



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1230

High-Temperature Alloy A286 (disk form)

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 consists of a disk approximately 32 mm × 19 mm.

Certified Values: The measurand is the certified value for each constituent in SRM 1230. Certified values are provided in Table 1 and are reported as mass fractions [1]. The uncertainty listed with the value is an expanded uncertainty, $U = k_{uc}$, based on a 95 % confidence level [2] and is calculated according to the method in the ISO/JCGM 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 the constituents are identified in Appendix A and the accompanying key. Metrological traceability is to the derived SI unit for mass fraction (expressed as a percent).

Reference Values: The measurands are the reference values for niobium and tungsten provided in Table 2 as determined by the indicated methods. Reference values for 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/JCGM Guide [3]. Metrological traceability is to the derived SI unit for mass fraction (expressed as a percent).

Information Values: Information values are provided for additional 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. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1230** 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”). Periodic 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.

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 the NIST Chemical Sciences Division.

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

Statistical consultation for this SRM was provided by D.D. Leber of the NIST Statistical Engineering Division.

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

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 Appendix A.

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

Element	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor, <i>k</i>
Aluminum (Al)	0.249	0.015	2.8
Boron (B)	0.00519	0.00050	2.8
Carbon (C)	0.0428	0.0046	2.8
Chromium (Cr)	14.65	0.29	2.6
Cobalt (Co)	0.151	0.023	2.8
Copper (Cu)	0.137	0.011	2.8
Iron (Fe)	55.6	2.0	4.3
Manganese (Mn)	0.652	0.023	2.8
Molybdenum (Mo)	1.15	0.10	2.8
Nickel (Ni)	24.08	0.26	2.8
Phosphorus (P)	0.0239	0.0026	2.8
Silicon (Si)	0.411	0.085	3.2
Sulfur (S)	0.00095	0.00032	2.8
Titanium (Ti)	2.18	0.24	2.8
Vanadium (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 Mass Fraction Values for SRM 1230, High-Temperature Alloy A286

Element	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor, <i>k</i>
Niobium (Nb)	0.067	0.024	3.2
Tungsten (W)	0.0695	0.0094	3.2

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

Element	Mass Fraction (%)
Arsenic (As)	< 0.005
Bismuth (Bi)	< 0.0001
Lead (Pb)	< 0.0003
Nitrogen (N)	0.003
Silver (Ag)	0.000025
Tantalum (Ta)	< 0.001
Tin (Sn)	< 0.033
Zirconium (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://www.nist.gov/pml/pubs/sp811/index.cfm> (accessed July 2016).
- [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.; *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 July 2016).
- [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/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed July 2016); 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 July 2016).
- [4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

Certificate Revision History: 13 July 2016 (Editorial changes); 25 June 2014 (Editorial changes); 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 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>.

Appendix A. Analytical Methods

Constituent	Methods*	Constituent	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 furnace
3. Optical emission spectrometry
4. Colorimetric – dianthrimide
5. Colorimetric - diethyldithiocarbamate – extraction
6. Colorimetric – dithizone
7. Colorimetric - Molybdenum Blue
8. Colorimetric – 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 – H₂O₂
22. Photometric - Molybdenum Blue
23. Photometric – periodate
24. Photometric - p-hydroxy phenylarsonic acid precipitation, peroxide color-photometric
25. Titrimetric - AgNO₃ - NaCN titration
26. Titrimetric - Ion exchange, K₂Cr₂O₇ titration
27. Titrimetric - perchloric – KMnO₄ titration
28. Titrimetric - Persulfate oxidation, sodium arsenite titration
29. Titrimetric – KMnO₄ - FeSO₄-(NH₄)₂S₂O₈ titration
30. Titrimetric - redox titration
31. Volumetric - Ion exchange – EDTA
32. X-ray fluorescence spectrometry
33. X-ray fluorescence spectrometry after borate fusion