NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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24 June 2024

Standard Reference Material® 1548b

Typical Diet

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended for the evaluation of methods for the determination of proximates, sugars, elements, amino acids, fatty acids, and herbicides in mixed diets and food matrices and for quality assurance when assigning values to in-house control materials.

Description: A unit of SRM 1548b consists of two packets, each containing approximately 5 g of the freeze-dried homogenate of mixed foods.

Certified Values: These NIST certified values are traceable to the International System of Units (SI) derived unit of mass fraction, expressed as milligrams per kilogram. The values are reported on an as-received basis [1].

Table 1. Certified Values for Elements in SRM 1548b

Measurand	Mass Fraction[(a)] (mg/kg)	Measurand	Mass Fraction[(a)] (mg/kg)
Aluminum (Al)	67.8 ± 1.2	Lead (Pb)	0.0144 ± 0.0015
Arsenic (As)	0.0355 ± 0.0075	Magnesium (Mg)	538 ± 41
Barium (Ba)	0.85 ± 0.11	Manganese (Mn)	6.30 ± 0.21
Calcium (Ca)	1604 ± 83	Phosphorus (P)	2410 ± 120
Cadmium (Cd)	0.0327 ± 0.0022	Potassium (K)	5950 ± 430
Chlorine (Cl)	10770 ± 740	Sodium (Na)	6980 ± 760
Copper (Cu)	2.03 ± 0.16	Strontium (Sr)	3.69 ± 0.16
Iron (Fe)	28.9 ± 4.1	Zinc (Zn)	19.1 ± 1.1

[(a)] 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. 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 $U_{95\%}(x)/2$ [2–4].

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Values of potential interest and additional information are provided in Appendices B and C.

Period of Validity: The certified values delivered by **SRM 1548b** are valid within the measurement uncertainty specified until **01 July 2030**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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Safety: SRM 1548b IS INTENDED FOR RESEARCH USE; not for human consumption.

Storage: SRM 1548b should be stored under refrigeration at a temperature between 2 °C and 8 °C, sealed in its original package, and should **NOT** be exposed to intense direct light or ultraviolet radiation. The package can be opened for removal of test portions and resealed until the material reaches its expiration date.

Use: The contents of the package should be thoroughly mixed before each use and should be allowed 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 [5], users must estimate their measurement uncertainty and use minimum test portion masses as described in the relevant NIST method section(s) of Appendix B: 0.1 g for nitrogen (N); 1 g for boron (B), chloride (Cl), and sulfur (s); 4 g for aluminum (Al) and silicon (Si); 0.5 g for remaining elements; 1 g for ash and solids. Test portions should be taken by gently tapping to remove the material needed from the SRM packet into a secondary container.

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 (NIST SP) 260-136, 2021 Edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jun 2024).

[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 2024).

[3] 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/committees/jc/jcgm/publications (accessed Jun 2024); 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 2024).

[4] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to Expression of Uncertainty in Measurement" — Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jun 2024).

[5] Sharpless, K.E.; Lippa, K.A.; Duewer, D.L.; Rukhin, A.L.; *The ABCs of Using Standard Reference Materials in the Analysis of Foods and Dietary Supplements: A Practical Guide*; NIST Special Publication 260-181; U.S. Government Printing Office: Washington, DC (2014); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-181.pdf (accessed Jun 2024).

[6] SRM 1548a; *Typical Diet;* National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (06 October 2020); available at https://tsapps.nist.gov/srmext/certificates/archives/1548a.pdf (accessed Jun 2024).

[7] Searle, S.R.; Casella, G.; McCulloch, C.L.; *Variance Components;* John Wiley; Hoboken, NJ (1992).

[8] Rukhin, A.L.; Possolo, A.; *Laplace Random Effects Models for Interlaboratory Studies;* Computational Statistics and Data Analysis, Vol. 55, pp. 1815–1827 (2011).

[9] Bryan Sallee, C.E.; Phillips, M.M.; Barber, C.A.; Burdette, C.Q., Kotoski, S.P.; Wood, L.J. (2023) *Food Nutrition and Safety Measurements Quality Assurance Program: Exercise 1 Final Report*. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Internal Report (IR) NIST IR 8447r1. Available at https://doi.org/10.6028/NIST.IR.8447r1 (accessed Jun 2024).

Certificate Revision History: 24 June 2024 (Non-certified value for glyphosate added to Table A3; silicon value of potential interest moved to appendix B; updated format; editorial changes); **03 May 2021** (Original certificate date).

Certain commercial equipment, instruments or materials may be identified in this Certificate of Analysis 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.

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 100 Bureau Drive, Stop 2300, Gaithersburg, Maryland 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * * * * * * End of Certificate of Analysis* * * * * * * * * * * *

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not meet the NIST criteria for certification [1] nor provide metrological traceability to the International System of Units (SI). They are the best estimates of the true values based on available data. The values are provided with an uncertainty that may reflect only measurement reproducibility, may not include all sources of uncertainty, and/or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Information about methods used to determine non-certified values is summarized in Appendix B.

Table A1. Non-Certified Values for Elements in SRM 1548b

Element	Mass Fraction[(a)] (mg/kg)			Element	Mass Fraction[(a)] (mg/kg)		
Boron (B)	4.422	±	0.091	Rubidium (Rb)	3.567	±	0.052
Cesium (Cs)	0.0113	±	0.0015	Sulfur (S)	1640	±	490
Iodine (I)	0.587	±	0.027	Selenium (Se)	0.177	±	0.051
Molybdenum (Mo)	0.266	±	0.013	Tin (Sn)	1.0937	±	0.0073
Nickel (Ni)	0.265	±	0.022				
Nitrogen (N)	245.9	±	8.0				

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence.

Table A2. Non-Certified Values for Amino Acids and Proximates in SRM 1548b

Measurand	Mass Fraction[(a)] (g/100 g)			Measurand	Mass Fraction[(a)] (g/100 g)		
Alanine	0.687	±	0.017	Ash	3.55	±	0.18
Arginine	0.728	±	0.042	Carbohydrates	48.2	±	2.2
Aspartic Acid	1.29	±	0.12	Extracted Fat	26.5	±	1.2
Cystine	0.174	±	0.044	Total Fatty Acids	24.3	±	1.5
Free Methionine	2.86	±	0.19	Protein	14.9	±	1.0
Isoleucine	0.654	±	0.021	Solids	99.55	±	0.39
Valine	0.758	±	0.060	Total Sugars	24.0	±	2.5
				Fructose	5.63	±	0.60
				Sucrose	9.0	±	1.1
				Glucose	5.27	±	0.59
				Total Fiber	4.80	±	0.67

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence.

Table A3. Non-Certified Value for Glyphosate in SRM 1548b

	Mass Fraction[(a)] (ng/g)
Glyphosate	78.8 ± 5.5

[(a)] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence.

Table A4. Non-Certified Values for Fatty Acids, Cholesterol, and Calories in SRM 1548b

Measurand		Mass Fraction[a] (g/100 g)
Hexanoic Acid (C6:0)	Caproic Acid	0.073 ± 0.008
Octanoic Acid (C8:0)	Caprylic Acid	0.0948 ± 0.0089
Decanoic Acid (C10:0)	Capric Acid	0.147 ± 0.005
Dodecanoic Acid (C12:0)	Lauric Acid	0.728 ± 0.037
Tetradecanoic Acid (C14:0)	Myristic Acid	0.657 ± 0.060
Pentadecanoic Acid (C15:0)		0.044 ± 0.006
Hexadecanoic Acid (C16:0)	Palmitic Acid	4.92 ± 0.80
(*Z*)-9-Hexadecenoic Acid (C16:1 n-7)	Palmitoleic Acid	0.162 ± 0.040
Heptadecanoic Acid (C17:0)	Margaric Acid	0.049 ± 0.012
Octadecacanoic Acid (C18:0)	Stearic Acid	1.53 ± 0.24
(*Z,Z*)-9,12-Octadecadienoic Acid (C18:2 n-6)	Linoleic Acid	6.16 ± 0.89
Eicosanoic Acid (C20:0)	Arachidic Acid	0.074 ± 0.018
Tetracosanoic Acid (C24:0)	Lignoceric Acid	0.028 ± 0.005
Total Trans C18:1 and C18:2 Fatty Acids		0.252 ± 0.063
Total Polyunsaturated Fatty Acids		6.99 ± 0.69
Total Saturated Fatty Acids		8.31 ± 0.39
Total Trans Fatty Acids		0.336 ± 0.087
Total Omega-3 Fatty Acids		0.930 ± 0.072

	Mass Fraction[a] (mg/100 g)
Cholesterol	40.4 ± 2.7

	Mass Fraction[a] (kcal/100 g)
Calories[b]	490 ± 20

[a] These values are expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence.

[b] The value for calories is the weighted median of lab mean caloric calculations from the interlaboratory comparison exercise. If the mean proximate values are used for calculation, with caloric equivalents of 9, 4, and 4 for fat (total), protein, and carbohydrate, respectively, the mean caloric content is 491 kcal/100 g.

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **01 July 2030**. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Table B1. Value of Potential Interest for Silicon in SRM 1548b

	Mass Fraction[a] (mg/kg)
Silicon (Si)[a]	80

[a] The mass fraction value for silicon may be of interest to the SRM user; however, insufficient information is available to assess the uncertainty associated with this value and therefore no uncertainty is provided.

Source and Preparation: SRM 1548b is a blended freeze-dried homogenate of foods served in a typical cafeteria, based loosely on the recipe used for SRM 1548a Typical Diet [6]. Each food item was individually frozen prior to blending and freeze drying by Apex Lyo, Inc (Eugene, OR). The bulk material was cryogenically homogenized at the NIST Reference Material Production Facility (Charleston, SC). The material was blended, aliquoted, and heat-sealed inside nitrogen-flushed 4 mil polyethylene bags by High-Purity Standards (Charleston, SC), then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel each. The packaged material was irradiated at Neutron Products, Inc. (Dickerson, MD) by ^{60}Co to an absorbed dose of 6.8 kGy to 7.5 kGy.

Elements: Value assignment of the mass fractions of the elements in SRM 1548b was based on the combination of results from measurements made by NIST and collaborating laboratories, where available, as described in Table B2.

Table B2. Methods Used in Value Assignment for Elements in SRM 1548b[a]

Element	NIST Method(s)	Method(s) Reported by Collaborating Laboratories[b]
Aluminum (Al)	INAA	ICP-OES
Arsenic (As)	ICP-MS	ICP-MS[b]
Barium (Ba)	ICP-MS, ICP-OES, INAA	
Boron (B)	PGAA	
Cadmium (Cd)	ICP-MS, ID ICP-MS	ICP-MS[b]
Calcium (Ca)	ICP-OES, WDXRF	ICP-OES, ICP-MS[b]
Cesium (Cs)	INAA	
Chlorine (Cl)	PGAA, WDXRF	Other
Copper (Cu)	ICP-MS, ICP-OES, WDXRF	ICP-OES, ICP-MS
Iron (Fe)	ICP-OES, INAA, WDXRF	ICP-OES, ICP-MS[b]
Iodine (I)	ICP-MS	[b]
Lead (Pb)	ICP-MS, ID ICP-MS	ICP-MS[b]
Magnesium (Mg)	ICP-OES, WDXRF	ICP-OES, ICP-MS[b]
Manganese (Mn)	ICP-OES, WDXRF	ICP-OES, ICP-MS
Molybdenum (Mo)	ICP-MS	ICP-MS
Nitrogen (N)	Combustion	
Nickel (Ni)	ICP-MS	ICP-MS
Phosphorus (P)	ICP-OES, WDXRF	ICP-OES, ICP-MS[b]
Potassium (K)	ICP-OES, WDXRF	ICP-OES, ICP-MS[b]
Rubidium (Rb)	INAA	
Sulfur (S)	PGAA, WDXRF	
Selenium (Se)	ICP-MS, INAA	ICP-MS[b]
Silicon (Si)	WDXRF	
Sodium (Na)	ICP-OES, WDXRF	ICP-OES, ICP-MS[b]
Strontium (Sr)	ICP-MS, ICP-OES, INAA	
Tin (Sn)	ICP-MS	
Zinc (Zn)	ICP-OES, INAA, WDXRF	ICP-OES, ICP-MS[b]

[a] ICP-OES: inductively coupled plasma optical emission spectrometry; ICP-MS: inductively coupled plasma mass spectrometry; ID ICP-MS: isotope dilution inductively coupled plasma mass spectrometry; INAA: instrumental neutron activation analysis; PGAA: prompt gamma-ray activation analysis; WDXRF: wavelength dispersive X-ray fluorescence spectrometry

[b] Not all laboratories reported methods used; for iodine, no method information was reported.

NIST Analysis for N by Combustion: The mass fraction of nitrogen was determined by combustion with a CHNOS Elemental Analyzer Elementar vario MACRO cube in the CHNS-S mode using a thermal conductivity detector with helium gas as a carrier to aid in the combustion of the samples. Duplicate 0.1 g aliquots were taken from each of six packets and SRM 143d *Cystine (L-Cystine)* was used as a calibrant.

NIST Analyses for As, Ba, Ca, Cd, Cu, Fe, I, K, Mg, Mn, Mo, Ni, P, Pb, Na, Se, Sr, Sn, and Zn by ICP-OES and/or ICP-MS: Mass fractions of barium, calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, strontium, and zinc were determined by ICP-OES from duplicate 0.5 g test portions taken from each of ten packets of SRM 1548b. Samples were digested in nitric acid in closed vessels in a microwave digestion system. For the determination of mass fractions of arsenic, barium, cadmium, copper, lead, molybdenum, nickel, selenium, strontium, and tin by ICP-MS, duplicate 0.5 g test portions were taken from each of ten packets of SRM 1548b and were digested in a nitric acid/hydrofluoric acid mixture in closed vessels in a microwave digestion system. For the determination of iodine by ICP-MS, duplicate 0.5 g test portions were taken from each of ten packets of SRM 1548b. Iodine was extracted from the samples using ammonium hydroxide. Quantification for all elements was based on the method of standard additions using the SRM 3100 series single element standard solutions.

NIST Analyses for Cd and Pb by ID ICP-MS: Mass fractions of cadmium and lead were determined by ID ICP-MS using duplicate, nominal 1 g test portions taken from each of four to six packets of SRM 1548b. Samples were spiked with isotopically enriched ^{111}Cd and ^{206}Pb and were digested in a nitric/hydrofluoric acid mixture using a microwave sample preparation system. For lead, sample digests were evaporated to near dryness and a portion was reconstituted in dilute nitric acid for analysis in standard mode. Cadmium was separated from the matrix using anion exchange chromatography and measured in collision cell/kinetic energy discrimination mode. Quantification was based on standard solutions prepared from SRM 746 *Cadmium -Vapor Pressure*, SRM 3108 *Cadmium (Cd) Standard Solution*, SRM 3128 *Lead (Pb) Standard Solution*, and from Pb metal (Johnson Matthey, 0.99999 % pure).

NIST Analyses for Al, Ba, Cs, Fe, Rb, Se, Sr, and Zn by INAA: Mass fractions of barium, cesium, iron, rubidium, selenium, strontium, and zinc were determined by INAA from single 0.2 g test portions taken from each of 12 packets of SRM 1548b. Samples, standards, and controls were packaged individually in clean polyethylene bags and irradiated individually at 20 MW for 8 h with a 180-degree inversion after 4 h. Nuclides were counted for 24 h after decays of more than 14 d for cesium, iron, rubidium, selenium, strontium, and zinc; for 2 h after a decay of 7 d for barium; and for 5 min after a decay of 5 min for aluminum. Quantification was based on pure elements or compounds on compressed filter papers and foil fluence monitors and on the SRM 3100 series single element standard solutions.

NIST Analyses for B, Cl, and S Using PGAA: Mass fractions of boron, chlorine, and sulfur were determined by PGAA from individual pellets prepared from duplicate 1 g to 1.5 g test portions taken from each of six packets of SRM 1548b. Samples, controls, and standards (prepared from SRM 3107 *Boron (B) Standard Solution*, SRM 143d *Cystine (L-Cystine)*, SRM 920 *D-Mannitol*, SRM 912a *Urea*, sodium sulfate (Alpha Puratronic 99.9955 %, metals basis), and graphite (Spectrographic Services, 200 mesh)) were packaged individually in clean Teflon bags and irradiated individually for less than 1 h. Gamma-ray spectra up to 10 MeV were collected, and the gamma-ray signal was monitored at 477 keV for boron, at 841 keV for sulfur, and at 785 keV + 788 keV (a doublet) for chlorine. These signals were compared to that of the standards to determine the mass fractions of the analytes.

NIST Analyses for Ca, Cl, Cu, Fe, K, Mg, Mn, Na, P, S, Si, and Zn using WDXRF: Mass fractions of calcium, chlorine, copper, iron, magnesium, manganese, phosphorous, potassium, sulfur, silicon, sodium, and zinc were determined using WDXRF analyses by weighing single, nominal 4.0 g test portions taken from each of 12 packets of SRM 1548b and pressing the portions into briquettes. Calibration standards in briquette form were prepared from a suite of NIST SRMs for food and vegetable matter. The $KL_{2,3}$ characteristic Xray lines of all elements were measured for quantification with net count rates used for Al, Cl, Cu, Fe, Mg, Mn, Na, and Zn and gross count rates for Ca, K, P, S, and Si. Quantification of all elements was based on calculated calibration curves with appropriate matrix absorption correction factors.

Amino Acids: Value assignment of the mass fractions of amino acids in SRM 1548b was based on the combination of measurements made by collaborating laboratories. Collaborating laboratories reporting amino acids data used acid digestion and hydrolysis followed by liquid chromatography with absorbance detection.

Ash and Solids: Value assignment of the mass fractions of ash and solids in SRM 1548b was based on the results from measurements made by NIST.

NIST Analyses for Ash: The mass fraction of ash was determined by TGA (LECO Model 701) and muffle furnace from duplicate, nominal 1 g test portions taken from each of six packets of SRM 1548b. Samples were held for 3 h at 575 °C and the residual mass determined. Quantitation was based on the standard mass set maintained by the NIST Inorganic Chemical Metrology Group.

NIST Analyses for Solids: The mass fraction of solids was determined by vacuum oven from nominal 1 g test portions taken from each of 12 packets of SRM 1548b. Samples were held for 24 h at room temperature in a vacuum oven and measurements were repeated three times over a one year period. The mass fraction of solids was also determined by freeze dryer to constant mass over 7 d using duplicate, nominal 2 g test portions taken from each of 6 packets. Quantitation was based on the standard mass set maintained by the NIST Inorganic Chemical Metrology Group.

Additional Proximates, Calories, Cholesterol, Fatty Acids, and Glyphosate: Value assignment of the mass fractions of remaining measurands in SRM 1548b was based on the combination of results from measurements made by collaborating laboratories, as described in Table B3.

Table B3. Methods Used in Value Assignment for Additional Measurands in SRM 1548b[(a)]

Proximate	Method(s) Reported by Collaborating Laboratories[(b)]
Carbohydrates	Calculated as solids – (protein + fat + ash)
Extracted Fat	Acid digestion/hydrolysis, Mojonnier[(b)]
Total Fatty Acids	Acid digestion/hydrolysis, derivatization, sum of fatty acids[(b)]
Protein	Calculated from nitrogen results (N x 6.25) by combustion, Kjeldahl[(b)]
Total Sugars	Extraction with LC-ELSD or LC-RI
Fructose	Extraction with LC-ELSD or LC-RI
Sucrose	Extraction with LC-ELSD or LC-RI
Glucose	Extraction with LC-ELSD or LC-RI
Total Fiber	[(b)]
Calories	Calculated as (9 x fat) + (4 x protein) + (4 x carbohydrate)
Cholesterol	Saponification, extraction, and derivatization with GC-FID[(b)]
Fatty Acids	Acid digestion/hydrolysis with GC-FID[(b)]
Glyphosate	Acid hydrolysis, QuPPe, solvent extraction/SPE/derivatization; LC-MS or LC-MS/MS [9]

[(a)] GC-FID: gas chromatography with flame ionization detection; LC-ELSD: liquid chromatography with evaporative light scattering detection; LC-MS: liquid chromatography mass spectrometry; LC-MS/MS: liquid chromatography with tandem mass spectrometry LC-RI: liquid chromatography with refractive index detection; QuPPe: quick polar pesticides; SPE: solid phase extraction

[(b)] Not all laboratories reported methods used; for total fiber, no method information was reported.

Collaborating Laboratories' Analyses: The collaborating laboratories were asked to use their usual methods to make duplicate measurements on test portions taken from each of two packets of SRM 1548b. Because of the variability among data provided by laboratories participating in an interlaboratory comparison exercise, the weighted median of the individual laboratory means is used based on a Laplace random effects model [8]. The uncertainty is estimated using a bootstrap procedure based on Laplace random effects model for the between-lab and within-lab effects [4,7,8]. Collaborating laboratories' data alone were used to assign non-certified values for proximates, sugars, fatty acids, amino acids, and glyphosate.

Homogeneity Assessment: The homogeneity of elements and select proximates was assessed at NIST using the methods and test portion sizes described above; analysis of variance with 5 % significance showed possible inhomogeneity for moisture, protein, ash, molybdenum, and nitrogen. The uncertainty for these analytes incorporates a component for inhomogeneity based on the standard deviation. Homogeneity of constituents measured solely by collaborating laboratories (e.g., fatty acids, amino acids) was not assessed, although the data were treated as though these analytes were homogeneously distributed.

Value Assignment: For calculation of assigned values for analytes that were measured only by NIST, the mean of the mean values from NIST results were used. For calculation of assigned values for analytes that were measured only by the collaborating laboratories, the weighted median of the laboratory means was used. For analytes that were also measured by NIST, the means of the individual sets of NIST data were averaged with the weighted median of the individual collaborating laboratory means, as appropriate.

* * * * * * * * * * End of Appendix B * * * * * * * * * *

APPENDIX C

Contributors to Value Assignment of SRM 1548b

Coordination of Technical Measurements

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Analytical Measurements at NIST

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Collaborating Laboratories

NIST Total Nutrients Quality Assurance Program Interlaboratory Comparison Exercise

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Eurofins Nutritional Analysis Center (Des Moines, IA)
Eurofins Steins (Vejen, Denmark)
Krueger Food Laboratories, Inc. (Chelmsford, MA)
Land O'Lakes (Arden Hills, MN)
Mérieux NutriSciences (Crete, IL)
Nestle Quality Assurance Center (Dublin, OH)
Silliker JR Laboratories (Burnaby, BC, Canada)

NIST Food Nutrition and Safety Measurements Quality Assurance Program (FNSQAP) Exercise 1

Canadian Food Inspection Agency (Calgary, AB, Canada)
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University of Hawaii at Manoa (Honolulu, HI)

High-Purity Standards (Charleston, SC)

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