National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 368

Steel (AISI 1211)

This Standard Reference Material (SRM) is intended primarily for use in the validation of chemical and instrumental methods of analysis. SRM 368 is in the form of chips sized to pass sieve openings between 0.5 mm and 1.18 mm (35 and 16 mesh) and is packaged in a glass bottle. The mass of the unit is approximately 150 grams.

Certified Mass Fraction Values: Certified mass fraction values are provided in Table 1 [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

Reference Mass Fraction Values: Reference mass fraction values 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 reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods [2].

Expiration of Certification: The certification of **SRM 368** 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 Storage, and 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, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Overall coordination of the technical measurements leading to certification was performed under the direction of R.E. Michaelis of the NIST Office of Reference Materials and J.I. Schultz, Research Associate, ASTM International. Review and revision of value assignments were performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 368 were performed by S.A. Wicks, T.S.M. Lee, and R.K. Bell of the NIST Chemical Sciences Division; J.E. Joyce, Inland Steel Company, East Chicago, IN; R.W. Jones, Republic Steel Corporation, Canton, OH; N.J. Williams, Sharon Steel Corporation, Sharon, PA; V.M. Chapman, Timken Company, Canton, OH; and J.D. Selvaggio, J.B. Ferrons, H.R. Frisbee, D.T. Glaser, F.T. Hornak, and H.S. Karp, United States Steel Corporation, Monroeville, PA.

Statistical analysis 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 Office of Reference Materials.

Carlos A. Gonzalez, Chief
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Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Material

INSTRUCTIONS FOR STORAGE AND USE

The chip form material should be sampled for analysis as-is without additional preparation. Store the material in its original container in a cool, dry location. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 1.0 g should be used. It is recommended to mix the contents of the bottle prior to sampling by turning the bottle end over end for one minute.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 368 was provided by the United States Steel Corporation, Lorain, OH. Homogeneity testing of the key elements carbon and sulfur was performed at NIST. Material variability was determined to be within the method imprecision. Quantitative determinations were performed at NIST and at collaborating laboratories using the test methods listed in Table 3.

Certified Mass Fraction Values: The values in Table 1 are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3] and the DerSimonian-Laird procedure [4,5]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the JCGM Guide and its Supplement 1 [6–9]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c represents, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The coverage factor, k, corresponds to an approximately 95 % confidence level.

Table 1. Certified Mass Fraction Values for SRM 368 Steel (AISI 1211)

Element	Mass Fraction (%)	Coverage Factor, k
Chromium (Cr)	0.0295 ± 0.0012	2.00
Copper (Cu)	0.00984 ± 0.00078	1.97
Manganese (Mn)	0.8238 ± 0.0053	2.01
Molybdenum (Mo)	0.00311 ± 0.00058	1.98
Nickel (Ni)	0.00783 ± 0.00059	1.98
Nitrogen (N)	0.01030 ± 0.00017	1.98
Phosphorus (P)	0.0827 ± 0.0017	2.00
Silicon (Si)	0.0067 ± 0.0013	2.01

Reference Mass Fraction Values: The values in Table 2 are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3] and the DerSimonian-Laird procedure [4,5]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the JCGM Guide and its Supplement 1 [6–9]. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c represents, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The coverage factor, k, corresponds to an approximately 95 % confidence level.

Table 2. Reference Mass Fraction Values for SRM 368 Steel (AISI 1211)

Element	Mass Fraction (%)	Coverage Factor, k
Carbon (C)	0.090 ± 0.002	1.97
Sulfur (S)	0.1324 ± 0.0013	2.00
Vanadium (V)	0.0013 ± 0.0004	1.99

[(1)] Certain organizations, commercial equipment, 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. Analytical Methods Used for SRM 368 Steel (AISI 1211)

Method	Elements Determined
Combustion with infrared or thermal conductivity detection	C
Potentiometric titration	Cr
Diphenylcarbazide photometric method	Cr
Flame atomic absorption spectrometry	Cr, Cu, Mn, Mo, Ni, V
Carbamate-butylacetate photometric method	Cu
Neocuprine photometric method	Cu
Thiosulfate-iodide titration	Cu
Sodium arsenite titration	Mn
Thiocyanate – stannous chloride photometric method	Mo
Dimethylglyoxime photometric method	Ni
Inert gas fusion with thermal conductivity detection	N
Kjeldahl titration	N
Gravimetry	P
Molybdenum blue photometric method	P, Si
Molybdic acid precipitation and potassium hydroxide titration	P
Gravimetric determination after dehydration with perchloric acid or sulfuric acid	Si
Combustion with infrared detection of iodate titration	S
Phenylbenzohydroxamic acid photometric method	V
Ferrous ammoniumsulfate reaction and permanganate titration	V

REFERENCES

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[7] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed June 2013).

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Certificate Revision History: 19 June 2013 (Revised assignments and values for all constituents based on re-evaluation of the original analytical results; editorial changes); 01 January 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 at: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.