

# NanoValid D.5.47 Annex 3

Inter-laboratory comparison on measurand Specific Surface Area (BET)  
of nanoparticulate Titania (#15 BAM Titania)

## Report of the Results

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A number (12) of external laboratories participated, too, because it was a comparison organized to underpin the certification of the titania test sample #15 BAM Titania (see D5.41/42) as a BAM CRM.

## Summary

This Report describes an inter-laboratory comparison aiming on the establishment of the used method (BET) as a reference method. Another purpose was the certification of the porous reference material #15 BAM Titania as CRM BAM-P110 (cf. D 5.41/42). The certified values determined by nitrogen adsorption at 77.3 K according to the international standards ISO 15901-2 and ISO 9277 are summarized in the Table below.

Property <sup>a</sup>	Value	Uncertainty <sup>b</sup>	Unit
BET Specific Surface Area <sup>c</sup> $A_{\text{BET}}$	107.44	To be delivered after testing the long time stability as part of the formal certification procedure.	m <sup>2</sup> /g

<sup>a</sup> Certified in accordance with the Guidelines for the Production of BAM Reference Materials [1]

<sup>b</sup> Uncertainty  $U = k u_c$  calculated according to ISO Guide 35 [2] and ISO/IEC Guide 98 [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 each certified property includes both an uncertainty contribution resulting from the interlaboratory testing and a second one taking into account possible inhomogeneities of the material

<sup>c</sup> Specific surface area calculated according to the BET model [4] as described in ISO 9277 [5]

The listed value for the surface area are method-defined (model dependent) parameters.

## List of abbreviations

(as far as not explained in particular sections of this Report)

SEM	Scanning Electron Microscopy
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)
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 observations
$N$	number of bottles selected for testing the homogeneity
$s_{\text{between}}^2$	variance between the bottles (homogeneity testing)
$s_{\text{within}}^2$	variance within one bottle (homogeneity testing)
$U$	expanded standard uncertainty of a property value
$u_{\text{bb}}$	standard uncertainty due to between-bottle (in)homogeneity
$u_{\text{char}}$	standard uncertainty due to characterization
$u_{\text{c}}$	combined standard uncertainty of a property value
$u_{\text{Its}}$	standard uncertainty due to long-term (in)stability
$x$	property value of a candidate material
$x_{\text{cert}}$	certified property value of a CRM
$x_i$	result of a single measurement in the experiment

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## 1. Inter-laboratory comparison for certification of #15 BAM Titania as BAM P110 CRM

The certification of this new CRM has been carried out on the basis of BAM Guidelines for the Development of Reference Materials [1] and relevant ISO Guides [2], [3], [9], [10].

## 2. Description of the material

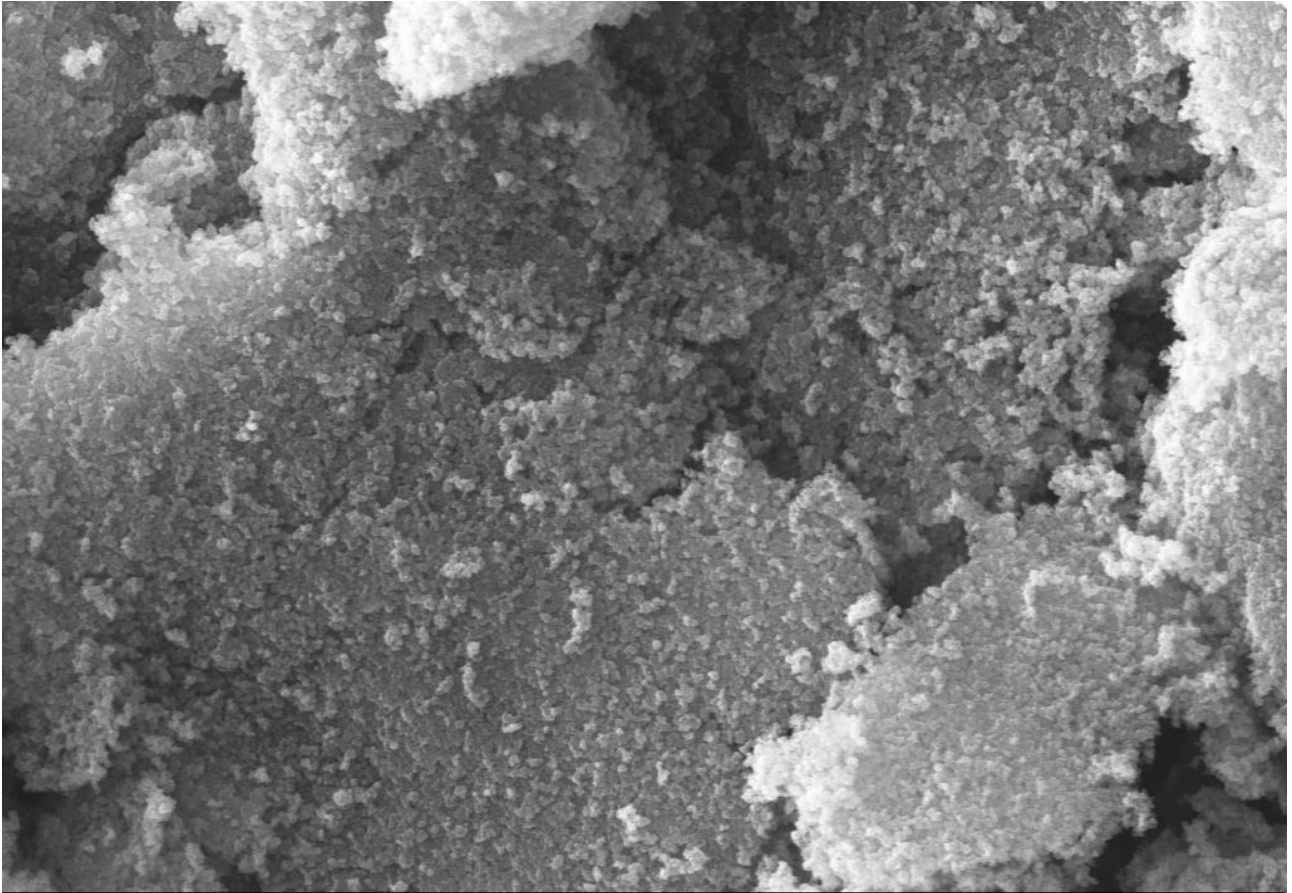
### 2.1 Selection, source and characterization of the candidate material

In comparison to the predecessor material BAM-PM-106, which was mesoporous titania, the successor material is a nanoscaled titania ( $\text{TiO}_2$ , also called titania), #15 BAM Titania (cf. D 5.41/42). It was selected from a number of tested candidate materials. The titania candidate material NO-0058-HP was manufactured and delivered by Iolitec (Germany). In Fig. 1 representative SEM images of the powderous candidate material are shown.

As a result of XRD measurements at BAM, Division 1.3, it could be confirmed that the crystal modification of NO-0058-HP is pure anatase. Although anatase is metastable i.e. thermodynamically less stable than rutile (the other main modification of  $\text{TiO}_2$ ), the phase conversion rate of anatase into rutile is virtually zero at temperatures up to about 600 °C. Therefore, the long term stability of anatase is not affected in the temperature range between room temperature and the recommended degassing temperature of 180 °C (see 2.2). After degassing the sample material in a vacuum (often carried out at elevated temperatures), the first step of BET analysis using the gas adsorption method is the measurement of a low temperature physisorption isotherm (see Fig. 2). By means of appropriate models, a number of porosity parameters can be calculated from the isotherm data such as

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

The degassing of #15 BAM Titania has to be carried out in vacuum. After heating the sample with a rate of about 5 K/min to 180 °C, further heating at this temperature for at least 3 hours is necessary. Afterwards, the sample is allowed to cool slowly back to ambient temperature. The final pressure should be < 0.1 Pa.



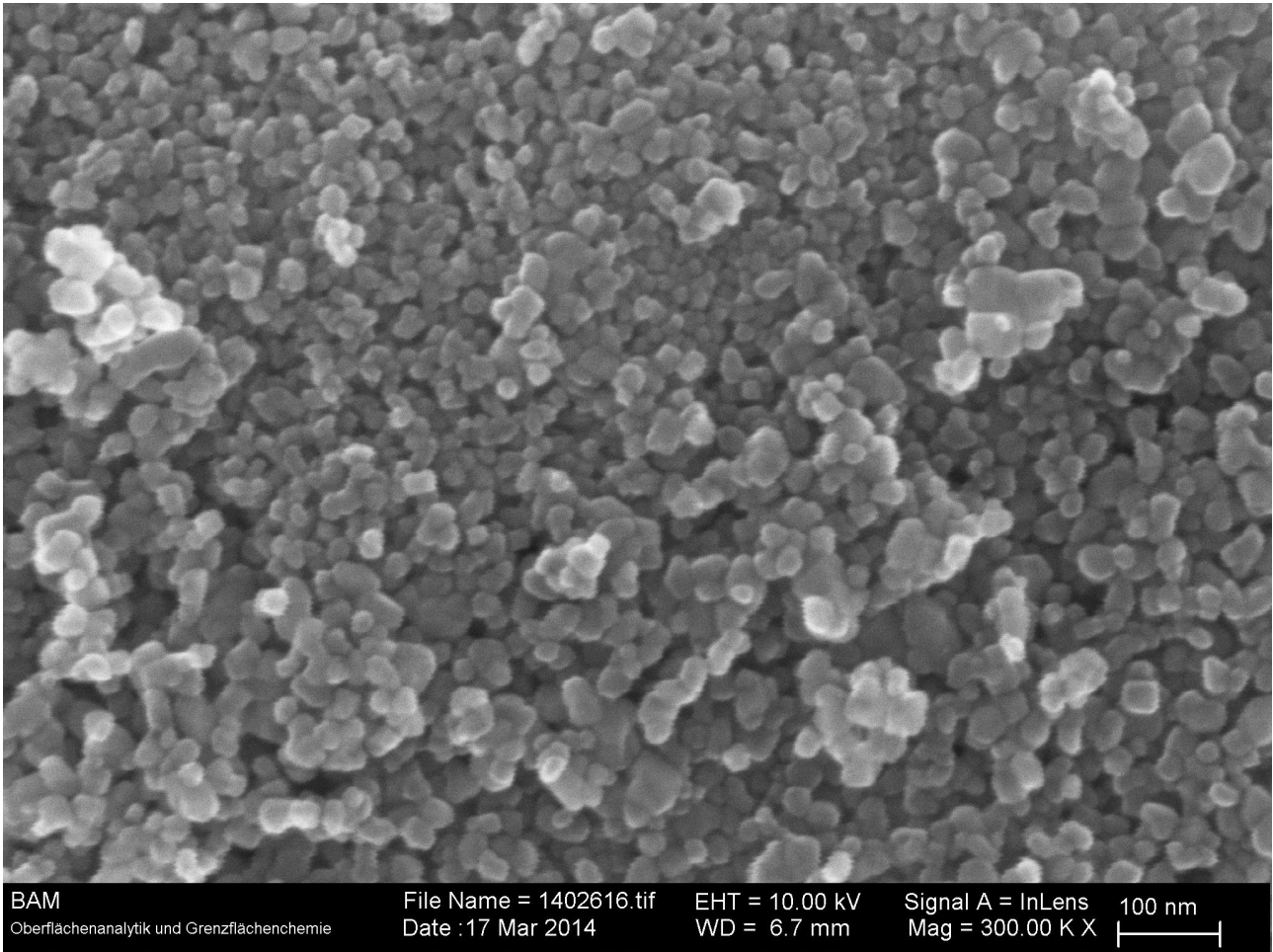
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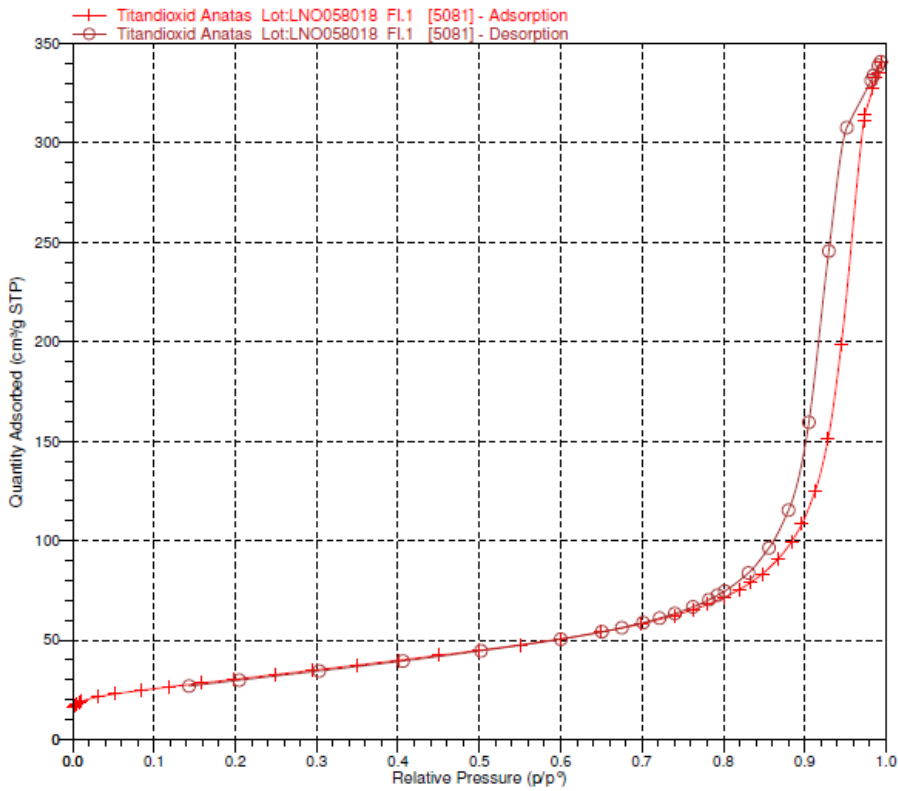
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**Fig. 1:** Scanning electron micrographs of anatase (#15 BAM Titania) at two different magnifications

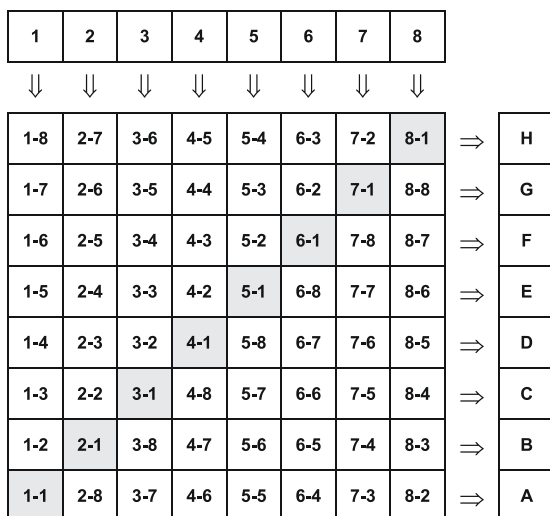




**Fig. 2:** Isotherm of titania NO-0058-HP (#15 BAM Titania)

### 3. Homogenization and subdividing of the candidate material

Homogenization and subdividing of the candidate material were carried out by means of a 8 port rotary sample divider PT 100 (Retsch, Germany) using the cross riffing scheme [12] seen in Fig. 3.



**Fig. 3** Cross riffing scheme

The initial amount of anatase was subdivided into single units of at least 10 g packaged in glass bottles of 30 mL volume. The total number of units was 448, 21 units of them have been used for testing the homogeneity and stability and for the interlaboratory comparison as well.

#### **4. Homogeneity and stability testing**

##### **4.1 Homogeneity**

1. For testing the homogeneity, 20 individual units of BAM-P110 were randomly selected. Two replicate measurements per bottle were carried out under repeatability conditions. To detect the within-bottle standard deviation, 6 replicates from one additional, also randomly selected bottle were measured. The test results are summarized in Tables 1 and 2.

**Table 1: Results of homogeneity testing between bottles (2 replicates for each bottle)**

Random sample no.	Bottle	Data file	$A_{BET}$
			m <sup>2</sup> /g
1	C06-05	ANA-H802	110,1822
		ANA-H803	109,4039
2	D05-02	ANA-H804	110,0236
		ANA-H805	109,7119
3	B04-01	ANA-H806	110,0161
		ANA-H807	109,3451
4	A02-03	ANA-H809	110,2388
		ANA-H810	110,1425
5	E03-06	ANA-H812	110,2012
6	G08-07	ANA-H815	109,2988
		ANA-H816	109,5056
		ANA-H813	109,7370
7	F08-04	ANA-H818	110,0530
		ANA-H819	110,0964
8	B01-03	ANA-H820	109,5593
		ANA-H822	109,8074
9	E07-02	ANA-H828	109,3330
		ANA-H830	109,5428
10	G06-05	ANA-H833	109,9592
		ANA-H834	108,0446
11	F05-01	ANA-H841	109,2363
		ANA-H842	109,6792
12	D02-04	ANA-H843	108,3097
		ANA-H844	109,7685
13	F04-01	ANA-H845	108,4965
		ANA-H846	109,5109
14	A07-01	ANA-H847	110,1145
		ANA-H848	109,8115
15	E06-08	ANA-H849	108,6341
		ANA-H850	109,9420
16	D04-08	ANA-H851	108,5144
		ANA-H852	109,7779
17	A05-07	ANA-H853	110,1397
		ANA-H854	107,9612
18	G04-02	ANA-H855	109,8997

		ANA-H856	109,9441
19	C01-05	ANA-H863	109,2838
		ANA-H864	109,7771
20	C04-03	ANA-H865	109,2520
		ANA-H866	109,6509

**Table 2: Results of replicate measurements with samples from a single bottle**

Bottle	Data file	$A_{BET}$
		$m^2/g$
B08-08	ANA-H857	109,7498
	ANA-H858	109,9620
	ANA-H859	109,9195
	ANA-H860	109,7302
	ANA-H861	109,5444
	ANA-H862	109,7741

To obtain the inhomogeneity contribution  $u_{bb}$  to be included into the total uncertainty budget of each porosity parameter, a 1-way Analysis of Variances (ANOVA) was carried out with the experimental homogeneity data. The  $u_{bb}$  values for #15 BAM Titania (see Table 3) were calculated according to ISO Guide 35 [2] as the maximum of the values obtained from the Equations (1) and (2).

$$u_{bb} = \sqrt{\frac{s_{\text{between}}^2 - s_{\text{within}}^2}{n}} \quad (1)$$

$$u_{bb} = \frac{\sqrt{s_{\text{within}}^2}}{\sqrt{n}} \sqrt[4]{\frac{2}{N(n-1)}} \quad (2)$$

**Table 3: Inhomogeneity contributions  $u_{bb}$  of #15 BAM Titania**

Property	$u_{bb}$	Unit
$A_{BET}$	0.25094834	$m^2/g$

The statistical evaluation of the homogeneity testing results indicated that no significant inhomogeneity for the BET surface area of anatase could be determined and therefore this candidate material has been suitable for the certification as CRM BAM-P110.

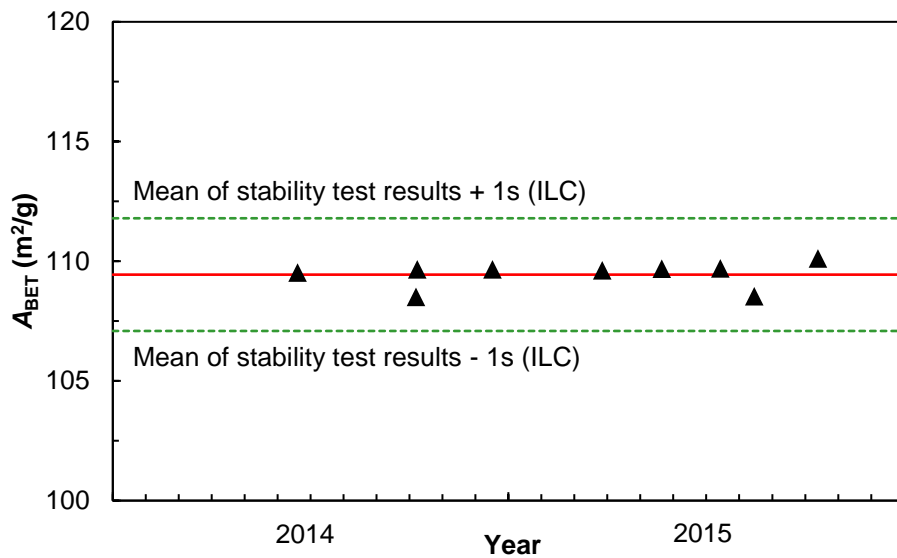
From the homogeneity testing measurements a recommendation for the minimum sample intake of #15 BAM Titania can be derived because these measurements were carried out using sample masses of about 0.8 g. This means that the calculated value of  $\mu_{bb}$  is based on this sample mass and the recommended minimum sample intake for future usage of CRM BAM-P110 also should be 0.8 g.

## 4.2 Stability testing

The numerical results of the measurements to monitor the stability of the #15 BAM Titania are listed in Table 4 for the period between June 2014 and October 2015. The stability measurements were carried out with the same automated surface area and porosity analyzer ASAP 2020 (Micromeritics, Norcross USA). The respective diagrams for BET surface area are depicted in Fig. 4.

**Table 4: Numerical results of stability monitoring**

Data file	Test date	$A_{\text{BET}}$
		m <sup>2</sup> /g
ANA-749.smp	18.06.2014	109.52
ANA-795.smp	06.10.2014	108.50
ANA-795.smp	07.10.2014	109.66
ANA-H866.smp	16.12.2014	109.65
ANAT-899.smp	26.03.2015	109.62
ANA-918.smp	20.05.2015	109.68
ANA-941.smp	13.07.2015	109.70
ANA-955.smp	14.08.2015	108.53
ANA-989.smp	12.10.2015	110.10
	$\bar{x}_{\text{Stab}}^{\text{a}}$	109.44
	$\bar{x}_{\text{ILC}}^{\text{b}}$	107.44
	$s_{x,\text{ILC}}$	2.35
	$\bar{x}_{\text{Stab}} + 1 \cdot s_{x,\text{ILC}}$	111.79
	$\bar{x}_{\text{Stab}} - 1 \cdot s_{x,\text{ILC}}$	107.09
<sup>a</sup> Stab = stability monitoring, <sup>b</sup> ILC = interlaboratory comparison (certification round robin)		



**Fig. 4:** Stability monitoring for the porosity property  $A_{\text{BET}}$  (BET specific surface area)

The results of the statistical evaluation of the stability data indicate that no instability could be detected for the porosity parameter BET specific surface area but the contribution  $u_{\text{ITS}}$  to the uncertainty of the certified values is not negligible and should be included in the final calculation of the combined uncertainty.

## 5. Alphabetical list of participating laboratories

AQura GmbH, Hanau (Germany)

AQura GmbH, Marl (Germany)

Bundesanstalt für Materialforschung und –prüfung (BAM), Fachbereich 1.3, Berlin (Germany)

Bayer Technology Services GmbH, Leverkusen (Germany)

Bayerisches Zentrum für Angewandte Energieforschung e.V., Würzburg (Germany)

BEL Japan Inc., Osaka (Japan)

Centro Ricerche FIAT S.C.p.A., Orbassano (Italy)

Delft Solids Solutions, Delft (The Netherlands)

Fraunhofer-Institut für Keramische Technologien und Systeme (IKTS), Hermsdorf (Germany)

INMETRO, Duque de Caxias (Brazil)

Micromeritics Instrument Corp., Norcross, GA (USA)

Micromeritics GmbH, Aachen (Germany)

Nanologica AB, Södertälje (Sweden)

Oerlikon Metco WOKA GmbH, Barchfeld-Immelnborn (Germany)

POROTEC GmbH, Hofheim (Germany)

PTT Global Chemical Public Company Limited, Noenphra (Thailand)

Quantachrome GmbH, Odelzhausen (Germany)

Quantachrome Instruments, Boynton Beach (USA)

ThermoFisher Scientific, Milano (Italy)

University of Alicante (Spain)

Instituto de Nanociencia de Aragón – University of Zaragoza, Zaragoza (Spain)

Veneto Nanotech, Rovigo (Italy)

The majority of the laboratories had already participated in previous interlaboratory comparisons in the field of gas adsorption measurements organized by BAM. Eight laboratories took part for the first time. All of them had to report about their calibration and reference material used.



**Table 6: Types of instruments used by the participants**

Type of instrument	Number	Manufacturer
Surfer	2	ThermoFisher Scientific, Waltham (USA)
ASAP 2000	1	Micromeritics Instrument Corporation, Norcross, GA (USA)
ASAP 2010	1	
ASAP 2020	4	
ASAP 2420	1	
TriStar 3000	3	
TriStar 3020	1	
TriStar II PLUS	1	
TriStar Gemini 2360	1	
Autosorb-1	2	Quantachrome Instruments Corporation, Boynton Beach, FL (USA)
Autosorb-6B	1	
Autosorb-iQ	2	
Nova 3200e	1	
Nova 2000e	1	
BELSORP-mini II	1	BEL Japan Inc., Osaka (Japan)
BELSORP-max	1	
non-branded (self made)	1	University of Alicante (Spain)
Total *	25	

\* Three laboratories participated with two instruments each

## 6. Results of the interlaboratory testing and statistical uncertainty estimation

### 6.1 Experimental results

The interlaboratory comparison for the certification of #15 BAM Titania as BAM-P110 was performed on the basis of the Guidelines for the Production of BAM Reference Materials [1], data evaluation and statistical tests were carried out using the software package SoftCRM [13]. Each participating laboratory received a bottle containing about at least 10 g of the candidate material together with the instructions for running the tests and the data evaluation according to ISO standards 9277 [5] and 15901-2 [8]. The laboratories had to perform 6 replicate measurements with each participating instrument. The mean values for the BET specific surface area gained by each instrument are shown in Table 7 and displayed graphically in Fig. 5. The error

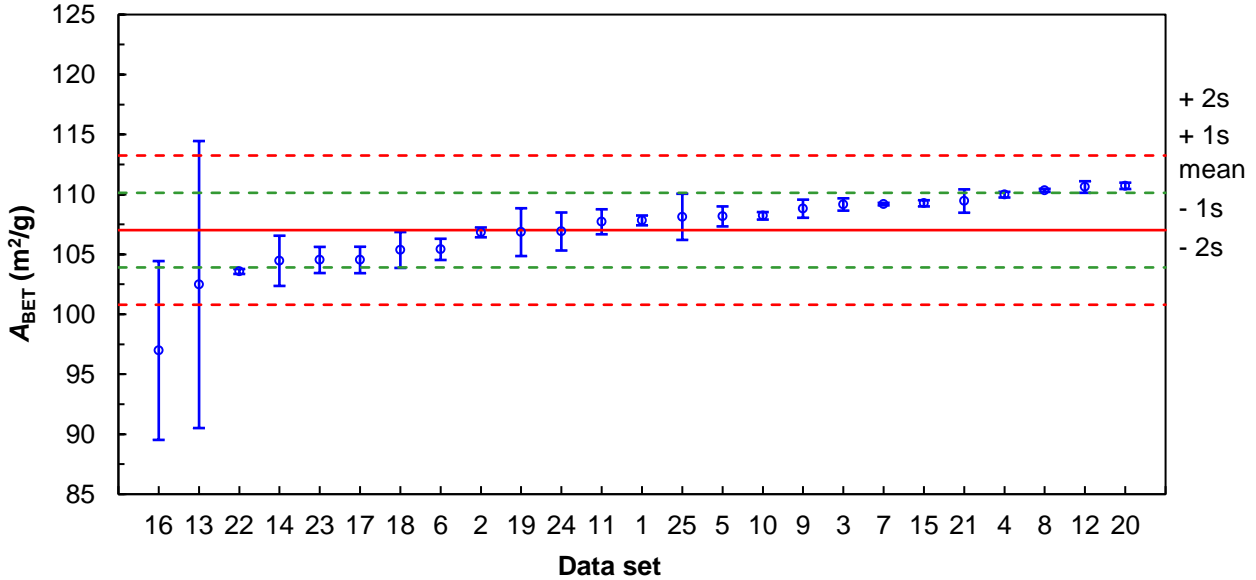
bars at the data points for the data set means represent the standard deviation of the 6 certification measurements per data set.

**Table 7: Data set means of the participants in the interlaboratory comparison (ILC)**

Property $x \rightarrow$	$A_{\text{BET}}$
Data set no.* $\downarrow$	$\text{m}^2/\text{g}$
01	107.83
02	106.83
03	109.16
04	109.98
05	108.17
06	105.42
07	109.19
08	110.34
09	108.81
10	108.22
11	107.72
12	110.62
13	102.48
14	104.46
15	109.26
16	96.98 <sup>a</sup>
17	104.53
18	105.37
19	106.85
20	110.72
21	109.45
22	103.57
23	104.53
24	106.91
25	108.13
$\bar{x}$ <sup>b</sup>	107.44
$s_x$ <sup>c</sup>	2.35
$l$	24
$u(x) = \frac{s_x}{\sqrt{l}}$	0.48
$\bar{x} + 1 \cdot s_x$	109.79
$\bar{x} - 1 \cdot s_x$	105.09
$\bar{x} + 2 \cdot s_x$	112.15
$\bar{x} - 2 \cdot s_x$	102.73
<p>* data set numbers do not correlate to the alphabetical list of participants!  <sup>a</sup> insufficient data set mean for the particular property statistically detected as outlier and therefore have not been included in the evaluation  <sup>b</sup> average of the accepted data set means for the particular property on the basis of 6 single isotherm measurements each</p>	

<sup>c</sup> standard deviation of the data set means for the particular property

**Fig. 5: Result of inter-laboratory comparison: Calculated data set means for  $A_{\text{BET}}$**



## 6.2 Statistical evaluation

An important aspect for the statistical treatment of the experimental data according to ISO Guide 35 to obtain the uncertainties of the certified values was the fact that different instruments were used by the participating laboratories (see Table 6). Moreover, although all participants in the intercomparison 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 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 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 an outlier was detected for the parameter  $A_{\text{BET}}$  data set No. 16.

This instrument mean value was excluded from the calculations of the certified values and their uncertainties. The results of the calculations with the evaluation software SoftCRM [13] after deleting the outlying instrument mean are presented in Table 8.

**Table 8: Statistical evaluation of the ILC data using the software program SoftCRM**

Property $x$	$\bar{x}$	$s(x)$	$u(x)$	CI	TI	Unit	Pooling	$l$
$A_{\text{BET}}$	107.4395	2.3537	0.4804			m <sup>2</sup> /g	no	24

The plausibility of the obtained means of the instrument means has been checked by the comparison between the results of the homogeneity measurements, the stability study, and the ILC measurements (see Table 9).

**Table 9: 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
$A_{\text{BET}}$	109.56	109.44	107.44	

The combined uncertainty  $u_c(x)$  for each certified value was calculated according to Equation (3) using the numerical values summarized in Table 10 as the 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). The last contribution also serves as an estimate for possible inhomogeneities within the bottles.

$$u_c^2(x) = u_{\text{char}}^2 + u_{\text{bb}}^2 + u_{\text{Its}}^2 + \frac{1}{l^2} \sum_{i=1}^l s_i^2 \quad \text{with} \quad u_{\text{char}}^2 = \frac{s_x^2}{l} \quad (3)$$

**Table 10: Values of the uncertainty components for the parameter BET specific surface area of #15 BAM Titania (BAM-P110)**

Property	$\bar{x}$	$u_{\text{char}}(x)$	$u_{\text{bb}}(x)$	$u_{\text{Its}}(x)$	$\frac{1}{l} \sqrt{\sum_{i=1}^l s_i^2}$	$u_c(x)$	$U(x)$	$l$	Unit

$A_{\text{BET}}$	107.4395	0.4804	0.2509		0.5396			24	m <sup>2</sup> /g
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The certified value of BET surface area with a reasonable number of digits and the respective expanded uncertainty (rounded according to DIN 1333 [14]) are summarized in Table 11.

**Table 11: Final values for the certified porosity properties of #15 BAM Titania (BAM-P110)**

Property	Certified value $x_{\text{cert}}$	Expanded uncertainty $U = k \cdot u_c$ (with $k = 2$ )	Unit
$A_{\text{BET}}$	107.44		m <sup>2</sup> /g

The formal BAM certificate and certification report for P110 are accessible at:

[https://www.webshop.bam.de/show\\_blob\\_data.php?filename=pdf/certificates/bam\\_p110e.pdf](https://www.webshop.bam.de/show_blob_data.php?filename=pdf/certificates/bam_p110e.pdf)

and

[https://rrr.bam.de/RRR/Content/EN/Downloads/RM-certificates/RM-cert-porous-materials/bam\\_p110repe.pdf?\\_blob=publicationFile](https://rrr.bam.de/RRR/Content/EN/Downloads/RM-certificates/RM-cert-porous-materials/bam_p110repe.pdf?_blob=publicationFile)

## 7. References

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