2008 年 3 月 31 日　本文のタイトルと付属文書のタイトル、Table1、AnnexB の TableB,TableC、Commentary の 5. を修正。2008 年 7 月 28 日　AnnexB の Table B3 のタイトルを修正。

Standard accepted by the Association of Powder Process, Industry and Engineering, JAPAN

SAP 11-05

Reference Powder for Specific Surface Area

Established March 16, 2006

The Association of Powder Process, Industry and Engineering, JAPAN

1. Scope

This standard specifies the reference powder for calibration of the specific surface area analyzer using gas adsorption method.

Note 1. This standard is based on the following standards.

JIS K 5116 Titanium dioxide(pigment)

JIS K 6216-2 Carbon black for rubber industry－General matters－
Part 2：Standard reference blacks

JIS K 6217-1 Carbon black for rubber industry－Fundamental characteristics－
Part 1：Determination of iodine adsorption number－Titrimetric method

JIS K 6217-4 Carbon black for rubber industry－Fundamental characteristics－
Part 4：Determination of dibutylphthalate absorption number

JIS Z 8103 Glossary of terms used in measurement

JIS Z 8830 Determination of the specific surface area of powders (solids) by gas adsorption method

JIS Z 8901 Test powders and test particles

2. Definition of technical terms

The main technical terms used in this standard are defined by **JIS K 5116, JIS K 6216-2, JIS K 6217-1, JIS K 6217-4, JIS Z 8103, JIS Z 8830** and **JIS Z 8901**.

3. The kind of reference powders

Reference powders for specific surface area are defined as the three kinds listed in Table 1.

Table 1 Three kinds of reference powders for specific surface area

Class	Raw material	Nominal specific surface area(m^2/g)
1	Titanium dioxide(rutile)	10
2	Carbon black	25
3	Carbon black	110

4. Quality The quality of reference powders for specific surface area is specified as followings.

4.1 Class 1 Titanium dioxide(rutile) The quality of Titanium dioxide(rutile) should be characterized by the method which is regulated in **JIS K 5116**, and should be restricted for the value listed **in Table 2**.

Table 2 The properties of Class 1 Titanium dioxide(rutile)

Item	Regulated value
Purity	＞97.0%
Water soluble content	＜0.6%
Volatile content at 105℃	＜1.0%

4.2 Class 2 Carbon black and Class 3 Carbon black The quality of Class 2 Carbon black and Class 3 Carbon black should be characterized by the following method.

(1) Chemico-physical properties Chemico-physical properties should be measured by the method which is described **in JIS K 6217-1** and **JIS K 6217-4**, and should be restricted for the value listed in **Table 3**.

Table 3 Chemico-physical properties of Class 2 and Class 3 Carbon black

	Regulated value	
Item	Class 2	Class 3
Iodine adsorption(g/kg)	27.5 (±1.5)	118.0 (±2.0)
DBP adsorption($10^{-5}m^3/kg$)	28.0 (±1.5)	112.0 (±2.0)

5. Specific surface area The specific surface area should be measured according to the procedure which is described in **JIS Z 8830**, and using the volumetric method or the carrier gas method. The measured specific surface area should be restricted for the value listed in **Table 4**.

Table 4 Specific surface area for reference powders

Kind of reference powder	Class 1	Class 2	Class 3
Specific surface area (m^2/g)	8.93	23.5	110.7
Uncertainty*	0.26	1.10	5.76

*Expanded uncertainty $U=ku_c$ (95% level of confidence, coverage factor k=2) in m^2g^{-1}, where u_c is a combined standard uncertainty for the average, calculated according to the [GUM][1].

6. Notice of handling

6.1 Storage The reference powder should be stored under the dry condition after opening a seal of vial.

6.2 Limit of storage term The reference powder which has elapsed more than five years after opened the vial should not be used.

6.3 Repeat use In the case of repeat use, the reference powder which has elapsed more than 24hours after the first use should not be used.

7. Indication label Indication label of the reference powders for specific surface area should include the information of "The Association of Powder Process, Industry and Engineering, JAPAN SAP 11-05" and following items.

(1) Kind of s reference powder for specific surface area

Example　Class 2　Reference Powder for Specific Surface Area

(2) Low material

Example　Carbon black

(3) Lot number

(4) Range of the specific surface area (Displaying to decimal one column)

Example　Maximum value　26.0m²/g,　Minimum value　22.5m²/g

(5) Net mass

(6) Manufacturer

Bibliography

(1) ISO/IEC Guide 98: Guide to the Expression of Uncertainty in Measurement ［GUM］, International Organization for Standardization, 2nd Ed., Geneva (1995)

Annex A
(Informative)
Experimental Conditions of Reference Powders for Specific Surface Area

The reference powders for calibration of the specific surface area specified in this standard are following three kinds of powders.

① Class 1　Titanium dioxide(rutile)
② Class 2　Carbon black
③ Class 3　Carbon black

In this Annex, the pre-treatment condition, measuring condition and calculating condition, in the case of using the reference powders for specific surface area, are described.　In addition, these conditions almost submit to **JIS Z 8830** .

1. Pre-treatment conditions　The reference powder should be prepared under the following conditions before maesurement.

① Drying method:　Purging by gas flow
② Kind of flow gas:　Nitrogen gas
③ Gas flow rate:　Approximately 20 cm^3/min
④ Gas pressure:　Approximately 100 kPa
⑤ Heating temperature:　200℃
⑥ Treating time:　2 hours

2. Measuring conditions　Analyzers have the each suitable operational conditions. These conditions should be adopted, when the reference powder is analyzerd.　The measuring conditions being common to analyzers are shown as below.

① Adsorntive gas:　For volumetric method, the nitrogen gas having more than 99.99% purity.
For carrier gas method, the mixture gas of nitrogen gas and helium gas (at the mixing ratio being assigned to the analyzer).
② Adsorbing temperature:　Boiling temperature of the liquid nitrogen. (Approximately 77K)
③ The mass of sample should be weighed to a figure of mg at least.

3. Calculation conditions　The **Eq.(1)** in **JIS Z 8830** is used to calculate data.　This equation is incorporated in all marketed analyzers to evaluate the surface area automatically. The calculation conditions are showed as below.

1) Multipoint determination: Calculating surface area by using plural BET plots within relative pressure range of 0.05 to 0.3.
2) Singlepoint determination: Calculating surface area by using a BET plot around 0.3 of relative pressure.
3) Calculate surface area by using 0.162nm^2 of the molecular cross sectional area of nitrogen.

Annex B
(Informative)
Result of Round-Robin Test for Reference Powders for Specific Surface Area

In 2004, the technical group of the Measurement and Control in the Association of Powder Process, Industry and Engineering, JAPAN performed a round-robin test of the reference powder based on the conditions explained in Annex A. To this test, three manufacturers and four dealers who market the specific surface area analyzer in Japan participated. The name of participators and their instrument are shown in following Table B1.

Table B1　Participator and analyzer in the round-robin test

Participator	Instrument name
BEL Japan,Inc.	Belsorb-mini
Shimadzu corporation	TriStar 3000
MOUNTECH Co.,Ltd.	Macsorb HM-1201MP
YUASA IONICS CO.,LTD.	Autosorb 1 Monosorb NOVA 4200 Quadrasorb SI,Kr/MP Multisorb-16
MICRO.DATA CO.	MS4242Ⅱ
AMCO	Q-surf
Beckman Coulter KK.	SA3100

(Order of participation application)

In the test program, each instrument analyzed three kind of the reference powders and performed three times analysis for one kind of powder. The summary of test results are shown in Table B2 and Table B3.

Table B2　Summary of test results (Singlepoint determination)

Class of RP	Average(m^2/g)	Uncertainty*
1	8.83	0.55
2	23.6	1.78
3	111.8	7.54

Table B3　Summary of test results (Multipoint determination)

Class of RP	Average(m^2/g)	Uncertainty*
1	8.88	0.55
2	23.8	1.10
3	111.7	8.62

* Expanded uncertainty $U=ku_c$ (95% level of confidence, coverage factor k=2) in m^2g^{-1}, where u_c is a combined standard uncertainty for the average, calculated according to the [GUM][1].

Bibliography

(1) ISO/IEC Guide 98: Guide to the Expression of Uncertainty in Measurement ［GUM］, International Organization for Standardization, 2nd Ed., Geneva (1995)

SAP 11-05
Reference Powder for Specific Surface Area
COMMENTARY

This commentary explains the items regulated and described in the standard and annex and also comments the related items , but is not a part of the standard .

This commentary is edited and issued by the Association of Powder Process, Industry and Engineering, JAPAN, and it is wished that the inquiry relating to the description is asked to the Association.

1. Purpose of standardization The specific surface area is one of the most important properties for the powder. In 1995, **ISO 9277** "Determination of the specific surface area of powders (solids) by gas adsorption method" was standardized by ISO TC24/SC4. On the other hand, in Japan, in 1996, **JIS R 1626** "Measuring methods for the specific surface area of fine ceramic powders by gas adsorption using the BET method" was standardized, and, in 2001, **JIS Z 8830** "Determination of the specific surface area of powders (solids) by gas adsorption method" which translated **ISO 9277** was also standardized.

However, these standards have described the procedure and instrument in detail comparatively, but have not mentioned concretely about the reference material, which is used to verify the reliability of analyzers. Therefore, in the field relating to powder, especially in the field considering the importance of specific surface area of powder, the standardization of the reference materials, that can verify the reliability and performance of the analyzer, has been desired.

2. Process of standardization In the powder and particle industry field, the reference materials to calibrate the particle size distribution analyzer were discussed previously, and the Standard of the Association of Powder Process, Industry and Engineering, JAPAN, **SAP 10-03** "Reference Particles" were regulated in 2003. For the specific surface area, the standardization of the reference materials which can verify the analyzer was also requested, so APPIE decided to standardize the specific surface area reference materials in the same way as **SAP 10-03**.

3. Considered matter during deliberation

3.1 Kind of material From the necessity of avoiding a change by the elapsed time and having suitable quality to verify the performance of the specific surface area analyzer, the titanium dioxide and the carbon black were selected.

Range of application At present, the air-permeability method and gas adsorption methods are used practically. In these both methods, the gas adsorption method is

used more widely and the specific surface area reference material for the gas adsorption method is demanded more frequently than the air-permeability method. By these reasons, the object of the reference material discussing here was directed to the analyzers using the gas adsorption method. This method includes volumetric method, carrier gas method and gravimetric method. But, in the round-robin test using the reference powder, the volumetric method analyzer and the carrier gas method analyzer participated but the gravimetric method analyzers did not, so the gravimetric method analyzers were not discussed in this standard.

3.3 Adsorptive gas In the measurement of specific surface area, the nitrogen gas, argon gas or krypton gas is used, however, because this standard describes the reference materials to verify analyzers, the nitrogen gas being most generally used has been standardized as the adsorptive gas.

3.4 Evaluation method of specific surface area The BET equation is used to evaluate the specific surface area, and single-point determination method and multipoint determination method are used to measure the specific surface area. At present, both the single-point determination analyzer and the multipoint determination analyzer are marketed. Generally, on the multipoint analyzer, it is also possible to evaluate the specific surface area by the single-point determination method. Because, both evaluation methods were used, in early stage of discussion, two kinds of standard were planed. However, the result of round robin test showed insignificant deference between both determinations, so it was decided to make single standard covering both determinations (**Table 4** in main body of the standard). For this reason, the interval between maximum value and minimum value became slightly large.

4. Problem for real specific surface area The most significant matter in this standard is the real value of specific surface area in the reference materials. The gas permeability method, gas adsorption method, liquid adsorption method, heat of immersing method etc. are mentioned as the measuring method of the specific surface area of materials, however these methods are not traceable to the international standard and the national standard. Furthermore, the liquid adsorption method and heat of immersing method are almost not used now. Therefore, it may be said that there is no way to measure the specific surface area other than the gas permeability method and gas adsorption method under the present situation. In this sense, it is said that the values of specific surface area described in this standard don't have the traceability, but these values have been obtained from majority of analyzers.

5. How to use reference powder As described above, there are several way to measure the specific surface area in the gas adsorption method, and as the evaluation

method, there are the single-point determination and the multipoint determination. Additionally, each evaluation method includes several variations, such as a relative pressures where BET plot being read out in single-point determination, or such as the number of BET plots should being taken in the multipoint determination. Each analyzer marketed presently adopts different evaluation methods. From the standpoint of hardware, different analyzers are consist of different components, and the arrayment of these components is not same among different analyzers. Therefore, even if using the same reference material, there is possiblity that different results are obtained from different analyzers and the measured result does not agree with the regulated value.

Though, the reference powders regulated in this standard have been prepared to have the specific surface area not exceeding the limit of uncertainty described in **table 4**, there is possibility that the result deviating from the regulated specific surface area is obtained occasionally from the analyzer, which has a habit giving a result near the limit of uncertainty. Then, in the case that the result of analysis is judged with the reference material regulated in this standard, the following procedure is recommended.

1) The reference powder should be prepared under the conditions described in Annex A.
2) The analysis should be performed more than three times. And each result should be within the limit of uncertainty described in Table B2 or Table B3 in Annex B.
3) The average value of analysis results should be compared with “Range of specific surface area” labeled on the vial.