



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2206

#### Controlled Pore Glass - BET Specific Surface Area

(Nominal Pore Diameter 300 nm)

This Standard Reference Material (SRM) is intended for the calibration and performance testing of instruments used for the determination of the Brunauer-Emmett-Teller (BET) specific surface area (SSA) by the static volumetric gas sorption technique. A unit of SRM 2206 consists of one bottle containing approximately 5.1 g of high-purity granulated controlled-pore glass with a nominal pore diameter of 300 nm. This SRM is certified for BET SSA determined by nitrogen gas sorption at liquid nitrogen temperature (77.3 K) using commercial instrumentation.

**Certified Values:** Certified values for the single-point (SP) and multiple-point (MP) BET analysis of specific surface area are listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. Certified values are based on measurements performed at NIST and validated by qualified collaborating laboratories [2].

Table 1. Certified Values for BET Specific Surface Area<sup>(a)</sup>

| Measurement Technique | Specific Surface Area Value<br>(m <sup>2</sup> /g) |
|-----------------------|--|
| MP                    | 10.99 ± 0.68                                       |
| SP                    | 10.73 ± 0.68                                       |

<sup>(a)</sup> The assigned value is a weighted mean of the results from measurements of ten bottles of the material using a Gaussian, linear mixed effects model [2]. The uncertainty listed with the value is an expanded uncertainty with coverage factor  $k = 2$ , corresponding to a 95 % confidence interval, calculated by Monte-Carlo simulation of uncertainty components using methods from the ISO Guide or its Supplement [3,4]. It includes between-bottles as well as within-bottle heterogeneity components, plus an additional type B evaluated component.

**Expiration of Certification:** The certification of **SRM 2206** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recertification of this SRM is not required. However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor the certified values of this SRM; if substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements for certification was accomplished under the direction V.A. Hackley of the NIST Ceramics Division. Certification measurements were performed at NIST by V.A. Hackley. Material selection, procurement, and production aspects were carried out by W. Haller, V.A. Hackley, and J.F. Kelly of the NIST Ceramics Division.

Statistical consultation for this SRM was provided by B. Toman of the NIST Statistical Engineering Division.

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Validation measurements were provided by P. Klobes from Bundesanstalt für Materialforschung und -prüfung (Berlin, Germany); R. Ahmad of Quantachrome Corporation (Boynton Beach, FL, USA) and J. Kenvin of Micromeritics Instrument Corporation (Norcross, GA, USA).

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

**Heterogeneity Assessment:** Ten units were selected using a stratified random sampling procedure and used for certification and heterogeneity testing at NIST based on BET SSA measurements. Analysis of these ten units was randomized, and two samples were tested from each of the ten units.

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Handling:** SRM 2206 is granular with a grain size principally larger than 130  $\mu\text{m}$ , resulting in good flowability to enable ease of sampling and transfer operations. The material is composed principally of silica (mass fraction >99 %).

**Storage:** SRM 2206 should be stored in the original bottle with screw cap tightly sealed. Bottles can be stored at normal laboratory ambient temperature and humidity. In order to avoid possible contamination, material removed for testing should not be returned to the original bottle but can be reused indefinitely if stored separately under similar conditions in a clean sealed bottle or measurement tube; non-compliance with the certified values indicates that a reused sample should be replaced with a new sample taken from the original bottle.

**Sampling Procedure:** Before removing a sample for testing, the bottle containing SRM 2206 (with screw cap in place) should be gently and alternately rotated and inverted several times to ensure mixing. Using a clean stainless steel spatula or similar device, remove sufficient material for analysis, and transfer to an appropriate-size pre-weighed glass sample tube; the mass to be analyzed should be between 0.5 g and 0.9 g. A funnel can be utilized to deliver powder more efficiently to the bottom of the sample tube. Gentle tapping is applied to the funnel and/or sample tube in order to ensure that the powder is delivered to the bulb area at the bottom. If powder persists on the inner surface of the tube stem (above the bulb), a pipe cleaner can be used to remove this material.

**Gravimetric Procedure:** Sample mass for gas sorption analysis should be measured using a properly calibrated analytical balance that reads to  $\pm 0.1$  mg or better. The mass of the dry empty tube plus fill rod (if used) and sealing device (e.g., stopper or seal frit) should then be recorded (mass = M1). The fill rod and sealing device should then be removed and the balance tared with the empty tube to facilitate sample transfer. After transferring an appropriate mass of sample (see above), the mass of the tube + sample + fill rod + sealer should be recorded (mass = M2). After weighing operations are complete, the instrument manufacturer's instructions or accepted practice should be followed to seal and install the sample tube for outgassing (see below). Once outgassing is complete and the sample is cooled to room temperature, measure and record the mass of the tube + outgassed sample + fill rod (if used) + sealing device (mass = M3). Subtract M1 from M3 to obtain the mass of the outgassed sample to  $\pm 0.1$  mg; use this value for calculation of BET SSA. Subtract mass M3 from mass M2 to obtain the mass loss resulting from outgassing (moisture removal); the mass loss is typically 1.5 % to 4 %, but can exceed this range depending on ambient humidity and storage conditions.

**Outgassing Procedure:** The sample should be outgassed under vacuum. Heat the sample to 110  $^{\circ}\text{C}$  at a rate not to exceed 10  $^{\circ}\text{C}$  per minute. If automated evacuation control is available, the sample tube should be evacuated at a maximum rate of 0.667 kPa/s (5 Torr per second); if the rate exceeds this value then the heating ramp should be suspended until the pressure drops into the safe range. This procedure prevents sample powder uptake into the evacuation system. Once the pressure drops below 0.667 kPa (5 Torr), unrestricted evacuation should proceed. In the absence of automated evacuation control, a slower temperature ramp (e.g., 1  $^{\circ}\text{C}$  per minute) is recommended. Hold sample temperature at the first set point (110  $^{\circ}\text{C}$ ) for approximately 10 minutes. Then raise the sample temperature to 300  $^{\circ}\text{C}$  at a rate not to exceed 10  $^{\circ}\text{C}$  per minute and hold at the maximum set point temperature *for at least 2 hours and no more than 6 hours*. After completion of outgassing, turn off heat, backfill with nitrogen gas, and allow the sample to cool to room temperature. If helium is used for backfill instead of nitrogen, a gravimetric error of order 3 % to 5 % can be expected due to the density difference between helium and air; this error can be reduced by using a fill rod or increasing the sample volume (i.e., reducing the dead space), but it is preferable to backfill with nitrogen.

**Analysis Procedure:** Sample analysis should be initiated as soon as possible following completion of outgassing and weighing operations. Follow the instrument manufacturer's recommendations or accepted practice to measure BET SSA using nitrogen gas as the adsorptive at liquid nitrogen temperature. The certified values were determined

using a discontinuous manometric technique as described in ISO 9277 [5]; use of other measurement techniques may result in values that deviate beyond the certified uncertainty range. The following parameters should be used for measurements. For multi-point analysis, choose at least four measurement points (preferably more) evenly distributed over the relative pressure ( $p/p_0$ ) range from 0.05 to 0.3 (where  $p$  and  $p_0$  are the equilibrium and the saturation pressure of the adsorptive, respectively). The intercept for the linear BET plot must be positive and the correlation coefficient ( $r^2$ ) for linear regression must be at least 0.999. For single-point analysis, use a  $p/p_0$  value close to but not exceeding 0.3. A value of  $0.162 \text{ nm}^2$  must be used for the molecular cross-sectional area of adsorbed nitrogen gas. The purity of helium gas used to calibrate measurement volumes (e.g., free space determination) should be at least 99.999 %. The purity of the adsorptive nitrogen gas should be at least 99.99 %. The liquid nitrogen level should be maintained at least 50 mm above the sample and constant to within 1 mm. The analysis procedure should include a leak test.

For informational purposes, a representative multipoint BET plot is shown in Figure 1.

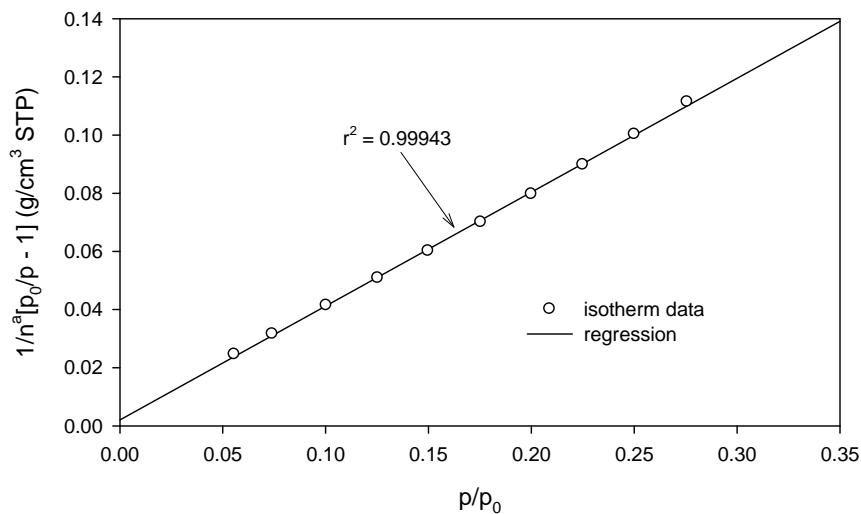


Figure 1. Representative nitrogen sorption isotherm for SRM 2206 plotted in the linear form of the BET equation. The specific amount of gas adsorbed ( $n^a$ ) is reported at standard temperature and pressure (STP).

## REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/publications.cfm> (accessed April 2012).
- [2] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Inc.: New York (1992).
- [3] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at [http://www.bipm.org/utlis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utlis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed April 2012); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/> (accessed April 2012).
- [4] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement; Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (2008); available at [http://www.bipm.org/utlis/common/documents/jcgm/JCGM\\_101\\_2008\\_E.pdf](http://www.bipm.org/utlis/common/documents/jcgm/JCGM_101_2008_E.pdf) (accessed April 2012).
- [5] ISO 9277:2010; *Determination of the Specific Surface Area of Solids by Gas Adsorption — BET Method*; International Organization for Standardization: Geneva, Switzerland (2010).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.