National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1224

Carbon Steel
(AISI 1078)

This Standard Reference Material (SRM) is intended primarily for use in the validation of chemical and instrumental methods of analysis. A unit of SRM 1224 consists of one disk, 32 mm diameter and 19 mm thick.

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 1224** is valid indefinitely, within the measurement uncertainty 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, 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 1224 were performed by B.I. Diamondstone, J.A. Norris and R.K. Bell of the NIST Chemical Sciences Division; W.L. Wright, Great Lakes Steel Division of National Steel Corp., Detroit, MI; L. Chalker and C.R. Vinyard, Jones & Laughlin Steel Corp., Cleveland, OH and Youngstown, OH; and V.M. Chapman, P. Conley, J. Coobatis, F. Cuenot, G. Hanni, N. Pierko, P. Smith, and D. Wolfe, Timken Company, Canton, OH.

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
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 November 2013
Certificate Revision History on Last Page

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

INSTRUCTIONS FOR USE

The test surface is the side that is not labeled with the SRM number and the diamond-shaped logo. The entire thickness of the unit is certified. Each packaged disk has been prepared by finishing the test surface using a milling machine. The user must determine the correct surface preparation procedure for each analytical technique. The user is cautioned to use care when either resurfacing the disk or performing additional polishing as these processes may contaminate the surface. The material should be stored in its original container in a cool, dry location. This material was tested using both solid disks and chips prepared from disks.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 1224 was provided by the Bethlehem Steel Corp., Bethlehem, PA. Three billets were fabricated at the Puget Sound Naval Shipyard, Bremerton, WA, where the billets were forged into slabs and portions of doubtful homogeneity were cut and discarded. The remaining slab sections were forged and swaged to rods. The rods were given a sub-critical anneal and were centerless ground to the final size of 32 mm diameter. The metallurgical structure of the specimens is that resulting from hot working followed by annealing. Extensive homogeneity testing was performed at NIST by optical emission spectrometry and by a rapid carbon/sulfur analyzer. Only that material exhibiting satisfactory homogeneity was accepted. Quantitative determinations were performed at NIST and at collaborating laboratories using the test methods listed in Table 3.

Certified Mass Fraction Values: The measurand is the total mass fraction for each analyte listed in Table 1 and the certified value is metrologically traceable to the SI unit of mass. The values 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 1224 Steel (AISI 1078)

Constituent	Mass Fraction (%)	Coverage Factor, k
Carbon (C)	0.7518 ± 0.0035	2.0
Chromium (Cr)	0.0710 ± 0.0031	2.0
Copper (Cu)	0.0711 ± 0.0016	2.0
Manganese (Mn)	0.4098 ± 0.0021	2.0
Molybdenum (Mo)	0.01320 ± 0.00027	2.1
Nickel (Ni)	0.0537 ± 0.0014	2.3
Phosphorus (P)	0.00884 ± 0.00087	2.0
Silicon (Si)	0.1725 ± 0.0020	2.2
Sulfur (S)	0.0395 ± 0.0022	2.0

Reference Mass Fraction Values: The measurands in Table 2 are reference mass fraction values obtained by the methods indicated in Table 3 and are metrologically traceable to the SI unit of mass. The values 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.

[(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 1224 Steel (AISI 1078)

Constituent	Mass Fraction (%)	Coverage Factor, *k*
Aluminum (Al)	0.0598 ± 0.0005	2.1
Vanadium (V)	0.0016 ± 0.0007	2.0

Table 3. Analytical Methods Used for Characterization of SRM 1224 Steel (AISI 1078)

Method	Elements Determined
Flame atomic absorption spectrometry	Al, Cr, Cu, Mn, Mo, Ni, Si, V
Mercury cathode, 8-hydroxyquinoline method	Al
Combustion with infrared or thermal conductivity detection	C, S
Combustion with gravimetric determination	C
$NaHCO_3$ precipitation, peroxydisulfate oxidation, potentiometric titration with $Fe(NH_4)_2(SO_4)_2$	Cr
$HClO_4$ oxidation, $FeSO_4$ – $KMnO_4$ titration	Cr
Carbamate-butylacetate photometric method	Cu
Neocuprine photometric method	Cu
Sodium bismuthate oxidation and sodium arsenite titration	Mn
Peroxydisulfate oxidation and sodium arsenite titration	Mn
Thiocyanate-stannous chloride photometric method	Mo
Dimethylglyoxime gravimetric method	Ni
Molybdenum blue photometric method	P
Molybdic acid precipitation and KOH titration	P
Gravimetric determination after dehydration with $HClO_4$	Si
Combustion with iodate titration	S
HNO_3 oxidation and potentiometric titration with $Fe(NH_4)_2(SO_4)_2$	V
H_2O_2 photometric method	V

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 Nov 2013).

[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 Nov 2013).

[3] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Hoboken, NJ (2006).

[4] Dersimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).

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

[6] 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* http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2013); 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); avail*able at* http://www.nist.gov/pml/pubs/index.cfm (accessed Nov 2013).

[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 Nov 2013).

[8] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).

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

Certificate Revision History: 18 November 2013 (Revision based on re-evaluation of original analytical results, update of certified mass fraction values and uncertainties, addition of coverage factors; change of Vanadium and Aluminum from certified to reference values with revised uncertainties and addition of coverage factors,; editorial changes); 24 February 1981 (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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.