National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1568b

Rice Flour

This Standard Reference Material (SRM) is intended primarily for validation of methods for determining elements in rice flour and similar materials. This SRM can also be used for quality assurance when assigning values to in-house reference materials. This SRM is rice flour prepared from a single lot of river rice by a commercial manufacturer. A unit of SRM 1568b consists of a single bottle containing approximately 50 g of material sealed inside an aluminized pouch.

Certified Mass Fraction Values: The certified mass fraction values of selected elements in SRM 1568b are provided in Tables 1 and 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 [1]. Analyses for value assignment were performed by NIST and the National Metrology Institute of Japan (NMIJ). Values are reported on a dry-mass basis in mass fraction units [2].

Reference Mass Fraction Values: Reference mass fraction values are provided for additional elements (Table 3). A NIST reference value is a non-certified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification [1] and is provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. The reference mass fraction values were derived from results reported by NIST. Values are reported on a dry-mass basis in mass fraction units [2].

Expiration of Certification: The certification of **SRM 1568b** is valid, within the measurement uncertainty specified, until **20 June 2025**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by W.C. Davis and L.J. Wood of the NIST Chemical Sciences Division and S.E. Long formerly of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by S. Christopher, W.C. Davis, A.F. Marlow, A.J. Moors, K.E. Murphy, L.J. Wood, T.W. Vetter, and L.L. Yu of the NIST Chemical Sciences Division and B.M. Adair, S.E. Long, R. Oflaz, and J.R. Sieber, formerly of NIST. Analyses for value assignment were also performed by T. Narukawa of the NMIJ. Original analyses were performed by E.S. Beary, T.A. Butler, M.S. Epstein, J.D. Fassett, R.R. Greenberg, L.B. Jassie, W.R. Kelly, H.M. Kingston, J.R. Moody, P.J. Paulsen, T.C. Rains, T.A. Rush, S.F. Heller-Zeisler, R.L. Watters, Jr., and L.J. Wood.

Statistical analysis was provided by J.H. Yen 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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Certificate Revision History on Last Page

Steven J. Choquette, Director
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NOTICE AND WARNING TO USERS

SRM 1568b IS INTENDED FOR LABORATORY USE ONLY, NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored at controlled room temperature (20 °C to 25 °C) in the original unopened bottle. For elemental analyses, the bottle can be re-capped, stored at controlled room temperature (20 °C to 25 °C), and test portions removed and analyzed until the material reaches its expiration date.

Use: Before use, the contents of the bottle should be mixed thoroughly. Allow the contents to settle for one minute prior to opening to minimize the loss of fine particles. To relate analytical determinations to the certified values in this Certificate of Analysis, the sample test portion indicated in the description of each group of analytes below should be used. Results obtained in analyses should include their own estimates of uncertainty and can be compared to the certified values using procedures described in reference [3].

INSTRUCTIONS FOR DRYING

Residual moisture content should be determined on a separate sample for converting analytical results to a dry-mass basis. The recommended drying methods are (1) freeze drying at 25 °C for 24 h; (2) oven drying at 90 °C for 2 h; or (3) desiccator drying over magnesium perchlorate for 28 days. The observed moisture content ranges from 6.1 % to 6.4 % (mass fraction).

SOURCE, PREPARATION, AND ANALYSIS[1]

Source and Preparation: Material existing from production of SRM 1568a was used to produce SRM 1568b. SRM 1568b is rice flour produced from 100 % long grain river rice grown in Arkansas. The original rice flour was ground to pass through a 425 μm (40 mesh) sieve, blended, and bottled at NIST. The reprocessed material was dried for 24 h at 101 °C and then double blended using a ceramic-lined cone blender for 30 min. Using a Teflon-lined hopper and trough, 50 g quantities of material were placed in 4 oz amber bottles. The bottles were capped and individually sealed in aluminized bags. Prior to bottling, the rice flour was irradiated with 2.5 megarads of ^{60}Co by Neutron Products, Inc. (Dickerson, MD).

Analytical Approach for Determination of Elements: Assigned mass fraction values for the elements in SRM 1568b were based on the combination of measurements from two different analytical methods at NIST and NMIJ, where available. Where certified values from SRM 1568a were considered scientifically valid, the combined estimator is the mean of two methods, one from the new data and the other from the certified value from SRM 1568a. NIST provided measurements by using inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS), instrumental neutron activation analysis (INAA), liquid chromatography triple quadrupole mass spectrometry (LC/MS/MS), ion exchange chromatography inductively coupled plasma mass spectrometry (IC/ICP-MS), sector field inductively coupled plasma mass spectrometry (SF-ICP-MS), and wavelength dispersive X-ray fluorescence spectrometry (WDXRF).

NIST Analyses for Al, As, Cu, Mn, Mo, Rb, and Se Using ICP-OES, SF-ICP-MS, and/or ICP-MS: Aluminum, arsenic, copper, manganese, molybdenum, rubidium, and selenium were measured by ICP-OES, SF-ICP-MS or ICP-MS using duplicate 0.5 g test portions taken from each of eight bottles of SRM 1568b. Samples were digested in nitric acid using a microwave sample preparation system. Quantification was based on the method of standard additions.

NIST Analyses for Pb Using Isotope Dilution ICP-MS: Lead was measured by ID-ICP-MS using duplicate 1.0 g test portions taken from each of six bottles of SRM 1568b. Samples were spiked with isotopically enriched ^{206}Pb and were digested in nitric acid using a microwave sample preparation system. Sample digests were evaporated to near dryness and a portion was reconstituted in dilute nitric acid for Pb analysis.

NIST Analyses for Al, Br, Ca, Cl, Fe, Mg, Mn, P, K, Rb, S, and Zn Using WDXRF: Aluminum, bromine, calcium, chlorine, iron, magnesium, manganese, phosphorus, potassium, rubidium, sulfur, and zinc were measured by WDXRF

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[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.

using duplicate or triplicate 2.5 g test portions taken from each of 24 bottles of SRM 1568b. Briquettes were prepared for each sample. The K-L$_{2,3}$ characteristic X-ray lines of all elements were used for quantification.

NIST Analyses for Br, Mn, and K Using INAA: Bromine, manganese, and potassium were measured by INAA using duplicate 0.25 g test portions taken from each of six bottles of SRM 1568b. Powders were pressed into cylindrical pellets and irradiated in the pneumatic tube RT-2 of the NIST reactor at a reactor power of 20 MW. Samples, standards, and controls were packaged individually in clean polyethylene bags for analysis using gamma-ray spectroscopy. The count was done after several hours decay at a sample-to-detector distance of 5 cm for 30 min counting time.

NIST Analyses for As Species Using LC/MS/MS and IC/ICP-MS: Dimethylarsinic acid (DMA), monomethylarsonic acid (MMA), and As (III) and As (V), reported as inorganic arsenic (iAs), were measured by IC/ICP-MS using duplicate 1 g test portions taken from each of eight total bottles of SRM 1568b. DMA was also analyzed by LC/MS/MS using duplicate 1 g test portions taken from each of six bottles of SRM 1568b. Samples were prepared in a methanol/water mixture using a microwave sample preparation system. Quantification was based on the method of standard additions. NMIJ reported individual results for DMA, MMA, As (III), and As (V) using IC/ICP-MS. For analytes that were measured by NIST and NMIJ, the combination of the mean results from analyses by NIST and the mean results provided by NMIJ were averaged, as appropriate.

Homogeneity Assessment: The homogeneity of elements was assessed at NIST from methods and test portion sizes described above. For the elements measured by NIST, analyses of variance with 5 % significance level did not show statistically significant heterogeneity with the exception of Pb. A pattern of heterogeneity in the Pb data was evident, so the uncertainty for Pb incorporates an uncertainty component for possible heterogeneity.

Value Assignment: The SRM 1568a material was re-examined and the original certified values were determined to be scientifically valid in some cases. Where the data were determined to be valid, new NIST values were combined with values from SRM 1568a. The combined estimator is the mean of the new NIST data and the certified value for SRM 1568a. In the case of cadmium and mercury, the data from the original SRM 1568a were considered valid and the combination of the mean of results from analyses by NIST for SRM 1568a were used for certification. Original SRM 1568a data were determined using the following methods: heated graphite atomizer (electrothermal) atomic absorption spectrometry (ETAAS), flame atomic absorption spectrometry (FAAS), flame emission spectrometry (FES), flow injection analysis cold vapor atomic absorption spectrometry (FIA-CV-AAS), hydride generation atomic absorption spectrometry (Hyd-AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), thermal ionization mass spectrometry (TIMS), isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS), instrumental neutron activation analysis (INAA), radiochemical neutron activation analysis (RNAA), and spectrophotometry (SPECTRO).

Certified Mass Fraction Values for Selected Elements: Each certified mass fraction value is the mean from the combination of the mean of results from analyses by NIST or the combination of mean results from analyses by NIST and the certificate value from SRM 1568a treated as a method estimate, where appropriate. 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 [4–6]. The true value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$.

Metrological Traceability: The measurand in each case is the total concentration of each analyte as described in the text. Metrological traceability is to the International System of Units (SI) unit indicated for each analyte in Tables 1–3, respectively.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1568b

Element	Mass Fraction (mg/kg)		
Aluminum[a,b,c]	4.21	±	0.34
Arsenic[a,d]	0.285	±	0.014
Bromine[b,e]	8.31	±	0.61
Cadmium[f,g]	0.0224	±	0.0013
Calcium[b,h,i]	118.4	±	3.1
Chlorine[b,e]	301.1	±	3.8
Copper[a,e,g,h]	2.35	±	0.16
Iron[b,e,h,j]	7.42	±	0.44
Magnesium[b,e,h,i]	559	±	10
Manganese[b,e]	19.2	±	1.8
Mercury[e,l]	0.00591	±	0.00036
Molybdenum[a,e,k,m]	1.451	±	0.048
Phosphorus[b,i,m,n]	1530	±	40
Potassium[b,e]	1282	±	11
Rubidium[a,b]	6.198	±	0.026
Selenium[a,e,o]	0.365	±	0.029
Sulfur[b,j]	1200	±	10
Zinc[b,e,h]	19.42	±	0.26

[a] NIST ICP-MS
[b] NIST WDXRF
[c] NIST SF ICP-MS
[d] NIST IC-ICP-MS
[e] NIST INAA
[f] NIST ETAAS
[g] NIST RNAA
[h] NIST FAAS
[i] NIST FES
[j] NIST TIMS
[k] NIST ID-ICP-MS
[l] NIST FIA-CV-AAS
[m] NIST ICP-OES
[n] NIST SPECTRO
[o] NIST Hyd-AAS

Certified Mass Fraction Values for Selected Arsenic Species: Each certified mass fraction value is the mean from the combination of the mean results from analyses by NIST and the mean results provided by NMIJ, where appropriate. 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 [2–4]. The true value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [4–6].

Table 2. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Arsenic Species in SRM 1568b

Arsenic Species	Mass Fraction (mg/kg, as As)		
Dimethylarsinic acid (DMA)[a,b,c]	0.180	±	0.012
Monomethylarsonic acid (MMA)[a,c]	0.0116	±	0.0035
Inorganic arsenic (iAs)[a,c,d]	0.092	±	0.010

[a] NIST IC/ICP-MS
[b] NIST LC/MS/MS
[c] NMIJ IC/ICP-MS
[d] As (III) and As (V), reported as total inorganic arsenic (iAs)

Reference Mass Fraction Values for Selected Elements: Each reference mass fraction value is the mean result of NIST analyses using one method. 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 [4,5]. The uncertainty for lead incorporates an uncertainty component for possible heterogeneity.

Table 3. Reference Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1568b

Element	Mass Fraction (mg/kg)		
Cobalt[a]	0.0177	±	0.0005
Lead[b]	0.008	±	0.003
Tin[c]	0.005	±	0.001

[a] NIST INAA
[b] NIST ID-ICP-MS
[c] NIST RNAA

REFERENCES

[1] 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 Jun 2021).

[2] 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 2021).

[3] Sharpless, K.E.; Duewer, D.L.; *Standard Reference Materials for Analysis of Dietary Supplements*; J. AOAC Int., Vol. 91, pp. 1298–1302 (2008).

[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 (2008); available at https://www.bipm.org/en/publications/guides (accessed Jun 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 Jun 2021).

[5] 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/en/publications/guides (accessed Jun 2021).

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

Certificate Revision History: 22 June 2021 (Removal of sodium values in Table 1 due to inhomogeneity; uncertainty statements updated; change of expiration date; editorial changes); 23 October 2013 (Analyst added; editorial changes); 07 August 2013 (Original 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.