National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 613

Trace Elements in Glass

This Standard Reference Material (SRM) is intended to facilitate development of chemical methods of analysis for trace elements in glass. The nominal mass fractions of 61 elements added to the glass matrix are in the range of 10 mg/kg to 80 mg/kg. A unit of SRM 613 consists of four wafers, sliced to 1 mm thickness from a hand-pulled rod. The wafers are of oval to circular cross-section with nominal diameter of 12 mm to 14 mm.

Certified Mass Fraction Values: Certified values for 15 elements of SRM 613 are reported in Table 1 as mass fractions [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 investigated or taken into account [2].

Reference Values: Reference values for four elements are reported in Table 2 as mass fractions [1]. The normalized isotope atom ratio for strontium is also included. Reference values are non-certified values that are the best estimates of the true values based on available data; however, the values do not meet the NIST criteria for certification [2] and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Values: Information values for 13 elements are reported in Table 3 as mass fractions. Also reported is the isotope atom fraction of uranium-235. An information value is considered a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

Expiration of Certification: The certification of **SRM 613** 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 Handling, Storage, and Use"). Accordingly, 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 material over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of technical measurements for certification of SRM 613 was performed by W.R. Shields of what is now the NIST Analytical Chemistry Division. Coordination of technical measurements for updates of values was performed by W.C. Davis and J.R. Sieber of the NIST Analytical Chemistry Division.

Analyses for the original characterization were performed by R.W. Burke, T.E. Gills, E.J. Maienthal, L.W. Masters, T.C. Rains, and B.A. Thompson of what is now the NIST Analytical Chemistry Division. Analyses for the initial update of values were performed by E.L. Garner, J.W. Gramlich, L.A. Machlan, J.R. Moody, L.J. Moore, T.J. Murphy, P.J. Paulsen, and K.M. Sappenfield of what is now the NIST Analytical Chemistry Division. Analyses for the current update of values were performed by W.C. Davis and J.R. Sieber of the NIST Analytical Chemistry Division.

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Certificate Revision History on Last Page

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Analyses for the original characterization were performed by the following collaborating laboratories and analysts: United States Geological Survey, Denver, CO, C. Hedge and M. Tatsumoto; Australian National University, Canberra, ACT, Australia, W. Compston; and University of Ghent, Ghent, Belgium, F. Bellemans.

Statistical consultation for this SRM was provided by S.D. Leigh, A.L. Pintar, and A.M. Possolo of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To relate analytical determinations to certified values, a minimum sample quantity of 250 mg is recommended (see "Preparation and Analysis"). Each wafer surface should be cleaned before use. To prepare a wafer for analysis, wipe it clean with ethanol, then give it a mild surface cleaning (not etch) in dilute (1:10) HNO_3. The acid wash is recommended to remove potential copper contamination from cutting with a copper-bonded diamond wheel. The material should be stored in its original container in a cool, dry location.

Preparation and Analysis: Sixty-one trace elements were added to the glass support matrix, which has a nominal composition of 72 % SiO_2, 14 % Na_2O, 12 % CaO, and 2 % Al_2O_3 (mass fractions). A list of 29 elements that were added but for which no values have been assigned is provided in Table 4. The material was prepared in rod form and then sliced into wafers. Considerable effort was invested in the manufacturing of the material to ensure sufficient homogeneity to yield a ≤2 % relative repeatability of measurement when an entire wafer is used. Spatial heterogeneity exists within each wafer, which may adversely affect repeatability of microanalysis techniques. Values were assigned using the analytical methods listed in Table 5.

Certified Value Assignment: For antimony, arsenic, barium, cadmium, chromium, manganese, and selenium, the certified value and uncertainty were determined during recertification. Their certified values are weighted means of the mass fractions determined using the methods listed in Table 5. The form of the weights was introduced in reference 3 and described further in reference 4. Their expanded uncertainties are the half widths of symmetric 95% parametric bootstrap confidence intervals [5] with expansion factor $k = 1.96$, and are consistent with the ISO Guide [6].

The certified values for iron, lead, nickel, rubidium, silver, strontium, thorium, and uranium are as assigned in the original certificate of this material. These values have not been updated, and are qualified with the original statement of measurement uncertainty, which is equal to the entire range of values measured for individual samples or to the 95 % confidence interval, whichever is greater. The user can treat such uncertainty assessments as half widths of 95 % confidence intervals based on Gaussian, Type A evaluations using no more than five measured values each.

Table 1. Certified Mass Fraction Values for SRM 613

Constituent	Mass Fraction (mg/kg)
Antimony	34.9 ± 2.2
Arsenic	37.4 ± 2.2
Barium	38.6 ± 2.6
Cadmium	29.9 ± 4.2
Chromium	35.0 ± 3.3
Iron	51 ± 2
Lead	38.57 ± 0.2
Manganese	37.7 ± 3.8
Nickel	38.8 ± 0.2
Rubidium	31.4 ± 0.4
Selenium	16.1 ± 1.6
Silver	22.0 ± 0.3
Strontium	78.4 ± 0.2
Thorium	37.79 ± 0.08
Uranium	37.38 ± 0.08

Reference Value Assignment: Each reference value is the equally weighted mean of the results from the methods listed in Table 5. The reference values for cobalt, copper, thallium, titanium, and strontium isotope ratio are as assigned in the original certificate of this material. These values have not been updated, and are qualified with the original statement of measurement uncertainty, which is equal to the entire range of values measured for individual samples or to the 95 % confidence interval, whichever is greater. The user can treat such uncertainty assessments as half widths of 95 % confidence intervals based on Gaussian, Type A evaluations using no more than five measured values each.

Table 2. Reference Values for SRM 613

Constituent	Mass Fraction (mg/kg)
Cobalt	35.5 ± 1.2
Copper	37.7 ± 0.9
Thallium	15.7 ± 0.3
Titanium	50.1 ± 0.8
Ratio of Isotope Atomic Abundances	
$^{87}Sr/^{86}Sr$ (normalized)	0.7089 ± 0.0002

Table 3. Information Values for SRM 613

Constituent	Mass Fraction (mg/kg)
Boron	32
Cerium	39
Dysprosium	35
Erbium	39
Europium	36
Gadolinium	39
Gold	5
Lanthanum	36
Lithium	40
Neodymium	36
Potassium	64
Samarium	39
Ytterbium	42
Ratio of Isotope Atomic Abundances	
$^{235}U/U_{Total}$	2.392×10^{-3}

Table 4. Additional Elements Incorporated in SRM 613 for Which No Values Are Assigned

Beryllium	Holmium	Praseodymium	Thulium
Bismuth	Indium	Rhenium	Tin
Cesium	Lutetium	Scandium	Tungsten
Chlorine	Magnesium	Sulfur	Vanadium
Fluorine	Molybdenum	Tantalum	Yttrium
Gallium	Niobium	Tellurium	Zinc
Germanium	Phosphorus	Terbium	Zirconium
Hafnium			

Table 5. Test Methods Used for Characterization of SRM 613 by NIST and Collaborating Laboratories

Isotope dilution mass spectrometry	Ag, B, Ba, Ce, Cu, Dy, Er, Eu, Gd, K, La, Nd, Ni, Pb, Rb, Sm, Sr, Th, Tl, U, Yb
Nuclear track counting	B
Neutron activation analysis	Ag, Au, Co, Li, Mn
Spectrophotometry	Au, Fe, Mn, Ni
Polarography	Fe, Ti
Flame emission spectrometry	K, Rb, Sr
Inductively-coupled plasma mass spectrometry	As, Ba, Cd, Cr, Sb, Se
Isotope dilution inductively-coupled plasma mass spectrometry	Cr, Se
Standard additions borate fusion X-ray fluorescence spectrometry	Ag, As, Ba, Cd, Cr, Mn, Sb

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 http://www.nist.gov/pml/pubs/index.cfm/ (accessed Apr 2012).

[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed Apr 2012).

[3] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*, Control. Clin. Trials, Vol. 7, pp. 177–188 (1986).

[4] Rukhin, A.L.; *Weighted Mean Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

[5] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).

[6] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Cor*rections); Joint Committee for Guides in Metrology (JCGM) *(2008); available at* http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2012); 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); avail*able at http://www.nist.gov/pml/*pubs/index.cfm (accessed Apr 2012).

Certificate Revision History: 06 April 2012 (New values added for antimony, arsenic, cadmium, chromium, and selenium and revised values for barium and manganese based on new analytical determinations; Changed information values for cobalt, copper, thallium, and titanium to reference values; Added information value for lithium; Changed unit size from 6 wafers to 4 wafers; Editorial changes); 27 January 1992 (Editorial changes); 04 January 1982 (Editorial changes); 08 August 1972 (Revised values for strontium, thorium, and titanium based on new analytical determinations); 05 August 1970 (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 http://www.nist.gov/srm.