National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 368

Carbon Steel (AISI 1211)
(chip form)

This Standard Reference Material (SRM) is intended primarily for use in the validation of chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials. 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 containing approximately 150 grams.

Certified Mass Fraction Values: Certified values for constituents in SRM 368 are provided in Table 1 as mass fractions of the elements in steel [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 368 Steel (AISI 1211)

Element	Mass Fraction (%)	Expanded Uncertainty (%)	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

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 Handling, 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 or register online) will facilitate notification.

Coordination of the technical measurements for certification was performed by 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 July 2017
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Material

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.

INSTRUCTIONS FOR HANDLING 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.

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 368.

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]. The measurands are the concentrations of the constituents listed in Table 2 as determined by the methods indicated in Table 3. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent).

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

Element	Mass Fraction (%)	Expanded Uncertainty (%)	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

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.

The certified mass fraction 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, and the coverage factor, k, corresponds to an approximately 95 % confidence level.

The reference mass fraction 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, and the coverage factor, k, corresponds to an approximately 95 % confidence level.

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,

[(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.

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.

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 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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[5] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

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[9] Davison, A.C.; Hinkley, D.V.; *Bootstrap Methods and Their Application*; Cambridge University Press, New York (1997).

Certificate Revision History: 31 July 2017 (Update title; editorial changes); 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, email srminfo@nist.gov; or via the Internet at http:/www.nist.gov/srm.