NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 612
Trace Elements in Glass
CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended to facilitate development of chemical methods of analysis for trace elements in glass.

Description: A unit of SRM 612 consists of four wafers, sliced to 3 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 and an approximate mass of 1.4 g. The nominal mass fractions of 61 elements added to the glass matrix are in the range of 10 mg/kg to 80 mg/kg.

Certified Values: Certified values for 15 elements of SRM 612 are reported in Table 1 as mass fractions [1]. These values are traceable to International System of Units (SI). 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]. The certified values are weighted means of the mass fractions determined using the methods listed in Table B4. 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/JCGM Guide [6].

Table 1: Certified Mass Fraction Values for SRM 612

Constituent	Mass Fraction[(a)] (mg/kg)		
Antimony (Sb)	34.9	±	2.2
Arsenic (As)	37.4	±	2.2
Barium (Ba)	38.6	±	2.6
Cadmium (Cd)	29.9	±	4.2
Chromium (Cr)	35.0	±	3.3
Iron (Fe)	51	±	2
Lead (Pb)	38.57	±	0.2
Manganese (Mn)	37.7	±	3.8
Nickel (Ni)	38.8	±	0.2
Rubidium (Rb)	31.4	±	0.4
Selenium (Se)	16.1	±	1.6
Silver (Ag)	22.0	±	0.3
Strontium (Sr)	78.4	±	0.2
Thorium (Th)	37.79	±	0.08
Uranium (U)	37.38	±	0.08

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. 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.

Period of Validity: The certification of **SRM 612** is valid indefinitely, within the measurement uncertainty specified. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified. Periodic recertification of this SRM is not required.

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Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Values of potential interest to users and additional information are provided in Appendix B.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Storage: The material should be stored in its original container in a cool, dry location.

Use: To relate analytical determinations to certified values, a minimum sample quantity of 250 mg is recommended (see "Source and Preparation"). 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.

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/special-publication-811 (accessed Apr 2024).

[2] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Apr 2024).

[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*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Apr 2024); 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/nist-technical-note-1297 (accessed Apr 2024).

Certificate Revision History: 03 April 2024 (Revised non-certified value for titanium and updated to value of potential interest to users; updated format; editorial changes); **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)

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system. Non-certified mass fraction values are provided below.

Table A1. Non-Certified Mass Fraction Values for SRM 612

Constituent	Mass Fraction[(a)] mg/kg		
Cobalt (Co)	35.5	±	1.2
Copper (Cu)	37.7	±	0.9
Thallium (Tl)	15.7	±	0.3

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

Table A2. Non-Certified Ratio of Isotopic Abundances for SRM 612[(a)]

$^{87}Sr/^{89}Sr$ (normalized)	0.7089	±	0.0002

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. While the best estimate of measurand mass fraction lies within the interval $x \pm 2u(x)$, neither the purity nor the identity of the calibrants used have been determined by NIST. For purposes of harmonization and process control, the imprecision and homogeneity components of uncertainty can be propagated as $u(x)/x$ relative standard deviations.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Source and Preparation: 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). Mass fraction values are provided in Table B1. The ratio of isotopic abundance is provided in Table B2. A list of 29 elements that were added but for which no values have been assigned is provided in Table B3. 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 $\leq$ 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 B4.

Table B1. Mass Fraction Values of Potential Interest for SRM 612

Constituent	Mass Fraction mg/kg
Boron (B)	32
Cerium (Ce)	39
Dysprosium (Dy)	35
Erbium (Er)	39
Europium (Eu)	36
Gadolinium (Gd)	39
Gold (Au)	5
Lanthanum (La)	36
Lithium (Li)	40
Neodymium (Nd)	36
Potassium (K)	64
Samarium (Sm)	39
Titanium (Ti)	41
Ytterbium (Yb)	42

Table B2. Ratio of Isotopic Abundance Value of Potential Interest for SRM 612

$^{235}U/U_{total}$	2.392×10^{-3}

Table B3. Additional Elements Incorporated in SRM 612 for Which No Values Are Assigned

Beryllium (Be)	Holmium (Ho)	Praseodymium (Pr)	Thulium (Tm)	Bismuth (Bi)
Indium (In)	Rhenium (Re)	Tin (Sn)	Cesium (Cs)	Lutetium (Lu)
Scandium (Sc)	Tungsten (W)	Chlorine (Cl)	Magnesium (Mg)	Sulfur (S)
Vanadium (V)	Fluorine (F)	Molybdenum (Mo)	Tantalum (Ta)	Yttrium (Y)
Gallium (Ga)	Niobium (Nb)	Tellurium (Te)	Zinc (Zn)	Germanium (Ge)
Phosphorus (P)	Terbium (Tb)	Zirconium (Zr)	Hafnium (Hf)	

Table B4. Test Methods Used for Characterization of SRM 612 by NIST and Collaborating Laboratories

Isotope dilution mass spectrometry (IDMS)	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 (NAA)	Ag, Au, Co, Li, Mn
Spectrophotometry	Au, Fe, Mn, Ni
Polarography	Fe, Ti
Flame emission spectrometry (FES)	K, Rb, Sr
Inductively-coupled plasma mass spectrometry (ICP-MS)	As, Ba, Cd, Cr, Sb, Se
Isotope dilution inductively-coupled plasma mass spectrometry (ID-ICP-MS)	Cr, Se
Standard additions borate fusion X-ray fluorescence spectrometry	Ag, As, Ba, Cd, Cr, Mn, Sb

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