National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1244

Nickel Alloy UNS N06600
(disk form)

This Standard Reference Material (SRM) is intended primarily for use in optical emission and X-ray fluorescence spectrometric methods of analysis of nickel alloys and similar matrices. It can be used to validate value assignment of in-house reference materials. A unit of SRM 1244 is a wrought disk 35 mm diameter and 19 mm thick.

Certified Mass Fraction Values: Certified values for constituents in SRM 1244 are reported in Table 1 as mass fractions of the elements in a nickel matrix [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 taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction, expressed as percent of as milligrams per kilogram. The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %.

Table 1. Certified Mass Fraction Values for SRM 1244 Nickel Alloy UNS N06600

Element	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor *k*
Aluminum (Al)	0.252	0.007	1.97
Cobalt (Co)	0.0602	0.0017	1.98
Chromium (Cr)	15.74	0.14	1.99
Copper (Cu)	0.255	0.005	1.98
Iron (Fe)	9.63	0.12	1.97
Manganese (Mn)	0.288	0.011	1.99
Molybdenum (Mo)	0.204	0.007	1.98
Nickel (Ni)	73.09	0.20	1.91
Vanadium (V)	0.0327	0.0016	1.99

Element	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
Boron (B)	28.3	1.6	1.97
Magnesium (Mg)	138.3	2.6	1.99
Lead (Pb)	2.27	0.03	2.30
Thallium (Tl)	0.0029	0.0002	2.21

Expiration of Certification: The certification of **SRM 1244** 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 Handling, Storage, and Use"). Periodic recalibration or recertification of this SRM is not required. However, the certification will be nullified if the SRM is damaged, contaminated or otherwise modified.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 July 2017
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

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 or register online) will facilitate notification.

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 Office of Reference Materials.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Each disk of SRM 1244 is certified through its entire thickness. NIST has created the surfaces on each disk using a machine cutting technique. Users should apply the surface finished called for by their laboratory test methods.

Store the SRM in a cool, dry location, preferably in its original container. The alloy is expected to remain stable provided adequate precautions are taken to protect it from contamination, extremes of temperature, and moisture.

To relate the results of analysis to the assigned values and associated uncertainty estimates for SRM 1244, use the following recommendations. For X-ray fluorescence spectrometry (XRF), a measurement area $\geq 2\ cm^2$ is expected to be adequate for this material. For arc-spark and glow discharge optical emission spectrometers, the use of the average of multiple measurements is recommended for a single determination. User experience indicates that three or more “burns,” each ≥ 3 mm diameter, are necessary to adequately sample the alloy.

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 1244.

Reference Mass Fraction Values: Reference values for constituents in SRM 1244 are reported in Table 2 as mass fractions of the elements in a nickel matrix. 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 [2]. The coverage factor (*k*) corresponds to approximately 95 % confidence level for each analyte. Metrological traceability is to the SI derived units for mass fraction, expressed as percent or as milligrams per kilogram.

Table 2. Reference Mass Fraction Values for SRM 1244 Nickel Alloy UNS N06600

Element	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor, *k*
Carbon (C)	0.063	0.002	1.97
Niobium (Nb)	0.126	0.013	1.99
Phosphorus (P)	0.011	0.002	2.00
Sulfur (S)	0.0028	0.0003	2.26
Silicon (Si)	0.114	0.019	1.97
Titanium (Ti)	0.251	0.004	1.99

Element	Mass Fraction (mg/kg)	Expanded Uncertainty (mg/kg)	Coverage Factor *k*
Arsenic (As)	19.0	6.1	1.97
Tin (Sn)	7.4	0.4	2.29
Zirconium (Zr)	3.7	1.2	2.00

Information Mass Fraction Values: Information values for constituents of SRM 1244 are reported 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. Information values cannot be used to establish metrological traceability.

Table 3. Information Mass Fraction Values for SRM 1244 Nickel Alloy UNS N06600

Element	Mass Fraction (%)
Silver (Ag)	<0.0001
Bismuth (Bi)	0.00001
Calcium (Ca)	<0.0001
Gallium (Ga)	0.003
Nitrogen (N)	0.01
Oxygen (O)	0.004
Antimony (Sb)	0.0001
Selenium (Se)	<0.001
Tantalum (Ta)	<0.001
Tellurium (Te)	<0.0001
Tungsten (W)	<0.002

PREPARATION AND ANALYSIS(1)

The material for SRM 1244 was prepared by Huntington Alloys, Inc., Huntington, WV. Homogeneity testing was performed at NIST on the rods received from the supplier by using optical emission spectrometry, X-ray fluorescence spectrometry, and chemical methods of analysis. Quantitative analyses were performed at NIST and the collaborating laboratories using the test methods listed in Table 4.

The certified mass fraction values in Table 1 were derived from the combination of results provided by NIST and collaborating laboratories. The values are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [4] and the DerSimonian-Laird procedure [5,6]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the GUM Supplement 1 [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 approximately 95 % confidence level for each analyte. The values for Pb and Tl in Table 1 were derived based upon a single NIST primary method and confirmed by values provided by a collaborating laboratory using an alternate method. The certified value is the mean of nine NIST measurements. The associated uncertainty is expressed as an expanded uncertainty, U, calculated as $U = ku_c$, where k is the coverage factor and u_c is the combined standard uncertainty, at the level of one standard deviation, calculated according to the JCGM Guide [3]. Included in u_c are the uncertainty components associated with measurement repeatability, blank correction, spike calibration, and isotope ratio measurement uncertainty. The value of k is obtained from the Student's t-distribution using the effective degrees of freedom, ν_{eff} and controls the approximate level of confidence associated with U, which, for this SRM is approximately 95 %. For Pb, $\nu_{eff} = 8.2$ and for Tl, $\nu_{eff} = 10.5$.

The reference mass fraction values are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [4] and the DerSimonian-Laird procedure [5,6]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the GUM Supplement 1 [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.

Analytical determinations for value assignment of SRM 1244 were performed at NIST by E.S. Beary, R.K. Bell, K.A. Brletic, D.E. Brown, B.I. Diamondstone, M.S. Epstein, R.M. Lindstrom, J.A. Norris, and P.A. Pella; at Allegheny Ludlum Steel Corp., Brackenridge, PA, by C.W. Hartig, R.M. Chybrzynski, and A.I. Fulton; at ATI Allegheny Ludlum, Natrona Heights, PA, by S. Bissell-Seymour and G.A. Witt; at ATI Allvac, Monroe, NC, by P.M. Cole; at Carpenter Technology Corp., Reading, PA, by T.R. Dulski; at Huntington Alloy, Inc., Huntington, WV, by R.L. Blake, D.A. Damron, D.E. Howells, M. Kirk, L.J. Lundy, R.D. Laishley, G.T. Marshall, A.H. Roberts, K.S. Roberts, F.H. Robinson, W.L. Stickler, and F.A. Blair; at Pratt & Whitney Aircraft Group, Middletown, CT, and East Hartford, CT, by J.Y. Marks, G. Welcher, D. Fornwall, and R. Spellman; at United Technologies Corporation,

(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.

East Hartford, CT, by G.S. Golden; at Universal-Cyclops, Bridgeville, PA, and Titusville, PA, by F.F. Liberato, W.S. Harbin, S.J. Staron, S.L. Kelley, D.K. Luoni, and R. Hall; and at Wyman Gordon Co., North Grafton, MA, by H. Ackerman.

Table 4. Analytical Test Methods Applied to SRM 1244 Nickel Alloy UNS N06600

Method	Elements
Combustion with Infrared or Thermal Conductivity Detection	C, N, O, S
Direct Current Plasma Optical Emission Spectrometry	Ag, Al, As, Cu, Fe, Mg, Mn, Ni, Zr
Graphite Furnace Atomic Absorption Spectrometry	Ag, Al, As, Co, Cu, Fe, Mn, Mo, Se, Sn, Te, Ti
Gravimetry	Cr, Ni, Si
Inductively Coupled Plasma Mass Spectrometry	Ag, Bi, Ga, Pb, Sb, Sn, Ta, Tl, V, Zr
Inductively Coupled Plasma Optical Emission Spectrometry	B, Cu, Mg, Nb, P, Ta, V, W, Zr
Instrumental Neutron Activation Analysis	Al, As, Cu, Ga, Mn, Mo, V, W
Isotope Dilution Thermal Ionization Mass Spectrometry	Pb, Tl
Laser Enhanced Ionization Spectrometry	Co
Potentiometry	Cr, Mn
Prompt Gamma-Ray Activation Analysis	B
Spark Source Optical Emission Spectrometry	B, Mg, P
Spectrophotometry	Al, B, Co, Cu, Mo, Nb, P, Ti
Titrimetry	Fe
X-Ray Fluorescence Spectrometry	Al, Co, Cr, Cu, Fe, Mn, Mo, Nb, Ni, Si, Ti, V

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 https://www.nist.gov/physical-measurement-laboratory/special-publication-811 (accessed July 2017).

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[6] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

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Certificate Revision History: 31 July 2017 (Updated title; editorial changes); 20 July 2012 (Values for all elements updated; editorial changes); 05 May 1984 (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 at: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet at http:/www.nist.gov/srm.