National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 195

Ferrosilicon (75 % Si – High-Purity Grade)

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 195 consists of a bottle containing approximately 75 g of pulverized, fine powder (< 0.15 mm). A companion material, SRM 58a Ferrosilicon (73 % Si – Regular Grade), is also available.

Certified Values: Certified values for seven constituents in SRM 195 are provided in Table 1. All values are reported as mass fractions [1]. The uncertainty listed with the value is an expanded uncertainty, $U = ku_c$, based on a 95 % confidence level [2] and is calculated according to the method in the ISO Guide [3]. 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 investigated or taken into account [4]. A certified value is the present best estimate of the "true" value based on the results of analyses performed at NIST and collaborating laboratories. Test methods used to determine these elements are identified in the appendix and the accompanying key.

Reference Values: Reference values for six constituents are provided in Table 2. Reference values are non-certified values that are the present best estimates of the true values; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all components of uncertainty [4]. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence level [4] and is calculated according to the method in the ISO Guide [3].

Information Values: Information values are provided for five constituents in Table 3. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. They are intended to provide additional information on the matrix.

Expiration of Certification: The certification of **SRM 195** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). 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, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The original characterization of this material was performed in 1976 under the direction of O. Menis and J.I. Shultz of the National Bureau of Standards (NBS, now NIST). Homogeneity assessment was performed by S.D. Rasberry, J. McKay, D. Reid, and K.M. Sappenfield of NBS.

Review and revision of value assignments was performed by J.R. Sieber and W.R. Kelly of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by D.D. Leber of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 October 2009

Robert L. Watters, Jr., Chief
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Certification analyses were performed by the following: NBS: K.M. Sappenfield, E.L. Garner, and R.K. Bell. Allegheny Ludlum Steel Corporation, Research Center, Brackenridge, PA: R.B. Fricioni and M.A. McMahon; Armco Steel Corporation, Research Center, Middletown, OH: M. Dannis, E.C. Schmidt, and R.J. Bendure; Carpenter Technology Corporation, Reading, PA: A.L. Sloan; Interlake, Inc., Globe Metallurgical Division, Beverly, OH: J.C. Cline and R.A. Pontello; Union Carbide Corporation, Ferroalloys Division, Marietta, OH: H.H. Hall, J.J. Armour, and G. Porter.

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum test portion of 200 mg is recommended. The material should be stored in its original container in a cool, dry location.

PREPARATION, TESTING, ANALYSIS[1]

The material for this SRM was supplied in pulverized form (< 0.15 mm) by the Union Carbide Corporation, Ferroalloys Division.

Following sieving and blending operations at NBS, homogeneity testing was performed using X-ray fluorescence. Certification analyses were performed using the method provided in the appendix.

Table 1. Certified Values for SRM 195 Ferrosilicon (75 % Si – High-Purity Grade)

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
B	0.00105	0.00016	3.2
Cr	0.0474	0.0039	3.2
Cu	0.0468	0.0024	3.2
Fe	23.62	0.12	2.6
Mn	0.1710	0.0050	3.2
Ni	0.0318	0.0020	3.2
Si	75.32	0.13	2.4

Table 2. Reference Values for SRM 195 Ferrosilicon (75 % Si – High-Purity Grade)

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
Al	0.0460	0.0090	4.3
C	0.03445	0.00084	3.2
Ca	0.054	0.019	12.7
P	0.0190	0.0088	4.3
Ti	0.0367	0.0038	4.3
Zr	0.0110	0.0066	4.3

[1] 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.

Table 3. Information Values for SRM 195 Ferrosilicon (75 % Si – High-Purity Grade)

Constituent	Mass Fraction (%)
Co	< 0.01
Mo	0.01
O	< 1
S	< 0.002
Sn	< 0.005

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 http://physics.nist.gov/Pubs/.

[2] May, W., Parris, R., Beck, C., Fassett, J., Greenberg, R., Guenther, F., Kramer, G., Wise, S., Gills, T., Colbert, J., Gettings, R., MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm).

[3] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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.physics.nist.gov/Pubs/contents.html.

[4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

Certificate Revision History: 22 October 2009 (Corrected reference to Certificate Revision History on page 1); 08 September 2009 (This revision reports revised assignments and values for all constituents based on re-evaluation of the original analytical results and updates the entire certificate to current NIST standards); Revision 25 April 1978; 07 January 1976 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Appendix. Analytical Methods

Element	Methods*
Al	1, 20
B	4, 9, 10, 12
C	2
Ca	1, 21
Co	1, 4
Cr	1, 14, 22
Cu	1, 9, 13, 18
Fe	24
Mn	1, 23
Ni	1, 9, 19
P	17
S	3, 11
Si	5, 6, 7, 8
Ti	15
Zr	16, 17, 25

*Key to Methods in Table 4:

1. Atomic Emission Spectrometry
2. Combustion – Conductometric
3. Combustion - Titration
4. Emission Spectrometry
5. Gravimetric – Fusion with Na_2CO_3 + Na_2O_2, dehydration with HCl, second dehydration with $HClO_4$.
6. Gravimetric – Fusion with Na_2CO_3 and KNO_3
7. Gravimetric – Fusion with H_2O_2, dehydration with HCl
8. Gravimetric - Perchloric acid dehydration
9. Isotope dilution mass spectrometry
10. Photometric – Azure C
11. Photometric – Combustion, paraosaniline
12. Photometric – Curcumin complex
13. Photometric – Diethyldithiocarbamate
14. Photometric - Diphenylcarbazide
15. Photometric - H_2O_2
16. Photometric – Ion exchange, phenylfluorone
17. Photometric
18. Photometric - Neocuproine
19. Titrimetric - $AgNO_3$ - NaCN titration
20. Titrimetric - CDTA titration
21. Titrimetric - EDTA titration
22. Titrimetric – Peroxydisulfate oxidation - ferrous ammonium sulfate titration
23. Titrimetric – Persulfate oxidation, sodium arsenite titration
24. Volumetric – $SnCl_2$ – $K_2Cr_2O_7$
25. X-ray spectrometry