National Institute of Standards & Technology

Report of Investigation

Reference Material 8435

Whole Milk Powder

A Joint Material of Agriculture Canada and NIST

Distributed by the National Institute of Standards and Technology

Reference Material (RM) 8435 is intended for use in evaluating analytical methods and instruments used for the determination of major, minor, and trace constituent elements, as well as proximates, fatty acids, calories, and vitamins in milk, dairy products, and other similar food, agricultural, and biological materials. This material can also be used for quality assurance when assigning values to in-house control materials. RM 8435 consists of 40 g of dry powdered whole milk packaged in a glass bottle sealed in an aluminum-nylon pouch.

Reference Concentration Values: Reference concentration values for major, minor, and trace constituent elements are provided in Table 1. Reference concentration values for proximates, calories, and fatty acids are provided in Table 2. Reference concentration values for selected vitamins are provided in Table 3. The reference values in Tables 1 through 3 were derived from results reported in three separate interlaboratory comparison exercises. Reference values are noncertified values that are the best estimates of the true values; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: Information concentration values for additional elements, fatty acids, and selected vitamins are provided in Tables 4 through 6, respectively. These are noncertified values with no reported uncertainties as there is insufficient information to assess uncertainties. The information values are given to provide additional characterization of the material. Use of this RM to quantitatively monitor method performance for analytes other than those with reference concentration values in Tables 1 through 3 is not recommended.

Expiration of Certification: The certification of RM 8435 is valid, within the measurement uncertainty specified, until **25 February 2011**, provided the RM is handled in accordance with instructions given in this report (see "Instructions for Use"). This certification is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM Value Assignment: NIST will monitor this RM over the period of its value assignment. If substantive technical changes occur that affect the value assignment before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Statistical support was provided by M.S. Wolynetz of the Statistical Research Section, Research Program Service, Agriculture Canada and L.M. Gill of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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See Report Revision History on Page 10

RM 8435 was prepared at Agriculture Canada under the direction of M. Ihnat, Centre for Land and Biological Resources Research (CLBRR). Coordination of the technical measurements leading to the value assignment of this RM was performed by M. Ihnat of CLBRR, Agriculture Canada, and K.E. Sharpless and S.A. Wise of the NIST Analytical Chemistry Division. Following the original analyses for elemental value assignment by the laboratories listed in Appendix A, the material was distributed by NIST to Covance Laboratories (Madison, WI), Lancaster Laboratories (Lancaster, PA), Medallion Laboratories (Minneapolis, MN), and Southern Testing and Research Laboratories (Wilson, NC) for the measurement of proximates, calories, and fatty acids. RM 8435 was also distributed in an interlaboratory comparison exercise in 1995; reference and information values for the concentrations of selected vitamins have been assigned based on results reported by the laboratories listed in Appendix C.

NOTICE AND WARNING TO USERS

Storage: Until required for use, RM 8435 should be refrigerated between 2 °C and 8 °C in its original bottle, tightly-capped, and not exposed to intense direct light or ultraviolet radiation.

Warning: For laboratory use only. Not for human consumption.

Instructions for Use: The bottle and contents should be allowed to warm up to room temperature prior to opening. Prior to each use, contents of the bottle should be well mixed by gentle shaking and rolling of the bottle. A minimum subsample size of 0.5 g should be taken for analysis. Moisture content should be determined on a separate subsample for conversion of analytical results to a dry-mass basis. The recommended method of drying to relate analytical results to the reference values listed in the tables is drying for 4 h in an air oven at 85 °C. Dissolution procedures for elemental analyses should be capable of rendering a completely dissolved sample appropriate to the method and should be designed to avoid losses of elements by volatilization or by retention on decomposition and processing containers and measuring equipment. Analytical methods should be capable of measuring total levels of elements for comparison with reference values.

PREPARATION AND ANALYSIS

Preparation: The source of material for RM 8435 was spray-processed whole milk powder (Fedeco brand manufactured by Cooperative Federe de Quebec, Montreal, Quebec) obtained from Cooperative Agricole de la Cote Sud, La Pocatiere, Quebec, Canada. All preparatory work following acquisition of the commercial product was performed at the facilities of Agriculture Canada, Ottawa [1,2]. The dry bulk powder was sterilized with cobalt-60 gamma radiation to 2.0 megarads by Atomic Energy of Canada Ltd. Material sieving was through nylon monofilament sieve cloths supported in high- density white polyethylene holders. Pairs of sieves with openings of approximately 250 µm and 90 µm were used to yield a middle-cut fraction for use as the RM. This fraction was blended in a polymethylmethacrylate V-configuration blender and packaged into clean 150 mL brim capacity, colorless glass bottles with triseal (polyethylene)-lined white polypropylene screw caps. A total of 144 randomly selected units were used for physical and chemical characterization in the original analyses. Units were individually hermetically sealed in aluminum-nylon pouches to enhance long-term stability.

Homogeneity Assessment: Homogeneity testing was performed on randomly selected units for ten elements in three laboratories [2-4]. Subsamples of 0.5 g were taken from a total of four units and analyzed by M. Ihnat, Agriculture Canada, for calcium, potassium, magnesium, sodium, and zinc using acid digestion flame atomic absorption spectrometry [5,6]. Subsamples of 0.7 g to 3.0 g each, taken from a total of six units, were analyzed by R.W. Dabeka, Health and Welfare Canada, for cadmium, cobalt, nickel, and lead by graphite furnace atomic absorption spectrometry following acid digestion and separation and preconcentration of the analytes using coprecipitation with ammonium pyrrolidine dithiocarbamate (cadmium, cobalt, and nickel) or palladium and ascorbic acid for lead [7-9]. Fluoride was determined by the same analyst in 0.2 g subsamples from six units by an acid-facilitated microdiffusion ion specific electrode method [10]. In addition, the analytical results obtained from a large number of analysts (Appendix A) participating in the interlaboratory comparison exercise were assessed to provide homogeneity estimates for other elements [3,4]. No statistically significant heterogeneity was found for aluminum, barium, boron, bromine, calcium, chlorine, copper, iodine, iron, lead, magnesium, manganese, molybdenum, nitrogen, phosphorus, potassium, selenium, sodium, strontium, sulfur, and zinc in sample sizes required by the analytical technique, ranging from 0.2 g to 3 g. Data for all analytes (including the proximates and fatty acids) have been statisticallly treated as though they are homogeneous, although the homogeneity of other analytes has not been investigated.

Value Assignment: Chemical analyses to establish reference concentrations of elements were conducted in an interlaboratory comparison exercise involving Agriculture Canada and selected analysts in other laboratories (Appendix

A) using analytical methods listed in Table 7. Analyses were performed by each participant on duplicate subsamples from randomly selected (typically four) units of material; subsample sizes and methods were left to the discretion of the analyst. Subsample sizes ranged from 0.001 g to 5 g, typically 0.5 g. Elemental determinations were performed on the material "as received," with conversion of results to a dry-mass basis using moisture values determined on separate 2 g subsamples by the drying procedure specified in the "Instructions for Use" section of this report.

Following the original elemental determinations, NIST distributed RM 8435 to four laboratories (Appendix B) for measurement of proximates, fatty acids, and calories. Each laboratory analyzed one portion from each of three bottles of RM 8435 using their routine methods (Table 8). Determinations were performed on the material "as received," with conversion of results to a dry-mass basis using moisture values determined on separate subsamples taken from each of the three bottles. Standard Reference Material (SRM) 1846 Infant Formula was analyzed for quality assurance. RM 8435 was also analyzed by several laboratories participating in an interlaboratory comparison exercise in 1995; several of these laboratories (Appendix C) reported values for vitamins, and these results are provided as reference and information values in Tables 3 and 6.

Table 1. Reference Concentrations of Constituent Elements

Major Constituents

Element	Mass Fraction (%)[a]	Methods[b]
Nitrogen[c]	4.187 ± 0.043	I01, J01
Potassium	1.363 ± 0.047	A01, B02, B04, E01
Calcium	0.922 ± 0.049	A01, B02, B04, B05
Chlorine	0.842 ± 0.044	D01, D04, F02, K01, K02
Phosphorus	0.780 ± 0.049	B02, B04, B05, F01, F02, M01
Sodium	0.356 ± 0.040	A01, A03, B02, B03, D01, D04
Sulfur	0.265 ± 0.035	B02, B03, D04, F04, J02, J03, J04, M02

Minor and Trace Constituents

Element	Mass Fraction (mg/kg)[a]	Methods[b]
Magnesium	814 ± 76	A01, A03, B02, B03, B04, B05, D01
Zinc	28.0 ± 3.1	A01, A03, B02, B03, B04, B05, D02, D03, E01
Bromine	20 ± 10	D01, E01
Strontium	4.35 ± 0.50	B02, B03, B04, B05, C03, E01
Iodine	2.3 ± 0.4	D01, D05, F02
Iron	1.8 ± 1.1	B02, B03, B05, D02, E01, E02
Boron	1.1 ± 0.23	B02, B03, B04, B05, C03
Copper	0.46 ± 0.08	A05, B02, B05, C03, C06, D03, E01
Molybdenum	0.29 ± 0.13	B02, C03, C06, C07, D03, F01, H06
Manganese	0.17 ± 0.05	A05, A06, B02, B05, D03
Selenium	0.131 ± 0.014	B06, C01, C04, G01
Lead	0.11 ± 0.05	A05, A16, C03

[a] Reference values, expressed as mass fractions, are based on the dry material, dried according to instructions in this report, and are equally weighted means of results from generally at least two, but typically several, different analytical methods applied by analysts in different laboratories. Uncertainties are imprecision estimates expressed either as a 95 % confidence interval or occasionally (B, Ca, Cl, Se) as an interval based on the entire range of accepted results for a single future determination based on a sample weight of at least 0.5 g. These uncertainties, based on among-method, among-laboratory, among-unit, and within-unit estimates of variances, include measures of analytical method and laboratory imprecisions and biases and material homogeneity. (NIST has replaced the previously used term "best estimate" with "reference value.")

[b] Analytical method codes and descriptions are provided in Table 7.

[c] The reference value for nitrogen has been updated to include results from four additional collaborating laboratories (Appendix B).

Table 2. Reference Concentrations of Proximates, Selected Fatty Acids (as Triglycerides), and Calories

Analyte	Mass Fraction, as received (%)[a,b]	Mass Fraction, dry-mass basis (%)[a]
Moisture	3.54 ± 0.57	0 (by definition)
Solids	96.46 ± 0.57	100 (by definition)
Ash	5.97 ± 0.11	6.19 ± 0.13
Protein[b]	25.86 ± 0.67	26.81 ± 0.67
Carbohydrate	43.4 ± 3.0	45.0 ± 2.9
Fat	21.3 ± 2.4	22.0 2.7
Butanoic Acid (C4:0) (Butyric Acid)	1.03 ± 0.11	1.07 ± 0.11
Decanoic Acid (C10:0) (Capric Acid)	0.70 ± 0.18	0.73 ± 0.19
Dodecanoic Acid (C12:0) (Lauric Acid)	0.81 ± 0.13	0.84 ± 0.14
Tetradecanoic Acid (C14:0) (Myristic Acid)	2.72 ± 0.28	2.83 ± 0.31
Pentadecanoic Acid (C15:0)	0.302 ± 0.030	0.313 ± 0.033
Hexadecanoic Acid (C16:0) (Palmitic Acid)	7.11 ± 0.67	7.38 ± 0.73
Heptadecanoic Acid (C17:0) (Margaric Acid)	0.191 ± 0.020	0.198 ± 0.020
Octadecanoic Acid (C18:0) (Stearic Acid)	2.51 ± 0.19	2.60 ± 0.21
(Z)-9-Octadecenoic Acid (C18:1) (Oleic Acid)	3.54 ± 0.72	3.67 ± 0.76
9-Octadecenoic Acid (C18:1) (Z-Elaidic Acid)	0.417 ± 0.058	0.432 ± 0.060
Eicosanoic Acid (C20:0) (Arachidic Acid)	0.051 ± 0.005	0.053 ± 0.005
Calories(c)	(470 ± 10) kcal/100 g	(490 ± 10) kcal/100 g

[a] Each reference concentration value, expressed as a mass fraction on an as-received or dry-mass basis, is an equally weighted mean of results from the laboratories shown in Appendix B. The uncertainty in the reference values is expressed as an expanded uncertainty, *U*, at the 95 % level of confidence, and is calculated according to the method described in the *ISO Guide to the Expression of Uncertainty in Measurement* [11]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, *k*, is determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte. Analytical methodology information is provided in Table 8.

[b] The "as received" values are based on the moisture content at the time the measurements for value assignment were performed. The amount of moisture in this material may change if moisture is transferred to or absorbed from the atmosphere during storage.

[c] The protein concentration was calculated from the nitrogen values reported by the laboratories shown in Appendix B using a conversion factor of 6.38; subsequent calculation of carbohydrates and calories were also based on these protein concentrations. The nitrogen values reported by the laboratories shown in Appendix B were combined with the original data for calculation of the reference value for nitrogen provided in Table 1.

[d] The value for calories is the mean of the individual caloric calculations. If the mean proximate values are used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively, the mean caloric content is 460 kcal/100 g and 490 kcal/100 g on an as-received and dry-mass basis, respectively.

Table 3. Reference Concentrations of Selected Vitamins

Analyte	Mass Fraction, as received (mg/kg)[a]	Mass Fraction, dry-mass basis (mg/kg)[a]
Vitamin B_1	1.80 ± 0.52	1.87 ± 0.54
Vitamin B_2	10.6 ± 3.2	11.0 ± 3.4
Vitamin B_6	1.86 ± 0.58	1.94 ± 0.61
Vitamin B_{12}	0.017 ± 0.003	0.018 ± 0.003
Niacin	7.35 ± 0.86	7.65 ± 0.93
Pantothenic Acid	25.6 ± 5.4	26.7 ± 5.7

[a] Each reference concentration value, expressed as a mass fraction on an as-received or dry-mass basis, is an equally weighted mean of results from the laboratories shown in Appendix C. The uncertainty in the reference values is expressed as an expanded uncertainty, *U*, at the 95 % level of confidence, and is calculated according to the method described in the *ISO Guide to the Expression of Uncertainty in Measurement* [11]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, *k*, is determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte. Analytical methodology information is provided in Table 9.

Table 4. Information Concentrations of Constituent Elements

Element	Mass Fraction (mg/kg)[a]	Methods[b]
Aluminum	0.9	A05, A06, B02
Arsenic	0.001	D03
Cadmium	0.0002	A16
Cobalt	0.003	A16, D02
Chromium	0.5	B02, B04
Fluorine	0.17	H04
Nickel	0.01	A14, A16
Rubidium	16	D01, D02, E01
Titanium	4	B02, C08, E02
Tungsten	0.002	C07, H06

[a] These analytical values, on a dry-mass basis, are estimates given strictly for information only, as they are based on results of limited determinations or only one method; no uncertainties are provided.
[b] Analytical method codes and descriptions are provided in Table 7.

Table 5. Information Concentrations of Selected Fatty Acids (as Triglycerides)

Analyte	Mass Fraction, as received (%)[a]	Mass Fraction, ry-mass basis (%)[a]
Tridecanoic Acid (C13:0)	0.030	0.031
9-Tetradecenoic Acid (C14:1) (Myristoleic Acid)	0.22	0.23
9-Hexadecenoic Acid (C16:1) (Palmitoleic Acid)	0.30	0.31
Docosanoic Acid (C22:0) (Behenic Acid)	0.030	0.31
Tetracosanoic Acid (24:0) (Lignoceric Acid)	0.021	0.022

[a] These information values, reported on an as-received or dry-mass basis, are the equally weighted means of results reported by the laboratories shown in Appendix B. These values are based on results from determinations by two to four of the laboratories and are included to provide additional characterization of the material; no uncertainties are provided. Analytical methodology information is provided in Table 8.

Table 6. Information Concentrations of Selected Vitamins

Analyte	Mass Fraction, as received (mg/kg)[a]	Mass Fraction, dry-mass basis (mg/kg)[a]
Vitamin A (as *trans*-retinol)	1.9	2.0
Vitamin D	0.039	0.041
Biotin	0.15	0.16
Choline	940	980
Inositol	310	320

[a] These information values, reported on an as-received or dry-mass basis, are the equally weighted means of results reported by the laboratories shown in Appendix C. These values are based on results from determinations by one to four laboratories, and are included to provide additional characterization of the material; no uncertainties are provided. Analytical methodology information is provided in Table 9.

Table 7. Analytical Methods Used by Collaborating Laboratories (Appendix A) to Determine Reference and Information Concentration Values of Elements[a]

Analytical Method	Code	Elements Determined
Acid digestion flame atomic absorption spectrometry	A01	Ca, K, Mg, Na, Zn
Dry ashing flame atomic absorption spectrometry	A03	Mg, Na, Zn
Closed vessel acid digestion electrothermal atomic absorption spectrometry	A05	Al, Cu, Mn, Pb
Dry ashing electrothermal atomic absorption spectrometry	A06	Al, Mn
Acid digestion solvent extraction flame atomic absorption spectrometry	A14	(Ni)
Acid digestion coprecipitation electrothermal atomic absorption spectrometry	A16	(Cd), (Co), (Ni), Pb
Acid digestion inductively coupled plasma atomic emission spectrometry	B02	Al, B, Ba, Ca, (Cr), Cu, Fe, K, Mg, Mo, Na, P, S, Sr, (Ti), Zn
Closed vessel acid digestion inductively coupled plasma atomic emission spectrometry	B03	Ba, Fe, Mg, Na, S, Sr, Zn
Dry ashing inductively coupled plasma atomic emission spectrometry	B04	B, Ba, Ca, (Cr), K, Mg, P, Sr, Zn
Acid digestion dry ashing inductively coupled plasma atomic emission spectrometry	B05	Ba, Ca, Cu, Fe, Mg, Mn, P, Sr, Zn

Acid digestion hydride generation inductively coupled plasma atomic emission spectrometry	B06	Se
Acid digestion isotope dilution mass spectrometry	C01	Se
Closed vessel acid digestion isotope dilution inductively coupled plasma mass spectrometry	C03	Ba, Cu, Mo, Pb, Sr
Acid digestion dry ashing hydride generation isotope dilution inductively coupled plasma mass spectrometry	C04	Se
Acid digestion isotope dilution inductively coupled plasma mass spectrometry	C06	Cu, Mo
Dry ashing inductively coupled plasma mass spectrometry	C07	Mo, (W)
Acid digestion inductively coupled plasma mass spectrometry	C08	(Ti)
Instrumental neutron activation analysis	D01	Br, Cl, I, Mg, Na, (Rb)
Instrumental neutron activation analysis with acid digestion	D02	(Co), Fe, (Rb), Zn
Neutron activation analysis with radiochemical separation	D03	(As), Cu, Mn, Mo, Zn
Neutron capture prompt gamma activation analysis	D04	B, Cl, Na, S
Epithermal instrumental neutron activation analysis	D05	I
Particle induced X-ray emission spectrometry	E01	Br, Cu, Fe, K, (Rb), Sr, Zn
X-ray fluorescence	E02	Fe, (Ti)
Acid digestion light absorption spectrometry	F01	Mo, P
Dry ashing light absorption spectrometry	F02	Cl, I, P
Combustion light absorption spectrometry	F04	S
Acid digestion fluorometry	G01	Se
Extraction ion selective electrode	H04	(F)

Dry ashing catalytic adsorption Polarography	H06	Mo,(W)
Kjeldahl method for nitrogen - volumetry	I01	N[(b)]
Combustion elemental analysis - thermal conductivity	J01	N[(b)]
Combustion elemental analysis with chromatographic separation - thermal conductivity	J02	S
Combustion elemental analysis - infrared spectrometry	J03	S
Combustion elemental analysis - fluorometry	J04	S
Extraction volumetry	K01	Cl
Dry ashing volumetry	K02	Cl
Acid digestion gravimetry	M01	P
Dry ashing gravimetry	M02	S

[(a)] Letter codes refer to classes of similar methods; number codes refer to specific variants. Elements in parentheses have only information values in this RM.
[(b)] See Table 8 for additional information.

Table 8. Methods Used by Collaborating Laboratories (Appendix B) for the Determination of Proximates, Calories, and Fatty Acids

Ash	mass loss after ignition in a muffle furnace
Calories	calculated; [(9 × fat) + (4 × protein) + (4 × carbohydrate)
Carbohydrate	calculated; [solids – (protein + fat + ash)]
Fat	sum of individual fatty acids
Fatty acids	hydrolysis followed by gas chromatography
Moisture	mass loss after drying in a vacuum oven (3 laboratories); mass loss after drying in a forced-air oven (1 laboratory)
Nitrogen	Dumas (1 laboratory); modified Dumas (1 laboratory); Kjeldahl (2 laboratories). Note that in the original elemental determinations, 8 laboratories provided results using Kjeldahl, combustion - thermal conductivity, and combustion - chromatographic separation - thermal conductivity.
Protein	calculated from nitrogen using a factor of 6.38
Solids	calculated; (sample mass – moisture)

Table 9. Methods Used by Collaborating Laboratories (Appendix C) for the Determination of Vitamins

Vitamin A	saponification – normal-phase liquid chromatography (NPLC) – absorbance detection (1 laboratory) saponification – reversed-phase liquid chromatography (RPLC) – absorbance detection (1 laboratory)
Vitamin D	saponification – NPLC – absorbance detection (2 laboratories) saponification – RPLC – absorbance detection (2 laboratories)
Vitamin B_1	microbiological (1 laboratory)

	digestion with fluorescence detection (3 laboratories) extraction – RPLC – fluorescence detection (3 laboratories)
Vitamin B_2	microbiological (1 laboratory) digestion with fluorescence detection (3 laboratories) extraction – RPLC – fluorescence detection (2 laboratories)
Vitamin B_6	microbiological (5 laboratories)
Vitamin B_{12}	microbiological (6 laboratories)
Niacin	microbiological (5 laboratories) acid digestion – colorimetry (1 laboratory)
Folic Acid	microbiological (5 laboratories)
Pantothenic Acid	microbiological (5 laboratories)
Biotin	microbiological (5 laboratories)
Choline	acid digestion – colorimetry (2 laboratories) acid digestion – LC – enzymatic reaction – Pt electrode (1 laboratory) acid digestion – choline oxidase membrane (1 laboratory)
Inositol	LC - amperometry (1 laboratory)

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Users of this RM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

Collaborating Analysts for Elemental Determinations

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APPENDIX B

Collaborating Laboratories for Proximate, Fatty Acid, and Caloric Determinations

Covance Laboratories, Madison, WI, USA.
Lancaster Laboratories, Lancaster, PA, USA.
Medallion Laboratories, Minneappolis, MN, USA.
Southern Testing and Research Laboratories, Wilson, NC, USA.

APPENDIX C

Collaborating Laboratories for Vitamin Determinations

Abbott Laboratories, Ross Products Division, Columbus, OH, USA.
Bristol Meyers Squibb, Mead Johnson Nutritionals, Zeeland, MI, USA.
Food and Drug Administration, Center for Food Safety and Applied Nutrition, Washington, DC, USA.
Covance Laboratories, Madison, WI, USA.
Lancaster Laboratories, Lancaster, PA, USA.
Nestle USA, Dublin, OH, USA.
Southern Testing and Research Laboratories, Wilson, NC, USA.