



National Institute of Standards & Technology

# Certificate of Analysis

Standard Reference Material<sup>®</sup> 348a

High-Temperature Alloy A286

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 348a consists of a bottle containing approximately 150 g of chips sized between 0.50 mm and 1.18 mm sieve openings (35 mesh and 16 mesh). Material from the same lot is available in disk form as SRM 1230 intended primarily for use in optical emission and X-ray spectrometric methods.

**Certified Values:** Certified values for 15 constituents in SRM 348a 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 two constituents 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 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 348a** 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 at 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.

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Gaithersburg, MD 20899  
Certificate Issue Date: 08 September 2009  
*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. Wlodarczyk; 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

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The material should be stored in its original container in a cool, dry location.

## PREPARATION AND ANALYSIS<sup>1</sup>

The material for this SRM was furnished by Crucible Metals Corporation, Specialty Metals Division, Syracuse, NY. Analytical methods used for certification are provided in the appendix.

Table 1. Certified Values for SRM 348a High-Temperature Alloy A286

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, <i>k</i>
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

Table 2. Reference Values for SRM 348a High-Temperature Alloy A286

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

<sup>1</sup> 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 3. Information Values for SRM 348a 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.; Beck II, C. M.; Fassett, J. D.; Greenberg, R. R.; Guenther, 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](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/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/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); 30 March 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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*

## Appendix. Analytical Methods

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

\*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 – H<sub>2</sub>O<sub>2</sub>
22. Photometric – molybdenum blue
23. Photometric – periodate
24. Photometric – p-hydroxy phenylarsonic acid precipitation, peroxide color-photometric
25. Titrimetric – AgNO<sub>3</sub> - NaCN titration
26. Titrimetric – ion exchange, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> titration
27. Titrimetric – perchloric – KMnO<sub>4</sub> titration
28. Titrimetric – persulfate oxidation, sodium arsenite titration
29. Titrimetric – potassium permanganate - ferrous ammonium sulfate titration
30. Titrimetric – redox titration
31. Volumetric – Ion exchange – EDTA
32. X-ray fluorescence spectrometry
33. X-ray fluorescence spectrometry after borate fusion