National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 898

Nickel-Base High-Temperature Alloy
(Tracealloy B)

This Standard Reference Material (SRM) is intended for use in the validation of chemical and instrumental methods for trace elemental analysis of high-temperature alloys. A unit of SRM 898 consists of a single bottle containing approximately 35 g of fine millings.

Certified Mass Fraction Values: Certified mass fraction values for constituents of SRM 898 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 for constituents of SRM 898 are provided in Table 2. Reference values are non-certified values that are the 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 sources of uncertainty [2].

Information Mass Fraction Values: Information mass fraction values for constituents of SRM 898 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 898** 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"). 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.

Coordination of technical measurements for certification of this SRM was performed by I.L. Barnes of the NIST Chemical Sciences Division. Review and revision of value assignments were performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 898 were performed by J.W. Gramlich, H.M. Kingston, L.A. Machlan, E.J. Maienthal, J.D. Messman, P.J. Paulson, L.P. Powell, and T.C. Rains of the NIST Chemical Sciences Division; P. Cole of ATI Allvac (Monroe, NC); G. Infantino of Evans Analytical Group (Syracuse, NY); L. Dilks of Laboratory Testing, Inc. (Hatfield, PA); B. Guidoboni of Northern Analytical Laboratories, (Londonderry, NH); L. Somrack of NSL Analytical (Cleveland, OH); T. LeRoy and J. Capobianco of Vale Inco (Mississauga, Ontario, Canada); G. Witt of ATI Allegheny Ludlum (Brackenridge, PA); S. Damp of Alcoa Howmet (Whitehall, MI); C. Polinko of Carpenter Technologies (Reading, Pennsylvania); A. O'Connell of Haynes International (Kokomo, IN); J. Jordan of Huntington Alloys Corp. (Huntington, WV); and D. Dorn of ATI Wah Chang (Albany, OR).

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

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Certificate Issue Date: 05 February 2014
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Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR USE

The material must be sampled directly from its original container and used without additional preparation. The recommended minimum sample mass is 1.0 g. SRM 898 should be stored in its original, tightly capped container in a cool, dry location.

PREPARATION AND ANALYSIS[(1)]

The preparation of material for SRM 898 was carried out by the Subcommittee on Trace Elements in Superalloys of the Gas Turbine Panel of the Joint Committee on the Effect of Temperature on the Properties of Metals, consisting of members from ASTM International, the American Society of Mechanical Engineers, and the Metals Properties Council. The nominal composition of the nickel-base alloy contains all elements found in turbine blade applications and was designated “Tracealloy”. Approximately 135 kg of the “Tracealloy” composition was vacuum induction melted. Trace element master alloys were added under partial pressure, and a set of ingots, 7.6 cm diameter and 76 cm long, were statically cast. Homogeneity studies showed no detectable segregation of the trace elements from top to bottom of the ingots. At NIST, material from the lot was selected that exhibited minimal piping. Following complete removal of scale and cold shuts from the periphery of the ingots, the material was converted to fine millings, sieved, and thoroughly blended to form SRM 898. Quantitative determinations were performed at NIST and at collaborating laboratories using the test methods listed in Table 4. Laboratories and analysts participating in the development and preparation of the materials are listed in Table 5.

Certified Mass Fraction Values: The values in Table 1 were derived from the combination of results provided by NIST and collaborating laboratories. The measurands are the total mass fractions of selected elements in the nickel-base alloy. The certified values are metrologically traceable to the SI unit of kilogram and are expressed as milligrams as per kilogram. The values are the weighted means of the individual sets of measurements made by the laboratories estimated using a Gaussian random effects model [3] and the DerSimonian-Laird procedure [4,5]. Each associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [6,7]. The uncertainty is expressed as an expanded uncertainty, U, represented as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The coverage factor (k) corresponds to an approximately 95 % confidence level for each analyte.

Table 1. Certified Mass Fraction Values for SRM 898

Constituent	Mass Fraction (mg/kg)	Coverage Factor, k
Lead (Pb)	2.08 ± 0.12	2.0
Selenium (Se)	2.00 ± 0.10	2.0
Tellurium (Te)	0.622 ± 0.067	2.0
Thallium (Tl)	2.85 ± 0.14	2.0

Reference Mass Fraction Values: The values in Table 2 were derived from the combination of results provided by collaborating laboratories. The measurands are the mass fractions of selected elements in the nickel-base alloy, as determined by the listed test methods in Table 4. The reference values are metrologically traceable to the SI unit of kilogram and are expressed as milligrams as per kilogram. The values are the weighted means of the individual sets of measurements made by the laboratories estimated using a Gaussian random effects model [3] and the DerSimonian-Laird procedure [4,5]. Each associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [6,7]. The uncertainty is expressed as an expanded uncertainty, U, represented as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The coverage factor (k) corresponds to an approximately 95 % confidence level for each analyte.

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(1) Certain commercial organizations, services, equipment, or materials are identified in this certificate in order 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 organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

Table 2. Reference Mass Fraction Values for SRM 898

Constituent	Mass Fraction (mg/kg)	Coverage Factor, *k*
Antimony (Sb)	1.49 ± 0.09	2.0
Bismuth (Bi)	1.03 ± 0.05	2.0
Gallium (Ga)	6.3 ± 0.4	2.0
Silver (Ag)	0.9 ± 0.6	2.0
Tin (Sn)	4.1 ± 0.3	2.0

Information Mass Fraction Values: In Table 3, the values for all listed constituents are intended to provide the base composition of the "Tracealloy" material. The values were provided by the committee that designed the alloy.

Table 3. Information Mass Fraction Values for SRM 898

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
Aluminum (Al)	2	Niobium (Nb)	0.9
Boron (B)	0.01	Nickel (Ni)	Balance
Carbon (C)	0.12	Tantalum (Ta)	1.75
Cobalt (Co)	8.5	Titanium (Ti)	2
Chromium (Cr)	12	Tungsten (W)	1.75
Hafnium (Hf)	1.2	Zirconium (Zr)	0.1

Table 4. Methods Used for Analysis of SRM 898

Constituent	Methods[a]	Constituent	Methods[a]
Silver (Ag)	3	Selenium (Se)	2, 3, 4
Bismuth (Bi)	3, 4	Tin (Sn)	3
Gallium (Ga)	3	Tellurium (Te)	2, 4
Lead (Pb)	1, 3, 4	Thallium (Tl)	1, 3
Antimony (Sb)	3		

[a] Key to Methods:
1. Isotope dilution thermal ionization mass spectrometry
2. Isotope dilution spark source mass spectrometry
3. Inductively coupled plasma mass spectrometry [8]
4. Graphite furnace atomic absorption spectrometry [9]

Table 5. Laboratories Collaborating in the Development of "Tracealloy" Materials

Acculabs – R. Brown	Cannon Muskegon – R. Schwer	Cartech – R. Whitney
Falconbridge – L. Morris	Howmet Alloy Division – J. White	Huntington Alloys – D. Carlson
INCO – C. David	J. Dirats & Co. – E. Dirats	Lycoming – R. Sussman
Misco-Howmet – D. Preston	Pratt & Whitney – J. Marks	SNECMA – M. Frerre
Special Metals – M. Harrington	Stellite-Cabot – J. McLafferty	Teledyne Allvac – W. Thomas
TRW – L. Harper	Union Carbide Metals – C. Brown	Union Carbide - UK – I. Nolleth
Universal-Cyclops – L. Lherbier, – A. Mirarchi	Westinghouse – G. Beck	Wyman Gordon – R. Sparks

REFERENCES

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[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 Feb 2014).

[8] ASTM E1834–11; *Standard Test Method for Analysis of Nickel Alloys by Graphite Furnace Atomic Absorption Spectrometry*; Annu. Book of ASTM Stand., Vol. 03.05 (2011).

[9] ASTM E2823–11; *Standard Test Method for Analysis of Nickel Alloys by Inductively Coupled Plasma Mass Spectrometry (Performance-Based Method)*; Annu. Book of ASTM Stand., Vol. 03.05 (2011).

Certificate Revision History: 05 February 2014 (Revision of certified values and uncertainties for Se, Te, Tl, and Pb and new reference values for Ga, Ag, Sn, Sb, and Bi based on re-evaluation of the original and new analytical results; editorial changes); 31 August 1983 (Revision of value and uncertainty for lead); 08 August 1980 (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.