National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 89

Lead-Barium Glass

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis for element contents of lead-barium glass and materials of similar matrix. It can be used to validate value assignment of in-house reference materials. A unit of SRM 89 consists of one bottle containing approximately 45 g of powder.

Certified Mass Fraction Values: Certified values for constituents of SRM 89 are reported in Table 1 as mass fractions of the element or oxide compound in a lead-barium glass matrix on a dry basis [1]. The values for oxide forms assume stoichiometry of the form listed per industry convention. 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 International System of Units (SI) derived unit for mass fraction (expressed as percent). The expanded uncertainty estimates are expressed at a confidence level of approximately 95 %.

Expiration of Certification: The certification of **SRM 89** 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.

Maintenance of SRM Certification: NIST will monitor this SRM. 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 certification was performed by G.E.F. Lundell, formerly of NIST. Review and revision of values and uncertainty estimates were coordinated by J.R. Sieber of the NIST Chemical Sciences Division.

Analyses leading to the certification of this SRM were performed at NIST by G.E.F. Lundell and H.B. Knowles, formerly of NIST. Analytical determinations were also performed by P.E. Corbin, Corning Glass Works (Corning, NY); R.W. Goodwin, General Electric Co. (Cleveland, OH); D.W. Moore III, Libbey-Owens-Ford Glass Co. (Charleston, WV); M.O. Lamar, Norton Co. (Worcester, MA); R.H. Lardin, Pittsburgh Plate Glass Co. (Creighton, PA); and The Sharp-Schurtz Co. (Lancaster, OH).

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

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

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Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Director
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Table 1. Certified Mass Fraction Values for SRM 89 Lead-Barium Glass
(Values expressed on a dry sample basis.)

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Aluminum oxide (Al_2O_3)	0.155	0.023
Arsenic (III) oxide (As_2O_3)	0.0440	0.0035
Arsenic (V) oxide (As_2O_5)	0.363	0.030
Barium oxide (BaO)	1.423	0.033
Calcium oxide (CaO)	0.197	0.027
Chlorine (Cl)	0.0517	0.0052
Iron (III) oxide (Fe_2O_3)	0.0487	0.0029
Lead oxide (PbO)	17.436	0.063
Magnesium oxide (MgO)	0.0332	0.0051
Manganese (II) oxide (MnO)	0.081	0.015
Phosphorus pentoxide (P_2O_5)	0.233	0.027
Potassium oxide (K_2O)	8.323	0.087
Silicon dioxide (SiO_2)	65.33	0.20
Sodium oxide (Na_2O)	5.73	0.11
Sulfur trioxide (SO_3)	0.034	0.014
Titanium dioxide (TiO_2)	0.0136	0.0077

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 1 g should be used. Before sampling, it is recommended to mix the powder by inverting and rotating the bottle by hand for at least one minute. Prior to analysis, samples of the lead-barium glass powder must be dried for 1 h at 105 °C to 110 °C. A bottle containing unused material should be recapped immediately and stored at room temperature away from light.

The SRM slowly absorbs carbon dioxide and water. Therefore, the analyst must determine the loss on ignition at 900 °C to 1000 °C and correct all determinations for the difference between the new result and the original value shown in Table 2.

To use the uncertainty estimates given in this certificate, in propagation of uncertainty, divide the expanded uncertainty by $k = 2$ to obtain the combined standard uncertainty.

PREPARATION AND ANALYSIS[1]

The material for SRM 89 was prepared by NIST in collaboration with the Standards Committee of the Glass Division of the American Chemical Society, Chairman A.R. Payne, Fostoria Glass Co. (Moundsville, WV). The glass was ground to pass a 150 µm (100 mesh) sieve size in a porcelain mill with flint quartz pebbles by B.F. Drakenfeld & Co. Inc. (Washington, PA). At NIST, the material was put through magnetic separation, blended and bottled.

Each certified value is an unweighted mean of the results from classical, gravimetric methods performed independently by two to seven analysts. The test methods are listed in Table 3. The uncertainty listed with each certified value is an expanded uncertainty about the mean, with coverage factor, $k = 2$, calculated following the ISO/JCGM Guide [3,4].

[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.

ADDITIONAL CONSTITUENTS

Non-certified values are provided for the following additional constituents in SRM 89. These values are not certified, because NIST cannot vouch fully for the calibrations of the test methods and other details.

Reference Mass Fraction Values: Reference values for the properties of SRM 89 are reported in Table 2 as mass fractions, expressed as percent. A NIST reference value is a non-certified value that is the present best estimate based on available data; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2]. The values are the unweighted means of results obtained at several laboratories. The expanded uncertainty, U, is an expanded uncertainty about the mean, with coverage factor, $k = 2$ [3,4]. Results derived from the use of this value are considered by NIST to be traceable only to the value itself.

Table 2. Reference Mass Fraction Values for SRM 89 Lead-Barium Glass

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)
Loss on Ignition (LOI)	0.321	0.078
Zirconium dioxide (ZrO_2)	0.0045	0.0013

Table 3. Analytical Methods Used in Value Assignment of SRM 89

Element	Test Methods
Al_2O_3	Gravimetric as $AlPO_4$; Gravimetric as Al_2O_3
As_2O_3	Method given in reference [5]
As_2O_5	Method given in reference [5]
BaO	Electrolysis followed by gravimetry as $BaSO_4$
CaO	Spectrometric method; Gravimetric after double dehydration with NH_4OH; Gravimetric after precipitation as oxalate from acetic acid solution
Cl	Method given in reference [5]
Fe_2O_3	Gravimetric as Fe_2O_3; Titration with $KMnO_4$ after reduction with Zn; Electrometric titration with $K_2Cr_2O_7$ after $SnCl_2$ reduction
PbO	Electrolysis followed by gravimetry as $PbSO_4$; Gravimetric as $PbCrO_4$; Gravimetric by precipitation as $PbSO_4$ followed by dissolution in ammonium acetate with H_2SO_4 and weighing by difference
MgO	Spectrometric method; Gravimetric after double precipitation with $NaNH_4PO_4$ and ignition to $Mg_2P_2O_7$
MnO	Bismuthate oxidation and titration with Fe_2SO_4-$KMnO_4$
P_2O_5	Precipitate as phosphor-molybdate and determine gravimetrically as $Mg_2P_2O_7$; Gravimetrically as ammonium phospho-molybdate
K_2O	Gravimetric after separation and precipitation as K_2PtCl_6 (method given in reference [6]); Gravimetric after precipitation with $HClO_4$ and absolute ethyl acetate
SiO_2	Fusion with $Na_2(CO_3)$, dissolution in HCl, SiO_2 determined by double evaporation; Fusion with soda and niter, double dehydration with HNO_3, ignition to constant weight
Na_2O	Gravimetric after separation and precipitation of K with H_2PtCl_6 (method given in reference [6])
SO_3	Method given in reference [5]
TiO_2	Colorimetric method after cupferron separation
ZrO_2	Gravimetric as $ZrPO_4$ after cupferron separation
LOI	Heating in open crucible over a Tirrill burner; Heating in a muffle furnace and summing loses at 965 °C, 1100 °C and 1200 °C; Heating to constant weight in a Pt crucible at 1000 °C over a Fisher burner

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 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 Apr 2021).

[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.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sharpless, K.E.; Sieber, J.R.; 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; U.S. Government Printing Office: Washington, DC (2020); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2020.pdf (accessed Apr 2021).

[3] Sieber, J.R.; Possolo, A.M.; Epstein, M.S.; *A Retuned Horwitz Procedure for Upgrading Certificates of Older Standard Reference Materials*; NIST Special Publication 260-198; available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-198.pdf (accessed Apr 2021).

[4] 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/publications/guides (accessed Apr 2021); 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 2021).

[5] Lundell, G.E.F; Knowles, H.B.; *The Analysis of Soda-Lime Glass*; J. Am. Ceram. Soc., Vol. 10(11), pp. 829−849 (1927).

[6] Smith, J.L.; In *Select Methods of Chemical Analysis (Chiefly Inorganic)*, 3rd ed.; Crookes, W.; Eds.; Longmans, Greene, and Co.: New York, NY, p. 26 (1894).

Certificate Revision History: 26 April 2021 (Recommended percentages replaced by certified values for constituents in Table 1; uncertainty estimates provided for Table 1 constituents; reassigned LOI and ZrO_2 as reference values with recommended percentages replaced by non-certified values in Table 2; uncertainty estimates provided for non-certified values in Table 2; editorial changes); 20 December 1990 (Editorial changes); 27 August 1932 (Original certificate date); 02 March 1928 (Provisional 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; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.