National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Materials

612, Trace Elements in a Glass Matrix (3 mm Wafer)
613, Trace Elements in a Glass Matrix (1 mm Wafer)

(Nominal Trace Element Concentration 50 mg/kg (ppm))

These Standard Reference Materials (SRMs) were produced and certified to facilitate the development of chemical methods of analysis for trace elements and are one of a series of four pairs of SRMs. For both SRMs, 612 and 613, the nominal trace element concentration is 50 mg/kg for each of the sixty-one elements that have been added to the glass support matrix. The two SRMs differ only in the thickness of the glass wafer. Units of SRMs 612 and 613 are issued as sets of six wafers.

(Certified Values are Listed on Page 2)

These materials were prepared in rod form and have been sliced into wafers. The rods were hand-pulled, and therefore are not uniform over their length. Each wafer is oval to circular in cross-section, with a nominal diameter of 12-14 mm. The certified values are for an entire wafer (no fragment thereof). The debris from wafering has been only partially removed and each wafer should be surface cleaned before use. The first step in preparing the wafer for analysis is to wipe it clean with alcohol, and then to give it a mild surface cleaning (not etch) in dilute (1:10) nitric acid. The wafers were cut with a copper-bonded diamond wheel and the nitric acid step is included to remove any possible copper contamination.

Considerable care and effort have gone into the manufacturing of these SRMs to ensure homogeneity. The target level of precision and accuracy for certification of these materials was 5 percent or better. To date no element has been proven to be heterogeneous outside this limit for the SRM wafer used in its entirety. However, spatial inhomogeneity does exist within each wafer. For certification, two or more methods or laboratories must agree to at least the target level.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W.R. Shields.

The technical and support aspects involved in the original preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Standard Reference Materials Program by J.L. Hague. Revision of this certificate was coordinated through the Standard Reference Materials Program by J.S. Kane.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented in this certificate.

Gaithersburg, MD 20899
January 27, 1992
(Revision of certificate dated 1-4-82)

William P. Reed, Chief
Standard Reference Materials Program

(over)

A listing of the 61 elements added and the present status of the analytical certification are given in the following table. An asterisk before the element indicates a certified concentration for that element. The indicated limits on the concentration are equal to the entire range of observed results among sample points and/or the 95 percent confidence interval, whichever is larger. Values in parentheses are information values and are not certified, for the reasons given in the footnotes. Nominal composition of the support matrix is 72% SiO_2, 12% CaO, 14% Na_2O, and 2% Al_2O_3.

Element	Value	Notes	Element	Value	Notes
Antimony	--		Boron	(32)	2,b,d
Arsenic	--		Cadmium	--	
Barium	(41)	1,a	Cerium	(39)	1,a
Beryllium	--		Cesium	--	
Bismuth	--		Chlorine	--	
Chromium	--		Europium	(36)	1,a
Cobalt	(35.5±1.2)	3,c	Fluorine	--	
Copper	(37.7±0.9)	4,d	Gadolinium	(39)	1,a
Dysprosium	(35)	1,a	Gallium	--	
Erbium	(39)	1,a	Germanium	--	
Gold	(5)	5,c,e	Lanthanum	(36)	1,a
Hafnium	--		*Lead	38.57±0.2	7,d,g
Holmium	--		Lithium	--	
Indium	--		Lutetium	--	
*Iron	51±2	6,e,f	Magnesium	--	
Manganese	(39.6±0.8)	8,e	Phosphorus	--	
Molybdenum	--		Potassium	(64)	10,d,h
Neodymium	(36)	1,a	Praseodymium	--	
*Nickel	38.8±0.2	9,d,e	Rhenium	--	
Niobium	--		*Rubidium	31.4±0.4	11,d,h,j,k
Samarium	(39)	1,a	Sulfur	--	
Scandium	--		Tantalum	--	
Selenium	--		Tellurium	--	
*Silver	22.0±0.3	12,c,d	Terbium	--	
*Strontium	78.4±0.2	13,d,h,j,k	Thallium	(15.7±0.3)	4,d
*Thorium	37.79±0.08	14,d,g	*Uranium	37.38±0.08	16,d,g
Thulium	--		Vanadium	--	
Tin	--		Ytterbium	(42)	1,a
Titanium	(50.1±0.8)	15,f	Yttrium	--	
Tungsten	--		Zinc	--	
			Zirconium	--	

(All values given in table are in mg/kg (ppm) by weight.)

NOTES:

1. Isotope dilution: interim data from only two sample points.
2. Nuclear track counting plus two sample points by isotope dilution, insufficient precision and accuracy for certification.
3. Neutron activation: one method only.
4. Isotope dilution: one method only, observed range caused by sample variability.
5. Spectrometry and neutron activation give grossly different results, value included only to indicate that the gold was not all lost in the processing of the glass rods.
6. Pooled value from data by spectrophotometry and polarography.
7. Pooled isotope dilution data: NIST and USGS data weighed equally.
8. Spectrophotometry: one method only.
9. Isotope dilution data accepted for certification, substantiated by spectrophotometry.
10. Interim data: isotope dilution and atomic absorption (both troubled with high blanks).
11. NIST isotope dilution data accepted for certification, cooperating analysts' data have a much larger uncertainty statement (range).
12. NIST isotope dilution data accepted for certification, substantiated by neutron activation.
13. Pooled data: NIST isotope dilution data accepted and substantiated by USGS and Australian National University. The normalized $^{87}Sr/^{86}Sr$ ratio = 0.7089±0.0002.
14. Pooled isotope dilution data: NIST data accepted for certification and substantiated by USGS.
15. Polarographic: one method only.
16. Isotope dilution: NIST substantiated by USGS. Uranium in glass depleted in ^{235}U. The atom percent ^{235}U = 0.2392.

a. Isotope dilution - N.J. Hubbard, NASA, Manned Spacecraft Center, Houston, Texas
b. Nuclear track counting - B.S. Carpenter
c. Neutron activation - B.A. Thompson
d. All isotope dilution analysis at NIST by staff, Analytical Mass Spectrometry Group
e. Spectrophotometry - R.W. Burke
f. Polarography - E.J. Maienthal
g. Isotope dilution - M. Tatsumoto, Isotope Geology Branch, USGS, Federal Center, Denver
h. Flame emission - T.C. Rains
j. Isotope dilution - C. Hedge, Isotope Geology Branch, USGS, Federal Center, Denver
k. Isotope dilution - W. Compston, Australian National University, Canberra