National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1567b

Wheat Flour

This Standard Reference Material (SRM) is intended primarily for validation of methods for determining elements in wheat flour and similar materials. This SRM can also be used for quality assurance when assigning values to in-house reference materials. A unit of SRM 1567b 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 1567b are provided in Table 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 investigated or taken into account [1]. 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 2). A NIST reference value is a noncertified 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 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 [1]. Values are reported on a dry-mass basis in mass fraction units [2].

Information Mass Fraction Values: Information mass fraction values for several elements determined using a single method at NIST are provided in Table 3. A NIST 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 or only a limited number of analyses were performed. Values are reported on a dry-mass basis in mass fraction units [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1567b** is valid, within the measurement uncertainty specified, until **01 November 2023**, 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) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by M.S. Epstein and L.J. Wood of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by S.J. Christopher, W.C. Davis, A.F. Marlow, A.J. Moors, K.E. Murphy, R. Oflaz, J.R. Sieber, T.W. Vetter, L.J. Wood, and L.L. Yu of the NIST Chemical Sciences Division. Original analyses of existing material were performed by E.S. Beary, M.S. Epstein, J.D. Fassett, R.R. Greenberg, S.F. Heller-Zeisler, L.B. Jassie, W.R. Kelly, H.M. Kingston, J.R. Moody, P.J. Paulsen, T.C. Rains, T.A. Rush, 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 Issue Date: 12 March 2014

Robert L. Watters, Jr., Director
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NOTICE AND WARNING TO USERS

SRM 1567b 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 test portion mass indicated in the description of the NIST analyses for 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]. A minimum sample mass related to the NIST analytical determination is described in the "Source, Preparation, and Analysis" section.

INSTRUCTIONS FOR DRYING

Residual moisture content should be determined on a separate sample for conversion of 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 d. The observed moisture content ranges from 6.6 % to 8.5 % (mass fraction).

SOURCE, PREPARATION, AND ANALYSIS[(1)]

Source and Preparation: Material existing from production of SRM 1567a was reprocessed in 2013 to produce SRM 1567b. SRM 1567b is wheat flour milled from a blend of hard red spring wheat and hard red winter wheat grown primarily in South Dakota. The flour was taken from the mill packer during the middle of a run to obtain a homogeneous material. The flour has been bleached and brominated in accordance with standard treatment for commercial bakery use before 1991. The original wheat flour was ground to pass through a 425 μm (40 mesh) sieve, blended, and bottled at NIST. The original bottled material was then irradiated to an absorbed dose of 25 kGy of ^{60}Co by Neutron Products, Inc. (Dickerson, MD). The reprocessed material was dried for 24 h at 101 °C and then double-blended using a ceramic-lined cone blender for 30 min. Fifty grams of material were placed in 4 oz amber bottles. The bottles were capped and individually sealed in aluminized bags.

Analytical Approach for Determination of Elements: Value assignment of the mass fractions of the elements in SRM 1567b was based on the combination of measurements from two different analytical methods at NIST where available. Where certified values from SRM 1567a were considered scientifically valid, the combined estimator is the mean of two methods, one from the new data and the other being from the certified value from SRM 1567a. 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), sector field inductively coupled plasma mass spectrometry (SF-ICP-MS), and wavelength dispersive X-ray fluorescence spectrometry (WDXRF).

NIST Analyses for Al, As, Cu, Mo, Rb, Se, and Na Using ICP-OES, SF-ICP-MS, and/or ICP-MS: Aluminum, arsenic, copper, molybdenum, rubidium, selenium, and sodium 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 1567b. 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 1567b. 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 lead analysis.

[(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.

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 using duplicate 2.5 g test portions taken from each of 24 bottles of SRM 1567b. 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, K, Mn, and Na Using INAA: Bromine, manganese, potassium, and sodium were measured by INAA using duplicate 0.25 g test portions taken from each of six bottles of SRM 1567b. 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.

Homogeneity Assessment: The homogeneity of elements was assessed at NIST from methods and test portion sizes described above. For the elements measured from bottles of SRM 1567b, analyses of variance with 5 % significance level did not show statistically significant heterogeneity. The data for Pb indicated a pattern of heterogeneity so the uncertainty for Pb incorporates an uncertainty component for possible heterogeneity.

Value Assignment: For elements for which the certified values for SRM 1567a remained valid, new NIST data and these values were combined. The combined estimator is the mean of the new NIST data and the original certified value for SRM 1567a. For elements for which the certified values for SRM 1567a were no longer valid, the values are the mean from the combination of the means of results from analyses by NIST. In the case of cadmium, the certified value for SRM 1567a was used for SRM 1567b. Original values for SRM 1567a were determined using the following methods: graphite furnace atomic absorption spectrometry (GFAAS), flame atomic absorption spectrometry (FAAS), flame emission spectrometry (FES), hydride generation atomic absorption spectrometry (HydAAS), inductively coupled plasma optical emission spectrometry (ICP-OES), direct-current plasma atomic emission spectrometry (DCP-AES) 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 means of results from analyses by NIST or the combination of mean results from analyses by NIST and the certificate value from SRM 1567a treated as a method estimate, where appropriate. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c incorporates the observed difference between the results from the methods and their respective uncertainties and an uncertainty component for moisture correction, consistent with the ISO/JCGM Guide, and k is a coverage factor corresponding to an approximately 95 % level of confidence [4–6]. Since two or more methods were used for each analyte, the measurand is the total mass fraction for each analyte listed. The certified values are metrologically traceable to the SI unit of milligram per kilogram on a dry-mass basis.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1567b

	Mass Fraction (mg/kg)			Coverage Factor (k)	NIST Method
Cadmium	0.0254	±	0.0009	2.0	GFAAS, RNAA
Calcium	191.4	±	3.3	2.0	FAAS, FES, WDXRF
Chlorine	565.3	±	8.6	2.0	INAA, WDXRF
Copper	2.03	±	0.14	2.0	FAAS, ICP-MS, INAA, RNAA
Iron	14.11	±	0.33	2.0	FAAS, INAA, TIMS, WDXRF
Lead	0.0104	±	0.0024	2.2	ID-ICP-MS
Magnesium	398	±	12	2.0	DCP-AES, FAAS, INAA, WDXRF
Manganese	9.00	±	0.78	2.0	INAA, WDXRF
Molybdenum	0.464	±	0.034	2.0	ICP-MS, ICP-OES, ID-ICP-MS, INAA
Phosphorus	1333	±	36	2.0	DCP-AES, ICP-OES, SPECTRO, WDXRF
Potassium	1325	±	20	2.0	INAA, WDXRF

(continued on next page)

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1567b (Continued)

	Mass Fraction (mg/kg)			Coverage Factor (k)	NIST Method
Rubidium	0.671	±	0.012	2.0	ICP-MS, WDXRF
Selenium	1.14	±	0.10	2.0	HydAAS, ICP-MS, INAA
Sodium	6.71	±	0.21	2.0	ICP-OES, INAA, SF-ICP-MS
Sulfur	1645	±	25	2.0	TIMS, WDXRF
Zinc	11.61	±	0.26	2.0	FAAS, INAA, WDXRF

Reference Mass Fraction Values for Selected Elements: Each reference mass fraction value is the mean result of a NIST analysis using a single method or the mean from the combination of the mean of results from analyses by NIST. The uncertainty provided is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c represents the combined uncertainty, incorporating an uncertainty component for moisture correction, consistent with the ISO/JCGM Guide, and k is a coverage factor corresponding to an approximately 95 % level of confidence [4,5]. Since one or more methods were used for each analyte, the measurand is the total mass fraction for each analyte listed. The reference values are metrologically traceable to the SI unit of milligram per kilogram on a dry-mass basis.

Table 2. Reference Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1567b

	Mass Fraction (mg/kg)			Coverage Factor (k)	NIST Method
Aluminum	4.4	±	1.2	2.0	ICP-MS, SF-ICP-MS, WDXRF
Arsenic	0.0048	±	0.0003	2.1	ICP-MS
Bromine	6.45	±	0.97	2.0	INAA, WDXRF

Information Mass Fraction Values for Selected Elements: Each information mass fraction value is the mean result of a NIST analysis using a single method.

Table 3. Information Mass Fraction Values (Dry-Mass Basis) for Selected Elements in SRM 1567b

	Mass Fraction (mg/kg)	NIST Method
Mercury	0.0005	RNAA
Tin	0.003	RNAA
Vanadium	0.01	INAA

REFERENCES

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[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed March 2014).

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

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 http://www.nist.gov/srm.