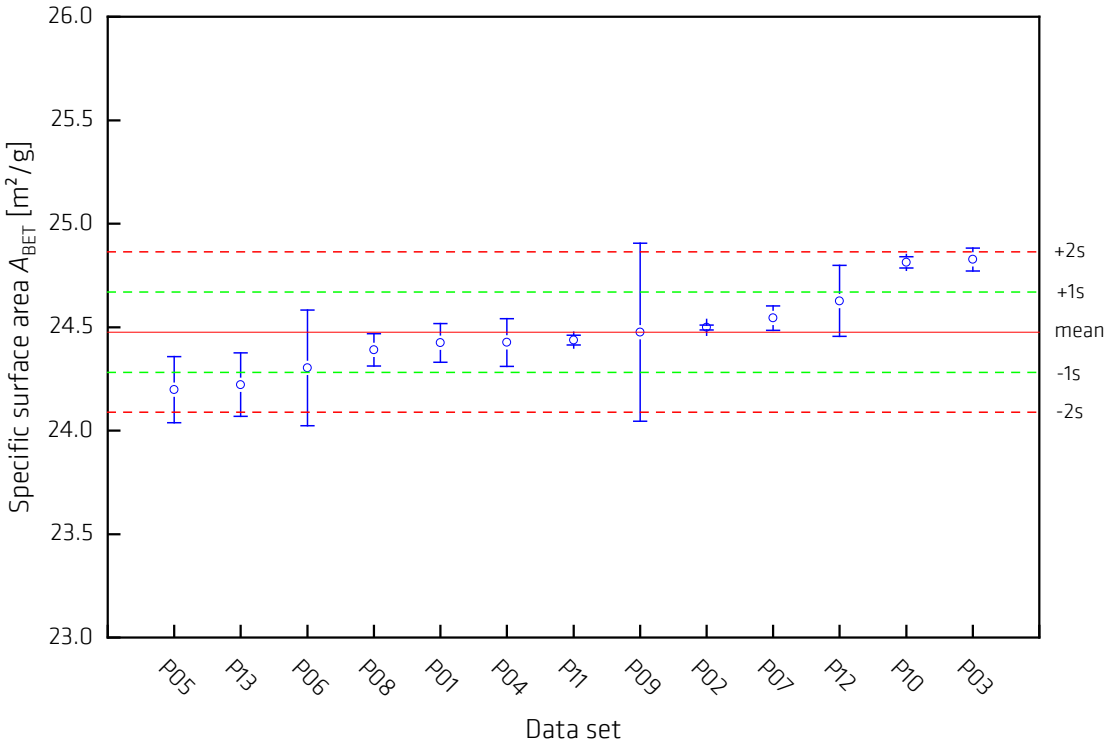

Fig. 9: Results received from ILC participants (l = 13) for A_{BET} (means and standard deviations n = 5)

6.2 Statistical evaluation

An important aspect for the statistical treatment of the experimental data according to ISO Guide 35 to obtain the uncertainty of the certified value was the fact that different instruments were used by the participating laboratories (see Table 6). Furthermore, although all participants of the interlaboratory comparison followed the same standardized procedure, significant differences caused by different implementations in different laboratories were to be expected. This has been confirmed by the observation of 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 value (see Table 9) certification to certify the specific surface area.

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

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

As a result of the statistical analysis (see Table 8) only few outliers were detected by testing of variance for the parameter A_{BET}. The outliers were data sets P09 and P06 which were not removed. The results of the calculation are presented in Table 9.

Table 8: Statistical tests results

Number of accepted Data sets		13
Scheffe's multiple t-test All Data Sets compatible two by two?		yes
TESTING FOR OUTLYING LAB MEANS		
Dixon Test	(α = 0.05) (α = 0.01)	Outliers NOT Detected Outliers NOT Detected
Nalimov t-test	(α = 0.05) (α = 0.01)	Outliers NOT Detected Outliers NOT Detected
Grubbs test (Single)	(α = 0.05) (α = 0.01)	Outliers NOT Detected Outliers NOT Detected
Grubbs test (Double)	(α = 0.05) (α = 0.01)	Outliers NOT Detected Outliers NOT Detected
TESTING OF VARIANCES		
Cochran Test	(α = 0.05) (α = 0.01)	Outliers: P09, P06 Outliers: P09, P06
POSSIBILITY TO POOL THE DATA		
Snedecor-F-Test and Bartlett-Test		Pooling not allowed
Normality of Lab means distribution (In case of NO POOLING)		
Kolmogorov-Smirnov-Lilliefors test	(α = 0.05) (α = 0.01)	Normal Normal

Table 9: Evaluation of the ILC data

Property x	$\overline{x}$	s_x	u_{char}	Unit	Pooling	l
A_{BET} in m^2/g	24.4758	0.1938	0.0537	m^2/g	no	13

Before the combined uncertainty is calculated, the calculated relative uncertainties from u_{hom_r} and u_{Stab_r} are used to adapt the uncertainty budget for the homogeneity (u_{hom_max} to u_{hom}) and stability (u_{Stab} to u_{lts}) for the accepted data set of the interlaboratory comparison (ILC) participants.

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

Mean value from ILC	u_{hom_r}	u_{hom}	u_{Stab_r}	u_{lts}
24.4758	0.00370	0.09046	0.00364	0.08909

The combined uncertainty $u_c(x)$ was calculated according to Equation (4) using the numerical values summarized in Table 10. This equation is a combination of the standard uncertainty of the mean of the instrument means, the contribution of the variation between the bottles, the long-term stability contribution, and the uncertainty contribution due to the measurement result variations of the single instruments (mean data set precision).

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

Table 11: Values of the uncertainty components for the specific surface area of BAM-P114

Property	$\overline{x}$	u_{char}	u_{hom}	u_{lts}	$\frac{1}{l}\sqrt{\sum_{i=1}^{l} s_i^2}$	u_c	U	l	Unit
A_{BET}	24.4758	0.05374	0.09046	0.08909	0.04728	0.14575	0.29151	13	m^2/g

The certified value of the specific surface area and the respective expanded uncertainty (rounded according to DIN 1333 [13]) are summarized in Table 12.

Table 12: Final value for the certified specific surface area of BAM-P114

Property	Certified value x_{cert}	Expanded uncertainty $U = k \cdot u_c$ (k = 2)	Unit
A_{BET}	24.48	0.30	m^2/g

7. Metrological traceability

The certified specific surface area based on the BET method described in ISO 9277 and is 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 a vacuum with a final pressure below 10 Pa. Heat the sample for degassing in a vacuum with a rate of about 5 K/min to 453.15 K, then hold temperature at 453.15 K for at least three hours. During sample pre-treatment, a mass loss of 1.2 % is to be expected. After this period, the sample must be allowed to cool down slowly to ambient temperature.

The adsorption branch of the N_2 isotherm must be measured at 77.3 K. Perform the analysis following the instrument manufacturer's instructions. The recommended sample intake is 1.5 g.

The sample preparation station should have a separate vacuum circuit in addition to the analysis station or the preparation should be carried out at 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

BAM-P114 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 opened as briefly as possible to avoid deterioration of the certified value. 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:

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9. Information on and purchase of the CRM

Certified reference material CRM BAM-P114 is supplied by

Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 1.6: Inorganic Reference Materials
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-P114 will be distributed together with a detailed certificate containing the certified value and their uncertainties, the names of the participating laboratories, the mean values of all accepted data sets and information on the analytical method used.
Information on certified reference materials can be obtained from BAM,
https://www.bam.de.
www.webshop.bam.de

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11. Annexes

Annex 1: Homogeneity study (measurement results)

Annex 2: Measurement results of ILC-participants

Annex 1: Calculation of uncertainty contribution of potential inhomogeneity

	TiO_2 - HOMBIKAT-4							
	Value:	**A_BET**						
	$0.05 \leq p/p_0 \leq 0.3$							
	Homogeneity:	**10 samples x 3 replicates**						
		Replicates						
	Bottle-No.	**1**	**2**	**3**				
		m^2/g	**m^2/g**	**m^2/g**				
	D4(08) - 128	24.3638	24.3049	24.3060				
	C1(04) - 068	24.4664	24.4819	24.4117				
	F3(03) - 179	24.2290	24.2150	24.1854				
	H3(04) - 244	24.2769	24.1937	24.2499				
	B2(06) - 046	24.3168	24.2073	24.2849				
	E3(01) - 145	24.4434	24.4010	24.4991				
	D2(06) -110	24.4157	24.4109	24.4166				
	A3(05) - 021	24.5221	24.4390	24.4661				
	G2(07) - 207	24.1506	24.3928	24.3564				
	C3(02) - 082	24.3730	24.4041	24.3387				
	1-way ANOVA							
	Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*	
	Between Groups	0.24597	9	0.02733	9.01482	2.506E-05	2.39281	
	Within Groups	0.06063	20	0.00303				
	Total	6.33686	29					
	s_bb	0.09000			mean_total	24.35077		
	u'_bb	0.01788						
	u_hom_max	0.09000						
	u_hom_r	0.00370						
	u_hom_max: Maximum uncertainty of s_bb and u'_bb							
	u_hom_r: Relative uncertainty							

Annex 2: Measurement results of ILC-participants

Inter-laboratory comparison for the certification of BAM-P114								
Data set no.	Specific surface area A_{BET} [m²/g]						Mean value	Standard deviation
	Replicate							
	1	**2**	**3**	**4**	**5**			
P01	24.5483	24.4925	24.3490	24.3989	24.3304		24.4238	0.0938
P02	24.4891	24.5028	24.5102	24.5056	24.4841		24.4984	0.0112
P03	24.8439	24.7987	24.8726	24.8753	24.7445		24.8270	0.0555
P04	24.2575	24.3952	24.5565	24.4103	24.5094		24.4258	0.1157
P05	24.1310	24.0894	24.4079	24.3233	24.0354		24.1974	0.1601
P06	24.4950	23.9770	24.1720	24.1940	24.6780		24.3032	0.2796
P07	24.5417	24.5411	24.6263	24.4598	24.5506		24.5439	0.0590
P08	24.2753	24.3571	24.4077	24.4347	24.4771		24.3904	0.0777
P09	24.3514	24.9487	24.0240	24.9094	24.1464		24.4760	0.4300
P10	24.7741	24.8401	24.8178	24.8322	24.8006		24.8130	0.0264
P11	24.4302	24.4632	24.4562	24.4376	24.4031		24.4381	0.0237
P12	24.4919	24.4133	24.6561	24.7617	24.8118		24.6270	0.1711
P13	24.3108	24.4459	24.0786	24.1098	24.1659		24.2222	0.1536