National Institute of Standards and Technology
U.S. Department of Commerce

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Reference Material® 8983
Silicon Nitride Powder
REFERENCE MATERIAL INFORMATION SHEET

Purpose: The non-certified values delivered by this Reference Material (RM) are 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].

Description: One unit of RM 8983 consists of a bottle of approximately 4.5 g of silicon nitride powder.

Non-Certified Values: The non-certified values for total nitrogen, oxygen, and carbon are provided in Table 1 and are expressed as mass fractions on an as-received basis. Non-certified 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 [2].

Table 1. Non-Certified 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 non-certified 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 [3]. 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.

Additional Information: Additional information is available in Appendix A.

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **31 December 2030.** The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Reference Material Information Sheet and notify registered users. RM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the RM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Dave Holbrook, Chief
Materials Measurement Science Division
Information Sheet Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Storage: 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.

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].

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] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jun 2023).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jun 2023); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jun 2023).

[4] 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).

[5] 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).

Report Revision History: 22 June 2023 (Change of period of validity; updated format; editorial changes); **12 February 2013** (Extension of the expiration date; editorial changes); **21 February 2008** (Extension of the expiration date and editorial changes); **07 January 2000** (Original report date).

Certain commercial equipment, instruments, or materials may be identified in this Reference Material Information Sheet 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.

Users of this RM should ensure that the Reference Material Information Sheet in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

The non-certified values are based on results from three of the cooperating laboratories listed in Table A1. Each laboratory used the modified ASTM E 1019 high temperature combustion test procedure [4] 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 [5].

Table A1. 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.

Production and certification were coordinated by P.T. Pei formerly of the NIST Materials Measurement Science Division.

Statistical analysis of the certification data was performed by N.F. Zhang and L.M. Gills formerly of the NIST Statistical Engineering Division.

Technical and support aspects involved in the preparation, certification, and issuance of this RM were coordinated by R. Gettings formerly of the NIST Standard Reference Materials Program.

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