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The certification of SRM 1546 – Meat Homogenate, a new reference material for nutrients in a high protein, high fat matrix

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Abstract In response to reference material needs expressed by the food industry and government regulators, the National Institute of Standards and Technology (NIST) has developed a new Standard Reference Material (SRM) consisting of a canned meat product with certified and reference values for a large number of constituents. SRM 1546 Meat Homogenate consists of a mixture of finely ground pork and chicken prepared and canned by a commercial process. NIST determined the concentration levels of cholesterol, sodium, calcium, iron, and seven fatty acids in this SRM using well defined methods and procedures. These analytes as well as 34 other constituents or properties were determined in an interlaboratory comparison exercise involving 21 laboratories, most of which are associated with the National Food Processors Association (NFPA) Food Industry Analytical Chemists Subcommittee (FIACS). From statistical analysis of the data, NIST assigned certified concentrations for the eleven analytes measured at NIST and reference concentrations for the proximates, six additional fatty acids, seven minerals, and seven water-soluble vitamins. Information values without uncertainties are provided for the concentrations of six additional constituents for which the uncertainties could not adequately be assessed. SRM 1546 will provide laboratories with a means to evaluate the accuracy of the methods they use to assign nutrient levels to processed meats and similar products.

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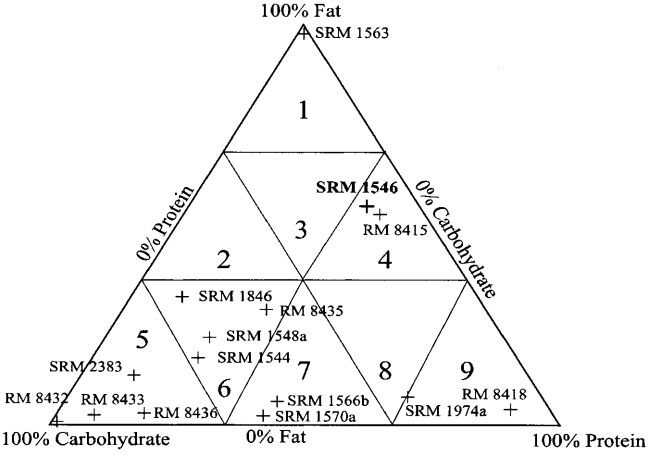
Supplementary material (Table 1 and 3–9) is available in electronic form on Springer Verlag's server at http://link.springer.de/journals/fjac

Introduction

Accurate and traceable nutritional measurements are important for a number of reasons. Nutritional labeling with accurately determined quantities of important nutrients helps consumers make better choices about the benefits and risks of various foods. Accurate assessment of the nutritional qualities of foods used in studies that evaluate the role of foods or food components in promoting or inhibiting disease states enables scientists to make more definitive conclusions. The export of foods to other countries relies heavily upon the validity of the nutritional data supplied with the food and the confidence regulators have in the data. Consequently, if the nutritional measurements are traceable to internationally recognized standards, they are more likely to be accepted.

To address the needs for accuracy and traceability in nutritional measurements, the National Institute of Standards and Technology (NIST) is developing a series of food-matrix Standard Reference Materials (SRMs) to help laboratories validate their methods and demonstrate accuracy and traceability in their measurements. Available SRMs include SRM 1544 Cholesterol and Fatty Acids in a Frozen Diet Composite, SRM 1548 Typical Diet, SRM 1563 Cholesterol and Fat-soluble Vitamins in Coconut Oil [1], SRM 1845 Cholesterol in Whole Egg Powder, and SRM 1846 Infant Formula [2], SRM 2383 Baby Food Composite [3, 4].

The red meat industry is the largest sector in the U.S. processed food and beverage industry [5]. The value of red meat shipments was estimated to be $79B in 1998. Because of the nutritional importance of this type of food in the U.S. diet, NIST undertook development of an SRM for nutrients in a canned meat material, SRM 1546 Meat Homogenate, with input and support from the nutritional measurement community and scientists at the US Department of Agriculture (USDA) and the Food and Drug Administration (FDA). Such a material is high in fat and protein, while low in carbohydrate, in contrast to most of the other available food-matrix reference materials. The relationship of this SRM to other food-matrix reference mate-



Food Triangle showing relative amounts of fat, carbohydrate, and protein, with ash and moisture not included, for food-based reference materials.

Examples: Region 1 represents foods having 67 -100% fat, 0 - 33% carbohydrate, and 0 - 33% protein. SRM 1563 Coconut Oil with nearly 100% fat falls in this range.

Region 4 represents foods having 33 - 67% fat, 0 - 33% carbohydrate, and 33 - 67% protein. SRM 1546 Meat Homogenate with 54% fat, 5 % carbohydrate, and 41 % protein falls in this range.

Fig. 1 Food triangle showing where various SRMs and RMs available from NIST fall in terms of their relative percentages of protein, fat, and carbohydrate. More information about the other SRMs and RMs can be found at http://www.nist.gov/srm

rials from NIST in terms of fat, protein, and carbohydrate composition is shown in Fig. 1.

A commercially available meat product was identified that had the necessary characteristics. This material, prepared from finely-ground and blended chicken and pork, was quite uniform, it had high levels of protein and fat, and it had a long shelf life. A single batch of this material was purchased, in the form of cans, each containing 85 grams (3 oz) of the product. Measurements were performed at NIST to determine the concentration levels for cholesterol, major fatty acids, sodium, calcium, and iron. Outside laboratories, primarily those affiliated with the National Food Processors Association (NFPA) Food Industry Analytical Chemists Subcommittee (FIACS), performed measurements of proximates, caloric content, cholesterol, fatty acids, minerals, and vitamins. Based upon statistical analysis of the combined measurement results of NIST and the collaborating laboratories, certified concentration values for eleven analytes, reference concentration values for 24 analytes, a reference value for caloric content, and information values for six other analytes were determined.

This paper describes the measurement methods used, the results obtained, and the statistical treatment of the data for the value assignment of this SRM.

Experimental

Measurement protocol. During the filling process, each can was numbered sequentially according to the fill order, thus permitting sampling from across the run. A stratified random sampling plan was developed that assured that representative samples from throughout the run would be measured. Each of the participants in the interlaboratory study received four cans of the SRM from across the filling sequence. The participants were instructed to take one sample from each can for a single analysis. Although each participant received enough cans for four analyses for each constituent that they measured, some of the participants only performed one or two measurements per analyte. This stratified random sampling plan was also applied to the selection of the cans used in the measurements performed at NIST.

Materials. The canned meat material for the SRM was obtained from Hormel, Inc.[1] (Austin, MN) and was from one batch. The material consists of a mixture of pork, mechanically separated chicken, ham, salt, sugar, water, spices, and sodium nitrite as a preservative. The material is finely ground and heat sterilized. It is vacuum sealed in aluminum cans with pull top lids.

Primary standards for NIST analyses included SRM 911b Cholesterol from NIST and fatty acid standards from Nu-Chek-Prep, Inc. (Elysian, MN). Cholesterol-25,26,27-$^{13}C_3$ was from Isotec, Inc. (Miamisburg, OH). Deuterated fatty acids were from Cambridge Isotope Laboratories (Andover, MA), CDN Isotopes (Quebec, Canada), and DSR Laboratories (Englewood Cliffs, NJ). Primary standards for calcium, sodium, and iron were SRM 915a Calcium Carbonate, SRM 2201 Sodium Chloride, and iron metal from Alfa Aesar (Ward Hill, MA), respectively.

Control materials used included NIST SRMs 1544 Fatty Acids and Cholesterol in a Frozen Diet Composite, 1845 Cholesterol in Whole Egg Powder, 1846 Infant Formula, and LGC Certified Reference Material (CRM) 7002, Pork/Chicken Meat, available from LGC, Teddington, Middlesex, U.K.

NIST analyses for cholesterol and fatty acids

Cholesterol was measured using the isotope dilution/gas chromatography/mass spectrometry (ID/GC/MS) method developed at NIST for serum cholesterol [6] and modified for the determination of cholesterol in food matrices using AOAC Method 43.235 for hydrolysis [7]. Three sets of samples were prepared. Each set consisted of duplicate samples from each of three cans of SRM 1546, one jar of SRM 1544 Fatty Acids and Cholesterol in a Frozen Diet Composite, and one jar of SRM 1845 Cholesterol in Whole Egg Powder. These last two materials were used as controls. Each can of SRM 1546 was opened, thoroughly stirred with a spatula, and two approximately 1 g samples were withdrawn and accurately weighed into round-bottomed flasks. An aliquot of a solution containing a known mass of the internal standard, cholesterol-$^{13}C_3$, was added to each flask. Hydrolysis of cholesterol esters was accomplished by refluxing the samples in a solution consisting of 15 mL of ethanol/methanol/2-propanol (95:5:5 by volume) and 3 g of KOH dissolved in 2 mL water for 1 h in a boiling water bath. Water (15 mL) was added and 30 mL of hexane was then used to extract the cholesterol. A portion of the hexane extract was evaporated to dryness and N,O-bis(trimethylsilyl)-acetamide was added to convert cholesterol to its trimethylsilyl (TMS) derivative. Analyses were performed on a GC/MS system operated in the electron ionization mode with selected ion monitoring at m/z 458 and 461 for the unlabeled and labeled cholesterol-TMS, respec-

[1] Certain commercial equipment, instruments, or materials are identified in this report to specify adequately 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 the best available for the purpose.

tively. The GC was equipped with a non-polar fused silica column (DB-5MS from J &W Scientific, Folsom, CA), 30 m in length with 0.25 mm i.d. and 0.25 μm film thickness, directly interfaced to the ion source. Standards consisting of mixtures of known quantities of pure unlabeled cholesterol (SRM 911b) and cholesterol-$^{13}C_3$ were run before and after the samples to generate composite linear regressions for calculation of the quantity of cholesterol in the samples.

Fatty acids (FAs) were also determined by ID/GC/MS. Three sets of samples were prepared. Each set consisted of duplicate samples from each of three cans of SRM 1546 and two jars of SRM 1544 Fatty Acids and Cholesterol in a Frozen Diet Composite. Two solutions of deuterated fatty acids were prepared. One, containing major components, consisted of C18:1 (oleic acid)-d_2, C16:0 (palmitic acid)-d_3, and C18:0 (stearic acid)-d_3. The minor component solution consisted of C10:0 (capric acid)-d_3, C12:0 (lauric acid)-d_3, C14:0 (myristic acid)-d_3, and C20:0 (arachidic acid)-d_3. These labeled solutions were used for the preparation of standards and for spiking of samples.

Each can of meat homogenate was opened and mixed well in a plastic bag by squeezing repeatedly. Quantities of about 1 g were weighed and combined with approximately 1.6 g of diatomaceous earth and then loaded into a pressurized fluid extraction (PFE) cell. The cells were spiked with C13:0 triglyceride (tritridecanoin) and C19:0 triglyceride (trinonadecanoin) in chloroform as extraction recovery surrogates. The meat homogenate sample materials were subjected to semi-static fluid extraction with hexane dichloromethane methanol (70:25:5 by volume) at 125 °C and 10 300 kPa (1500 psig) for 5 min. After PFE, the extracts were diluted to known volume (50 mL), and a 5 mL aliquot was spiked with deuterated internal standard mixture, allowed to equilibrate, and subjected to alkaline hydrolysis for 1 h in an aqueous 1 mol/L sodium hydroxide solution at 60 °C. After hydrolysis, the samples were acidified with 1.0 mL of 6 mol/L HCl and buffered with 2.5 mL of pH 4 buffer. The FAs were subsequently extracted three times into hexane (5 mL). A 1.0 mL aliquot of this material was treated with 50 μL of 1,1-dimethoxytrimethylamine to form the corresponding fatty acid methyl esters (FAMEs). Analysis of the resultant FAME mixture was performed on an ion trap mass spectrometer. Separation was accomplished on a 30-m column of 0.25 mm i.d. with a polyethylene glycol stationary phase of 0.25 μm thickness (AT-Wax from Alltech Associates, State College, PA), followed by electron impact ionization and full-scan mass spectrometric detection.

NIST analyses for calcium, iron, and sodium

Eight cans of SRM 1546 and one can of LGC CRM 7002 were opened and placed in separate plastic bags. The contents of each of the nine bags were thoroughly mixed by squeezing the bags repeatedly. Two 3.5 g portions were taken from each bag and weighed into teflon beakers. The samples and accompanying blanks were digested in $HNO_3/HClO_4$ (1:1 by volume) at 160 °C until solutions were clear. The acids were evaporated and the residue redissolved in water/HNO_3 and transferred to 50 mL volumetric flasks with addition of water. Separate aliquots from these solutions were taken for measurements of each element. For each element, from each solution, two aliquots were taken, one of which was spiked with a known concentration of the element, and the aliquots diluted to a final volume at a final acid volume fraction of 3.2% HNO_3. Measurements were performed using inductively coupled plasma optical emission spectrometry (ICP-AES). Emission wavelengths monitored were: 393.366 nm (Ca); 238.204 nm (Fe); and 589.592 nm (Na). Each solution was measured four times and the results averaged. Spike recoveries were measured to correct for matrix effects.

Methods used by the NFPA FIACS participants

The participating laboratories are listed in Table 1 (see Supplementary Electronic Material) and a summary of the methodologi-

Table 2 Methods used by the participating laboratories

Solids	Gravimetry (oven drying)
Ash	Gravimetry (ignition in muffle furnace)
Extractable fat	Acid digestion/extraction (solvents, PFE, SFE)
Fat by summation of fatty acids	Gas chromatography (GC) with flame ionization detection (FID)
Nitrogen	Kjeldahl; thermal conductivity; neutron activation
Protein results	Calculated; a factor of 6.25 was used to calculate protein from nitrogen
Carbohydrates	Calculated; carbohydrate = solids – (protein + fat + ash)
Cholesterol	GC-FID; GC/MS
Caloric content	Calculated; caloric content = 9(fat) + 4(protein) + 4(carbohydrate)
Sugars	LC-UV; GC-FID
Vitamins $B_1 \cdot HCl$, B_2	Digestion/extraction and LC-fluorescence; microbiological
Vitamin B_6	Microbiological; extraction and LC-fluorescence
Vitamin B_{12}	Microbiological
Niacin	Microbiological; acid digestion – absorption spectrophotometry
Folic acid, inositol	Microbiological
Pantothenic acid, biotin	Microbiological
Choline	Acid digestion – absorption spectrophotometry; microbiological
Boron	Neutron activation
Ca, Cu, Fe, Mg, Mn, K	Flame atomic absorption spectrometry (FAAS); inductively coupled plasma atomic emission spectrometry (ICP-AES); direct current plasma atomic emission spectrometry (DCP-AES)
Chloride	Colorimetric titration; electrochemical titration; ICP-AES
Iodine	Colorimetric titration; ICP-AES
Phosphorus	FAAS; ICP-AES; colorimetric titration
Sodium	Flame atomic emission spectrometry; ICP-AES, FAAS, DCP-AES; neutron activation
Sulfur	Neutron activation
Zinc	FAAS; ICP-AES; DCP-AES; ICP-MS

cal approaches used is shown in Table 2. Most of these are Official Methods as designated by the AOAC [8]. Each participant was asked to make single measurements from each of four cans for each analyte they chose to measure. The laboratories also analyzed SRM 1544, SRM 1846, and LGC CRM 7002 as controls for quality assurance.

Methods used by other laboratories

Two laboratories not part of the NFPA-FIACS provided results on the fat content of the material. One of these laboratories used supercritical fluid extraction for the determination of extractable fat and individual fatty acids; the other used pressurized fluid extraction to measure the extractable fat. One additional non-NFPA-FIACS laboratory performed neutron activation analysis for a number of minerals in this material.

Results

Certified values

A NIST certified value represents data for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for. The results of the determination of cholesterol and fatty acids by NIST, the mean results for these analytes from each of the participating laboratories, and the certified values are shown in Table 3 (see Supplementary Electronic Material). The ID/MS method for cholesterol used at NIST is highly repeatable [6], so most of the imprecision shown is a measure of the inhomogeneity of the lipids in the material as shown in Fig. 2. Even thorough mixing cannot overcome all of the inhomogeneity. When whole cans were processed, the imprecision was reduced significantly, demonstrating that the overall fat content of a can is more uniform than is the distribution within a can. To account for the within-can inhomogeneity determined for cholesterol, all of the certified values have a variance component of 3% for material inhomogeneity, even though some of the non-lipid analytes may be distributed differently.

Similar inhomogeneity is apparent in the results for fatty acids, shown in the NIST data in Table 3. However, between-laboratory imprecision is also a major factor, as shown in the participants' data in this table. Fatty acids are typically reported in one of three ways: as free fatty acids, as fatty acid methyl esters, and as triglycerides. The participants were allowed to submit data using any of these, as long as they specified which they used. The most commonly reported form for this study was the free fatty acid approach, and, therefore, is the form reported in the SRM 1546 certificate of analysis. All of the data was converted to this form using published conversion tables [9]. The change is relatively small, with values decreasing by 4 to 8% when going from FAMEs or triglycerides to free fatty acids, with the largest percentage changes for the shortest chain acids. Because of the scatter, the collaborating laboratory data overlaps well with the NIST data for six of the seven fatty acids measured by NIST. The one exception is arachidic acid (C20 : 0) for which there is little overlap. The uncertainty limits as calculated for this fatty acid include most of the data from both sources.

For sodium, calcium, and iron, agreement was good between the means of the collaborating laboratories and the NIST results, with considerable overlap between the two sets of data for each element. Spike recovery factors were measured at NIST to correct for matrix effects caused by differences between samples and standards. Measured data were multiplied by the spike recovery factors of .9784, .8865, and 1.0493 for Ca, Fe, and Na, respectively, to achieve the NIST results shown in Table 4 (see Supplementary Electronic Material), which also has the collaborating laboratories' results and the certified values.

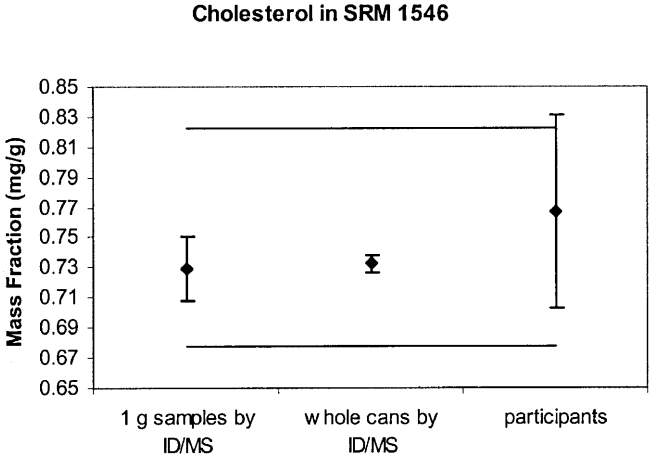


Fig. 2 Mean values ± 1 s.d. for cholesterol in SRM 1546. The two columns on the left are the NIST results by ID/MS and the third column shows the distribution of results among the participating laboratories. The two dark lines represent the range of the certified value

Reference values

Proximates (protein, fat, solids, carbohydrates, ash) were not measured by NIST. The collaborating laboratories provided all of the data, generally using methods approved by the AOAC [8]. These are well-established methods with which the participants have had considerable experience. Consequently, the agreement among the laboratories is quite good for most of the constituents. These results (Table 5, Supplementary Electronic Material) are considered to be reference values because results have not been confirmed by an independent analytical technique as required for certification and/or no NIST measurements were made [10]. Total protein determinations involve measurement of the nitrogen content of the material and multiplying the result by 6.25 [11]. The interlaboratory comparison data are shown in Fig. 3. Of the 20 laboratories submitting data, only one had a mean result that fell outside of the 95% confidence limits around the reference value. Total fat is measured in two ways. One method is the traditional gravimetric determination

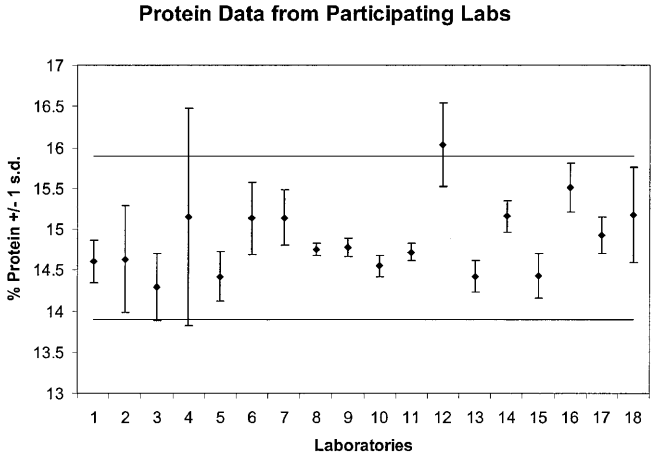


Fig. 3 Mean values ± 1 s.d. for protein reported by the participating laboratories. The two dark lines represent the range of the reference value for protein calculated from these data

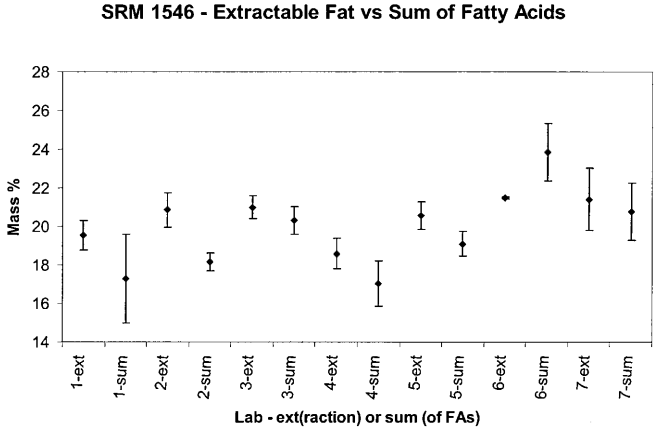


Fig. 4 A comparison of extractable fat versus sum of fatty acids for participating laboratories that reported fat content both ways. The lines are mean values ± 1 s.d.

of extractable fat; the second is the sum of the individual fatty acids (as triglycerides) measured. The former approach generally gives results that are larger than those from the latter approach, as some non-lipid material is probably extracted in the gravimetric method and some low-level fatty acids may be missed in the summation of the fatty acids. Figure 4 shows how the seven laboratories that reported both extractable fat and total fatty acids compared for the two approaches. Six of the seven had higher results for the extractions, averaging 6% less for the total fatty acids, overall.

The mass fraction of carbohydrates is not measured. It is calculated by assuming that any solid not accounted for by ash, protein, or fat must be carbohydrate. This material is very low in carbohydrate, such that the values are highly scattered and sometimes calculate to less than zero when the sum of the ash, protein, and fat is subtracted from the total solids. Some of the laboratories used extractable fat while others used the sum of the fatty acids in this calculation for carbohydrate, thus further increasing the scatter. However, it is known that some sucrose is added to the material for flavor. A few laboratories measured the sucrose and reported a value. These values are in generally good agreement and well within the calculated carbohydrate range. Therefore, the SRM 1546 certificate of analysis reports the measured sucrose concentration as the reference value for carbohydrates.

The laboratories calculated the caloric content, using the standard caloric equivalents for fat, protein, and carbohydrate of 9, 4, and 4 kcal/g, respectively. The reference value is calculated from the means of the individual laboratories' results. Caloric content could also be calculated by using the reference values for fat, protein, and carbohydrate multiplied by the appropriate factor. If that were done, the mean caloric content would be 256 kcal/100 g if the extractable fat mean were used and 244 kcal/100 g if the mean sum of the fatty acids were used. These two values bracket the value shown in Table 5.

A number of additional fatty acids were measured by the collaborating laboratories but not measured by NIST. These are present at low levels and there is relatively high scatter among the laboratories. However, it was still possible to calculate reference values for six of these fatty acids. These results are shown in Table 6 (see Supplementary Electronic Material). Similarly, seven additional minerals (Table 7, Supplementary Electronic Material) and seven vitamins (Table 8, Supplementary Electronic Material) were measured by collaborating laboratories and provided as reference values.

Information values

The collaborating laboratories were given wide latitude in terms of what constituents they would measure. The major components were measured by a large number of laboratories. However, there were some minor constituents measured by a small number of laboratories. These concentrations are listed as information values because of larger disagreement of results among laboratories and/or a limited number of collaborating laboratories. Because of the quantity and quality of the data, it is not possible to assess the validity and uncertainty of the results. Therefore, these results, shown in Table 9 (see Supplementary Electronic Material) are reported for information only, without uncertainties.

Control data

Use of controls by NIST and the collaborating laboratories provided a means of evaluating the quality of the data for SRM 1546. Two SRMs, 1544 Fatty Acids and Cholesterol in a Frozen Diet Composite, and 1846 Infant Formula, were used as controls by the participating laboratories for determinations of lipids and vitamins, respectively. LGC CRM 7002, Pork/Chicken Meat, was used as a control for minerals. The control data were examined for cases where results from a particular laboratory for a

particular analyte were clearly discordant from the other laboratories' results. The criteria used were that the results had to be greater than three standard deviations from the mean of the other participants to be rejected. In such cases, the data for SRM 1546 from that particular laboratory for that analyte were rejected. There were two laboratories that reported results that were far beyond three standard deviations for most of the fatty acids in both SRM 1544 (control) and SRM 1546. The laboratories were given an opportunity to correct their data for blunders, but no corrections were reported. Therefore, all of the fatty acid results from these laboratories were excluded from the calculations. Otherwise, the incidence of rejected data was very low and had minimal effect on the certified and reference values.

Conclusion

With 11 certified values and 28 reference values, SRM 1546 provides the nutritional measurement community with a high-fat, high-protein material that is useful for evaluating the accuracy of methods used for nutrition labeling. This material provides a traceability link between routine nutritional measurements and national standards.

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