National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1155a

Stainless Steel (Cr 18-Ni 12-Mo 2)
(AISI 316)

This Standard Reference Material (SRM) is intended for use with test methods for elemental analysis based on both chemical processes and instrumental techniques. A unit of SRM 1155a is in the form of a solid disk 3.2 cm in diameter and 1.9 cm thick.

Certified Mass Fraction Values: Certified mass fraction values are provided in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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. 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 [2] and are provided with associated uncertainties that may reflect only measurement precision, not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Values: Information mass fraction values are provided 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 [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1155a** 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 SRM 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 technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 1155a were performed by A.F. Marlow, J.A. Norris, P.A. Pella, and J.R. Sieber of the NIST Chemical Sciences Division.

Analyses for certification were also performed by S. Arvich, M. Crooks, L. Dilks, G. Doerfer, S. Howitz, A. Phillips, J. Starr, and C. Weaver, Laboratory Testing Inc., Hatfield, PA; B. Cardenas, D. Dietz, G. Mann, and P. Schmidt, Anderson Laboratories, Greendale, WI; F. Nguyen, Element Materials Technology, Huntington Beach, CA; M. Smith and G. Witt, ATI Allegheny Ludlum, Natrona Heights, PA; and C.E. Bixler, M.A. Michielutti, and K.S. Smith, ESAB Welding & Cutting Products, Hanover, PA.

Statistical consultation for this SRM was provided by J.H. Yen of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR USE

The test surface is the side 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.

Results of homogeneity testing indicated that care should be taken when sampling this material. For test methods requiring chipping of the material, the recommended minimum sample quantity is 200 mg. For test methods that directly measure the surface of the disk, it is recommended that the measured area be carefully selected. Spark source and glow discharge measurements should be made at four or more separate locations on the surface; X-ray fluorescence methods should view at least one-quarter of the surface area of a unit of the SRM. The material has not been qualified for use with small-area, direct measurement methods such as micro X-ray fluorescence and laser ablation techniques.

PREPARATION AND ANALYSIS[(1)]

The material for this standard was prepared by U.S. Steel Corporation, Pittsburgh, PA. Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry. Results indicated that silicon, phosphorus, sulfur, titanium, manganese, and niobium exhibit inhomogeneity at levels that may be significant for some test methods with relative standard deviations for individual measurements of 1.4 % for Si, 1.2 % for P, 7.1 % for S, 4.9 % for Ti, 1.8 % for Mn, and 1.1 % for Nb. Care should be taken when sampling the material. See “Instructions for Use”. Analyses for value assignments of this SRM were performed using the methods listed in Table 4.

[(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.

Certified Mass Fraction Values: The measurand is the total mass fraction for each analyte listed in Table 1. The certified values, expressed in percent, are metrologically traceable to the SI unit of mass. The certified value for each analyte was calculated as the mean of the means from the individual methods/laboratories. The uncertainty listed with the value is an expanded uncertainty about the mean, $U = ku_c$, where u_c represents the combined uncertainty and k is a coverage factor corresponding to approximately 95 % confidence, and is calculated according to the method in the ISO/JCGM Guide [3]. The combined uncertainty includes contributions from inhomogeneity of the element within the steel and variability among sets of results from the test methods used by the laboratories.

Table 1. Certified Mass Fraction Values for SRM 1155a

Element	Mass Fraction (%)	Coverage Factor, k
Carbon (C)	0.0260 ± 0.0036	2.45
Cobalt (Co)	0.225 ± 0.018	2.26
Chromium (Cr)	17.803 ± 0.099	2.20
Copper (Cu)	0.2431 ± 0.0050	2.20
Iron (Fe)	64.71 ± 0.12	2.00
Manganese (Mn)	1.593 ± 0.060	2.06
Molybdenum (Mo)	2.188 ± 0.015	2.18
Niobium (Nb)	0.0082 ± 0.0014	3.18
Nickel (Ni)	12.471 ± 0.056	2.20
Phosphorus (P)	0.0271 ± 0.0012	2.11
Silicon (Si)	0.521 ± 0.017	2.03
Titanium (Ti)	0.0039 ± 0.0012	2.45
Vanadium (V)	0.0725 ± 0.0046	2.23
Tungsten (W)	0.0809 ± 0.0059	2.45

Reference Mass Fraction Values: Reference mass fraction values are listed in Table 2. The measurand is the mass fraction value determined using the methods indicated in Table 4. The reference values, expressed in percent, are metrologically traceable to the SI unit of mass. The reference value for each analyte was calculated as the mean of the means from the individual methods/laboratories. The uncertainty listed with the value is an expanded uncertainty about the mean, $U = ku_c$, where u_c represents the combined uncertainty and k is a coverage factor corresponding to approximately 95 % confidence, and is calculated according to the method in the ISO/JCGM Guide [3]. The combined uncertainty includes contributions from inhomogeneity of the element within the steel and variability among sets of results from the test methods used by the laboratories.

Table 2. Reference Mass Fraction Values for SRM 1155a

Element	Mass Fraction (%)	Coverage Factor, k
Arsenic (As)	0.007 ± 0.003	4.30
Nitrogen (N)	0.0428 ± 0.0024	3.18
Sulfur (S)	0.0020 ± 0.0009	2.78
Tin (Sn)	0.0069 ± 0.0013	3.18

Information Mass Fraction Values: The information value for each analyte is an estimate obtained from one or more NIST or collaborator test methods. No uncertainty is provided because there is insufficient information available for its assessment.

Table 3. Information Values for SRM 1155a

Element	Mass Fraction (%)
Aluminum (Al)	< 0.01
Boron (B)	0.002
Bismuth (Bi)	< 0.0001
Lead (Pb)	< 0.005
Oxygen (O)	0.003
Tantalum (Ta)	< 0.0001
Zirconium (Zr)	< 0.003

Table 4. Analytical Methods Used for SRM 1155a

Element	Methods[(a)]	Element	Methods[(a)]
Al	1, 2	Ni	1, 2, 5, 12
As	1, 2, 3	O	1, 2
B	1	P	1, 2, 5, 13
Bi	3	Pb	1, 3, 5
C	2, 4	S	4
Co	1, 2, 5	Si	1, 2, 5, 14
Cr	1, 2, 5, 6	Sn	1, 2
Cu	1, 2, 5, 7	Ta	3
Fe	5, 8	Ti	1, 2, 5
Mn	1, 2, 5, 9	V	1, 2, 5
Mo	1, 2, 5, 10	W	1, 2, 5
N	1, 4, 11	Zr	3, 5
Nb	1, 2, 5		

[(a)]Key to Methods in Table 4:

1. Inductively coupled plasma optical emission spectrometry
2. Spark source optical emission spectrometry
3. Inductively coupled plasma mass spectrometry
4. Direct combustion with infrared or thermal conductivity detection
5. X-ray fluorescence spectrometry
6. Peroxydisulfate oxidation and titration
7. Neocuprine photometric method
8. Stannous chloride reduction titrimetric method
9. Meta-periodate photometric method
10. Thiocyanate – stannous chloride photometric method
11. Inert gas fusion
12. Dimethylglyoxime gravimetric method
13. Spectrophotometric molybdenum-blue method
14. Gravimetric determination after dehydration with perchloric acid

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/sp811/index.cfm (accessed Aug 2014).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Aug 2014).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2014); 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 Aug 2014).

Certificate Revision History: 11 August 2014 (Iron value changed from reference to certified, with uncertainty updated; editorial changes); 25 April 2013 (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, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.