



Designation: C1494 – 13

Standard Test Methods for Determination of Mass Fraction of Carbon, Nitrogen, and Oxygen in Silicon Nitride Powder¹

This standard is issued under the fixed designation C1494; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of mass fraction % of carbon, nitrogen and oxygen in silicon nitride powder having chemical compositions within the following limits:

Element	Mass Fraction % Range
Carbon	0.05 to 5.0
Nitrogen	30 to 45
Oxygen	0.1 to 1.5

1.2 Two test methods appear in this standard.

1.2.1 Total Carbon by the Direct Combustion-Infrared Measurement Method.

1.2.2 Nitrogen by the Inert Gas Fusion-Thermal Conductivity Measurement Method and Oxygen by the Inert Gas Fusion-Infrared Measurement Method.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*²

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

¹ These test methods are under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and are the direct responsibility of Subcommittee C28.03 on Physical Properties and Non-Destructive Evaluation.

Current edition approved Aug. 1, 2013. Published January 2014. Originally approved in 2001. Last previous edition approved in 2007 as C1494 – 01 (2007). DOI: 10.1520/C1494-13.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

3. Significance and Use

3.1 These test methods are for the chemical analysis of mass fraction of carbon, nitrogen and oxygen in silicon nitride powder. They are used in research, development, production, acceptance, and quality control of silicon nitride powders used to produce ceramic components with silicon nitride content.

3.2 It is assumed that all who use these test methods will be trained analysts, capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus and Reagents

4.1 The procedure was written with commercial carbon and nitrogen/oxygen analyzers in mind. For any other analyzer, the instrument manual specific to that analyzer shall be consulted for instrument set-up.

4.2 Specific apparatus and reagents required for each determination are listed in separate sections preceding the procedure.

5. Sampling

5.1 Procedures for sampling the materials refer to those parts of Practice E1806 pertaining to solid form samples of the type used for instrumental analysis.

6. Hazards

6.1 For hazards to be observed in the use of certain reagents in this test method, refer to Practice E50.

6.2 Use care when handling hot crucibles and operating furnaces to avoid personal injury by either burn or electrical shock.

7. Total Carbon in Silicon Nitride Powder by Direct Combustion—Infrared Detection Method

7.1 *Scope*—This test method covers the determination of carbon in concentrations from 0.05 to 5.0 % mass fraction.

7.2 *Summary of Test Method*³—The carbon is converted to carbon dioxide by combustion in a stream of oxygen. The amount of carbon dioxide is measured by infrared (IR) absorption.

7.3 *Apparatus*—This test method is written for use with commercial carbon analyzers, equipped to carry out the analyses operations automatically and calibrated using steel standards with known concentrations of carbon. The operating principles, specifications and descriptions of commercial carbon analyzers are given in the Practice of E1019.

7.4 *Reagents and Materials:*

7.4.1 *Crucibles*—Expendable ceramic (alumina) or similar refractory crucibles as specified by commercial carbon analyzers' manufacturers. Both the crucible and cover, if used, must be prebaked for a sufficient time to produce constant blank values. Use the prebake schedule recommended by the instrument manufacturer.

7.4.2 *Crucible Tongs*—Capable of handling recommended crucibles with respect to their sizes, shape and temperature.

7.4.3 *Accelerators*—Carbon free (or containing a known amount of carbon) granular tungsten/tin and iron chip accelerators shall be used.

7.4.4 *Carbon Standard Reference Materials*—NIST SRM 8k (steel - 0.0806 % C mass fraction), NIST SRM 19h (steel - 0.215 % C mass fraction), NIST SRM 12h (steel - 0.407 % C mass fraction), and NIST RM 8983 (silicon nitride - 0.107 % C mass fraction).

7.4.5 *Oxygen*—Ultra High Purity (99.95 % minimum purity) or Regular grade (99.5 %) purified by passing over heated CuO and through CO₂/H₂O absorbents. (When the instrument has a built in purifier, regular grade oxygen can be used.)

7.5 *Preparation of Apparatus*—Follow the operating instructions for the specific equipment used. After having properly set the operating controls of the instrument system, condition the apparatus by combustion of several blanks prepared with sample crucible and accelerator in the amounts to be used with the test specimen analyses. Successive blanks should achieve a steady state value.

7.6 *Blank Determination:*

7.6.1 Prebake ceramic crucibles in a muffle or tube furnace at 1250° C for not less than 15 min or at 1000° C for not less than 40 minutes. The crucibles shall be removed from the furnace, allowed to cool for 1-2 min and placed in a desiccator for storage. If the crucibles are not used within four hours, they must be prebaked again. This prebaking procedure is to burn off any organic contaminates.

7.6.2 Prepare instrument as outlined in the operator's instruction manual.

7.6.3 Determine the instrument blank.

(a) Enter 1.000 g weight into weight stack.

(b) Add 1.000 g (± 0.005 g) of tungsten/tin accelerator and 1.000 g (± 0.005 g) of iron chip accelerator.

(c) Place crucible on furnace pedestal and analyze.

(d) Repeat steps 7.6.3a through 7.6.3c a minimum of three times.

(e) Enter blank value following routine outlined in operator's instruction manual.

7.7 *Instrument Calibration Procedure :*

7.7.1 This procedure was written specially for a carbon analyzer. The type and amounts of accelerator to be added shall be adjusted according to the manufacturers recommendations for the other instrumentation.

7.7.2 Weigh 0.1 to 0.5 g of calibration standard to the nearest mg into a prebaked ceramic or similar refractory crucible and enter appropriate weight into weight stack.

7.7.3 Add approx. 1.0 ± 0.005 g of tungsten/tin accelerator and approx. 1.0 ± 0.005 g of iron chips accelerator.

7.7.4 Place crucible on pedestal and analyze.

7.7.5 Repeat the above steps 7.7.2 – 7.7.4 a minimum of three times for each standard, and calibrate the instrument following the auto calibration procedure as outlined in the operator's instruction manual.

7.7.6 Check calibration by analyzing the calibration standard again if it is not within the reported range. If it is not, repeat steps 7.7.2 – 7.7.4.

7.8 *Sample Analysis Procedure:*

7.8.1 Weigh 0.1 to 0.5 g of sample to the nearest mg into a prebaked expendable ceramic or a similar refractory crucible and add appropriate weight to the weight stack.⁴

7.8.2 Repeat steps 7.7.3 and 7.7.4 in the calibration procedure.

7.8.3 Each sample shall be analyzed in triplicate and record the integral values of the sample.

7.9 *Calculation*—Most commercially available instruments calculate percent concentration directly. If the instrument does not give percent concentration, please follow the manufacturer's directions to ensure all the essential variables in the calculation of analysis results have been included.

Or perform the following calculation to determine percent concentration (% mass):

a. Calibration Constant:

$$K = \frac{G \times P / 100}{A_c - A_b} \quad (1)$$

where:

K = calibration constant (g/integral value),

G = mass of calibration sample (g),

P = total carbon content of the calibration sample (% mass),

A_c = integral value of the calibration sample (7.7.6), and

A_b = integral value of the blank (7.6.3e).

b. Total Carbon Content:

$$C = \frac{(A_s - A_b) \times K \times 100}{m} \quad (2)$$

³ The test method procedure was adapted from (a) ASTM E1019-94, "Standard Test Methods for Determination of C, S, N, and O in Iron, Nickel and Cobalt Alloys" and (b) Application Bulletin: "Carbon and Sulfur in Ceramic and Similar Materials," LECO Corp., St. Joseph, MI.

⁴ The weight of sample is chosen based on the expected amount of carbon present and so the CO₂ produced will fall within the detection range of the IR detector.