National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2387

Peanut Butter

This Standard Reference Material (SRM) is intended primarily for use in validating methods for determining proximates, fatty acids, calories, vitamins, elements, amino acids, and aflatoxins in peanut butter and similar matrices. This SRM can also be used for quality assurance when assigning values to in-house reference materials. The SRM is a creamy peanut butter prepared by a manufacturer of peanut butter products. A unit of SRM 2387 consists of three jars of peanut butter containing 170 g each.

Certified Mass Fraction Values: Certified mass fraction values for fat, elements, and tocopherols in SRM 2387 are provided in Tables 1 through 3. 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 [1]. Analyses for value assignment were performed by NIST and collaborating laboratories. Certified mass fraction values in this material were calculated as the mean of the mean values from NIST methods and the mean of the measurements made by collaborating laboratories, where appropriate. The associated uncertainties are expressed at an approximately 95 % level of confidence [2-4]. Values are reported on an as-received (not dry-mass) basis in mass fraction units [5].

Reference Mass Fraction Values: Reference mass fraction values for proximates, fatty acids, amino acids, calories, total dietary fiber, vitamins, and aflatoxins are provided in Tables 4 through 8. 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 [1] and is provided with an associated uncertainty 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. Reference mass fraction values were derived from results reported by NIST and collaborating laboratories. Values are reported on an as-received (not dry-mass) basis in mass fraction units [5].

Expiration of Certification: The certification of **SRM 2387** is valid, within the measurement uncertainty specified, until **31 December 2029**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). This 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 M.M. Phillips, K.E. Sharpless, and L.J. Wood of the NIST Chemical Sciences Division and H.B. Chin, I-P. Ho, and D.W. Howell of the National Food Processors Association (NFPA, Dublin, CA, and Washington, DC).

Analytical measurements at NIST were performed by C.Q. Burdette, C.S. Phinney, B.J. Porter, K.E. Sharpless, and L.J. Wood of the NIST Chemical Sciences Division.

Statistical analysis was provided by J.H. Yen of the NIST Statistical Engineering Division.

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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Analyses for value assignment were also performed by the following laboratories participating in a NFPA Food Industry Analytical Chemists Subcommittee (FIACS) interlaboratory comparison exercise: Beech-Nut Nutrition Corporation (Canajoharie, NY); Campbell Soup Company (Camden, NJ); Covance, Inc. (Madison, WI); General Mills, Inc. (Minneapolis, MN); Hormel Foods Corporation (Austin, MN); Kraft Foods (Glenview, IL); Krueger Food Laboratories, Inc. (Cambridge, MA); Nabisco, Inc. (East Hanover, NJ); Nestlé USA (Dublin, OH); Novartis Nutrition Corporation (St. Louis Park, MN); Ralston Purina Company (St. Louis, MO); U.S. Department of Agriculture, Food Composition Laboratory (Beltsville, MD); and Woodson-Tenent Laboratories (Memphis, TN). Additional laboratories providing measurements for value assignment of aflatoxins included: U.S. Food and Drug Administration (Atlanta, GA); Neogen Corporation (Lansing, MI); U.S. Department of Agriculture, Agricultural Marketing Service (Blakely, GA); and Trilogy Analytical Laboratory (Washington, MO).

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

NOTICE TO USERS

SRM 2387 IS INTENDED FOR LABORATORY USE ONLY, NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR STORAGE AND USE

Storage: Until required for use, the peanut butter should be frozen at –20 °C.

Use: Prior to removal of a test portion for analysis, a jar of peanut butter should be thawed under refrigeration overnight. The contents of the jar should be mixed thoroughly prior to removal of a test portion. The following masses used for NIST analyses should be used as the minimum sample size to ensure valid results: 5 g to 7 g for tocopherols; 1 g for fat and fatty acids; 0.5 g for elements.

SOURCE, PREPARATION, AND ANALYSIS[(1)]

Source and Preparation: SRM 2387 is creamy peanut butter containing roasted peanuts, sugar, partially hydrogenated vegetable oils (48 % rapeseed, 40 % cottonseed, and 12 % soybean oil), and salt, and was prepared for NIST as part of a larger production run. Raw, shelled Florunner (primarily) peanuts were received from several suppliers and were roasted. The skins were removed from the roasted peanuts, and discolored peanuts were discarded. The roasted peanuts were then ground, and the remaining ingredients were added. After mixing, the peanut butter was further ground to a fine particle size, air was removed, and the peanut butter was cooled and packed in colorless polyethyl tetraethylene (PETE) jars with white screw caps and foil liners.

NIST Analyses for Fat: The mass fraction of gravimetric fat was measured from one set of three samples of peanut butter. One-gram portions of peanut butter were mixed with diatomaceous earth. The mixture was then briefly chilled at 4 °C to improve handling. The fat was then extracted from the mixture by pressurized fluid extraction (PFE) using hexane:acetone (4:1 volume fraction). Extracts were evaporated under nitrogen and dried at 100 °C to constant mass.

NIST Analyses for Ca, Cu, Fe, K, Mg, Mn, Na, P, and Zn: Mass fractions of calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, and zinc were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) in eight jars of peanut butter. Two 0.5 g portions were taken from each jar and digested in a nitric, perchloric, and hydrofluoric acid mixture. Because of the high fat content, the samples were predigested on a hot plate before digestion in a microwave oven. Digests were transferred to plastic bottles and diluted with the appropriate volume of 1.5 % (volume fraction) nitric acid. To correct for matrix effects caused by differences between samples and calibrants, the method of standard additions was used; spikes were added to one aliquot prepared from each 0.5 g test portion. Four measurements using ICP-OES were made and averaged for each sample and each spiked solution. Results were corrected for spike recoveries. Quantitation was based on the method of standard additions using SRM 3100 series single element standard solutions.

NIST Analyses for Fatty Acids: Mass fractions of fatty acids were measured by gas chromatography with flame ionization detection (GC-FID) in two sets of six samples of peanut butter prepared on two different days. The fat was extracted from approximately 1 g samples of peanut butter by PFE using a mixture of hexane:acetone (4:1 volume fraction). Methyl nonadecanoate (C19:0 fatty acid methyl ester [FAME]) was used as an internal standard. A two-step process employing methanolic sodium hydroxide and boron trifluoride was used to convert fatty acids to their

[(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.

methyl esters. FAMEs were extracted into hexane and samples were analyzed by GC-FID. Calibrants were prepared gravimetrically from SRM 2377 *Fatty Acid Methyl Esters in 2,2,4 Trimethylpentane* at levels intended to approximate the levels of the fatty acids in the SRM following extraction. A single internal standard solution was used for the calibrants and samples. Calculations were based on average response factors for the calibrants.

NIST Analyses for δ-Tocopherol and γ- (plus β-) Tocopherol: Mass fractions of δ-tocopherol and γ- (plus β-) tocopherol were measured using liquid chromatography with absorbance (LC-Abs) and fluorescence (FL) detection in test portions taken from six jars of peanut butter over a seven-day period. The peanut butter may contain β-tocopherol, but the chromatographic system described below is incapable of resolving β- and γ-tocopherol. Samples of approximately 5 g to 7 g were homogenized and saponified using potassium hydroxide. Analytes were extracted into a mixture of diethyl ether and hexane, which was subsequently evaporated, and the analytes were redissolved in a mixture of ethanol and ethyl acetate. Samples were analyzed by LC using a C18 column using a gradient of acetonitrile, methanol, and ethyl acetate [6] and with absorbance monitored at 450 nm for measurement of *trans*-β-apo-10'-carotenal oxime (the internal standard) and fluorescence excitation at 295 nm and monitored at an emission wavelength of 335 nm for quantitation of the tocopherols. Calibrants of δ- and γ-tocopherol were prepared gravimetrically, at levels intended to approximate the levels of the vitamins in the SRM following extraction, and the concentrations were assigned spectrophotmetrically. A single internal standard solution was used for the calibrants and samples.

NIST Analyses for α-Tocopherol: Mass fractions of α-tocopherol were measured using LC-FL in test portions taken from six jars of peanut butter on a single day. Samples of approximately 5 g were homogenized and saponified using potassium hydroxide. Analytes were extracted into a mixture of ethyl acetate and hexane using rotary mixing and sonication, and the supernatants from five sequential extractions were combined. The solvent from the combined supernatant was subsequently evaporated, and the analytes were redissolved in a mixture of ethanol and ethyl acetate. Samples were analyzed by LC using a C18 column using an isocratic flow of 99:1 methanol:water and with fluorescence excitation at 295 nm and monitored at an emission wavelength of 330 nm for quantitation of α-tocopherol, using tocol as an internal standard. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the vitamins in the SRM following extraction, and the concentrations were assigned spectrophotmetrically. A single internal standard solution was used for the calibrants and samples.

Analyses by Collaborating Laboratories: Data from additional sources were used for value assignment, including an interlaboratory comparison exercise organized by the NFPA FIACS and four laboratories participating in an exercise in which only aflatoxins were measured. The NFPA FIACS laboratories were asked to use AOAC methods or their equivalent, to make single measurements from each of two jars, and to report the analytical method that was used. The laboratories measuring aflatoxins were asked to use their usual methods to make single measurements in each of three jars.

Homogeneity Assessment: The homogeneity of elements, fatty acids, and tocopherols was assessed at NIST using the methods described above. A small but statistically significant heterogeneity was found for some analytes, and an inhomogeneity component of approximately 5.6 % has been incorporated in the uncertainty for α-tocopherol and an inhomogeneity component of approximately 1 % has been incorporated in the uncertainty for all other analytes.

Value Assignment: The collaborating laboratories reported values for two to twelve analyses for a given analyte. The mean of each laboratory's results was then determined. For calculation of assigned values for analytes that were measured only by the collaborating laboratories, the mean of laboratory means was determined. For analytes that were also measured by NIST, this mean of the individual collaborating laboratory means and the means of the individual sets of NIST data were averaged.

Certified Mass Fraction Value for Fat: The certified mass fraction value is the combined mean from the mean of results from analyses by NIST and the mean of results provided by collaborating laboratories. The value is 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, the certified value should be treated as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2-3]. The measurand is the total mass fraction of extractable fat in peanut butter as listed in Table 1 on an as-received basis. Metrological traceability is to the measurement processes and standards used by NIST and collaborating laboratories.

Table 1. Certified Mass Fraction Value for Total Extractable Fat in SRM 2387

	Mass Fraction (g/100 g)
Fat (extractable)	51.6 ± 1.4

Certified Mass Fraction Values for Elements: Each certified mass fraction value is the combined mean from the mean of results from analyses by NIST and the mean of results provided by collaborating laboratories. 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, the certified value should be treated as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2-3]. The measurands are the total mass fractions of elements in peanut butter as listed in Table 2 on an as-received basis. Metrological traceability is to the SI measurement unit for chemical mass fraction, expressed as milligrams per kilogram.

Table 2. Certified Mass Fraction Values for Elements in SRM 2387

	Mass Fraction (mg/kg)
Calcium (Ca)	411 ± 18
Copper (Cu)	4.93 ± 0.15
Iron (Fe)	16.4 ± 0.8
Magnesium (Mg)	1680 ± 70
Manganese (Mn)	16.0 ± 0.6
Phosphorus (P)	3378 ± 92
Potassium (K)	6070 ± 200
Sodium (Na)	4890 ± 140
Zinc (Zn)	26.3 ± 1.1

Certified Mass Fraction Values for Tocopherols: Each certified mass fraction value is the combined mean from the mean of results from analyses by NIST and the mean of results provided by collaborating laboratories. 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 [2-3]. To propagate this uncertainty, the certified value should be treated as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/2$ [2-3]. The measurands are the total mass fractions of tocopherols in peanut butter as listed in Table 3, on an as-received basis, as determined by the method indicated. Metrological traceability is to the SI measurement unit for chemical mass fraction, expressed as milligrams per kilogram, through the molar absorptivities of the compounds.

Table 3. Certified Mass Fraction Values for Tocopherols in SRM 2387

	Mass Fraction (mg/kg)
δ-Tocopherol	10 ± 3
γ- + β-Tocopherol	100 ± 19

Reference Mass Fraction Values for Fatty Acids as Free Fatty Acids: Each reference mass fraction value is the combined mean from the mean of results from analyses by NIST and the mean of results provided by collaborating laboratories, as available. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2-3]. For fatty acids values containing NIST data, the uncertainty incorporates a component for possible inhomogeneity based on the standard deviation. The measurands are the total mass fractions of fatty acids in peanut butter as listed in Table 4, on an as-received basis, as determined by the method indicated. Metrological traceability is to the measurement processes and standards used by NIST and collaborating laboratories.

Table 4. Reference Mass Fraction Values for Fatty Acids as Free Fatty Acids in SRM 2387

	Common Name	Mass Fraction (g/100 g)
Tetradecanoic Acid (C14:0)[a,b]	Myristic Acid	0.024 ± 0.002
(Z)-9-Hexadecenoic Acid (C16:1 n-7)[a,b]	Palmitoleic Acid	0.044 ± 0.010
Heptadecanoic Acid (C17:0)[a]	Margaric Acid	0.048 ± 0.001
(Z)-9-Heptadecenoic Acid (C17:1 n-8) [a]	Margoleic Acid	0.033 ± 0.006
Octadecanoic Acid (C18:0)[a,b]	Stearic Acid	2.13 ± 0.08
(Z)-9-Octadecenoic Acid (C18:1 n-9)[a,b]	Oleic Acid	23.38 ± 0.90
(Z)-11-Octadecenoic Acid (C18:1 n-7)[a,b]	Vaccenic Acid	0.255 ± 0.016
(Z,Z)-9,12-Octadecadienoic Acid (C18:2 n-6)[a,b]	Linoleic Acid	13.15 ± 0.41
(Z,Z,Z)-9,12,15-Octadecatrienoic Acid (C18:3 n-3)[a,b]	α-Linolenic Acid	0.030 ± 0.001
Eicosanoic Acid (C20:0)[a,b]	Arachidic Acid	0.710 ± 0.029
(Z)-11-Eicosenoic Acid (C20:1 n-9)[a,b]	Gondoic Acid	0.643 ± 0.031
Eicosadienoic Acid (C20:2)[a]		0.016 ± 0.007
(Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic Acid (C20:4 n-6)[a]	Arachidonic Acid	0.024 ± 0.015
Docosanoic Acid (C22:0)[a,b]	Behenic Acid	1.81 ± 0.08
(Z)-13-Docosenoic Acid (C22:1 n-9)[a]	Erucic Acid	0.054 ± 0.012
Total Fat (as the sum of fatty acids as triglycerides)[a,b]		49.8 ± 1.9
Monounsaturated Fatty Acids[a,b]		24.4 ± 0.9
Polyunsaturated Fatty Acids[a,b]		13.2 ± 0.4

[a] Collaborating laboratories
[b] NIST GC-FID

Reference Mass Fraction Value for α-Tocopherol: The reference mass fraction value is the combined mean from the mean of results from analyses by NIST and the mean of results provided by a collaborating laboratory. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2-3]. The uncertainty also incorporates an additional uncertainty component for possible inhomogeneity. The measurand is the mass fraction of α-tocopherol in peanut butter as listed in Table 5, on an as-received basis, as determined by the methods indicated. Metrological traceability is to the measurement processes and standards used by NIST and collaborating laboratories.

Table 5. Reference Mass Fraction Value for α-Tocopherol in SRM 2387

	Mass Fraction (mg/kg)		
α-Tocopherol	73.7	±	8.4

Reference Mass Fraction Values for Proximates and Calories: Each reference mass fraction value is the mean of results provided by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2]. For proximates and fiber, the measurands are the mass fractions of proximates and fiber in peanut butter listed in Table 6, on an as-received basis, as determined by the methods indicated. For calories, the measurand is the caloric content in peanut butter listed in Table 6, on an as-received basis, as determined by the methods indicated. Metrological traceability is to the measurement processes and standards used by collaborating laboratories.

Table 6. Reference Mass Fraction Values for Proximates and Calories in SRM 2387

	Mass Fraction (g/100 g)		
Solids	99.2	±	2.1
Ash	3.10	±	0.10
Protein[(a)]	22.2	±	0.5
Carbohydrates	25.0	±	1.8
Total Dietary Fiber	5.57	±	0.42
	Energy (kcal per 100 g)		
Calories[(b)]	629	±	15

[(a)] A factor of 5.46 was used to convert nitrogen results to protein.

[(b)] The reference value for calories is the mean of mean caloric calculations from the interlaboratory comparison exercise. If the mean proximate values above are used for calculation, with caloric equivalents of 9, 4, and 4 for fat (as the sum of fatty acids as triglycerides), protein, and carbohydrate, respectively, the mean caloric content is 637 kcal per 100 grams.

Reference Mass Fraction Values for Amino Acids: Each reference mass fraction value is the mean of results provided by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2]. The measurands are the mass fractions of amino acids in peanut butter as listed in Table 7, on an as-received basis, as determined by the collaborating laboratories. Metrological traceability is to the measurement processes and standards used by collaborating laboratories.

Table 7. Reference Mass Fraction Values for Amino Acids in SRM 2387

	Mass Fraction (g/100 g)
Alanine	0.93 ± 0.10
Arginine	2.65 ± 0.31
Aspartic Acid	2.83 ± 0.19
Cystine	0.27 ± 0.01
Glutamic Acid	4.69 ± 0.26
Glycine	1.41 ± 0.12
Histidine	0.55 ± 0.06
Isoleucine	0.77 ± 0.07
Leucine	1.56 ± 0.09
Lysine	0.78 ± 0.08
Methionine	0.21 ± 0.04
Phenylalanine	1.21 ± 0.08
Proline	0.96 ± 0.08
Serine	1.16 ± 0.09
Threonine	0.54 ± 0.08
Tryptophan	0.21 ± 0.06
Tyrosine	0.81 ± 0.14
Valine	0.94 ± 0.09

Reference Mass Fraction Values for Aflatoxins: Each reference mass fraction value is the mean of results provided by collaborating laboratories. Values are expressed as $x \pm U_{95\%}(x)$, where x is the estimated value and $U_{95\%}(x)$ is the expanded uncertainty of the value. The method-specific value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with about a 95 % confidence [2]. The measurands are the mass fractions of aflatoxins in peanut butter as listed in Table 8, on an as-received basis, as determined by the collaborating laboratories. Metrological traceability is to the measurement processes and standards used by collaborating laboratories.

Table 8. Reference Mass Fraction Values for Aflatoxins in SRM 2387

	Mass Fraction (ng/g)
Aflatoxin B_1	4.2 ± 0.9
Aflatoxin B_2	0.7 ± 0.3
Total Aflatoxins[(a)]	5.0 ± 0.5

[(a)] The reference value for total aflatoxins is the mean of the laboratory means of the sum of aflatoxins B_1 and B_2.

REFERENCES

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Certificate Revision History: 15 January 2020 (Change of expiration date; removal of certified values for palmitic acid, lignoceric acid, and total saturated fatty acids based on observed instability; removal of reference value for acrylamide based on observed instability; removal of reference values for thiamine, riboflavin, niacinamide, niacin, total vitamin B_3, pantothenic acid, pyridoxamine, pyridoxal, pyridoxine, and total vitamin B_6 based on NIST's decision to no longer support these measurement capabilities in this matrix; certified values for fatty acids and total fats as the sum of fatty acids downgraded to reference values to properly reflect traceability and moved from Table 1 to Table 4; editorial changes); **21 July 2015** (Addition of water-soluble vitamin values; removal of certified value for α-tocopherol; addition of reference value for α-tocopherol; editorial changes); **23 September 2014** (Extension of certificate period; editorial changes); **30 September 2009** (Extension of certificate period); **12 January 2007** (editorial changes); **29 September 2004** (Addition of a reference value for acrylamide); **14 March 2003** (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; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.