National Institute of Standards and Technology

Certificate

Standard Reference Material® 1900

Silicon Nitride Powder-Specific Surface Area Standard

This Standard Reference Material is intended for use in the calibration of instruments used to measure specific surface area (SSA) in the range of 0.1 m^2/g to 1000 m^2/g. The SRM unit 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 (2σ) by Nitrogen BET

Measurement Technique	Specific Surface Area
Multipoint	2.85 $m^2/g \pm 0.09$ m^2/g
Single Point	2.79 $m^2/g \pm 0.07$ m^2/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. However, certification will be nullified if the SRM is contaminated or otherwise altered.

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.G. Malghan, S.J. Dapkunas, and G. Onoda 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.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Program by R.J. Gettings.

Gaithersburg, MD 20899
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Certification Technique and Uncertainties: The SSA of SRM 1900 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 [1]. The certified specific surface area values and the expanded uncertainties assigned, computed according to the ISO Guide [2], 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 Calcined Kaolin (surface area) was used as a reference in the evaluation of SRM 1900. Two main advantages of using SRM 1900 over RM 8570 are that the sample preparation method for SRM 1900 is easier, and bound water of hydration is not an issue [3].

Instructions for Use: The following instructions are based upon using a Quantachrome Autosorb 1, Model AS-1-11[1] 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. **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 [4].

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 2 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.

Additional Noncertified Information: A typical analysis of the nitrogen gas used during the certification of SRM 1900 is given in Table 2.

Table 2. Typical Gas Analysis

Gas	Nitrogen
Grade	Industrial
Assay	99.995 %
O_2	<20 mg/kg
H_2O	<16 mg/kg
Total Hydrocarbons	<5 mg/kg

[1]Certain commercial equipment, instruments, or materials are identified in this certificate to adequately describe 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 are the best available for the purpose.

REFERENCES

[1] Brunauer, S., Emmett, P., and Teller, E., J. Amer. Chem. Soc., **60**, p. 309, (1938).

[2] *Guide to Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).

[3] Colbert, J.C., Report of Investigation of Surface Area Reference Material 8570 Kaolin, National Institute of Standards and Technology, Gaithersburg, MD, (1994).

[4] Malghan, S.G., Hausner, H., Pompe, R., Tsubaki, J., and Hsu, S.M., *Development and Testing of Procedures for Characterization of Ceramic Powders,* IEA ANNEX 11, Subtask 6, prepared by NIST for the U.S. Department of Energy, Oak Ridge National Laboratory, (Sept. 1993).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the internet http://ts.nist.gov/srm.