National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1243

Ni-Cr-Co Alloy UNS N07001
(disk form)

This Standard Reference Material (SRM) is intended primarily for the evaluation or calibration of spectrometric methods for analysis of Ni-Cr-Co alloys of similar compositions. A unit of SRM 1243 consists of one disk approximately 34 mm diameter and 19 mm thick.

Certified Mass Fraction Values: Certified values for 14 constituents of SRM 1243 are reported in Table 1 as mass fraction values on an as-received basis [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]. Analyses for value assignment were performed by NIST and collaborating laboratories.

Reference Mass Fraction Values: Four reference mass fraction values are reported in Table 2. A NIST reference value is a noncertified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification and is provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2].

Information Mass Fraction Value: One information value is reported in Table 3. An information value is considered to be a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

Expiration of Certification: The certification of **SRM 1243** 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"). Accordingly, 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 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.

Homogeneity testing and analyses leading to the certification of this SRM were performed by A.F. Marlow, J.A. Norris, P.A. Pella, J.R. Sieber, and T.W. Vetter of the NIST Chemical Sciences Division. Analytical determinations were also performed by R.M. Crain, G.L. Bergstrom, S.A. Bissell, and C.B. Farrell of Allegheny Ludlum Corp, Brackenridge, PA; R.R. Buehrer, T.R. Dulski, M.H. Hannay, A.A. Mattiuz, and C.T. Polinko of Carpenter Technology Corp, Reading, PA; D.R. Neese of Cyclops Corp, Titusville, PA; and P. Cole of ATI Allvac, Monroe, NC.

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

Support aspects involved with the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR USE

The test surface is the side opposite from the labeled surface that includes the SRM number. The entire thickness of the unit is certified; however, the user is cautioned not to measure disks less than 2 mm thick when using X-ray fluorescence spectrometry. 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 take care when either resurfacing the disk or performing additional polishing as these processes may contaminate the surface. NIST found that abrasive paper must be changed frequently during surface grinding. Used paper loses its ability to remove contaminants from the surface of the alloy. When not in use, the material should be stored in its original container in a cool, dry location. The certified values are considered to be representative of the overall average composition of the material.

SOURCE, PREPARATION, AND ANALYSIS[(1)]

The material for SRM 1243 was provided by Carpenter Technology Corporation, Reading, PA. Alloy UNS 07001 is a precipitation hardenable nickel alloy for high-temperature service. Homogeneity testing was performed using spark source optical emission spectrometry and X-ray fluorescence spectrometry. Quantitative determinations were performed at NIST and at collaborating laboratories using the test methods listed in Table 4.

Certified Mass Fraction Values: The 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 [3] and the DerSimonian-Laird procedure [4,5]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO 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 effects 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 1243

Elements	Mass Fraction (%)	Coverage Factor, k
Co	12.39 ± 0.12	2.00
Cr	19.05 ± 0.29	2.00
Fe	0.776 ± 0.027	1.96
Mo	4.226 ± 0.039	1.99
Nb	0.0286 ± 0.0018	1.98
Ni	58.782 ± 0.035	1.98
Si	0.0192 ± 0.0033	2.00
Ti	3.054 ± 0.022	2.37
V	0.1043 ± 0.0031	1.99

Elements	Mass Fraction (mg/kg)	Coverage Factor, k
B	49.4 ± 2.8	1.98
Cu	63 ± 14	2.00
Mn	73.0 ± 4.3	2.01
P	31.7 ± 4.4	1.98
S	21.7 ± 7.5	2.00

[(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.

Reference Mass Fraction Values: The values for Al, C, and Zr in Table 2 were obtained during the original certification of SRM 1243 using methods employed at NIST and collaborating laboratories. Each value is the mean of multiple determinations ($n = 6$ for Al and C, $n = 5$ for Zr). 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. Included in u_c are uncertainty components associated with measurement repeatability and reproducibility among methods and laboratories. The value of k controls the level of confidence associated with U, which, for this SRM, is approximately 95 %. The value of k is obtained from the Student's t-distribution using the degrees of freedom, $v = n–1$.

The value for tungsten (W) in Table 2 was derived from the combination of results provided by two collaborating laboratories. The value is the weighted mean of the individual sets of measurements estimated using a Gaussian random effects model [3] and the DerSimonian-Laird procedure [4,5]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO 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.

Table 2. Reference Mass Fraction Values for SRM 1243

Elements	Mass Fraction (%)	Coverage Factor, k
Al	1.23 ± 0.05	2.57
C	0.024 ± 0.002	2.57
W	0.0139 ± 0.0006	1.98
Zr	0.053 ± 0.004	2.78

Information Mass Fraction Value for Tantalum: The mass fraction value is the mean result of a NIST analysis using a single method. No uncertainty is provided because there is insufficient information available for its assessment.

Table 3. Information Mass Fraction Value for SRM 1243

Element	Mass Fraction (%)
Ta	0.0003

Table 4. Methods[(a)] Used for Analysis of SRM 1243

Element	Methods	Element	Methods
Al	1, 5	Ni	1, 9
B	4, 5	P	1, 7
C	2	S	1, 2
Co	1, 5	Si	1, 10
Cr	1, 8	Ta	6
Cu	1, 3, 5	Ti	1, 7
Fe	1	V	1, 3, 4, 6
Mn	1, 3, 5	W	1, 4, 6
Mo	1, 7	Zr	1
Nb	1, 4, 6		

[(a)]Key to Methods in Table 4:

1. X-ray fluorescence spectrometry
2. Direct combustion followed by thermal conductivity or infrared detection
3. Flame atomic absorption spectrometry
4. Inductively coupled plasma optical emission spectrometry
5. Direct current plasma optical emission spectrometry
6. Inductively coupled plasma mass spectrometry
7. Photometric method
8. Oxidation with peroxydisulfate followed by titration with $KMnO_4$
9. Dimethyl glyoxime – titration
10. Gravimetric determination after $HClO_4$ dehydration

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 Nov 2012).

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

[6] 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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 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 Nov 2012).

[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 Nov 2012).

Certificate Revision History: 17 December 2012 (In Table 1, certified values and uncertainty estimates were updated and Nb was added; Al, C, Zr, and W were changed to reference values; Ta information value added; editorial changes); **10 January 1989** (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; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.