

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1258-I

Aluminum Alloy 6011 (Modified) (disk form)

This Standard Reference Material (SRM) is an aluminum alloy intended primarily for evaluation of methods for analysis of elements in aluminum alloys of similar composition. It can be used to validate value assignment of inhouse reference materials. A unit of SRM 1258-I consists of a wrought and annealed disk that is 35 mm diameter and 19 mm thick.

Certified Mass Fraction Values: Certified values for constituents in SRM 1258-I are provided in Table 1 as mass fractions of the total amounts of the elements in an aluminum 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 derived SI unit of mass fraction, expressed in %. The uncertainty listed with each value is an expanded uncertainty calculated by combining a between-method variance [3] with a pooled, within-method variance following the ISO/JCGM Guide [4]. The expanded uncertainty is expressed at the 95 % confidence level, using the coverage factor, k.

Table 1. Certified Values for SRM 1258-I

| Elements | Mass Fraction (%) | Expanded Uncertainty (%) | Coverage Factor k |
|----------------|-------------------|--------------------------|-------------------|
| Silicon (Si) | 0.80 | 0.04 | 2.4 |
| Iron (Fe) | 0.080 | 0.003 | 2.6 |
| Copper (Cu) | 0.848 | 0.010 | 2.4 |
| Manganese (Mn) | 0.481 | 0.010 | 2.1 |
| Magnesium (Mg) | 1.00 | 0.03 | 2.3 |
| Zinc (Zn) | 1.03 | 0.02 | 2.4 |

Expiration of Certification: The certification of **SRM 1258-I** 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 Storage, Handling and Use"). 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 or register online) will facilitate notification.

Coordination of technical measurements leading to certification was performed under the direction of J.I. Schultz of the ASTM/NIST Research Associate Program. Additional technical and analytical support was provided by J.R. Sieber of the NIST Chemical Sciences Division.

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

Carlos A. Gonzalez, Chief Chemical Sciences Division

Steven J. Choquette, Director Office of Reference Materials

Gaithersburg, MD 20899 Certificate Issue Date: 05 October 2018

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

INSTRUCTIONS FOR STORAGE, HANDLING AND USE

The test surface is the side opposite to the labeled surface, which has 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.

NOTICE AND WARNING TO USERS

CAUTION: Accurate determinations of copper (Cu), magnesium (Mg), and zinc (Zn) in the 6000 and 7000 series aluminum-base alloys by optical emission test methods may be sensitive to the metallurgical condition of the samples. Significantly large differences can be expected when wrought samples are compared to cast samples; however, small differences also may occur among wrought samples depending on the temperature and amount of working and annealing and/or tempering operations. Differences among cast samples may occur depending on the rate and type of solidification from the molten metal. This SRM is in the worked and fully annealed condition and is directly applicable to the analyses of samples in this same metallurgical condition.

X-ray fluorescence methods for the 6000 and 7000 series aluminum-base alloys are normally not sensitive to the metallurgical condition of the samples.

PREPARATION, TESTING, AND ANALYSIS(1)

SRM 1258-I was prepared under contract with NIST by the Aluminum Company of America (Alcoa Center, PA), coordinated by D.J. Levin. The material was melted to composition and continuously cast into three ingots, 13.7 cm in diameter and about 330 cm long. After cropping the ends and obtaining slices for homogeneity testing, each ingot was cut into four cylinders, 61.0 cm long; these were scalped to 11.6 cm in diameter. The cylinders were center-bored (3.7 cm diameter) and then sawed lengthwise into three pieces (one half-round and two quarter-round sections).

Selected half-round sections were upset-forged, forge-rolled, and annealed at the Naval Research Laboratory (Washington, DC), by T. Kissilnitkie. The material was processed to finished specimens at NIST.

Homogeneity testing was performed on both cast and finished specimens using optical emission spectrometry at the Aluminum Company of America by D.J. Levin, at the Reynolds Research Laboratory (Richmond, VA) by N. Christ, and at NIST by J.A. Norris.

Specimens representative of the accepted lot of material were chipped and blended to form a composite sample and portions were distributed for chemical analyses leading to certification.

Analytical measurements were performed by R.W. Burke, T.J. Brady, E.R. Deardorff, B.I. Diamondstone, M.S. Epstein, S. Hanamura, J.D. Messman, T.C. Rains, S.A. Wicks of the National Institute of Standards and Technology (Gaithersburg, MD); R.C. Obbink of Aluminum Company of America, Alcoa Technical Center; L. Girolami of Aluminum Company of Canada, Ltd. (Arvida, Quebec, Canada); H.J. Seim, R.C. Calkins, G.M. Calkins, R.C. Kinne, J.R. Skarset of Kaiser Aluminum and Chemical Corporation (Pleasanton, CA); M.E. Reed of Kaiser Aluminum and Chemical Corporation (Ravenswood, WV); and W.E. Pilgrim of Reynolds Aluminum, Reynolds Metals Company (Richmond, VA).

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 1258-I.

Reference Mass Fraction Values: Reference values for constituents of SRM 1258-I are provided in Table 2 as mass fractions of the total element in an aluminum matrix. A reference value is a non-certified value that is the present best estimate of the true value; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty 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 2]. The measurand is the mass fraction value

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⁽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.

determined using the methods indicated in Table 4. The reference values are metrologically traceable to the derived SI unit of mass fraction, expressed in %.

Table 2. Reference Values for SRM 1258-I

| Elements | Mass Fraction (%) | Expanded Uncertainty (%) | Coverage Factor k |
|---------------|-------------------|--------------------------|-------------------|
| Chromium (Cr) | 0.0011 | 0.0002 | 2.0 |
| Nickel (Ni) | 0.0006 | 0.0003 | 2.0 |
| Titanium (Ti) | 0.040 | 0.003 | 2.6 |

Information Mass Fraction Values: Information mass fraction values for constituents of SRM 1258-I are listed 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 Values for SRM 1258-I

| Elements | Mass Fraction (%) |
|----------------|-------------------|
| Beryllium (Be) | <0.0001 |
| Gallium (Ga) | 0.011 |

Table 4. Analytical Methods Used for SRM 1258-I

| Elements | Methods ^(a) | |
|----------|------------------------|--|
| Si | 1, 2 | |
| Fe | 1, 2, 3, 4 | |
| Cu | 1, 2, 5, 6 | |
| Mn | 1, 2, 7, 8 | |
| Mg | 1, 2, 10, 11 | |
| Zn | 1, 2, 10 | |
| Cr | 1, 2, 9 | |
| Ni | 1, 2, 13 | |
| Ti | 1, 2 | |
| Be | 12 | |

(a) Key to Methods in Table 4

- 1. Atomic absorption spectrophotometry (AAS)
- 2. X-ray fluorescence spectrometry (XRF)
- 3. 1,10 Phenanthroline spectrophotometric method
- 4. Bathophenanthroline disulfonic acid and solution spectrometry
- 5. Oxalyl dihydroxide spectrophotometric method
- 6. Zinc dibenzyldithiocarbamate and solution spectrometry
- 7. Periodate spectrophotometric method
- 8. Ammonium peroxydisulfate oxidation and solution spectrometry
- 9. Diphenylcarbazide spectrophotometric method
- 10. EDTA titration
- 11. CDTA titration
- 12. Fluorimetric with morin after extraction with acetylacetone-chloroform
- 13. 2,3 Quinoxaline dithiol and solution spectrometry

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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/index.cfm (accessed Oct 2018).
- [2] 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 260-136 (2000); available at https://www.nist.gov/srm/upload/SP260-136.pdf (accessed Oct 2018).
- [3] Hahn, G.J.; Meeker, W.Q; Statistical Intervals: A Guide for Practitioners; John Wiley & Sons, Inc.: New York (1991).
- [4] 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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2018); 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 https://www.nist.gov/pml/pubs/index.cfm (accessed Oct 2018).

Certificate Revision History: 05 October 2018 (Title update; k values added; editorial changes); 23 September 2014 (Extension of the certification period; editorial changes); 04 June 2004 (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 https://www.nist.gov/srm.

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