



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2383a

#### Baby Food Composite

This Standard Reference Material (SRM) is intended primarily for use in validating methods for determining proximates, calories, vitamins, and elements in food matrices. This SRM can also be used for quality assurance when assigning values to in-house control materials. The baby food composite is a mixture of fruits, vegetables, macaroni, rice flour, and milk powder. A unit of SRM 2383a consists of four jars, each containing approximately 70 g of material.

**Certified Mass Fraction Values:** The certified mass fraction values of selected vitamins and elements are provided in Tables 1 and 2, respectively. 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 collaborating laboratories. Certified values were calculated as the mean of the mean values from NIST methods and the median of the mean of results provided by collaborating laboratories, where appropriate. All values were combined without weighting. The associated uncertainties are expressed at the 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 are also provided for additional vitamins (Table 3); elements (Table 4); and proximates, sugars, total dietary fiber, and calories (Table 5). 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 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 and/or collaborating laboratories. Values are reported on an as-received (not dry-mass) basis in mass fraction units [5].

**Information Mass Fraction Values:** Information mass fraction values for several elements are provided in Table 6. 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, therefore no uncertainty is provided [1]. Values are reported on an as-received (not dry-mass) basis in mass fraction units [5].

**Expiration of Certification:** The certification of **SRM 2383a** is valid, within the measurement uncertainty specified, until **01 June 2018**, provided the SRM is handled and stored in accordance with 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 K.E. Sharpless and L.J. Wood of the NIST Analytical Chemistry Division and S. Ehling of the Grocery Manufacturers Association (GMA, Washington, DC).

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Analyses at NIST were performed by C. Bryan, W.C. Davis, R. Oflaz, D.J. O'Kelly, T.O. Okumu, Y. Nuevo Ordóñez, R.L. Paul, M.M. Phillips, B.E. Tomlin, and L.J. Wood of the NIST Analytical Chemistry Division.

Analysts at the following laboratories performed measurements that contributed to the value assignment of nutrients in SRM 2383a Baby Food Composite as part of the GMA interlaboratory comparison exercise: Campbell Soup Company, Camden, NJ; ConAgra Foods Analytical Laboratory, Omaha, NE; Covance Laboratories, Madison, WI; Del Monte Foods, Walnut Creek, CA; Eurofins Central Analytical Laboratories, Metairie, LA; Eurofins Scientific, Inc., Des Moines, IA; Eurofins Strasburger & Siegel, Hanover, MD; General Mills, Inc./Medallion Laboratories, Minneapolis, MN; Hormel Foods Corporation, Austin, MN; Krueger Food Labs, Billerica, MA; Land O' Lakes, Arden Hills, MN; Nestlé QA Center, Dublin, OH; Silliker, Inc., Chicago Heights, IL; The National Food Laboratory, Livermore, CA; and The Schwan Food Company, Salina, KS.

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 Measurement Services Division.

## NOTICE TO USERS

SRM 2383a IS INTENDED FOR LABORATORY USE ONLY, NOT FOR HUMAN CONSUMPTION.

## INSTRUCTIONS FOR STORAGE AND USE

**Storage:** The SRM should be stored under refrigeration at a temperature between 2 °C and 8 °C, in the dark, and in the original sealed jars. The certification does not apply to contents of previously opened jars that have been stored for more than one day as the stability of all analytes beyond this period has not been investigated.

**Use:** Before use, the contents of the jar should be mixed thoroughly. Test portions used for NIST vitamin analyses described below were 10 g. Test portions used for NIST elemental analyses ranged from 1 g to 3.5 g. Results obtained in users' analyses should include their own estimates of uncertainty and can be compared to the certified values using procedures described in reference 6.

## SOURCE, PREPARATION, AND ANALYSIS<sup>(1)</sup>

**Source and Preparation:** SRM 2383a is a mixture of the following ingredients (in order of decreasing mass): water, orange juice concentrate, corn, rice flour, papaya puree, spinach, macaroni, carrots, tomato paste, and non-fat milk powder. This material is different from its predecessor, SRM 2383; note that infant formula was not added to this material, therefore it contains no preformed vitamin A, nor does it contain other constituents that would be present in a fortified food product.

**Analytical Approach for Determination of Vitamins:** Value assignment of the mass fractions of the vitamins in SRM 2383a was based on the results of a single analytical method at NIST with confirmation by data provided by collaborating laboratories.

**NIST Analyses for Water-Soluble Vitamins:** Water-soluble vitamins were measured by using a liquid chromatography method with mass spectrometric detection (LC/MS). Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the vitamins in the SRM. In cases where an internal standard was employed, a single solution was used for the calibrants and samples.

Thiamine, riboflavin, niacinamide, niacin, pantothenic acid, pyridoxine, and pyridoxamine were measured by LC/MS in duplicate 10 g test portions taken from each of ten jars. Six internal standards were added: <sup>13</sup>C<sub>3</sub>-thiamine chloride; <sup>2</sup>H<sub>4</sub>-niacinamide; <sup>2</sup>H<sub>4</sub>-niacin; calcium <sup>13</sup>C<sub>3</sub>,<sup>15</sup>N-pantothenate; <sup>13</sup>C<sub>4</sub>-pyridoxine hydrochloride; and <sup>2</sup>H<sub>3</sub>-pyridoxamine dihydrochloride. The analytes and internal standards were extracted into dilute acetic acid for analysis by positive-ion mode LC/MS. A gradient method with an ammonium formate buffer/methanol mobile phase and a C<sub>18</sub> column were used. Thiamine and <sup>13</sup>C<sub>3</sub>-thiamine were measured at *m/z* 265 and *m/z* 268,

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<sup>(1)</sup> 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.

respectively. Niacinamide and  $^2\text{H}_4$ -niacinamide were measured at  $m/z$  123 and  $m/z$  127, respectively. Niacin and  $^2\text{H}_4$ -niacin were measured at  $m/z$  124 and  $m/z$  128, respectively. Pantothenic acid and  $^{13}\text{C}_3,^{15}\text{N}$ -pantothenic acid were measured at  $m/z$  220 and  $m/z$  224, respectively. Pyridoxine and  $^{13}\text{C}_4$ -pyridoxine were measured at  $m/z$  170 and  $m/z$  174, respectively. Pyridoxamine and  $^2\text{H}_3$ -pyridoxamine were measured at  $m/z$  169 and  $m/z$  171, respectively. Riboflavin was measured at  $m/z$  377, with  $^{13}\text{C}_4$ -pyridoxine as the internal standard.

**NIST Analytical Approach for Determination of Elements:** Value assignment of the mass fractions of the elements in SRM 2383a was based on the combination of measurements from at least two different analytical methods at NIST or a single NIST result and results obtained by collaborating laboratories, where available. NIST provided measurements by using inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), instrumental neutron activation analysis (INAA), and radiochemical neutron activation analysis (RNAA).

For the determination of calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, and zinc by ICP-OES, duplicate 3.5 g test portions were taken from each of ten jars of SRM 2383a and were digested in a nitric acid/hydrofluoric acid mixture in open beakers on a hotplate. For the determination of barium and strontium by ICP-OES, and the determination of barium, chromium, cobalt, molybdenum, nickel, strontium, and tin by ICP-MS, duplicate 1.5 g test portions were taken from each of ten jars of SRM 2383a and were digested in a nitric acid/hydrofluoric acid mixture in a microwave oven. For the determination of iodine by ICP-MS, single 1 g test portions were taken from six jars of SRM 2383a and from four other jars several months later. Samples were digested in aqueous tetramethylammonium hydroxide in a microwave oven. Quantitation for all of these analyses was based on the method of standard additions.

For the determination of chromium, cobalt, selenium, and zinc by INAA, material in ten jars of SRM 2383a was freeze dried and pulverized, and individual disks were prepared from 0.2 g test portions taken from the resulting powder. Standards were prepared from compounds of known purity. Samples, standards, and controls were packaged individually in clean polyethylene bags and irradiated individually at 20 MW for 8 hours. Nuclides were counted for 8 hours after decays of more than 120 days for chromium and 180 days for cobalt, selenium, and zinc. The following gamma-ray energies were used in the INAA analyses: 1173 keV and 1332 keV for the determination of cobalt, 320 keV for chromium, 136 keV and 265 keV for selenium, and 1115 keV for zinc.

For the determination of arsenic by RNAA, material in ten jars of SRM 2383a was freeze dried and pulverized, and individual disks were prepared from 0.2 g test portions taken from the resulting powder. Standards were prepared from SRM 3103a Arsenic (As) Standard Solution. Samples, standards, and controls were packaged individually in clean polyethylene bags and irradiated individually at 20 MW for 8 hours. Samples were combined with  $^{77}\text{As}$  prior to chemical separation. Samples were dissolved in a mixture of nitric and perchloric acids, and As separated from the matrix as described in reference 7. The 559 keV line from decay of  $^{76}\text{As}$  was used for quantitation. The 239 keV line from decay of  $^{77}\text{As}$  was evaluated for yield determination.

**Collaborating Laboratories' Analyses:** The GMA FIACC laboratories were asked to use their usual methods to make single measurements on test portions taken from each of two jars of SRM 2383a.

**Homogeneity Assessment:** The homogeneity of water-soluble vitamins and elements was assessed at NIST using the methods and test portion sizes described above; analysis of variance did not show statistically significant heterogeneity. All analytes have been treated as though they are homogeneously distributed in the material although the homogeneity of the other analytes that were measured by collaborating laboratories (e.g., proximates) was not assessed.

**Value Assignment:** The collaborating laboratories reported the individual results for each of their 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 median of the laboratory means was used. For water-soluble vitamins that were measured by NIST (except for riboflavin; see paragraph below), the average of the NIST values was used; results were confirmed by collaborating laboratories' data. For elements that were measured by NIST, the median of the individual collaborating laboratory means and the mean of the individual sets of NIST data were averaged, as appropriate.

**Certified Mass Fraction Values for Selected Water-Soluble Vitamins:** Each certified mass fraction value, except for riboflavin, is the mean from the analyses by NIST; these results were confirmed by data provided by collaborating laboratories. The variability of the riboflavin data was such that the collaborating laboratories' data did not confirm the veracity of the NIST mean, and the certified value for riboflavin is the mean of the NIST value and the median of the collaborating laboratories' means. 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, consistent with the ISO Guide and with its Supplement 1, and  $k$  is a coverage factor corresponding to approximately 95 % confidence [2-4].

Table 1. Certified Mass Fractions for Selected Water-Soluble Vitamins in SRM 2383a

Analyte	Mass Fraction (mg/kg)	$k$
Free Thiamine Chloride Hydrochloride	0.976 ± 0.014	2.09
Riboflavin	0.56 ± 0.15	2.00
Niacin	1.79 ± 0.04	2.09
Niacinamide	3.59 ± 0.06	2.09
Free Vitamin B <sub>3</sub> (as Niacinamide) <sup>(a)</sup>	5.36 ± 0.10	2.09
Pantothenic Acid	1.64 ± 0.02	2.09
Pyridoxine Hydrochloride	0.063 ± 0.002	2.09
Pyridoxamine Dihydrochloride <sup>(b)</sup>	0.228 ± 0.003	2.09
Free Vitamin B <sub>6</sub> (as Pyridoxine Hydrochloride)	0.330 ± 0.004	2.09

<sup>(a)</sup> Measured as the sum of niacinamide and niacin, which was mathematically converted to niacinamide by multiplication by the ratio of the relative molecular masses.

<sup>(b)</sup> Measured as the sum of pyridoxine and pyridoxal, which was mathematically converted to pyridoxine by multiplication by the ratio of the relative molecular masses.

**Certified Mass Fraction Values for Selected Elements:** Each certified mass fraction value is the mean from the combination of means of NIST data sets and the median of the mean results provided by collaborating laboratories. 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, consistent with the ISO Guide and with its Supplement 1, and  $k$  is a coverage factor corresponding to approximately 95 % confidence [2-4].

Table 2. Certified Mass Fractions for Selected Elements in SRM 2383a

Analyte	Mass Fraction (mg/kg)	$k$
Barium <sup>(a,b)</sup>	0.278 ± 0.020	2.00
Calcium <sup>(a,c)</sup>	342.6 ± 5.0	2.00
Cobalt <sup>(a,d)</sup>	0.048 ± 0.005	2.00
Copper <sup>(a,c)</sup>	0.758 ± 0.082	2.00
Iron <sup>(a,c)</sup>	4.42 ± 0.51	2.00
Magnesium <sup>(a,c)</sup>	212.2 ± 4.0	2.00
Manganese <sup>(a,c)</sup>	0.963 ± 0.064	2.00
Phosphorus <sup>(a,c)</sup>	453 ± 11	2.00
Potassium <sup>(a,c)</sup>	2910 ± 220	2.00
Sodium <sup>(a,c)</sup>	195 ± 29	2.00
Strontium <sup>(a,b)</sup>	4.445 ± 0.047	2.00
Zinc <sup>(a,c,d)</sup>	2.22 ± 0.18	2.00

<sup>(a)</sup> NIST ICP-OES

<sup>(b)</sup> NIST ICP-MS

<sup>(c)</sup> Collaborating laboratories

<sup>(d)</sup> NIST INAA

**Reference Mass Fraction Values for Selected Vitamins:** Each reference mass fraction value is the median of the mean results provided by collaborating laboratories using microbiological methods, thus the analytes' designations as 'total' vitamin. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  represents the combined uncertainty, consistent with the ISO Guide, and  $k$  is a coverage factor corresponding to approximately 95 % confidence [2].

Table 3. Reference Mass Fractions for Selected Water-Soluble Vitamins in SRM 2383a

Analyte	Mass Fraction (mg/kg)	$k$
Total Vitamin B <sub>5</sub> (Pantothenic Acid)	2.75 ± 0.42	2.78
Total Vitamin B <sub>6</sub> (Pyridoxine Hydrochloride)	0.710 ± 0.046	4.30

**Reference Mass Fraction Values for Selected Elements:** Each reference value is the mean of the results of a single NIST data set. 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  $u_c$  is the combined uncertainty, consistent with the ISO Guide, and  $k$  is a coverage factor corresponding to approximately 95 % confidence [2].

Table 4. Reference Mass Fractions for Selected Elements in SRM 2383a

Analyte	Mass Fraction (mg/kg)	$k$
Iodine <sup>(a)</sup>	0.0737 ± 0.0083	2.00
Selenium <sup>(b)</sup>	0.028 ± 0.001	2.09
Tin <sup>(a)</sup>	0.361 ± 0.019	2.10

<sup>(a)</sup> NIST ICP-MS

<sup>(b)</sup> NIST INAA

**Reference Values for Proximates and Calories:** Each reference mass fraction value is the median of the mean values provided by collaborating laboratories. The uncertainty provided with the 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  $u_c$  represents the combined uncertainty, consistent with the ISO Guide, and  $k$  is a coverage factor corresponding to approximately 95 % confidence [2].

Table 5. Reference Values for Proximates, Sugars, Total Dietary Fiber, and Calories in SRM 2383a

Analyte	Mass Fraction (%)	$k$
Solids	22.45 ± 0.68	2.16
Ash	0.705 ± 0.021	2.14
Protein	1.960 ± 0.060	2.16
Carbohydrates	19.41 ± 0.70	2.14
Total Sugars	12.05 ± 0.50	2.23
Sucrose	3.57 ± 0.12	2.23
Glucose	3.80 ± 0.11	2.23
Fructose	3.96 ± 0.09	2.26
Lactose	0.50 ± 0.10	2.36
Total Dietary Fiber	1.00 ± 0.22	2.31
	Energy (kcal per 100 g)	$k$
Calories <sup>(a)</sup>	89.0 ± 3.5	2.16

<sup>(a)</sup> The reference value for calories is the median of lab mean caloric calculations from the interlaboratory comparison exercise.

**Information Mass Fraction Values for Selected Elements:** Each information mass fraction value (except for chromium) is the mean result of a NIST analysis using a single method. The chromium value is the mean from two sets of NIST results. No uncertainty is provided because there is insufficient information available for its assessment.

Table 6. Information Mass Fraction Values for Selected Elements in SRM 2383a

Analyte	Mass Fraction (mg/kg)
Chromium <sup>(a,b)</sup>	0.2
Molybdenum <sup>(b)</sup>	0.06
Nickel <sup>(b)</sup>	0.2
Arsenic <sup>(c)</sup>	0.003

<sup>(a)</sup> NIST INAA

<sup>(b)</sup> NIST ICP-MS

<sup>(c)</sup> NIST RNAA

## REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Oct 2012).
- [2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at [http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Oct 2012); 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 <http://www.nist.gov/pml/pubs/tn1297/index.cfm> (accessed Oct 2012).
- [3] 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; Joint Committee for Guides in Metrology; International Bureau of Weights and Measures (BIPM), Sèvres, France (2008); available at [http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_101\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf) (accessed Oct 2012).
- [4] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall, London, UK (1993).
- [5] 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 <http://www.nist.gov/pml/pubs/sp811/index.cfm> (accessed Oct 2012).
- [6] Sharpless, K.E.; Duewer, D.L.; *Standard Reference Materials for Analysis of Dietary Supplements*; J. AOAC Int., Vol. 91, pp. 1298–1302 (2008).
- [7] Paul, R.L.; *Determination of Arsenic in Food and Dietary Supplement Standard Reference Materials by Neutron Activation Analysis*; Anal. Chem., Vol. 83, pp. 152-156 (2011).

*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 at: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*