



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 699

Alumina (Reduction Grade)

(In Cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is intended for use in checking chemical methods of analysis and in calibration of instrumental analyses. The certified values are based on methods that determine the mass fraction of the element in the material, which has been dried for 2 h at 300 °C. The values were calculated as the oxide compounds to reflect practices used by the aluminum industry. Each unit consists of 60 g alumina powder of which 95 % is less than 74 µm (No. 200).

The certified values listed in Table 1 are the present base estimates of the "true" values. The certified value listed for SiO₂ is based on the results of analyses performed at National Institute of Standards and Technology (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 SiO₂ 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 [2]. The values obtained by individual laboratories and methods used for analysis are given in Table 2.

The certified values for the other constituents listed in Table 1 are based on results of the cooperative analytical program with ASTM. 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 attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination. The values obtained by individual ASTM cooperating laboratories, and the methods used for analysis, are given in Table 3.

Table 1. Certified Values in SRM 699

Constituent	Mass Fraction (%)		
Calcium Oxide, CaO	0.036	±	0.002
Chromic Oxide, Cr ₂ O ₃	0.0002	±	0.0001
Ferric Oxide, Fe ₂ O ₃ (Total iron as)	0.013	±	0.001
Gallium Oxide, Ga ₂ O ₃	0.010	±	0.002
Lithium Monoxide, Li ₂ O	0.002	±	0.001
Magnesium Oxide, MgO	0.0006	±	0.0002
Manganous Oxide, MnO	0.0005	±	0.0001
Phosphorus Pentoxide, P ₂ O ₅	0.0002	±	0.0001
Silicon Dioxide, SiO ₂	0.0120	±	0.0008
Sodium Monoxide, Na ₂ O	0.59	±	0.01
Vanadium Pentoxide, V ₂ O ₅	0.0005	±	0.0002
Zinc Oxide, ZnO	0.013	±	0.002
Loss on Ignition, (LOI) (1000 °C)	0.69	±	0.08
(1200 °C)	0.86	±	0.08

Revision of this certificate was coordinated through the NIST Standard Reference Materials Group by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 17 October 2002
See Certificate Revision History on Last Page
SRM 699

John Rumble, Jr., Chief
Measurement Services Division

The support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Group by R.E. Michaelis.

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, ASTM/NIST Research Associate Program.

The overall direction and coordination of the technical measurements leading to the certification of SiO₂ were performed by J.R. Sieber of the NIST Analytical Chemistry Division.

The overall direction and coordination of the statistical consultation leading to the certification of SiO₂ were performed by S.D. Leigh of the NIST Statistical Engineering Division.

Coordination of the AWA TAT cooperating analyses for SiO₂ was performed by J. Elbicki of Alcoa Technical Center, Alcoa Center, PA.

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 subsamples which have been dried for 2 h at 300 °C and then cooled in a desiccator before weighing for analysis.

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.

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 SiO₂ 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 Alummar, 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

Table 2. Summary of Results for Silicon Dioxide, SiO₂
(values in mass fraction, %)

ASTM Cooperative Analytical Program Results					
Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Methods
0.016	0.015	0.010 0.013	0.015	0.014	a,i,j

NIST Standard Additions (Borate Fusion) X-ray Fluorescence Result: 0.0115

AWA TAT Collaborating Laboratory Results										
Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10	Methods
0.012	0.012	0.011	0.013	0.011	0.011	0.012	0.011	0.0125	0.012	a,d,j,o

Table 3. ASTM Cooperative Analytical Program Laboratory Results
(values in mass fraction, %)

Constituent	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Methods
Calcium Oxide, CaO	0.035	0.037	0.034	0.039	0.037	d,h,i,j,k
Chromic Oxide, Cr ₂ O ₃	0.0003	0.0002 0.0003	0.0002	----	----	f,i,j
Ferric Oxide, Fe ₂ O ₃ (Total iron as)	0.012	0.013	0.013	0.014	0.012	b,g,i,j,k
Gallium Oxide, Ga ₂ O ₃	0.014	0.011	0.009 0.010	0.010	----	d,i,h,j
Lithium Monoxide, Li ₂ O	----	0.002	0.003 0.002	0.002	----	h,i,j
Magnesium Oxide, MgO	----	0.0008	0.0005 0.0006	----	< 0.001	h,i,j,m
Manganese Oxide, MnO	0.0005	0.0007	0.0004 0.0006	0.0004	----	d,i,j,l
Phosphorus Pentoxide, P ₂ O ₅	0.0001	< 0.001	0.0004	0.0001	0.0002	e
Sodium Monoxide, Na ₂ O	0.60	0.58	0.57 0.59	0.60	----	c,h,i,j
Vanadium Pentoxide, V ₂ O ₅	0.0006	0.0008	0.0005 0.0006	0.0003	----	d,i,j,l
Zinc Oxide, ZnO	0.016	0.012	0.015 0.013	0.013	0.012	d,h,i,j
Loss on Ignition (LOI) (1000 °C)	0.62	0.76	0.72	0.85	0.67	
(1200 °C)	0.90	0.92	0.80	0.97	0.80	

--- not reported

Methods

- ^a Silicomolybdate photometric
- ^b 2,2' dipyridyl photometric
- ^c Flame emission spectrometry
- ^d X-ray spectroscopy
- ^e Molybdenum blue photometric
- ^f Diphenylcarbazide photometric
- ^g Ortho-phenanthroline photometric
- ^h Atomic absorption spectrometry
- ⁱ Optical emission spectroscopy

- ^j Emission spectroscopy (ICP)
- ^k Same value obtained by emission spectroscopy (ICP)
- ^l Same value obtained by 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

Elements other than those certified may be present in this material as indicated below. These concentrations are not certified, but are given as additional information on the composition of the SRM.

Table 4. Information Values for SRM 699

Constituent	Mass Fraction (%)
Beryllium Oxide, BeO	(0.0008)
Boron Oxide, B ₂ O ₃	(< 0.001)
Zirconium Dioxide, ZrO ₂	(0.0002)
Cupric Oxide, CuO	(0.0005)
Potassium Monoxide, K ₂ O	(0.005)
Titanium Dioxide, TiO ₂	(0.001)

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] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO, 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: 17 October 2002 (Updated SiO₂ 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 Group at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.