



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1899

#### Silicon Nitride Powder-Specific Surface Area Standard

This Standard Reference Material (SRM) is intended for use in the calibration of instruments used to measure specific surface area (SSA) in the range of 0.1 m<sup>2</sup>/g to 1 000 m<sup>2</sup>/g. A unit of SRM 1899 consists of a single bottle containing approximately 4 g of silicon nitride powder. The SSA of this material was measured using a static volumetric type Brunauer-Emmett-Teller (BET) (nitrogen gas) instrument using a pellet cell.

Table 1. Certified SSA Values and Uncertainties (2 $\sigma$ ) by Nitrogen BET

Measurement Technique	Specific Surface Area
Multipoint	10.67 m <sup>2</sup> /g $\pm$ 0.19 m <sup>2</sup> /g
Single Point	10.52 m <sup>2</sup> /g $\pm$ 0.62 m <sup>2</sup> /g

**Expiration of Certification:** The certification of this SRM is deemed to be indefinite within the stated uncertainties, provided the SRM is stored and handled in accordance with the Storage and Handling and Instructions for Use sections of this certificate. Accordingly, periodic recalibration or recertification of this SRM is not required. However, certification will be nullified if the SRM is contaminated or otherwise altered.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

#### NOTICE AND WARNINGS TO USERS

**Storage and Handling:** Silicon nitride is typically inert under ambient and testing conditions. Samples from the original bottle may be returned to the bottle and reused provided that the Instructions for Use, Sampling Procedure, and Outgassing Procedure are strictly followed.

**Cautions to User:** This SRM is **NOT** certified for pore size distribution, sample density, adsorption/desorption isotherm form, or particle size by any other method. While the SSA of this SRM was determined using a static volumetric BET type of instrument using nitrogen gas, it is possible that different methods of gas adsorption surface area analysis (e.g. flowing gas analysis/dynamic gas analysis) may be used. Its suitability for use with a different gas is undetermined. **The user must determine the effect of different methods and measurement dynamics on the SSA of this SRM.**

Coordination for this SRM was provided by S.E. Malghan and S.J. Dapkunas of the NIST Ceramics Division.

The BET measurement technique, development, and certification was performed by D.B. Minor of the NIST Ceramics Division.

Statistical analysis was performed by S.D. Leigh of the NIST Statistical Engineering Division.

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*See Certificate Revision History on Last Page*

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

## INSTRUCTIONS FOR USE<sup>1</sup>

The following instructions are based upon using a Quantachrome Autosorb 1, Model AS-1-11 using the pellet cell. Use of a large internal diameter cell will most likely yield an SSA, both single point and multipoint, higher than the stated value for this SRM. The effect of using a larger volume cell has not been determined by NIST. An instrument manufacturer (Quantachrome Corp.) has tested SRM 1899 using both a large diameter sample tube and the pellet tube. The results of these investigations are given in Table 2. **If using equipment other than that listed above, consult the equipment manufacturer's operating instructions for variations in testing procedure.** The sampling and outgassing procedures described below should always be followed. The sample preparation procedure was developed at NIST for the U.S. Department of Energy and the International Energy Agency [2].

**Sampling Procedure:** Pour the entire contents of the bottle onto a suitable piece of weighing paper and quarter the resulting pile. Scoop enough material from each of the quarters so that the entire amount is sufficient for analysis. The mass to be analyzed should be between 0.1 g and 0.25 g. Return the rest of the sample to the bottle, recap, and reserve for future use. The bulk sample need not be kept under vacuum or in a desiccator.

**Outgassing Procedure:** Pre-weigh an empty sample tube and filler rod and record this empty mass to the closest 0.0001 g. Place the sample into the analysis tube per manufacturer's instructions or accepted practice. Evacuate the sample tube to less than 1.3 Pa (10 mTorr) and heat the sample to 200 °C for two h. At the end of this time, turn off the heat, remove the heating mantle and allow the sample to come to ambient temperature. Back fill that sample with He gas, and weigh to the closest 0.0001 g. This is the filled mass. Calculate the sample mass by difference. Immediately perform the analysis following the manufacturer's directions or the user's particular method.

**Certification Technique and Uncertainties:** The SSA of SRM 1899 was determined on 20 randomly selected bottles from the production run. This SRM is certified for both multipoint (MP) and single point (SP) analysis using nitrogen gas as the adsorbate and using a static volumetric type of instrument based upon the BET method [3]. The certified specific surface area values and the expanded uncertainties assigned, computed according to the ISO and NIST Guides [4], are listed in Table 1. The expanded uncertainty at the 95 % level of confidence includes uncertainty due to measurement imprecision as well as material variation. The certified value and expanded uncertainty define a range of values within which the true specific surface area is expected to lie with at least 95 % confidence.

RM 8570 LGC GM Calcined Kaolin (surface area) was used as a reference in the evaluation of SRM 1899. The single point and multipoint values reported for SSA of RM 8570 are close to those determined for SRM 1899, thus RM 8570 and SRM 1899 can be directly related [5]. Two main advantages of using SRM 1899 over RM 8570 are that the sample preparation method for SRM 1899 is easier, and bound water of hydration is not an issue.

**Additional Noncertified Information:** Measurement using large diameter cells is compared with pellet cells in Table 2. A typical analysis of the nitrogen gas used during the certification of SRM 1899 is given in Table 3.

Table 2. Manufacturer's Information Values and Uncertainties (2σ) Comparison of SSA using Differing Cell Types

Measurement Technique	Cell Type	Specific Surface Area
Multipoint	Pellet	10.77 m <sup>2</sup> /g ± 0.15 m <sup>2</sup> /g
Single Point	Pellet	10.55 m <sup>2</sup> /g ± 0.14 m <sup>2</sup> /g
Multipoint	Large Diameter	11.13 m <sup>2</sup> /g ± 1.20 m <sup>2</sup> /g
Single Point	Large Diameter	10.85 m <sup>2</sup> /g ± 0.87 m <sup>2</sup> /g

<sup>1</sup>Certain commercial equipment, instruments or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 3. Typical Gas Analysis

Gas	Nitrogen
Grade	Industrial
Assay	99.995 %
O <sub>2</sub>	< 20 mg/kg
H <sub>2</sub> O	< 16 mg/kg
Total Hydrocarbons	< 5 mg/kg

#### REFERENCES

- [1] Malghan, S.G.; Hausner, H.; Pompe, R.; Tsubaki, J.; Hsu, S.M.; *Development and Testing of Procedures for Characterization of Ceramic Powders*, IEA ANNEX II, Subtask 6, prepared by NIST for the U.S. Department of Energy, Oak Ridge National Laboratory (1993).
- [2] Brunauer, S.; Emmett, P.; Teller, E.; *Adsorption of Gases in Multimolecular Layers*; J. Amer. Chem. Soc., Vol. 60, p. 309 (1938).
- [3] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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/>.
- [4] Colbert, J.C.; *Report of Investigation of Surface Area Reference Material 8570 Kaolin*, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (1994).

**Certificate Revision History:** 22 December 2008 (Editorial updates); 12 July 1997 (Original certificate date).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*