National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 2168

### High-Purity Iron

This Standard Reference Material (SRM) is high-purity iron in the form of chips and is intended primarily for evaluation of methods for analysis of trace elements in iron and materials of similar matrix. It should not be used for calibration. A unit of SRM 2168 consists of one bottle containing approximately 150 g of chips.

**Certified Mass Fraction Values:** A certified value for sulfur in SRM 2168 is reported in Table 1 as a mass fraction [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 investigated or taken into account [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using the test methods shown in Table 4.

**Reference Mass Fraction Values:** Reference values for 10 constituents are reported 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 sources of uncertainty [2].

**Information Mass Fraction Values:** Information values for 20 constituents are reported 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.

**Expiration of Certification:** The certification of **SRM 2168** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). Accordingly, periodic recalibration or 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 material 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) will facilitate notification.

Coordination of the original certification of SRM 2168 was performed by J.I. Schultz, ASTM/NIST Research Associate, and P.A. Lundberg of the NIST Standard Reference Materials Program through collaboration with ASTM International. Coordination of the reevaluation of boron and sulfur was performed by J.R. Sieber of the NIST Analytical Chemistry Division.

Analyses in the original characterization were performed by E.S. Beary and P.J. Paulsen of what is now the NIST Analytical Chemistry Division. Analyses for certification were also performed in the following laboratories: AB Sandvik Steel, Sandviken, Sweden; by B. Berglund; Analytical Associates, Inc., Detroit, MI, by C.K. Deak; Andrew S. McCreath & Son, Inc., Harrisburg, PA, by F.A. Pennington, Jr., R.F. Eakin, J.E. Fickel, R.M. Royer, and S.M. Goldinger; Armco Research & Technology, Middletown, OH, by C.C. Borland, D.E. Gillum, H.P. Vail, G.D. Smith, G.R. Doebler, and T.M. Minor; Hoogovens Groep BV, Ijmuiden, The Netherlands, by H. Umans; Ledoux & Co., Teaneck, NJ, by C.L. Maul, E.W. Hobart, and E.P. Kehoe; Luvak, Inc., Boylston, MA, by L.W. Ollila; Nippon Steel Corp., Kimitsu Works, Kawasaki, Japan; S. Kasai and M. Saeki. Analyses for the update were performed by W.R. Kelly, J.L. Mann, R.L. Paul, and R.D. Vocke of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
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Certificate Issue Date: 01 June 2011
*Certificate Revision History on Last Page*

Robert L. Watters, Jr., Chief
Measurement Services Division

Statistical consultation for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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

**INSTRUCTIONS FOR USE**

To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. Specimens may be used directly from the bottle without pre-treatment. The material should be stored in its tightly sealed, original bottle in a cool, dry location.

**Preparation and Analysis[1]:** The material for SRM 2168 was obtained in the form of rods provided by SCM Metals Products, Inc., Cleveland, OH. The material was chipped, blended, and bottled in the NIST Measurement Services Division. Homogeneity testing was performed at NIST using mass spectrometric methods. Quantitative determinations were performed at NIST and at collaborating laboratories using the test methods listed Table 4.

**Certified Value Assignment:** The certified value for sulfur was obtained by regression of results measured by NIST using isotope dilution thermal ionization mass spectrometry. The expanded uncertainty is calculated as $U = ku_c$ where $u_c$ is the combined uncertainty at the level of one standard deviation, and the coverage factor, $k = 2.31$, was determined from the Student's *t*-distribution corresponding to the associated degrees of freedom and 95 % confidence level [3].

Table 1. Certified Mass Fraction Value for SRM 2168

| Constituent | Mass Fraction (mg/kg) | | |
|---|---|---|---|
| Sulfur | 10.7 | ± | 2.8 |

**Reference Value Assignment:** Unless indicated otherwise, the reference values are the equally weighted means of results obtained by the collaborating laboratories using the test methods in Table 4. The expanded uncertainty, *U*, is calculated as $U = ku_c$ where $u_c$ is the combined standard uncertainty of the reported value and *k* is a coverage factor used to control the approximate confidence level for *U* [3]. The value of $u_c$ was taken to be the standard deviation of the mean of the laboratory results and includes contributions to the uncertainty from random measurement variability, potential systematic differences between laboratories and analytical methods, and possible material heterogeneity. The coverage factor, *k*, was determined from the Student's *t*-distribution corresponding to the degrees of freedom associated with $u_c$ and a confidence level of 95 %.

The reference value for boron is the mean of results obtained by NIST using prompt gamma-ray activation analysis. The expanded uncertainty is calculated as $U = ku_c$ where $u_c$ is the combined uncertainty at the level of one standard deviation [3].

Table 2. Reference Mass Fraction Values for SRM 2168

| Constituent | Mass Fraction (mg/kg) | | | Coverage Factor, *k* |
|---|---|---|---|---|
| Boron | 0.064 | ± | 0.026 | 2.43 |
| Carbon | 10 | ± | 6 | 2.57 |
| Chromium | 3 | ± | 2 | 3.18 |
| Cobalt | 6 | ± | 2 | 2.78 |
| Copper | 5 | ± | 2 | 2.78 |
| Manganese | 6 | ± | 4 | 2.36 |
| Nickel | 10 | ± | 5 | 2.57 |
| Nitrogen | 6 | ± | 4 | 2.78 |
| Oxygen | 110 | ± | 29 | 3.18 |
| Phosphorus | 14 | ± | 4 | 2.31 |

[1]Certain commercial organizations, services, equipment, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

**Information Value Assignment:** The values reported are estimates based on technical evaluation of the results reported from one or more test methods performed by the collaborating laboratories.

Table 3. Information Mass Fraction Values for SRM 2168

| Constituent | Mass Fraction (mg/kg) |
|---|---|
| Aluminum | 4 |
| Antimony | <3 |
| Arsenic | <1 |
| Bismuth | <3 |
| Cadmium | <1 |
| Calcium | <2 |
| Lead | <1 |
| Magnesium | 3 |
| Molybdenum | <7 |
| Niobium | <5 |
| Selenium | <5 |
| Silicon | <5 |
| Tantalum | <5 |
| Tellurium | <1 |
| Tin | <3 |
| Titanium | <3 |
| Tungsten | <10 |
| Vanadium | <1 |
| Zinc | <5 |
| Zirconium | <5 |

Table 4. Test Methods for SRM 2168

| | |
|---|---|
| Combustion with titration, infrared or thermal conductivity detection: | C, N, O, S |
| Direct current plasma optical emission spectrometry: | Ag, Al, Co, Cr, Cu, Mg, Mn, Mo, Nb, Ni, P, Si, Ti, V, Zr |
| Flame atomic absorption spectrophotometry: | Cr, Cu, Mn, Ni, Sn, Ti, V |
| Inductively coupled plasma optical emission spectrometry: | Ag, As, Co, Cr, Cu, Mn, Mo, Nb, Ni, P, Pb, Sb, Si, Sn, Ti, V |
| Inductively coupled plasma mass spectrometry: | Co, Cr, Cu, Mo, Ni, Sn, Zn |
| Inert gas fusion with infrared detection: | C, S |
| Isotope dilution thermal ionization mass spectrometry: | S |
| Photometric methods: | N, P, Si |
| Prompt gamma-ray activation analysis: | B |
| Spark source mass spectrometry: | As, Mn, Ni, P, S, Si, V |
| Zeeman atomic absorption spectrophotometry: | Ag, As, Bi, Cd, Pb, Sb, Se, Te |

## 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://www.nist.gov/pml/pubs/index.cfm/ (accessed Jun 2011).

[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: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed Jun 2011).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jun 2011); 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.nist.gov/pml/pubs/index.cfm (accessed Jun 2011).



**Certificate Revision History:** 01 June 2011 (Revised assignments and values for sulfur and boron based on new analytical determinations; revised assignments and values for remaining constituents based on reevaluation of original analyses; editorial changes); 20 December 1991 (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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.*