



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 864

Nickel Alloy UNS N06600

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of chemical and instrumental methods of analysis of nickel alloys and similar matrices. A unit of SRM 864 is a single bottle containing 50 g of chips. SRM 1244 is Nickel Alloy UNS N06600 in disk form.

Certified Mass Fraction Values: Certified values for 13 elements in SRM 864 are listed in Table 1. All values are reported as mass fractions [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 using the test methods identified below.

Reference Mass Fraction Values: Reference values for 9 elements 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 [2].

Information Mass Fraction Values: Information values for 11 elements are given 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.

Expiration of Certification: The certification of **SRM 864** 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.

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.

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

Analytical determinations for value assignment of SRM 864 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, 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.

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Gaithersburg, MD 20899
Certificate Issue Date: 20 July 2012
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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.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

A minimum test portion of 0.25 g of chips is recommended. Chips may be used in as-received form. 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.

PREPARATION AND ANALYSIS⁽¹⁾

The material for SRM 864 was prepared by Huntington Alloys, Inc., Huntington, WV. Homogeneity testing was performed at NIST on the original 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.

Certified Mass Fraction Values: 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 ISO 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, v_{eff} and controls the approximate level of confidence associated with U , which, for this SRM is approximately 95 %. For Pb, $v_{\text{eff}} = 8.2$ and for Tl, $v_{\text{eff}} = 10.5$.

The remaining 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.

⁽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 1. Certified Mass Fraction Values for SRM 864 Nickel Alloy UNS N06600

Element	Mass Fraction (%)			Coverage Factor k
Al	0.252	±	0.007	1.97
Co	0.0602	±	0.0017	1.98
Cr	15.74	±	0.14	1.99
Cu	0.255	±	0.005	1.98
Fe	9.63	±	0.12	1.97
Mn	0.288	±	0.011	1.99
Mo	0.204	±	0.007	1.98
Ni	73.09	±	0.20	1.91
V	0.0327	±	0.0016	1.99

Element	Mass Fraction (mg/kg)			Coverage Factor k
B	28.3	±	1.6	1.97
Mg	138.3	±	2.6	1.99
Pb	2.27	±	0.03	2.30
Tl	0.0029	±	0.0002	2.21

Reference Mass Fraction Values: The values in Table 2 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.

Table 2. Reference Mass Fraction Values for SRM 864 Nickel Alloy UNS N06600

Element	Mass Fraction (%)			Coverage Factor k
C	0.063	±	0.002	1.97
Nb	0.126	±	0.013	1.99
P	0.011	±	0.002	2.00
S	0.0028	±	0.0003	2.26
Si	0.114	±	0.019	1.97
Ti	0.251	±	0.004	1.99

Element	Mass Fraction (mg/kg)			Coverage Factor k
As	19.0	±	6.1	1.97
Sn	7.4	±	0.4	2.29
Zr	3.7	±	1.2	2.00

Table 3. Information Mass Fraction Values for SRM 864 Nickel Alloy UNS N06600

Element	Mass Fraction (%)
Ag	<0.0001
Bi	0.00001
Ca	<0.0001
Ga	0.003
N	0.01
O	0.004
Sb	0.0001
Se	<0.001
Ta	<0.001
Te	<0.0001
W	<0.002

Table 4. Analytical Test Methods Applied to SRM 864 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, Sb, Sn, Ta, 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; 2008 Ed. (April 2008); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed July 2012).
- [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).
- [3] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (ISO 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 2012); 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 June 2012).
- [4] Searle, S. R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Hoboken, NJ (2006).
- [5] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Controlled Clin. Trials, Vol. 7, pp. 177–188 (1984).
- [6] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).
- [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/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed July 2012).

Certificate Revision History: 20 July 2012 (Values for all elements updated; editorial changes); 05 May 1984 (Original certificate date).
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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 <http://www.nist.gov/srm>.