

# Certificate of Analysis

## Standard Reference Material® 196

Ferrochromium Low Carbon (powder form)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of ferrochromium and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 196 consists of one bottle containing approximately 100 g of powder.

**Certified Mass Fraction Values:** Certified values for constituents of SRM 196 are reported in Table 1 as mass fractions of the elements in a ferrochromium matrix [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %.

Table 1. Certified Mass Fraction Values for SRM 196

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Carbon (C)	0.0351	0.0046
Chromium (Cr)	70.81	0.21
Phosphorus (P)	0.0195	0.0064
Silicon (Si)	0.373	0.010

**Expiration of Certification:** The certification of **SRM 196** is valid indefinitely within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**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 or register online) will facilitate notification.

Coordination of technical measurements for certification was performed by O. Menis and J.I. Schultz, formerly of NIST. Review and revision of values and uncertainty estimates were coordinated by J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by A. Possolo of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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#### INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Ferrochromium powder may be analyzed in the as-received form. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 500 mg should be used. Before sampling, it is recommended to mix the powder by inverting and rotating the bottle by hand for at least one minute. A bottle containing unused material should be recapped immediately and stored at room temperature away from light.

To use the uncertainty estimates given in this certificate, divide the expanded uncertainty by k = 2 to obtain the combined standard uncertainty. The effective degrees of freedom of the combined standard uncertainty are  $\geq 60$ .

#### PREPARATION AND ANALYSIS(1)

The material for SRM 196 was furnished by the Union Carbide Corp. (Marietta, OH).

Each certified value is an unweighted mean of the results from the methods listed in Table 2. The uncertainty listed with each certified value is an expanded uncertainty about the mean, with coverage factor, k = 2, calculated following the ISO/JCGM Guide [3-10].

Analyses leading to the certification of this SRM were performed at NIST by E.D. Anderson, J.R. Baldwin, D.A. Becker, P.D. LaFleur, G. Marinenko, and S.A. Wicks, all formerly of the NIST Chemical Sciences Division. Analytical determinations were also performed by L.F. Risi, Shieldalloy Corp. (Newfield, NJ); J.C. Cline, Interlake, Inc. (Beverly, OH); J. Wallen, AB Ferrolegeringar (Stockholm, Sweden); A.C. Nickel and H.A. Patterson, United States Steel Corp. (Gary, IN); and J.R. McKaveney, Crucible Steel Co. of America (Pittsburgh, PA).

Table 2. Test Methods Employed in the Certification of SRM 196

Element	Test Methods Used at NIST and Collaborating Laboratories
Carbon	Combustion with infrared detection; Combustion with thermal conductivity detection (TCD)
Chromium	$Na_2O_2 \ fusion - AgNO_3 - (NH_4)_2S_2O_8 - coulometric \ titration; \ Na_2O_2 \ fusion - AgNO_3 - FeSO_4\text{-}KMnO_4 - (NH_4)_2S_2O_8 \ titration; \ Na_2O_2 \ fusion - FeSO_4\text{-}KMnO_4 \ titration; \ HClO_4 \ oxidation - FeSO_4\text{-}KMnO_4 \ titration$
Nickel	Dimethylglyoxime spectrometric method
Phosphorus	Molybdenum-blue photometric method; Alkali-molybdate method
Silicon	HClO <sub>4</sub> dehydration; H <sub>2</sub> SO <sub>4</sub> -HNO <sub>3</sub> dissolution; H <sub>2</sub> SO <sub>4</sub> dissolution, Na <sub>2</sub> O <sub>2</sub> fusion, and double dehydration; HCl dissolution, Na <sub>2</sub> O <sub>2</sub> fusion, and double dehydration; volumetric titration
Sulfur	Combustion with iodate titration

### NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

**ADDITIONAL CONSTITUENTS:** Noncertified values are provided for the following additional constituents in SRM 196.

**Reference Mass Fraction Values:** Reference values for two constituents of SRM 196 are reported in Table 3 as mass fractions of the total elements in a ferrosilicon matrix. A reference value is a non-certified value that is the present best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2]. The reference value is metrologically traceable to the derived SI unit for mass fraction expressed as

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<sup>(1)</sup> Certain commercial equipment, instruments or materials are identified in this certificate 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.

percent. The reference value is an unweighted mean of results obtained using the test method in Table 2. The expanded uncertainty, U, is an expanded uncertainty about the mean, with coverage factor, k = 2, calculated following the ISO/JCGM Guide [3-10].

Table 3. Reference Mass Fraction Value for SRM 196

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Nickel (Ni)	0.36	0.02
Sulfur (S)	0.003	0.001

**Information Mass Fraction Values:** Information values for constituents in SRM 196 are reported as mass fractions in Table 4. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability.

Table 4. Information Mass Fraction Values for SRM 196

Constituent	Mass Fraction (%)
Manganese (Mn)	0.28
Vanadium (V)	0.12

#### **REFERENCES**

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Mar 2019).
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- [10] 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 (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM\_100\_2008\_E.pdf (accessed Mar 2019); 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 Mar 2019).

**Certificate Revision History:** 05 March 2019 (Correction to revision history; editorial changes); 24 August 2018 (Revised values and uncertainties for C, Cr, P, and Si; reassignment of the sulfur value as a reference value, reassignment of vanadium value as an information value; addition of Ni reference value; title update; editorial changes); 9 November 1970 (Original certificate date); 14 July 1967 (Provisional certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.

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