National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1230

High-Temperature Alloy A286

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is in the form of a disk and is intended primarily for use in optical emission and X-ray spectrometric methods. A unit of SRM 1230 is a disk approximately 32 mm ×19 mm. Material from the same lot is available in the form of chips as SRM 348a intended primarily for use in validation of chemical and instrumental methods of analysis.

Certified Values: Certified values for 15 constituents in SRM 1230 are provided in Table 1. All values are reported as mass fractions [1]. The uncertainty listed with the value is an expanded uncertainty, $U = ku_c$, based on a 95 % confidence level [2] and is calculated according to the method in the ISO Guide [3]. 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 [4]. A certified value is the present best estimate of the "true" value based on the results of analyses performed at NIST and collaborating laboratories. Test methods used to determine these elements are identified in the appendix and the accompanying key.

Reference Values: Reference values for Nb and W are given 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 components of uncertainty [4]. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence level [4] and is calculated according to the method in the ISO Guide [3].

Information Values: Information values are provided for eight constituents 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. They are intended to provide additional information on the matrix.

Expiration of Certification: The certification of **SRM 1230** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). 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) will facilitate notification.

The original characterization of this material was performed in 1987 under the direction of J.I. Shultz of the National Bureau of Standards (NBS, now NIST). Homogeneity testing was performed by J.A. Norris and T.W. Vetter of NBS.

Review and revision of value assignments was performed by J.R. Sieber and W.R. Kelly of the NIST Analytical Chemistry Division.

Statistical consultation 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 Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 September 2009

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

See Certificate Revision History on Page 3

Analyses for certification were performed by the following: NBS: B.I. Diamondstone, R.C. Gauer, J.A. Norris, and T.W. Vetter; Allegheny Ludlum Steel Corporation, Analytical Services, Brackenridge, PA, R.M. Crain, G.L. Bergstrom, and C.M. Bottegal; Crucible Materials Corporation, Specialty Metals Division, Syracuse, NY, R.J. Wlodarzyk; Crucible Materials Corporation, Research Center, Pittsburgh, PA, G.L. Vassilaros and C.J. Byrnes; Cytemp Specialty Steels Division, Cyclops Corporation, Titusville, PA, F.F. Liberator and D.K. Luoni; General Electric Company, Cleveland, OH, J.W. Fulton; Ladish Company, Inc., Cudahy, WI, G. Bugalski and J.E. Rafalski.

INSTRUCTIONS FOR USE

The test surface is the side opposite to the surface labeled with the SRM number and the diamond-shaped NBS 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 the same material. The certified values are considered to be representative of the overall average composition of the material.

PREPARATION AND ANALYSIS[1]

The material for this SRM was furnished by Crucible Metals Corporation, Specialty Metals Division, Syracuse, NY. Certification analyses were performed using the methods provided in the appendix.

Table 1. Certified Values for SRM 1230, High-Temperature Alloy A286

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
Al	0.249	0.015	2.8
B	0.00519	0.00050	2.8
C	0.0428	0.0046	2.8
Co	0.151	0.023	2.8
Cr	14.65	0.29	2.6
Cu	0.137	0.011	2.8
Fe	55.6	2.0	4.3
Mn	0.652	0.023	2.8
Mo	1.15	0.10	2.8
Ni	24.08	0.26	2.8
P	0.0239	0.0026	2.8
S	0.00095	0.00032	2.8
Si	0.411	0.085	3.2
Ti	2.18	0.24	2.8
V	0.229	0.020	3.2

[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 Values for SRM 1230, High-Temperature Alloy A286

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
Nb	0.067	0.024	3.2
W	0.0695	0.0094	3.2

Table 3. Information Values for SRM 1230, High-Temperature Alloy A286

Constituent	Mass Fraction (%)
Ag	0.000025
As	< 0.005
Bi	< 0.0001
N	0.003
Pb	< 0.0003
Sn	< 0.033
Ta	< 0.001
Zr	< 0.018

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://physics.nist.gov/Pubs/.

[2] May, W. E.; Parris, R. M.; B*eck II, C. M.; Fassett, J. D.; Greenberg, R. R.; Guenthe*r, *F. R.; Kramer, G. W.; Wise, S. A.; Gills, T. E.; Colbert, J.* C.; Gettings, R. J.; MacDonald, B. S.; Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements; NIST Spec. Pub. 260-136, U.S. Government Printing Office, Washington, DC, p. 16 (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.

[3] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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.physics.nist.gov/Pubs/contents.html.

[4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

Certificate Revision History: 08 September 2009 (This revision reports revised assignments and values for all constituents based on re-evaluation of the original analytical results and updates the entire certificate to current NIST standards); 15 June 1987 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Appendix. Analytical Methods

Element	Methods*	Element	Methods*
Ag	2	Nb	1, 10, 13, 32
Al	1, 10	Ni	1, 10, 11, 12, 15, 25, 32
As	2, 10	P	1, 7, 10, 22, 32
B	1,3, 4, 10	Pb	2, 6, 10, 32
Bi	2, 10	S	1, 9
C	1, 9	Si	1, 10, 14, 17, 32
Co	1, 10, 16, 31, 32	Sn	2, 10
Cr	1, 10, 20, 27, 29, 30, 32	Ta	10
Cu	1, 5, 10, 32	Ti	1, 10, 21, 24, 32
Fe	1, 10, 26	V	1, 10, 32
Mn	1, 8, 10, 23, 28, 33	W	10, 20, 32
Mo	1, 10, 18, 19, 32	Zr	10
N	9		

*Key to Methods:

1. Atomic Emission Spectrometry
2. Atomic Absorption Spectroscopy - graphite furnance
3. Optical emission spectrometry
4. Colorimetric – dianthrimide
5. Colorimetric - diethyldithiocarbamate – extraction
6. Colorimetric – dithizone
7. Colorimetric - Molybdenum Blue
8. Colormetric – permanganate
9. Combustion – Infrared spectrophotometry
10. Direct Current Plasma Optical Emission Spectrometry
11. Gravimetric - dimethylglyoxime – titrator
12. Gravimetric - dimethylglyoxime precipitation
13. Gravimetric - ion exchange
14. Gravimetric - perchloric acid double dehydration
15. Ion Exchange – electrolytic
16. Ion Exchange – Nitroso R
17. Perchloric acid dehydration
18. Photometric - butyl acetate extraction
19. Photometric - ether extraction – thiocyanate photometric
20. Photometric – Hydroquinone
21. Photometric - Ion Exchange – H_2O_2
22. Photometric - Molybdenum Blue
23. Photometric – periodate
24. Photometric - p-hydroxy phenylarsonic acid percipitation, peroxide color-photometric
25. Titrimetric - $AgNO_3$ - NaCN titration
26. Titrimetric - Ion exchange, $K_2Cr_2O_7$ titration
27. Titrimetric - perchloric – $KMnO_4$ titration
28. Titrimetric - Persulfate oxidation, sodium arsenite titration
29. Titrimetric – $KMnO_4$ - $FeSO_4$-$(NH_4)_2S_2O_8$ titration
30. Titrimetric - redox titration
31. Volumetric - Ion exchange – EDTA
32. X-ray fluorescence spectrometry
33. X-ray fluorescence spectrometry after borate fusion