



Certificate of Analysis

Standard Reference Material[®] 861

Nickel-Base Superalloy PWA 1484 (chip form)

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques employed in the determination of sulfur and phosphorus in nickel-based alloys. A unit of SRM 861 consists of a glass jar containing 50 g of metal chips that have passed through a 600 μm (30 mesh) screen.

Certified Sulfur Mass Fraction Value: The certified mass fraction value for sulfur is based on a single NIST primary method, isotope dilution thermal ionization mass spectrometry (ID-TIMS) [1–4]. 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 [5].

Certified Value of Sulfur: 0.561 mg/kg \pm 0.078 mg/kg

The uncertainty in the certified value for sulfur is expressed as an expanded uncertainty, U , using the methods described in the ISO/JCGM Guide [6]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with measurement uncertainty and sulfur inhomogeneity; k is a coverage factor used to control the confidence level of the expanded uncertainty. The coverage factor, $k = 2.23$, is determined from the Student's t -distribution with 10 degrees of freedom and corresponds to an approximate 95 % confidence interval. The measurand is the mass fraction for sulfur. The certified value is metrological traceable to the SI unit of mass, expressed as milligrams per kilogram.

Expiration of Certification: The certification of **SRM 861** 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 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.

Coordination of the technical measurements leading to certification was performed by W.R. Kelly of the NIST Chemical Sciences Division.

Homogeneity testing of the chipped material was performed by R. Kunish of Howmet Corporation, Whitehall, MI.

Isotope dilution thermal ionization mass spectrometry analyses were performed by W.R. Kelly, J.L. Mann, and R.D. Vocke of the NIST Chemical Sciences Division. Radiochemical neutron activation analysis was performed by R.L. Paul of the NIST Chemical Sciences Division.

Statistical analysis was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

INSTRUCTIONS FOR USE

Use: The unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 100 mg should be used for analytical determinations to be related to the sulfur and phosphorus values provided in this certificate. It is recommended that when not in use, each unit of SRM 861 be stored tightly capped and isolated from the atmosphere to prevent contamination.

PREPARATION AND ANALYSIS⁽¹⁾

Source and Preparation of Material: The parent material for this SRM was purchased from Howmet Corporation and is a superalloy of a type that is commonly used to fabricate single-crystal high temperature turbine blades for jet aircraft engines. The original material was in the form of two bars (9 cm in diameter by 122 cm in length), each weighing about 45 kg. The bars were dry chipped at Analytical Reference Materials International (ARMI, Evergreen, CO) and the chips placed in 55 glass jars. Before blending the chips, two samples were taken from each jar and the sulfur determined by a combustion technique at Howmet Corporation under the direction of R. Kunish to check for the possibility of inadvertent contamination during the chipping process. All jars had essentially the same sulfur concentration. The blending of the contents of the 55 jars for distribution into SRM units was performed at NIST under the direction of D.G. Friend of the NIST Material Measurement Laboratory.

Reference Phosphorus Value and Uncertainty: The reference value for phosphorus is based on a radiochemical neutron activation analysis (RNAA) method developed at NIST [7—8]. A NIST reference value is a noncertified value and is the best estimate of the true value. The value does not meet NIST criteria for certification and is provided with an associated uncertainty that may not include all sources of uncertainty [5]. The measurand is the mass fraction for phosphorous listed below determined by the method listed above. The reference value is metrological traceable to the SI unit of mass, expressed as milligrams per kilogram.

Reference Value of Phosphorus: 12.71 mg/kg ± 0.71 mg/kg

The uncertainty in the reference value for phosphorus is expressed as an expanded uncertainty, U , using the methods described in the ISO/JCGM Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the effect of uncertainty components associated with measurement precision, and k is a coverage factor used to control the confidence level of the expanded uncertainty. The coverage factor, $k = 2$, is determined from the normal distribution.

⁽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.

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 Oct 2016).
- [2] Paulsen, P.J.; Kelly, W.R.; *Determination of Sulfur as Arsenic Mono-sulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry*; *Anal. Chem.*, Vol. 56, pp. 708–713 (1984).
- [3] Kelly, W.R.; Paulsen, P.J.; *Precise and Accurate Determination of High Concentrations of Sulfur by Isotope Dilution Thermal Ionization Mass Spectrometry*; *Talanta*, Vol. 31, pp. 1063–1068 (1984).
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- [5] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-36 (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Oct 2016).
- [6] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (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 Oct 2016); 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 Oct 2016).
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- [8] Paul, R.L.; *Measurement of Phosphorus in Metals by RNAA*; *J. Radioanal. Nucl. Chem.*, Vol. 245, pp. 11–15 (2000).

Certificate Revision History: 24 October 2016 (Title change; editorial changes) 09 July 2014 (Updated certification date to indefinite; editorial changes); 28 March 2001 (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>.