



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1107

Naval Brass UNS 46400

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of elemental analysis of brass alloys. SRM 1107 is a UNS 46400 naval brass alloy in wrought form. A unit of SRM 1107 consists of a single disk approximately 3.2 cm diameter and 1.9 cm thick.

Certified Mass Fraction Values: Certified mass fraction values for elements are provided in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories.

Reference Mass Fraction Value: A reference value for aluminum is provided 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 [2] and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Values: Information values for elements are provided 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 [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1107** is valid indefinitely, within the measurement uncertainties 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 of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the original certification of this SRM was performed by R.K. Bell and E.E. Maczkowske formerly of NIST. Review and revision of value assignments was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Analytical measurements were performed by R.K. Bell and E.E. Maczkowske. Additional measurements were performed by collaborating laboratories: O.P. Case, J.P. Irwin, and K.M. O'Brien of Anaconda American Brass, Waterbury, CT; A.E. LaRochelle, E.M. Penner, C.H. McMaster, and W.R. Inman, Dept. of Mines, Ottawa, Ontario, Canada; J. Gibson, S.C. Richards, R. Stevens, and A. Stuever, Mueller Brass Co., Port Huron, MI.; A.W. Young, Bridgeport Brass Co., Bridgeport, CT; H.J. Smith, Chase Brass and Copper Co., Waterbury, CT; F.V. Schatz, Olin Mathieson Brass, Inc., Rome, NY; and W.M. Rumberger, Titan Metal Manufacturing Co., Bellefonte, PA.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 25 September 2014
Certificate Revision History on Last Page

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

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 USE

The test surface for optical emission and X-ray fluorescence spectrometric methods is the side not labeled with the SRM number and the diamond-shaped logo. 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 or smear softer metals across the surface. For users requiring chip form material, the disk may be sampled using a machine cutting tool. It is recommended to perform cutting without lubricants or coolants other than water or alcohol. The recommended minimum mass of chips is 0.5 g, which was the minimum mass used in quantitative analyses for certification. The material should be stored in its original container in a cool, dry location.

PREPARATION AND ANALYSIS⁽¹⁾

The material was melted and cast at the Naval Research Laboratory, Washington DC. High-purity metals were used either directly or in the preparation of master alloys. Approximately 1400 kg heats were melted under a charcoal cover in a high-frequency induction furnace. The metal was cast on a massive water-cooled plate to provide rapid unidirectional solidification. The casting for each alloy was about 68.5 cm in diameter and 9 cm thick. The material for wrought form samples of SRM 1107 was obtained after removal of the chill-cast material (19 mm thick) and an additional 19 mm of material from the top of the slab. Strips of the remaining material were forged, fully annealed, and finished to samples 32 mm diameter and 19 mm thick.

The homogeneity of the material was investigated by metallographic studies, by optical emission spectrometry, and by chemical analyses at NIST, and by optical emission spectrometry and chemical analyses by ASTM International Committee E01 on Analytical Chemistry of Metals, Ores, and Related Materials. The homogeneity was found to be satisfactory. Samples for quantitative analyses requiring alloy dissolution were prepared in the form of millings taken from a cross-section of the finished samples of the wrought material. Test methods used in the certification of this SRM are listed in Table 4.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements. The certified values are metrologically traceable to the SI unit of mass, expressed as a percent. The values in Table 1 are the weighted means of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3–5] and the DerSimonian-Laird procedure [6,7]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [8–11]. The expanded uncertainty, U , is calculated 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.

Table 1. Certified Mass Fraction Values for SRM 1107 Naval Brass B

| Elements | Mass Fraction (%) | Coverage Factor k |
|-------------|-------------------|---------------------|
| Copper (Cu) | 61.183 ± 0.074 | 1.98 |
| Iron (Fe) | 0.0389 ± 0.0032 | 1.99 |
| Lead (Pb) | 0.1850 ± 0.0024 | 1.97 |
| Nickel (Ni) | 0.0946 ± 0.0037 | 1.99 |
| Tin (Sn) | 1.066 ± 0.015 | 1.98 |
| Zinc (Zn) | 37.396 ± 0.084 | 1.96 |

⁽¹⁾ Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institutes 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 Value: The measurand is the mass fraction of the element, as determined by the methods indicated. The reference value is metrologically traceable to the SI unit of mass, expressed as a percent. The value in Table 2 is the weighted mean of the individual sets of measurements made by NIST and collaborating laboratories estimated using a Gaussian random effects model [3–5] and the DerSimonian-Laird procedure [6,7]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guide and its Supplement 1 [8–11]. The expanded uncertainty, U , is calculated 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.

Table 2. Reference Mass Fraction Value for SRM 1107 Naval Brass B

| Element | Mass Fraction (%) | Coverage Factor k |
|---------------|-------------------|---------------------|
| Aluminum (Al) | 0.083 ± 0.013 | 2.00 |

Information Mass Fraction Values: In Table 3, the values for the listed elements represent the estimated limits of detection of the applied test methods.

Table 3. Information Mass Fraction Values for SRM 1107 Naval Brass B

| Elements | Mass Fraction (%) |
|----------------|-------------------|
| Manganese (Mn) | < 0.003 |
| Phosphorus (P) | < 0.001 |

Table 4. Methods Used for Analysis of SRM 1107 Naval Brass B

| Method | Elements |
|--|-----------------------------------|
| Optical emission spectrometry (spark or arc source) | Al, Cu, Fe, Mn, Ni, P, Pb, Sn, Zn |
| Double ammonium hydroxide precipitation and weighing as Al ₂ O ₃ | Al |
| Photometric method | Al |
| Phosphomolybdenum blue photometric method | P |
| Potassium iodate photometric method | Mn |
| Potassium dichromate titration | Fe |
| Ortho-phenanthroline photometric method | Fe |
| Thiocyanate photometric method | Fe |
| Dimethylglyoxime gravimetric method | Ni |
| Dimethylglyoxime photometric method | Ni |
| Bromine colorimetric method | Ni |
| Dimethylglyoxime-CHCl ₃ photometric method | Cu |
| ZnS-ZnO gravimetric method | Zn |
| Titration with potassium iodate | Sn |
| Weighed as PbMoO ₄ | Pb |
| Weighed as PbO ₂ | Pb |

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 Aug 2014).
- [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: Washington, DC (2000); available at: <http://www.nist.gov/srm/publications.cfm> (accessed Aug 2014).
- [3] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Hoboken, NJ (2006).
- [4] Pinheiro, J.C.; Bates, D.M.; *Mixed Effects Models in S and S-Plus*; Springer, New York, NY (2000).
- [5] Toman, B.; Possolo, A.; *Laboratory Effects Models for Interlaboratory Comparisons*; *Accredit. Qual. Assur.*, Vol. 14, pp. 553–563 (2009); see also Toman, B.; Possolo, A.; *Erratum to: Laboratory Effects Models for Interlaboratory Comparisons*; *Accredit. Qual. Assur.*, Vol. 15, pp. 653–654 (2010).
- [6] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; *Control Clin. Trials*, Vol. 7, pp. 177–188 (1986).
- [7] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; *Metrologia*, Vol. 46, pp. 323–331 (2009).
- [8] 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/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2014); 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 Aug 2014).
- [9] 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 Aug 2014).
- [10] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).
- [11] Davison, A.C.; Hinkley, D.V.; *Bootstrap Methods and their Application*; Cambridge University Press, New York (1997).

| |
|---|
| <p>Certificate Revision History: 25 September 2014 (Revised certified values for Fe, Ni, Cu, Zn, Sn, and Pb; reference value added for Al; information values added for P and Mn; editorial changes); 23 November 1981 (Editorial changes); 01 August 1979 (Certificate of analysis issued); 17 November 1969 (Provisional certificate reprinted); 17 August 1962 (Provisional certificate revised); 29 March 1961 (Provisional certificate issue date).</p> |
|---|

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>.