National Institute of Standards & Technology

Report of Investigation

Reference Material 8983

Silicon Nitride Powder

This Reference Material (RM) is intended primarily for use as an analytical standard for the determination of nitrogen, oxygen, and carbon in silicon nitride powder by the high temperature combustion method [1]. One unit of RM 8983 consists of a bottle of approximately 4.5 g of silicon nitride powder.

Reference Values: The reference values for total nitrogen, oxygen, and carbon are provided in Table 1, are expressed as mass fractions on an as-received basis. Reference values are noncertified values that are the best estimates of the true values. However, these values are based on determinations done by a single reliable method and do not meet the NIST criteria for certification. They are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

Table 1. Reference Values and Uncertainties for RM 8983 Silicon Nitride Powder

Element	Mass Fraction (%)
Nitrogen	39.23 ± 1.06
Oxygen	1.20 ± 0.14
Carbon	0.107 ± 0.015

The uncertainty in each reference value given in Table 1 is expressed as an expanded uncertainty U, at the 95 % level of confidence and is calculated according to the method described in the ISO and NIST Guides [2]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined uncertainty due to laboratory variability and measurement uncertainty. The coverage factor k, is determined from the Student's t-distribution corresponding to the degrees of freedom and a 95 % level of confidence.

Expiration of Value Assignment: RM 8983 is valid, within the measurement uncertainty specified, until **31 December 2022**, provided the RM is handled and stored in accordance with the instructions given in this Report of Investigation (see "Storage and Use"). Once a unit of RM 8983 has been opened, the material is valid for one year from opening the bottle. This report is nullified if the RM is damaged, contaminated or otherwise modified.

Maintenance of RM: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Storage and Use: Heating of the material may cause a change in oxygen mass fraction and should be avoided. The material should be kept in a vacuum desiccator, especially after opening, to avoid reaction with moisture in ambient air.

Material Preparation: The silicon nitride powder was procured commercially and spin riffled into bottles. The bottles were then sealed in aluminized pouches to protect the RM from atmospheric moisture.

The production, measurement coordination, and data evaluation of RM 8983 were coordinated by P.T. Pei of the NIST Materials Measurement Science Division.

Statistical consultation and analysis was performed by N.F. Zhang of the NIST Statistical Engineering Division.

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Gaithersburg, MD 20899
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Robert L. Watters, Jr., Director
Office of Reference Materials

Support aspects involved in the preparation of this RM were coordinated through of the NIST Office of Reference Materials.

Homogeneity Testing and Value Assignment: A random stratified sample of 15 bottles was selected for homogeneity testing. The homogeneity testing was performed according to NIST protocol by the LECO Corporation using the high temperature combustion method [1].

The reference values are based on results from three of the cooperating laboratories listed in Table 2. Each laboratory used the modified ASTM E 1019 high temperature combustion test procedure [3] with selected NIST Standard Reference Materials (SRMs): SRM 8j, SRM 11h, SRM 12h, SRM 15h, SRM 356, and SRM 656 as calibrants and/or control samples. A fourth laboratory, the University of Kentucky, provided confirmational analyses for oxygen using Fast Neutron Activation Analysis [4].

Table 2. Cooperating Analysts and Laboratories

D.A. Lawrenz, LECO Corporation, St. Joseph, MI.
T. Karlon and C. MacLaurin, Saint-Gobain Norton Industrial Ceramics, Northboro, MA.
M. Pohl and P.R. Fernando, Horiba Instruments Inc., Irvine, CA.
J.D. Robertson, University of Kentucky, Lexington, KY.

REFERENCES

[1] Pei, P.; Lum, L.; Onoda, G.; *Chemical Analysis of Carbon, Nitrogen and Oxygen in Silicon Nitride, Advances in Process Measurements of the Ceramic Industry*, Jillvenkatesa, A.; Onoda, G. Eds.; The American Ceramics Society: OH, p. 127 (1999).

[2] 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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2013); 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://www.nist.gov/pml/pubs/index.cfm (accessed Feb 2013).

[3] ASTM E 1019-94 *Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, and in Iron, Nickel and Cobalt Alloy*, American Society of Testing and Materials: Annual Book of ASTM Standards, V03.06 (1994).

[4] ASTM E 385 *Standard Test Method for Oxygen Content Using a 14 MeV Neutron Activation and Direct Counting Technique*, American Society of Testing and Materials: Annual Book of ASTM Standards, V12.02 (1999).

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Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-6776; Fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.