National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1412a

Multicomponent Glass
(disk form)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of glass. It can be used to validate value assignment of a laboratory's in-house reference materials. A unit of SRM 1412a consists of two disks, each about 40 mm diameter, about 3 mm thick, and weighing approximately 9.5 g.

Certified Mass Fraction Values: Certified values for constituents of SRM 1412a are reported in Table 1 as mass fractions of the elements in a glass matrix [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 taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction (expressed as percent). The expanded uncertainty intervals are expressed at a coverage level of approximately 95 %.

Table 1. Certified Mass Fraction Values for SRM 1412a

Element	Mass Fraction (%)	Uncertainty Interval (%)		
Aluminum (Al)	4.63	4.43	to	4.88
Antimony (Sb)	0.0138	0.0127	to	0.0150
Arsenic (As)	0.0084	0.0067	to	0.0099
Barium (Ba)	0.102	0.085	to	0.122
Boron (B)	1.23	1.13	to	1.35
Cadmium (Cd)	0.0072	0.0060	to	0.0086
Calcium (Ca)	2.85	2.61	to	3.04
Chromium (Cr)	0.0059	0.0047	to	0.0074
Iron (Fe)	0.00881	0.00778	to	0.00975
Potassium (K)	3.27	2.99	to	3.48
Lead (Pb)	0.0176	0.0136	to	0.0206
Lithium (Li)	1.86	1.67	to	2.11
Magnesium (Mg)	2.33	2.19	to	2.49
Silicon (Si)	27.68	27.22	to	28.19
Silver (Ag)	0.0080	0.0063	to	0.0101
Sodium (Na)	2.93	2.63	to	3.22
Strontium (Sr)	3.42	3.23	to	3.64
Zinc (Zn)	3.10	2.87	to	3.37

Expiration of Certification: The certification of **SRM 1412a** is valid indefinitely within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Carlos A. Gonzalez, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 11 June 2019

Steven J. Choquette, Director
Office of Reference Materials

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Statistical consultation for this SRM was provided by A.L. Pintar of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

The glass disks may be analyzed in the as-received form. If necessary, a disk may be cleaned by rinsing with ethanol. Grinding to refresh the surface of a disk should be used with care, due to the potential for contamination of the glass surface with grinding media.

For X-ray fluorescence methods and small sampling test methods that measure solid samples, it is recommended to measure an area > 50 mm^2. For the elements barium, iron and zinc, microscale heterogeneity exists in the glass, such that measurements of smaller areas may be biased.

For methods requiring powder, it is recommended to take at least one quarter of a disk for grinding. The minimum recommended sample quantity for powder is 0.25 g. Analysts should be aware that grinding methods have a high potential for contamination of the glass powder.

During dissolution of a powder sample, there is the potential to observe undissolved particles, because the cullet material was ground to powder using alumina grinding media. Not all dissolution approaches can dissolve alumina. The quantity of undissolved material observed during analyses was low enough that no bias was detected in the results of determinations of total aluminum using the flame atomic absorption spectrometry method.

To use the uncertainty intervals given in this certificate in comparisons and calculations, the user must be aware that most intervals are not symmetric about the assigned values. The use of the intervals depends on the context, and instructions for two common uses follow.

Direct comparison to a result of a user determination: For this case, directly compare a coverage interval calculated by the user for their result to the appropriate coverage interval provided in this certificate.

Propagation of uncertainty when using SRM 1412a as a calibration standard: When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. The asymmetric intervals in Table 1 cannot be interpreted as (value $\pm$ ku_c), where k is an expansion factor, and u_c is a combined standard uncertainty representative of both sides of the interval around the value. The user may choose one of the following two options for their uncertainty calculations. A first option is to use an approximation to u_c equal to one fourth of the width of the coverage interval given in this certificate (see note 8.1 in reference 3). A second and better option is to use a Monte Carlo method for propagation of probability distributions. This can be done using the NIST Uncertainty Machine at https://uncertainty.nist.gov. The user would enter the measurement equation and standard uncertainty estimates or probability distributions describing uncertainty for the terms of the equation. For the estimate of uncertainty of the calibration value, the user would choose the "Sample values" option and provide a file containing the actual distribution of the NIST analytical results of certification analyses. Plain ASCII files for the constituents may be found at https://www-s.nist.gov/srmors/view_detail.cfm?srm=1412A.

PREPARATION AND ANALYSIS[(1)]

The material for SRM 1412a was designed and obtained with the assistance of ASTM International Committee C14 on Glass and Glass Products, subcommittee C14.91 on Reference Materials, E.D. Spinosa, chair. Cullet of the required composition was manufactured by Kopp Glass, Inc. (Pittsburgh, PA). The cullet was ground, blended and sintered into disks by Community Glass, Inc. (Elmira, NY), under the direction of M. Crouse. The surfaces of the disks were ground by Swift Glass Company (Elmira, NY), under the direction of K. Wheeler, to remove surface contaminants from the sintering process.

[(1)] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify 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.

Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry to measure 21 disks for all elements, except Li. Suitability of this material for testing using low milligram quantities was evaluated using microbeam X-ray fluorescence spectrometry for all elements, except Li and B.

For each certified value, the estimate was derived by fitting a statistical model to the results of measurements using the test methods listed in Table 2. That is, a posterior probability distribution, describing knowledge of the measurand, was developed for each constituent. Its median was taken as the estimate of the measurand, and the 2.5th and 97.5th percentiles form the 95 % coverage interval. The Bayesian paradigm was used for statistical inference [4]. The probability distribution combines all recognized sources of uncertainty. This procedure is consistent with the ISO/JCGM Guide [5,6].

Measurements for value assignment of SRM 1412a were performed at NIST by A.F. Marlow, S.A. Rabb and J.R. Sieber. Additional analyses were performed by collaborating laboratories, including C. Bielski, B. Mitchell, S. Olson, and S. Weiser of American Glass Research (Maumee, OH) and M. Beercheck, J. Crandall, C. D'Agostino, R. O'Malley, D. Mauser, and R. Skinner of NSL Analytical Services, Inc. (Cleveland, OH).

Analytical methods used in value assignment of SRM 1412a are listed in Table 2. Test methods used to characterize the material used both disks and powder.

Table 2. Analytical Methods Used for SRM 1412a

Method	Elements Determined
Alkali Titration with Mannitol	B
Combustion with Infrared Detection	S
Flame Atomic Absorption Spectrometry (FAAS)	Al, Ca, K, Li, Mg, Na
Gravimetry	Si
Inductively Coupled Plasma Mass Spectrometry (ICPMS)	Ag, As, Ba, Cd, Cr, Fe, Ga, P, Pb, Sb, Se
Inductively Coupled Plasma Optical Emission Spectrometry (ICPOES)	Al, Ag, As, B, Ba, Ca, Cd, Cr, Fe, K, Li, Mg, Na, Pb, Sb, Sr, Zn
Wavelength Dispersive X-Ray Fluorescence Spectrometry (WDXRF)	Ag, Al, As, Ba, Ca, Cd, Cr, Fe, K, Mg, Na, Pb, Sb, Si, Sr, Zn

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 1412a.

Information Mass Fraction Values: Information values for constituents in SRM 1412a are reported as mass fractions in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability. Information values for phosphorus and gallium represent estimates of the limits of quantification for the test method as implemented at the collaborating laboratory.

Table 3. Information Mass Fraction Values for SRM 1412a

Constituent	Mass Fraction (%)
Gallium (Ga)	< 0.001
Phosphorus (P)	< 0.005
Selenium (Se)	0.004
Sulfur (S)	0.002

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and a laboratory's in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

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 Jun 2019).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition 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 https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF (accessed Jun 2019).

[3] Possolo, A.; *Simple Guide for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1900; U.S. Department of Commerce: Washington, DC (1994); available at https://nvlpubs.nist.gov/nistpubs/TechnicalNotes/NIST.TN.1900.pdf (accessed Jun 2019).

[4] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (Boca Raton, FL) (2013).

[5] 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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jun 2019); 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 Jun 2019).

[6] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Jun 2019).

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 https://www.nist.gov/srm.