National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 80a

Soda-Lime Glass (Beads)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials. SRM 80a is soda-lime glass beads manufactured for use in reflective highway markings and for abrasive bead blasting. A unit of SRM 80a consists of a bottle containing approximately 45 g of beads having nominal diameters in the range 0.05 mm to 0.15 mm.

Certified Mass Fraction Values: Certified mass fraction values for SRM 80a are listed in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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. A certified value is the present best estimate of the true value.

Reference Mass Fraction Values: Reference mass fraction values are given in Table 2. Reference values are non-certified values that are the present best estimates of the true values; however, the values do not meet the NIST criteria for certification [2] and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Values: Information mass fraction values are provided in Table 3. An information value is considered to be 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 80a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the 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.

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

Coordination of technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 80a were performed at NIST by J.R. Sieber. Additional measurements were performed by collaborating laboratories: K. Blanton, R. Embrey, Y. Gao, G. Hay, M. McDonald, S. Robertson, and L.M. Schurter, Owens Corning Science & Technology Center (Granville, OH); and L. Glaubach, E. Miller, and A.M. Ogura, ALS Minerals Division (North Vancouver, BC, Canada).

Statistical consultation for this SRM was provided by S. Lund of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 15 December 2015

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To relate analytical determinations to the values on this Certificate of Analysis, a minimum sample quantity of 250 mg is recommended. This minimum mass is sufficient for analyses of the glass matrix composition elements and of all constituent elements using methods with sample preparation by acid dissolution. However, certain measurement techniques may require a greater sample mass.

Direct analysis using X-ray fluorescence spectrometry (XRF) requires a minimum sample mass of 1.5 g, and the quality of measurements may benefit from using sufficient sample mass to create specimens that are infinitely thick with respect to the measured X-ray of highest energy. In the measurements made at NIST, the highest energy X-rays were Sb K-$L_{2,3}$ at 26.3 keV. At this energy, a sample mass of 3.25 g is required to prepare a 31 mm diameter specimen, for example as a pressed briquette or as loose powder in a liquid cell. In addition, it is recommended to base results for Pb, obtained using XRF measurements, on the average of at least two independent measurements.

When handled with care and used only for nondestructive XRF analyses, specimens of SRM 80a can be retained for reuse. Exposure to X-rays may darken the glass, but that does not change its composition. To avoid contamination of the original container of SRM 80a, it is recommended to store beads used for direct measurements in a separate, clean container. Do not return used material to the original SRM 80a bottle. Store the remaining material in its tightly-capped, original container in a cool, dry location.

NOTICE TO USERS

NIST encourages the use of its SRMs to establish metrological traceability for the user's measurement results, and NIST strives to maintain the SRM inventory supply. However, NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house reference materials and working standards. As such, SRMs should be used to validate or otherwise assign values to the more routinely used reference materials in laboratory. When the metrologically traceable values of such reference materials are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house reference material. When this SRM is used only to validate the measurement process used to assign a value to an in-house material, inclusion of the uncertainty of the certified value is not appropriate. Comparisons between NIST SRMs and 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.

PREPARATION AND ANALYSIS[(1)]

The material for the preparation of this SRM was provided by C. Davies, Potters Industries, Inc. (Conshohocken, PA). The material was blended and bottled at the NIST facilities in Gaithersburg, MD.

Homogeneity testing was performed at NIST using XRF. The homogeneity of the as-received beads was found to be satisfactory for use with test methods used for certification, except for the element Pb. The natural variance of Pb mass fraction in soda-lime glass beads has been found to be greater than for other elements. It has been shown that, for direct XRF measurements of glass beads of the type represented by SRM 80a, it is necessary to base test results on the average of at least two independent measurements. As an alternative, the variance of the Pb mass fraction can be reduced by grinding the beads to a fine powder, for example in a ball mill, prior to direct XRF measurements.

Test methods used by NIST and collaborating laboratories in the development of this SRM are listed in Table 4.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in soda-lime glass listed in Table 1. Metrological traceability is to the derived SI units for mass fraction (expressed as percent). The values in Table 1 come from fitting a statistical model to the measurements made on the SRM 80a material using multiple test methods. The Bayesian inference paradigm was used for statistical inference [3]. The expanded uncertainty is an interval calculated in a manner consistent with the ISO/JCGM Guide [4,5], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, differences among samples, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of

[(1)] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institutes of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The listed 95 % coverage intervals are provided with the following intended interpretation: the average of repeated mass fraction measurements of a single constituent in the contents of any one bottle will converge, as the number of repeated measurements increases, to a value within the interval listed below with 95 % probability, provided that the measurement system is unbiased.

Table 1. Certified Mass Fraction Values for SRM 80a Soda-Lime Glass Beads

Constituent	Mass Fraction (%)	95 % Coverage Interval (%)
Aluminum (Al)	0.921	0.839 to 1.006
Antimony (Sb)	0.0063	0.0045 to 0.0087
Arsenic (As)	0.040	0.029 to 0.052
Calcium (Ca)	5.80	5.28 to 6.38
Iron (Fe)	0.108	0.094 to 0.124
Lead (Pb)	0.0095	0.0080 to 0.0112
Magnesium (Mg)	1.66	1.53 to 1.80
Potassium (K)	0.552	0.508 to 0.598
Silicon (Si)	33.6	28.1 to 39.6
Sodium (Na)	9.95	9.07 to 10.85
Sulfur (S)	0.087	0.068 to 0.111
Tin (Sn)	0.0018	0.0013 to 0.0025

Reference Mass Fraction Values: The measurands are the mass fractions of the elements in soda-lime glass listed in Table 2 as determined by the indicated methods listed in Table 4. Metrological traceability is to the SI derived units for mass fraction (expressed as percent). The values in Table 2 come from fitting a statistical model to the measurements made on the SRM 80a material using multiple test methods. The Bayesian inference paradigm was used for statistical inference [3]. The expanded uncertainty is an interval calculated in a manner consistent with the ISO/JCGM Guide [4,5], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, differences among samples, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The listed 95 % coverage intervals are provided with the following intended interpretation: the average of repeated mass fraction measurements of a single constituent in the contents of any one bottle will converge, as the number of repeated measurements increases, to a value within the interval listed below with 95 % probability, provided that the measurement system is unbiased.

Table 2. Reference Mass Fraction Values for SRM 80a Soda-Lime Glass Beads

Constituent	Mass Fraction (%)	95 % Coverage Interval (%)
Barium (Ba)	0.11	0.10 to 0.13
Manganese (Mn)	0.005	0.003 to 0.007
Phosphorus (P)	0.006	0.004 to 0.009
Strontium (Sr)	0.009	0.006 to 0.012
Titanium (Ti)	0.021	0.017 to 0.027
Zinc (Zn)	0.019	0.015 to 0.024
Zirconium (Zr)	0.010	0.007 to 0.014

Information Mass Fraction Values: Information values that may be of interest and use to the SRM user are given in Table 3. The values for the listed elements represent the estimated limits of detection of the applied test methods listed in Table 4. Information values cannot be used to establish metrological traceability.

Table 3. Information Values for SRM 80a Soda-Lime Glass Beads

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
Beryllium (Be)	< 0.0002	Gallium (Ga)	0.0003
Bismuth (Bi)	< 0.002	Fluorine (F)	0.15
Boron (B)	0.04	Lithium (Li)	< 0.02
Cadmium (Cd)	0.0003	Mercury (Hg)	< 1 μg/g
Cerium (Ce)	0.025	Nickel (Ni)	0.0002
Cesium (Cs)	0.001	Rubidium (Rb)	0.006
Chromium (Cr)	< 0.002	Selenium (Se)	0.0003
Cobalt (Co)	< 0.0002	Vanadium (V)	0.0003
Copper (Cu)	< 0.003	Loss on Ignition at 1000 °C	0.27

Table 4. Analytical Methods Used for SRM 80a Soda-Lime Glass Beads

Constituent	Methods[(a)]	Constituent	Methods[(a)]	Constituent	Methods[(a)]
Aluminum	1, 3, 5	Copper	3, 5	Rubidium	5
Antimony	2, 4, 5	Fluorine	10	Selenium	4, 5
Arsenic	1, 4, 5	Gallium	5	Silicon	1
Barium	1, 3, 5	Iron	1, 3, 5	Sodium	1, 5
Beryllium	3, 5	Lead	1, 2, 4, 5	Strontium	1, 4, 5
Bismuth	3, 5	Lithium	5, 7	Sulfur	1, 5, 6
Boron	3	Magnesium	1, 3, 5	Tin	1, 2, 3, 5
Cadmium	3, 5	Manganese	1, 3, 5	Titanium	1, 3, 5
Calcium	1, 5	Mercury	9	Vanadium	3, 5
Cerium	5	Nickel	3, 5	Zinc	1, 3, 5
Cesium	5	Phosphorus	1, 3, 5	Zirconium	1, 3, 5
Chromium	1, 3, 5	Potassium	1, 5	Loss on Ignition	8
Cobalt	3, 5				

[(a)] Key to Methods in Table 4:

1. X-ray fluorescence spectrometry after borate fusion
2. X-ray fluorescence spectrometry with standard additions calibration after borate fusion
3. Inductively coupled plasma optical emission spectrometry
4. Graphite furnace atomic absorption spectrometry
5. Inductively coupled plasma mass spectrometry after digestion in a mixture of HNO_3, $HClO_4$, HF, and HCl
6. Combustion with infrared detection
7. Flame atomic absorption spectrometry
8. 2 g powder heated to 1000 °F (537.78 °C) for 1 h, cooled in a desiccator, and weighed
9. Direct mercury analyzer
10. Determination using ion selective electrode after fusion with $LiBO_2$ and acid dissolution

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://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Dec 2015).

[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/upload/SP260-136.PDF (accessed Dec 2015)

[3] Searle, S.R.; Casella, G.; McCulloch, C. E.; *Variance Components*; John Wiley & Sons, Hoboken, NJ. (2006).

[4] Pinheiro, J.C.; Bates, D.M.; *Mixed Effects Models in S and S-Plus*; Springer, New York, NY (2000).

[5] Toman, B.; Possolo, A.; *Laboratory Effects Models for Interlaboratory Comparisons*; Accredit. Qual. Assur., Vol. 14, pp. 553–563 (2009); and Toman, B.; Possolo, A.; *Erratum to: Laboratory Effects Models for Interlaboratory Comparisons*; Accredit. Qual. Assur., Vol. 15, pp. 653–654. (2010).

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

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

[8] 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 www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 2015); 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 www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Dec 2015).

[9] 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 www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Dec 2015).

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

[11] Davison, A.C.; Hinkley, D.V.; *Bootstrap Methods and their Application*; Cambridge University Press, New York (1997).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.