

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1258

Aluminum Alloy 6011 (Modified)

(In cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in optical emission and X-ray spectrometric methods of analysis. A unit of SRM 1258 consists of wrought and annealed disks, 35 mm (1 3/8 in) diameter and 19 mm (3/4 in) thick.

The certified values for five elements in SRM 1258 are listed in Table 1. Reference values for two elements are listed in Table 2. Information values for four elements are listed in Table 3. For all elements, 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]. Table 4 summarizes the analytical methods used by NIST and cooperating laboratories for characterization of the composition of this SRM.

Certified Values and Uncertainties: 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. The certified value listed for a constituent is the present best estimate of the true value based on the results of analyses performed at NIST and cooperating laboratories using test methods listed in Table 4. The uncertainty listed with each value is an expanded uncertainty calculated by combining a between-method variance [4] with a pooled, within-method variance following the ISO/NIST Guides [3]. The expanded uncertainty is expressed at approximately the 95 % confidence level, using a coverage factor, k = 2.

Reference Values: The reference values for the constituents of SRM 1258 are given 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 and are provided with associated uncertainties that are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability (No attempt was made to derive exact statistical measures of imprecision.)

Information Values: The information values for the constituents of SRM 1258 are given in Table 3. These are non-certified values with no uncertainty reported because there is insufficient information with which to make the appropriate statistical assessments.

Expiration of Certification: The certification of this SRM is valid until **01 August 2024**, 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.

Willie E. May, Chief Analytical Chemistry Division

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

Gaithersburg, MD 20899 Certificate Revision Date: 04 June 2004 See Certificate Revision History on Last Page The overall coordination of technical measurements leading to the certification of this SRM was performed under the direction of J.I. Schultz, Research Associate, ASTM/NIST Research Associate Program.

Additional technical and analytical support was provided by J.R. Sieber of the NIST Analytical Chemistry Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR 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.

Table 1. Certified Values for SRM 1258	Table 1.	Certified	Values fo	or SRM 1258
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Constituent	Mass Fraction (%)
Si	0.78 ± 0.04
Fe	0.079 ± 0.003
Cu	0.84 ± 0.01
Mn	0.48 ± 0.01
Zn	1.03 ± 0.02

Table 2. Reference Values for SRM 1258

Constituent	Mass Fraction (%)
Cr Ni	$\begin{array}{rrrr} 0.0011 \ \pm \ 0.0002 \\ 0.0006 \ \pm \ 0.0003 \end{array}$

Table 3. Information Values for SRM 1258

Constituent	Mass Fraction (%)
Be	<0.0001
Ga	0.011
Mg	1
Ti	0.04

Table 4. Methods of Analysis for SRM 1258

Element	Methods
Si	Atomic absorption spectrophotometry
Fe	Atomic absorption spectrophotometry 1,10 Phenanthroline spectrophotometric method Bathophenanthroline disulfonic acid and solution spectrometry
Cu	Atomic absorption spectrophotometry Oxalyl dihydroxide spectrophotometric method Zinc dibenzyldithiocarbamate and solution spectrometry
Mn	Atomic absorption spectrophotometry Periodate spectrophotometric method Ammonium peroxydisulfate oxidation and solution spectrometry
Cr	Atomic absorption spectrophotometry Diphenylcarbazide spectrophotometric method
Zn	Atomic absorption spectrophotometry EDTA titration
Mg	Atomic absorption spectrophotometry CDTA titration EDTA titration
Be	Fluorimetric with morin after extraction with acetylacetone-chloroform
Ti	Atomic absorption spectrophotometry
Ni	Atomic absorption spectrophotometry 2,3 Quinoxaline dithiol and solution spectrometry

NOTICE AND WARNING TO USERS

CAUTION: Accurate determinations of copper (Cu), magnesium (Mg), and zinc (Zn) in the 6000 and 7000 aluminum-base alloys by standard ASTM optical emission test methods are extremely 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 aluminum-base alloys are normally not sensitive to the metallurgical condition of the samples.

PLANNING, PREPARATION, TESTING, AND ANALYSIS¹

SRM 1258 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 (5 3/8 in) in diameter and about 330 cm (130 in) long. After cropping the ends and obtaining slices for homogeneity testing, each ingot was cut into four cylinders, 61.0 cm (24 in) long; these were scalped to 11.6 cm (4 9/16 in) 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, Alcoa Center, PA.; D.J. Levin at the Reynolds Research Laboratory, Richmond, VA.; N. Christ and J.A. Norris at NIST.

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.

Cooperative analyses for certification were performed in the following laboratories:

R.C. Obbink; Aluminum Company of America, Alcoa Technical Center; Alcoa Center, PA

- L. Girolami; Aluminum Company of Canada, Ltd.; Arvida, Quebec, Canada
- H.J. Seim, R.C. Calkins, G.M. Calkins, R.C. Kinne, J.R. Skarset; Kaiser Aluminum and Chemical Corporation; Pleasanton, CA
- M.E. Reed; Kaiser Aluminum and Chemical Corporation; Ravenswood, WV
- 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; National Institute of Standards and Technology; Gaithersburg, MD
- W.E. Pilgrim; Reynolds Aluminum, Reynolds Metals Company; Richmond, VA

REFERENCES

- [1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Spec. Pub. 811, U.S. Government Printing Office: Washington, DC (1995).
- [2] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Spec. Pub. 260-136, U.S. Government Printing Office: Washington, DC, p. 16 (2000).
- [3] ISO; Guide 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 <u>http://physics.nist.gov/Pubs/</u>.
- [4] Hahn, G.J.; Meeker, W.Q; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

Certificate Revision History: 04 June 2004 (The certificate for SRM 1258 has been revised to reflect the decertification of Mg, Cr, Ni, and Be. Mg and Be have been reduced to information values. Cr and Ni have been reduced to reference values. Uncertainty estimates for certified values have been recalculated to conform to the ISO/NIST Guides and to account for the findings of new homogeneity testing by X-ray fluorescence spectrometry. Editorial changes were also made.); 03 May 1978 (Original certification 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, email srminfo@nist.gov; or via the Internet at <u>http://www.nist.gov/srm</u>.

¹Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.