National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1249

Nickel-based Superalloy

(UNS N07718)

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis. A unit of SRM 1249 is in the form of a disk approximately 41 mm diameter and 19 mm thick.

The certified values for 9 elements in SRM 1249 are listed in Table 1. Reference values for 15 elements are listed in Table 2. Information values for 6 elements are listed in Table 3. Values are reported as mass fractions [1]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2].

Certified Values: 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 accounted for by NIST. 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 listed in Table 4. The uncertainty listed with the value is an expanded uncertainty (95 % confidence) [3] calculated according to the method in the ISO and NIST Guides [4].

Reference Values: 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 sources of uncertainty. The uncertainty listed with the value is an expanded uncertainty (95 % confidence) [3] calculated according to the method in the ISO and NIST Guides [4].

Information Values: 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 this SRM 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"). However, the certification will be nullified if the SRM is damaged or contaminated.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements for certification was under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Analytical measurements for homogeneity testing and certification of this SRM were performed by A.F. Marlow and P.A. Pella (retired) of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Revision Date: 07 March 2006
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

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

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

The test surface is the side opposite to the labeled surface, which includes the SRM number. The entire thickness of the unit is certified. 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 use care when either resurfacing the disk or performing additional polishing as these processes may contaminate the surface. The material should be stored in its original container in a cool, dry location. This material was tested using both solid disks and chips prepared from the disks. The certified values are considered to be representative of the overall average composition of the material.

Table 1. Certified Values for SRM 1249 Nickel-based Superalloy

Constituent	Value (mass fraction) (%)	Expanded Uncertainty (mass fraction) (%)
Al	0.5682	0.0065 [b]
Ti	0.959	0.015 [a]
Cr	18.472	0.034 [a]
Fe	17.693	0.064 [a]
Co	0.3371	0.0078 [a]
Ni	53.29	0.26 [a]
Cu	0.1402	0.0020 [a]
Nb	5.196	0.021 [a]
Mo	3.112	0.028 [a]

[a] The assigned value is a weighted mean of the results from two to seven analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with a coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [4,5].

[b] The assigned value is an unweighted mean of the results from two to five analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance with a pooled, within-method variance following the ISO and NIST Guides [4,6].

Table 2. Reference Values for SRM 1249 Nickel-based Superalloy

Constituent	Value (mass fraction) (%)	Expanded Uncertainty (mass fraction) (%)
B	0.0023	0.0003 [a]
C	0.0380	0.0014 [b]
Mg	0.0012	0.0001 [a]
Si	0.120	0.008 [a]
P	0.0134	0.0004 [a]
S	0.00064	0.00010 [a]
V	0.0338	0.0014 [a]
Mn	0.108	0.003 [a]
Ga	0.0019	0.0002 [a]
As	0.0013	0.0003 [a]
Zr	0.0029	0.0002 [a]
Sn	0.0024	0.0002 [a]
Sb	0.00030	0.00006 [a]
Ta	0.0027	0.0008 [b]
W	0.0846	0.0008 [a]

[a] The assigned value is a weighted mean of the results from two to seven analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with a coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [4,6].

[b] The assigned value is an unweighted mean of the results from two to five analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance with a pooled, within-method variance following the ISO and NIST Guides [4,6].

Table 3. Information Values for SRM 1249 Nickel-based Superalloy

Constituent	Value (mass fraction) (%)
N	0.007
Ca	0.0005
Zn	0.0006
Se	0.00003
Te	< 0.00005
Pb	0.00001

Table 4. Analytical Methods

Element	Methods
Al	DCP-OES; FAAS; ICP-OES; SS-OES; WDXRF
Ti	DCP-OES; FAAS; ICP-OES; MAS; SS-OES; WDXRF
Cr	ICP-OES; SS-OES; TITR; WDXRF
Fe	ICP-OES; WDXRF
Co	DCP-OES; FAAS; ICP-OES; SS-OES; WDXRF
Ni	GRAV; ICP-OES; SS-OES; TITR; WDXRF
Cu	DCP-OES; FAAS; ICP-OES; SS-OES; WDXRF
Nb	DCP-OES; EDXRF; FAAS; ICP-OES; SS-OES; WDXRF
Mo	DCP-OES; GRAV; FAAS; ICP-OES; SS-OES; WDXRF
B	GD-MS; ICP-MS; SS-OES
C	COMB-IR; SS-OES
Mg	DCP-OES; FAAS; GD-MS; ICP-OES; ICP-MS; SS-OES
Si	DCP-OES; FAAS; ICP-OES; SS-OES; WDXRF
P	DCP-OES; MAS; ICP-OES; SS-OES; WDXRF
S	COMB-IR; GS-MS
V	DCP-OES; FAAS; ICP-OES; SS-OES; WDXRF
Mn	DCP-OES; FAAS; ICP-OES; MAS; SS-OES; WDXRF
Ga	DCP-OES; GD-MS; GFAAS; ICP-MS; SA-ICP-MS
As	GD-MS; GFAAS; ICP-MS; SA-ICP-MS
Zr	GD-MS; ICP-MS; SS-OES; WDXRF
Sn	GD-MS; GFAAS; ICP-MS; SA-ICP-MS; SS-OES
Sb	GD-MS; GFAAS; ICP-MS; SA-ICP-MS
Ta	GD-MS; ICP-MS
W	ICP-OES; WDXRF
N	IGF
Ca	ICP-OES; SS-OES
Zn	DCP-OES; FAAS; ICP-MS
Se	GFAAS
Te	GFAAS
Pb	GFAAS; ICP-MS

Methods Key:

COMB-IR	(Combustion with Infrared Detection)
DCP-OES	(Direct Current Optical Emission Spectrometry)
EDXRF	(Energy Dispersive X-Ray Fluorescence Spectrometry)
FAAS	(Flame Atomic Absorption Spectrometry)
GD-MS	(Glow Discharge Mass Spectrometry
GFAAS	(Graphite Furnace Atomic Absorption Spectrometry)
GRAV	(Gravimetry)
ICP-OES	(Inductively-Coupled Plasma Optical Emission Spectrometry)
IGF	(Inert Gas Fusion)
MAS	(Molecular Absorption Spectrometry)
SA-ICP-MS	(Spark Ablation Inductively-Coupled Plasma Mass Spectrometry)
SS-OES	(Spark Source Optical Emission Spectrometry)
TITR	(Titrimetry)
WDXRF	(Wavelength Dispersive X-Ray Fluorescence Spectrometry)

Cooperating Laboratories: Analytical determinations for certification of this SRM were performed by the following laboratories:
Allegheny Ludlum, Technical Center (Brackenridge, PA, USA); R.M.Crain, S.A.Bissell-Seymour
ATI Allvac (Monroe, NC, USA); P.M.Cole, C.B.Wilson
ATI Allvac,(Lockport, NY, USA); T.A.Herdlein, P.S.Psutka
Carpenter Technology Corp. (Reading, PA, USA); C.T.Polinko, K.H.Dabbs, M.W.Teti
Howmet Corp. (Whitehall, MI, USA); R. DeHoff, R. Starr
Huntington Alloys Corp. (Huntington, WV, USA); D.McAnallen,
Inco Technical Services Ltd. (Mississauga, Ontario, Canada)
Shiva Technologies, Inc. (Syracuse, NY, USA); R. Balamut, M.Kasik

Material Preparation: The material for SRM 1249 was provided by Inco Alloys, Inc. (Huntington, WV)[1].

REFERENCES

[1] 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 (1995); available at http://www.physics.nist.gov/Pubs.

[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Gaithersburg, MD (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.

[3] Hahn, G. J., and Meeker, W. Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

[4] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

[5] Ruhkin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Mean Statistics*; J. Am. Statist. Assoc., Vol. 93, pp. 303–308 (1998).

[6] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

Certificate Revision History: 07 March 2006 (Revised and added certified, reference, and information values and their associated uncertainties following additional testing); 07 March 1996 (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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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.