National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 699

Alumina (Reduction Grade)

(In Cooperation with ASTM International and the Aluminum Association)

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical methods of analysis and in calibration of instrumental analyses. Each unit of SRM 699 consists of 60 g alumina powder of which 95 % passes a 74 µm (No. 200) sieve.

Certified values for 13 constituents and loss on ignition for SRM 699a are listed in Table 1. Information values for five constituents are listed in Table 2. The assigned values are based on methods that determine the mass fraction of the element in the material. Most values were calculated as the oxide compounds to reflect practices used by the aluminum industry.

Certified Values: 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. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using test methods listed in Table 3. With the exceptions of Be and SiO_2, the estimated uncertainties are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.5 g or more. No effort was made to derive exact statistical measures of imprecision because several methods were involved in the determination.

The certified value for Be is based on results of analyses at NIST and Alcoa Technical Center on behalf of the Aluminum Association. The uncertainty listed for Be is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [1] with a pooled, within-method variance following the ISO and NIST Guides [2].

The certified value listed for SiO_2 is based on the results of analyses performed at NIST, the cooperating laboratories of the Alcoa World Alumina (AWA) Technology Advancement Team (TAT), and the American Society for Testing and Materials (ASTM) cooperative analytical program. The uncertainty listed for SiO_2 is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [1] with a pooled, within-method variance following the ISO Guide and NIST Guides [2].

Information Values: 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.

Expiration of Certification: The certification of this SRM is valid indefinitely, 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.

Coordination of the technical measurements for certification of Be and SiO_2 was accomplished under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 29 November 2006
See Certificate Revision History on Last Page

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

Coordination of the AWA TAT cooperating analyses for SiO_2 was performed by J. Elbicki of Alcoa Technical Center [1], Alcoa Center, PA.

The overall coordination of the technical measurements provided by the ASTM cooperative analytical program was performed under the direction of J.I. Shultz, Research Associate (retired), ASTM/NIST Research Associate Program.

The overall direction and coordination of the statistical consultation leading to the certification of SiO_2 were performed by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

This material is hygroscopic and must be handled to minimize adsorption of moisture. When not in use, the bottle should be kept tightly capped and stored in a closed container with a suitable desiccant. To obtain certified values, analyses must be performed on sub-samples which have been dried for 2 h at 300 °C then cooled in a desiccator before weighing for analysis.

Table 1. Certified Values in SRM 699

Constituent*	Mass Fraction (%)		
Calcium Oxide, CaO [(d,h,i,j)]	0.036	±	0.002
Chromic Oxide, Cr_2O_3 [(f,i,j)]	0.0002	±	0.0001
Ferric Oxide, Fe_2O_3 (Total iron as) [(b,g,i,j)]	0.013	±	0.001
Gallium Oxide, Ga_2O_3 [(d,h,i,j)]	0.010	±	0.002
Lithium Monoxide, Li_2O [(h,i,j)]	0.002	±	0.001
Magnesium Oxide, MgO [(h,i,j,m)]	0.0006	±	0.0002
Manganous Oxide, MnO [(d,i,j,l)]	0.0005	±	0.0001
Phosphorus Pentoxide, P_2O_5 [(e)]	0.0002	±	0.0001
Silicon Dioxide, SiO_2 [(a,d,i,j,o)]	0.0120	±	0.0008
Sodium Monoxide, Na_2O [(c,h,i,j)]	0.59	±	0.01
Vanadium Pentoxide, V_2O_5 [(d,i,j,l)]	0.0005	±	0.0002
Zinc Oxide, ZnO [(d,h,i,j)]	0.013	±	0.002
Loss on Ignition, (LOI) (1000 °C) [(n)]	0.69	±	0.08
Loss on Ignition, (LOI) (1200 °C) [(n)]	0.86	±	0.08

Constituent*	Mass Fraction (mg/kg)		
Beryllium, Be [(k,p)]	2.81	±	0.14

*Superscripts following the constituent name indicate test methods listed in Table 3.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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. Information Values for SRM 699

Constituent	Mass Fraction (%)
Boron Oxide, B_2O_3	(< 0.001)
Zirconium Dioxide, ZrO_2	(0.0002)
Cupric Oxide, CuO	(0.0005)
Potassium Monoxide, K_2O	(0.005)
Titanium Dioxide, TiO_2	(0.001)

Table 3. Test Methods Employed at NIST and the Cooperating Laboratories

[a] Silicomolybdate photometric
[b] 2,2′ dipyridyl photometric
[c] Flame emission spectrometry
[d] X-ray fluorescence spectrometry
[e] Molybdenum blue photometric
[f] Diphenylcarbazide photometric
[g] Ortho-phenanthroline photometric
[h] Atomic absorption spectrometry
[i] Optical emission spectroscopy
[j] Inductively coupled plasma optical emission spectrometry (ICPOES)
[k] ICP-OES after Parr bomb digestion
[1] N-benzoyl-N-henylhydroxylamine photometric
[m] DC plasma emission spectroscopy
[n] The determination of LOI is based on methods given in ISO documents R803 and R806 (August 1968).
[o] DC arc
[p] ICP-OES with internal standard after Carius tube digestion

PLANNING, PREPARATION, TESTING, AND ANALYSIS

The material for this standard was provided by the Reynolds Aluminum Company, Bauxite, AR, through the courtesy of J.B. Ezell, Jr. It was produced by the Bayer process using Jamaican bauxite as the raw material.

The crushing, grinding, sieving, and homogeneity testing were performed by J.B. Ezell, Jr., Reynolds Aluminum Company. The material variability was determined to be within the imprecision of the methods used for homogeneity testing.

Homogeneity for Be was assessed during the quantitative analyses carried out at NIST by L.L. Yu, L.J. Wood, and W.R. Kelly of the NIST Analytical Chemistry Division.

ASTM cooperative analyses for certification were performed in the following laboratories:

Aluminum Company of America, Alcoa Center, PA; G.F. Leaz
Alcan International, Ltd., Arvida Laboratories, Jonquiere, Quebec, Canada; E. Van Dalen; L. Lepine
Kaiser Aluminum and Chemical Corporation, Pleasanton, CA; H.J. Seim; J.M. Winkler; R.C. Kinne; D.F.G. Marten; R.C. Calkins
Ormet Corporation, Burnside, LA; A.D. Lafleur
Reynolds Aluminum Company, Bauxite, AR; J.B. Ezell, Jr.

AWA TAT cooperative analyses for SiO_2 were performed in the following laboratories:

Alcan International Ltd., Jonquiere, Quebec, Canada; S. Pare; F. Picard
Alcoa of Australia Ltd., R&D, Kwinana, Western Australia, Australia; C. Dobbs
Alcoa Point Comfort Operations, Calhoun County TX; B. Chambless
Alcoa/Clarendon Refinery Works, Jamaica, West Indies; M. Wilson
Alcoa, Pocos de Caldas, MG, Brazil; E.L. Carvalho; P.R. Lazarin
Alcoa Paranam Operations/Suriname, Miami, FL; M. Boetius
Alcoa Alumar, Sao Luis, MA, Brazil; J.D. Costa
Alcoa Technical Center, Alcoa Center, PA, M. Ruschak; J. Elbicki
Kaiser Aluminum and Chemical Corporation, Gramercy, LA, J. Angier; D. Kirkpatrick
Sherwin Alumina Company, Corpus Christi, TX; C. Franz; D. Nutt

Cooperative analyses for Be were performed in the following laboratories:
Alcoa Technical Center, Alcoa Center, PA, M. Ruschak
Ultratrace Laboratories, Perth, Western Australia

REFERENCES

[1] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H-k.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, No. 4, p. 571 (2000).

[2] 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 http://physics.nist.gov/Pubs/.

Certificate Revision History: 29 November 2006 (Update Be value to certified status); 17 October 2002 (Updated SiO_2 certified value); 03 December 1993 (Editorial revision); 12 August 1981 (Original certificate 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; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.