



Certificate of Analysis

Standard Reference Material[®] 693

Iron Ore (Nimba)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for elemental contents of iron ore, iron ore concentrates, and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 693 consists of one bottle containing approximately 100 g of powder with < 74 μm (200 mesh) particle sizes.

Certified Mass Fraction Values: Certified values for constituents of SRM 693 are reported in Table 1 as mass fractions of the constituents in an iron oxide 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 International System of Units (SI) derived unit for mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %. Constituents listed as compounds are given according to industry convention, assuming perfect stoichiometry.

Table 1. Certified Mass Fraction Values for SRM 693 Iron Ore (Nimba)
(Values given on a dry basis, 1 h at 105 °C)

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Aluminum Oxide (Al_2O_3)	1.043	0.036
Calcium Oxide (CaO)	0.0158	0.0036
Iron (Total Fe)	65.08	0.20
Magnesium Oxide (MgO)	0.0143	0.0024
Manganese(II) Oxide (MnO)	0.0900	0.0040
Phosphorus (P)	0.0563	0.0021
Potassium Oxide (K_2O)	0.00283	0.00039
Silicon Dioxide (SiO_2)	3.860	0.012
Sodium Oxide (Na_2O)	0.0023	0.0011
Titanium Dioxide (TiO_2)	0.0345	0.0034

Expiration of Certification: The certification of **SRM 693** 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 21 April 2021
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Coordination of technical measurements for certification was performed by R.E. Michaelis 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.

Measurements for value assignment of SRM 693 were performed by A.F. Marlow, T.A. Rush, and J.R. Sieber of the NIST Chemical Sciences Division and R.K. Bell, T.J. Brady, J.D. Messman, and T.C. Rains, formerly of NIST. Additional measurements were performed by the Bethlehem Steel Corp. (Bethlehem, PA); Ledoux and Company (Teaneck, NJ); The Steel Company of Canada, Ltd. (Hamilton ON); and United States Steel Corp. (Monroeville, PA).

Statistical consultation for this SRM was provided by A.M. 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.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To dry samples, heat in an oven at 105 °C for 1 h. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 0.5 g 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.

PREPARATION AND ANALYSIS⁽¹⁾

The iron ore material for this SRM was prepared in powder form to pass a 74 µm sieve (200 mesh) by the Bethlehem Steel Corp. (Bethlehem, PA). At NIST, the material was sieved again and blended. Homogeneity testing of total iron content was performed at NIST, and the results indicated the material variability for 0.5 g samples was less than the test method repeatability. Additional testing for all elements, except sodium and sulfur, by borate fusion of 1.0 g samples and X-ray fluorescence spectrometry showed all tested elements, except nickel, arsenic, and zirconium, are sufficiently homogeneous within and among units for quantitative determinations.

Each certified value is a weighted average of the results from the methods listed in Table 3. The uncertainty listed with each certified value is an expanded uncertainty about the mean, with coverage factor, $k = 2$, calculated in accordance with the ISO/JCGM Guide [3–8].

ADDITIONAL CONSTITUENTS

Non-certified values are provided for the following additional constituents in SRM 693. These values are not certified, because NIST cannot vouch fully for the calibrations of the test methods and other details.

Reference Mass Fraction Values: Reference values for SRM 693 are reported in Table 2 as the mass fraction of the elements in an iron oxide matrix, expressed as percent. A NIST reference value is a non-certified value that is the present best estimate 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 value is the unweighted mean of results obtained at collaborating laboratories. The expanded uncertainty, U , is an expanded uncertainty about the mean, with coverage factor, $k = 2$ [3–8]. Results derived from the use of this value are considered by NIST to be traceable only to the value itself.

⁽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 2. Reference Mass Fraction Values for SRM 693 Iron Ore (Nimba)
(Values given on a dry basis, 1 h at 105 °C)

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Arsenic (As)	0.0012	0.0003
Chromium (Cr)	0.0048	0.0002
Nickel (Ni)	0.0009	0.0003
Sulfur (S)	0.005	0.002
Vanadium (V)	0.0043	0.0002
Zirconium (Zr)	0.0009	0.0003

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.

Table 3. Analytical Methods

Constituent	Method
Al ₂ O ₃	Chromeazurol S photometric method
	Flame atomic absorption spectrometry
	Flame atomic emission spectrometry
	Mercury cathode-NH ₄ OH-Cupferron-AlPO ₄
	X-ray fluorescence spectrometry after borate fusion
Arsenic (As)	X-ray fluorescence spectrometry after borate fusion
CaO	Flame atomic absorption spectrometry
	Flame atomic emission spectrometry
	Spectrographic method
	X-ray fluorescence spectrometry after borate fusion
Chromium (Cr)	X-ray fluorescence spectrometry after borate fusion
Total Fe	K ₂ Cr ₂ O ₇ titration after dissolution with reduction by SnCl ₂
	K ₂ Cr ₂ O ₇ titration after dissolution with reduction by H ₂ S
	Silver reductor method
	X-ray fluorescence spectrometry after borate fusion
K ₂ O	Flame atomic absorption spectrometry
	Flame atomic emission spectrometry
	Spectrographic method
	X-ray fluorescence spectrometry after borate fusion
MgO	Flame atomic absorption spectrometry
	Spectrographic method
	X-ray fluorescence spectrometry after borate fusion
MnO	Flame atomic absorption spectrometry
	Photometric method
	X-ray fluorescence spectrometry after borate fusion
Na ₂ O	Flame atomic absorption spectrometry
	Flame atomic emission spectrometry
	Spectrographic method
Nickel (Ni)	X-ray fluorescence spectrometry after borate fusion
Phosphorus (P)	Alkali-molybdate method
	Photometric method
	X-ray fluorescence spectrometry after borate fusion
SiO ₂	Fusion with Na ₂ CO ₃
	Gravimetry after HClO ₄ dehydration
	Photometric method
	X-ray fluorescence spectrometry after borate fusion
Sulfur (S)	Combustion and titration
TiO ₂	Chromotropic acid photometric method
	Flame atomic absorption spectrometry
	Flame atomic emission spectrometry
	H ₂ O ₂ photometric method
	4,4'-Methylenediantipyrine photometric method
	X-ray fluorescence spectrometry after borate fusion
Vanadium (V)	X-ray fluorescence spectrometry after borate fusion
Zirconium (Zr)	X-ray fluorescence spectrometry after borate fusion

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/special-publication-811> (accessed Apr 2021).
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- [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 (JCGM) (2008); available at <https://www.bipm.org/en/publications/guides> (accessed Apr 2021); 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 Apr 2021).
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- [8] Toman, B.; Possolo, A.; *Laboratory Effects Models for Interlaboratory Comparisons*; Accred. Qual. Assur., Vol. 14, pp. 553–563 (2009).

Certificate Revision History: 21 April 2021 (Revised values and uncertainties for all constituents; sulfur changed to a reference value; vanadium, chromium, nickel, arsenic, and zirconium added as reference values; title updated; editorial changes); 31 July 1990 (editorial changes); 24 October 1978 (Original 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; e-mail srminfo@nist.gov; or via the Internet at <https://www.nist.gov/srm>.