National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 158a

Silicon Bronze
(chip form)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of bronze alloys and materials of similar matrix. It can be used to validate value assignment of a laboratory's in-house reference materials. A unit of SRM 158a consists of one bottle containing approximately 150 g of chips produced by a milling machine.

Certified Mass Fraction Values: Certified values for constituents of SRM 158a are reported in Table 1 as mass fractions of the elements in a bronze alloy matrix [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 taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %, calculated following the ISO/JCGM Guide [3−10].

Table 1. Certified Mass Fraction Values for SRM 158a Silicon Bronze (chip form)

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Aluminum (Al)	0.4580	0.0094
Copper (Cu)	90.93	0.29
Iron (Fe)	1.228	0.015
Lead (Pb)	0.0973	0.0048
Manganese (Mn)	1.112	0.014
Phosphorus (P)	0.0263	0.0028
Silicon (Si)	3.026	0.021
Tin (Sn)	0.960	0.013
Zinc (Zn)	2.076	0.019

Expiration of Certification: The certification of **SRM 158a** is valid indefinitely within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Periodic 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for certification was performed by R.K. Bell, formerly of NIST. Review and revision of values and uncertainty estimates was coordinated by J.R. Sieber of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by A. Possolo of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 August 2018
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Silicon bronze chips may be analyzed in the as-received form. Test methods used to characterize the material used sample quantities of 2 g or more. While the material is believed to be homogeneous at lower quantities, it has not been tested in amounts < 2 g. Before sampling, it is recommended to mix bottle contents by inverting and rotating the bottle by hand for at least one minute. A bottle containing unused material should be recapped immediately and stored at room temperature away from light.

To use the uncertainty estimates given in this certificate, divide the expanded uncertainty by a coverage factor of $k = 2$ to obtain the combined standard uncertainty. The effective degrees of freedom of the combined standard uncertainty are ≥ 60.

PREPARATION AND ANALYSIS[(1)]

The material was sieved, blended and bottled at NIST. Homogeneity testing of samples from the final lot was performed J.E. Cumbo, Airco Alloys (Niagara Falls, NY) and J.C. Cline, Interlake, Inc. (Beverly, OH). Analyses for certification were performed using the test methods listed in Table 2.

Analyses leading to the certification of this SRM were performed at NIST by R.K. Bell and E.E. Maczkowske, formerly of NIST. Analytical determinations were also performed by A.W. Young, Bridgeport Brass (Bridgeport, CT); E.L. Smith, H.J. Smith, R.C. Burnham, and A.B. Feest, Chase Brass and Copper Co. (Waterbury, CT); O.P. Case, G.A. Reihl, W.T. Taras, and K.M. O'Brien, The American Brass Co. (Waterbury, CT); and K.H. Storks, E. Bloom, Jr., and E.K. Jaycox, Bell Telephone Laboratories (Murray Hill, NJ).

Table 2. Test Methods Employed in the Certification of SRM 158a Silicon Bronze (chip form)

Element	Test Methods Used at NIST and Collaborating Laboratories
Aluminum	• Cu in a 2 g sample removed by electrolysis of a HNO_3-HF solution. Mercury cathode separation then made in sulfate solution followed by H_2S separation in 0.01 *N* acidity. Sulfides filtered off, and MnO_2 removed with persulfate in dilute acid solution. Al precipitated twice with NH_4OH and ignited to Al_2O_3.; • Mercury cathode-Al_2O_3 method (see ASTM International method E54-49); • Mercury cathode 8-hydroxyquinoline gravimetric method; • Cu removed by electrolysis of a HNO_3-HF solution. Si in the electrolyte removed with H_2SO_4-HF treatment, Sn with HBr, and Mn with $(NH_4)_2S_2O_8$. Filtrate electrolyzed in a mercury cathode. Al precipitated with NH_4OH, ignited to Al_2O_3, and corrected for P.
Copper	• 5.0 g sample dissolved in 40 mL HNO_3 (1+1). Solution digested on steam bath overnight, filtered and the precipitate washed with hot HNO_3 (1+99). Precipitate treated with HNO_3-$HClO_4$-HF-HBr and residual solution combined with first filtrate. Two drops 0.1 *N* HCl added, solution diluted to 300 mL and electrolyzed overnight, using a current density of 0.5 A/dm^2. Residual Cu and Pb in electrolyte precipitated with H_2S and determined by electrolysis.; • ASTM International method E36-45; • Direct electrolysis of a 2 g sample in HNO_3-HF solution containing small amount of added Pb; • Direct electrolysis of a 2 g sample in HNO_3-HF solution.
Iron	• $SnCl_2$-$K_2Cr_2O_7$ method; • Fe titrated with $TiCl_3$; • Fe reduced in a silver redactor and titrated with $Ce(SO_4)_2$.
Lead	• Weighed as PbO_2; • Dithizone-photometric method; • Pb separated as $PbSO_4$ and weighed as $PbCrO_4$.

[(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 2. Test Methods Employed in the Certification of SRM 158a Silicon Bronze (chip form)

Element	Test Methods Used at NIST and Collaborating Laboratories
Manganese	• Persulfate-arsenite method with potentiometric titration; • KIO_4 photometric method (see ASTM International method E62-56); • $NaBiO_3$ method.
Phosphorus	• Phosphomolybdenum blue method; • Molybdivanadophosphoric acid method.
Silicon	• Double dehydration with $HClO_4$, and a Na_2CO_3 fusion followed by double dehydration with H_2SO_4; • Photometric method; • H_2SO_4 dehydration (see ASTM International method E54-49); • Double dehydration with H_2SO_4, and a Na_2CO_3 fusion followed by double dehydration with H_2SO_4; • Molybdisilicic acid-photometric method; • $HClO_4$ dehydration.
Tin	• Sn reduced with Pb and titrated with KIO_3 standardized with high-purity Sn; • Sn reduced with sodium hypophosphite and titrated with iodine; • Sn reduced with Al and titrated with KIO_3 standardized with high-purity Sn; • Sn reduced with Pb and titrated with KIO3 (see ASTM International method 54-49).
Zinc	• Cu removed by electrolysis and electrolyte evaporated to fumes of H_2SO_4. Solution treated with an excess of NaOH and filtered. Zn determined in filtrate by electrolysis.; • $ZnHg(CNS)_4$ method; • Zn extracted as thiocyanate and titrated with sodium ethylenediaminetetraacetate; • Zn in the electrolyte from the Cu determination precipitated twice with H_2S in formic acid solution and ignited to ZnO.

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 158a.

Information Mass Fraction Values: Information values for constituents in SRM 158a are reported as mass fractions in Table 3. An information value is 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]. Information values cannot be used to establish metrological traceability.

Table 3. Information Mass Fraction Values for SRM 158a Silicon Bronze (chip form)

Constituent	Mass Fraction (%)
Chromium (Cr)	0.001
Nickel (Ni)	0.001
Silver (Ag)	0.001

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and a laboratory's in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

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 https://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Aug 2018).

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Certificate Revision History: 23 August 2018 (Revised values and uncertainties for certified constituents; update of the certificate to current NIST standards; editorial changes); 8 August 1961 (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 https://www.nist.gov/srm.