

Summary of reference materials for the determination of the nutrient composition of foods

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Abstract Certified reference materials (CRMs) play a critical role in validating the accuracy of nutrient data for food samples. A number of available food CRMs of differing matrix composition have assigned concentrations for various nutrients, along with associated uncertainty intervals (UIs) for those values. These CRMs have been used extensively in the United States Department of Agriculture's (USDA) ongoing National Food and Nutrient Analysis Program (NFNAP) to monitor the accuracy of assays of key foods and nutrients consumed in the United States. A total of 690 assigned values for individual nutrients,

including proximates, vitamins, macroelements, microelements, fatty acids, amino acids, and selected phytochemicals (e.g., carotenoids), were compiled from the certificates of analysis for 63 CRMs, and the specified UI in each case was expressed as a percentage of the assigned certified or reference concentration. Across all nutrients, 63.5% of the UIs were less than 10% of the assigned value, 25.5% were 10–20%, and 11% were greater than 20% of the assigned value. The UIs for proximates, minerals, and trace elements were most consistently less than 10% of the assigned value. The relative uncertainties were significantly higher for vitamins, suggesting greater challenges in measuring and certifying these components. These high UIs (greater than 10% assigned value) in the best available reference materials are likely to be indicative of the precision and accuracy that can be obtained by current measurement systems for these components. These data suggest that care must be taken in choosing CRMs to monitor food composition analysis, including evaluating what levels of uncertainty are required in assigned values and which analytical measurement systems for food components need closer examination and improvement.

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Introduction

Certified reference materials (CRMs) play a critical role in validating the accuracy of nutrient data. Food-matrix CRMs are intended to mimic "real" samples that an analyst might encounter. CRMs are used for several purposes: to facilitate testing the accuracy of the entire assay system during the development or implementation of an analytical method (e.g., from extraction through quantita-

tion by instrumental analysis); for the determination of whether a method is in control during routine use; for the provision of traceability of values assigned to an in-house control material; or as a reference sample for the assessment of interlaboratory variability.

The United States Department of Agriculture's (USDA) continuing National Food and Nutrient Analysis Program (NFNAP) generates data for the USDA Nutrient Database for Standard Reference [1] based on statistical sampling and chemical analysis of the nutrient composition of key foods [2]. CRMs have been used extensively in the NFNAP to monitor the accuracy of nutrient assays for key foods and nutrients in the United States [2–4]. A range of food CRMs with assigned values and uncertainty intervals (UIs) for many nutrients are currently supplied by several organizations, primarily, the National Institute of Standards and Technology (NIST; Gaithersburg, MD, USA), the Institute for Reference Materials and Measurements (IRMM; Geel, Belgium), and LGC Promochem (Teddington, UK). The International Atomic Energy Agency (IAEA) maintains a database of CRMs for food and non-food matrices and organic and inorganic nutrients, contaminants, and other components (<http://www.naweb.iaea.org/nahu/nmrm/nmrm2003/default.htm>). Periodic publications discuss IAEA activities [5]. The Federal Institute for Materials Research and Testing (BAM; Berlin, Germany) has also maintained, for about 20 years, an international database of CRMs, the Code d'Indexation des Matériaux de Référence (COMAR; <http://www.comar.bam.de>). While COMAR presently contains information on over 11,000 CRMs, summarizes assigned values and UIs, and provides links to certificates of analysis for CRMs, it is difficult to use as a resource without prior knowledge of what CRMs are available for a particular nutrient. The IAEA database is searchable by nutrient, but it contains mainly information on CRMs with inorganic but not organic constituents. However, a consolidated reference table for CRMs, categorized by nutrient, matrix, and certification level, was needed and compiled for the NFNAP. The goal of this publication is to present this data set of food-matrix CRMs for organic and inorganic nutrients as a reference table, including certified and reference concentrations and UIs, in a format useful for selecting a CRM for a particular food analysis application.

Materials and methods

Information on available CRMs was obtained by searching supplier websites for current information, including: the National Institute of Standards and Technology (NIST; Gaithersburg, MD, USA; <http://www.nist.gov>); the Institute for Reference Materials and Measurements (IRMM;

Geel, Belgium; <http://www.irmm.jrc.be/html/home-page.htm>), supplier of BCR and European Reference Materials (ERM); LGC Promochem (Teddington, UK; <http://www.lgc.co.uk/>); the American Association of Cereal Chemists (AACC; St. Paul, MN, USA; <http://www.aaccnet.org>); the National Research Center for Certified Reference Materials (NRCCRM; Beijing, China; <http://www.nrccrm.org.cn>), supplier of GBW reference materials; the National Research Council Canada (NRC; Ottawa, Ontario, Canada; <http://inms-ienm.nrc-cnrc.gc.ca/>); as well as the International Atomic Energy Agency (IAEA) Database for Natural Matrix Reference Materials (<http://www.naweb.iaea.org/nahu/nmrm/nmrm2003/default.htm>). Consistent with the goals of the USDA's NFNAP, only nutrients found in the USDA Nutrient Database for Standard Reference [1] were included in this study; for example, CRMs for heavy metals and some other trace constituents were not considered. The nutrients evaluated are summarized in Table 1.

CRMs having assigned values for protein, moisture, and fat content were positioned in the appropriate sector of the existing AOAC food triangle model [6, 7]. For CRMs not supplied with values for fat, protein, and carbohydrate content, information for similar types of foods was obtained from the USDA National Nutrient Database for Standard Reference [1] or by analysis, to make an assignment to the proper sector.

Assigned nutrient values, UIs, and methods for the assignment of values for each CRM were obtained from the certificate of analysis accompanying each product. Various suppliers may define "certified" and "reference" values in different ways. For example, a certified value for a NIST standard reference material (SRM) is an assigned value for which the NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by the NIST (and the NIST has made measurements), while a reference value is a best estimate of the true value where all known or suspected sources of bias have not been fully investigated or accounted for by the NIST [8]. Both "certified" and "reference" values have assigned UIs, and could, therefore, be used in our comparison of analytical results with assigned values and their uncertainties. Units of concentration were standardized (e.g., assigned values in mg/kg were divided by 10 to convert to units of mg/100 g, which are customary in the food/nutrition industry). The specified UI for each nutrient concentration was also calculated as a percentage of the assigned certificate value (value) to yield the uncertainty interval percentage (UIP), as shown in Eq. 1:

$$\text{UIP} = \left(\frac{\text{UI}}{\text{value}} \right) \times 100 \quad (1)$$

Table 1 Nutrients evaluated in certified reference materials (CRMs)

Class	Nutrients
Proximates	Moisture, protein, ash, total fat
Carbohydrates	Total dietary fiber, glucose, fructose, sucrose, maltose, lactose
Minerals	Na, K, Ca, Mg, P
Trace elements	Mn, Cu, Zn, Se, Fe
Vitamins, water-soluble	Niacin, pantothenic acid, vitamin B1 (thiamin), vitamin B2 (riboflavin), vitamin B6, vitamin B12, folate, vitamin C
Vitamins, fat-soluble	Vitamin D, tocopherols ^a , vitamin A (retinol), vitamin K, carotenoids ^b
Fatty acids	C10:0, C12:0, C14:0, C16:0, C18:0, C16:1, C18:1, C18:2, C18:3, C20:0, C20:5, C22:6, C22:0, C22:5, C24:0
Amino acids	Alanine, arginine, aspartic acid, cystine, glutamic acid, glycine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, proline, serine, threonine, tryptophan, tyrosine, valine
Others	Cholesterol, choline, xanthines ^c , total procyanidins

^a α -tocopherol, β + λ -tocopherol, δ -tocopherol, λ -tocopherol

^b β -carotene, α -carotene, lutein, zeaxanthin, lycopene, β -cryptoxanthin

^c Caffeine, theobromine, theophylline

Results

Summary of the available food-matrix CRMs

A total of 690 assigned values and UIs for individual nutrients were compiled from the certificates of analysis for 63 CRMs. Reference tables summarizing assigned values, UIs, and UIPs for certified and reference nutrient concentrations in these CRMs, organized by nutrient, are included in full as supplementary online information for the nutrients listed in Table 1. The presentation of the data is illustrated by Table 2, which summarizes CRMs for water-soluble vitamins. CRM suppliers periodically update certificates of analysis (e.g., components may be added or removed, or the status of a nutrient value may change from certified to reference) or CRMs may become unavailable. Therefore, it is advisable to consult the supplier's current information prior to finalizing the selection of a material for an intended purpose.

Placement of CRMs in the AOAC triangle [6, 7] is shown in Fig. 1. The AOAC Task Force which developed this model anticipated that one or two foods in each sector would be representative of other foods in that sector, i.e., if a method worked properly for the analysis of one or two test foods, then it should also be useful for the analysis of other foods in that sector [6, 7]. At the NIST, the food triangle was used as a model for identifying where food-matrix CRMs were unavailable, and work was done to "fill in" the triangle, with other United States agencies and the food industry helping to set priorities [9]. While each sector does contain at least one CRM, additional materials are still needed because nutrient concentration as well as matrix composition, including factors other than proximate composition, may affect the perfor-

mance of specific assays; laboratories require a variety of CRMs to validate the applicability of an analytical method to specific foods.

Figure 2 expands upon the triangle/sector model by showing which CRMs in each sector have assigned values for specific nutrients. Based on this overview, it is clear that CRM availability is adequate for trace elements and minerals in foods in all sectors in which these nutrients are expected at nutritionally significant levels. In some cases, however, the available CRMs have only reference, not certified concentrations, in a given matrix (for example, Na in a high-sodium vegetable/mixed food material). No CRMs with assigned values are available for protein or amino acids in meats, fish, eggs, dairy products, or mixed foods (sectors 4, 6, 8, 9), where accurate data, particularly for phenylalanine, is needed for public health reasons. There is an overall shortage of CRMs for carbohydrates, especially sugars (and starch) in foods that are significant contributors of these nutrients (sectors 2, 3, 6), including sugars in a liquid or a high-moisture matrix (such as sweetened carbonated beverages). CRMs for vitamin B12 in meats are also lacking (sectors 4, 8, 9), as are CRMs for B vitamins in non-fortified grains (sector 5). Clearly, there are needs for CRMs with certified nutrient levels in particular matrices, and also for phytonutrients (e.g., phytosterols, flavonoids, phenolic acids) that are increasingly recognized to have health implications [10].

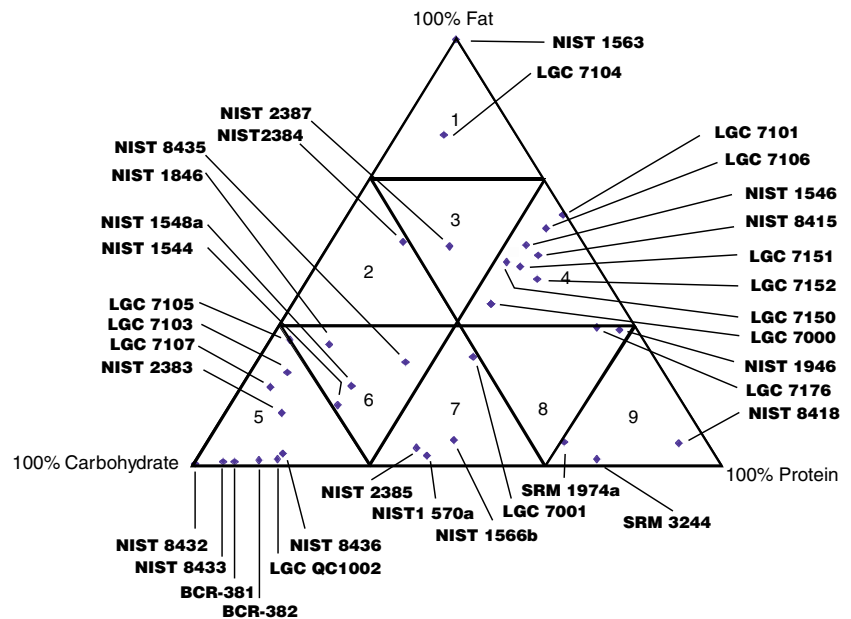
The NFNAP has included the analysis of more than 900 different foods and 100 components, with widely ranging matrix composition and nutrient levels for the USDA's Nutrient Database for Standard Reference [1]. During the NFNAP, CRMs were needed as controls for the analysis of key nutrients in foods that were significant contributors to

Table 2 Summary of food reference materials with assigned values and uncertainty intervals (UI) for water-soluble vitamins (similar tables for the nutrient classes listed in Table 1 are available as supplementary online material)

Sector ^a	Matrix	Supplier ^b CRM number	Assigned value (UI ^c ; UJP ^{d,e})															
			Total folate (mcg/100 g)	Folic acid (mcg/100 g)	Niacin (mg/100 g)	Pantothenic acid (mg/100 g)	Riboflavin (mg/100 g)	Thiamin (mg/100 g)	Vitamin B12 (mg/100 g)	Vitamin B6 (mg/100 g)	Vitamin C (mg/100 g)							
2	Baking chocolate	NIST 2384		1.21 (0.20, 16.5)				0.121 (0.016, 13.2)										
3	Peanut butter	NIST 2387		14.2 (0.6, 4.2)	1.08 (0.32, 29.6)			0.084 (0.017, 20.2)				0.466 (0.062, 13.3)						
4	Meat homogenate	NIST 1546		3.63 (0.38, 10.5)	0.576 (0.065, 11.3)			0.200 (0.059, 29.5)		0.6 (0.1, 16.7)		0.130 (0.061, 46.9)						
5	Wholemeal flour	BCR 121	50 (7, 14.0)					0.463 (0.039, 8.4)				0.410 (0.102, 24.9)						
5	Freeze-dried Brussels sprouts	BCR 431		4.3 (0.3, 7.0)				0.307 (0.034, 11.1)				0.48 (0.08, 16.7)						483 (24, 5.0)
5	Freeze-dried mixed vegetables	BCR 485	315 (28, 8.9)					0.270 (0.038, 14.1)		0.44 (0.19, 43.2)		0.151 (0.022, 14.6)						
5	Baby food	NIST 2383		1.81 (0.22, 12.2)	0.37 (0.14, 37.8)			5.97 (1.08, 27.3)		6.93 (0.87, 19.5)		8.5 (1.2, 18.2)						75.74 (13.04, 16.7)
5	Cereal	VMA 399	142 (14, 9.9)	1,395 (235, 16.1)				1.45 (0.06, 4.1)		3.4 (0.5, 14.7)		0.666 (0.085, 12.8)						76.9 (3.9, 5.1)
6	Milk powder	BCR 421		6.8 (0.2, 2.9)				1.74 (0.10, 5.7)		3.9 (0.3, 7.7)		0.84 (0.10, 11.9)						114.6 (6.6, 5.8)
6	Infant formula— powdered	NIST 1846		129 (28, 21.7)	4.87 (0.73, 15.0)			1.10 (0.34, 30.9)		1.8 (0.3, 16.7)		0.194 (0.061, 31.4)						
6	Whole-milk powder	NIST 8435		0.765 (0.093, 12.2)	2.67 (0.57, 21.3)			0.073 (0.005, 6.8)										
7	Stirred spinach	NIST 2385		0.299 (0.043, 14.4)														
9	Freeze-dried pigs liver	BCR 487	1,330 (130, 9.8)					10.68 (0.56, 5.2)		112 (9, 8.0)		1.93 (0.29, 15.0)						
9	Protein powder	NIST 3244		540 (120, 22.2)	17.2 (3.3, 19.2)			2.99 (0.23, 7.7)		10.7 (1.7, 15.9)		3.41 (0.22, 6.5)						89 (10, 11.2)

^a See [6, 7] and Fig. 1^b NIST National Institute of Standards and Technology (Gaithersburg, MD, USA; <http://www.nist.gov>); BCR=Institute for Reference Materials and Measurements (IRMM; Geel, Belgium; <http://www.irmm.jrc.be/html/homepage.htm>); AACC American Association of Cereal Chemists (AACC; St. Paul, MN, USA; <http://www.aaccnet.org>)^c ± UI specified on the certificate of analysis^d UI as a percentage of assigned value^e Blank cell indicates no certified or reference value is available; certified concentrations are in **bold** typeface; reference values are in regular typeface

Fig. 1 Sector locations [6, 7] of food-matrix CRMs for which fat, protein, and carbohydrate information is provided on the certificate of analysis. See Fig. 2 for the description of the matrices and suppliers



total intake [2]. A practical need for additional CRMs considering food type and nutrient level was encountered, including cases where composition beyond protein, fat, and carbohydrate content was an important factor in analytical measurements (e.g., considering the presence of a particular analyte and/or potential interferences in a given matrix, such as for carotenoids and vitamin C, naturally occurring forms of vitamins, or for vitamins and minerals in enriched foods). In addition to those mentioned above, CRMs were lacking for the following nutrients and matrices: amino acids in meat, fish, eggs, dairy products, and grains/cereals; iron in enriched grains and cereals; folate in non-enriched fruits, vegetables, and grains; *trans*-fatty acids in snack foods and margarines; vitamins A and D in fortified foods (e.g., dairy products and cereals); vitamin C in fruits, vegetables, and mixed foods; niacin in unenriched grains.

Another issue revealed during large-scale use of these CRMs was that some materials are supplied in packages containing a quantity much greater than that needed for typical analysis, and/or the certificate of analysis specifies that grinding/subsampling is required. For example, LGC7107 Madeira Cake is supplied as a 160-g individual cake in a sealed can; NIST 2384 Baking Chocolate is supplied in units of five 91-g individually wrapped bars. In such cases, a laboratory's homogenization and subsampling methods play a role (in addition to the analytical method performance itself) in the accuracy and precision of results if these procedures differ from those used during the certification process. Other CRMs are in lyophilized form, but if a laboratory does not routinely dry analytical samples, the nutrient concentrations and analyte extractability

could vary between samples and the CRM, even if the foods themselves are similar.

UIs for assigned values

There is also the question of uncertainty in certified values. Guidelines for the expression of uncertainty in measurements in general and in reference materials in particular (e.g., ISO Guide 35) are available [11–13]. However, many CRMs that are currently available were produced before these guidelines were developed. Therefore, the calculation of uncertainties may vary among materials, and are provided on the certificate of analysis for each CRM.

Figure 3 illustrates the UIPs (see Eq. 1) across all nutrients and CRMs (UIPs for assigned values in specific CRMs can be found in the supplementary online material). Regardless of the method of assigning the UIs, it is reasonable to assume that the values reflect the precision of the CRMs as metrology tools. The UIPs vary widely from less than 0.2% for moisture in some materials containing a high concentration of water to nearly 67% for copper in NIST RM 8432 Corn Starch, which has a certificate value for Cu of only 0.006 mg/100 g. Overall, 63.5% of the UIPs across all nutrients and CRMs were less than 10, 25.5% were 10–20, and 11% were greater than 20. The UIs for proximates, minerals, and trace elements were most consistently less than 10% of the assigned value, whereas the UIPs were significantly higher for vitamins (a little more than half of the fat-soluble and nearly three quarters of the water-soluble vitamin values have a UIP greater than 10). While concentration is also a factor in precision [14], as illustrated

◀ **Fig. 2** Overview of CRMs for major organic and inorganic components. Materials shown in **bold** font were used during the United States Department of Agriculture's (USDA) National Food and Nutrient Analysis Program (NFNAP) [2]. See Fig. 1 and [6, 7] for the description of the sectors. Abbreviations: *M* moisture; *Pr* protein; *Suc* sucrose; *Fru* fructose; *Glu* glucose; *Lac* lactose; *Mal* maltose; *Gal* galactose; *Fib* total dietary fiber; *St* starch; *B1* thiamin; *B1* riboflavin; *Nia* niacin; *Fol* folate; *PA* pantothenic acid; *Toc* tocopherols; *Car* carotenoids; *FA* fatty acids; *AA* amino acids; *Ch* choline; *X* xanthines; *Chl* cholesterol; *PrC* procyanidins. *NIST* National Institute of Standards and Technology (Gaithersburg, MD, USA; <http://www.nist.gov>); *BCR* Institute for Reference Materials and Measurements (IRMM; Geel, Belgium; <http://www.irmm.jrc.be/html/home-page.htm>); *AACC* American Association of Cereal Chemists (St. Paul, MN, USA; <http://www.aaccnet.org>); *LGC* LGC Promochem (Teddington, UK; <http://www.lgc.co.uk/>); *GBW* National Research Center for Certified Reference Materials (NRCCRM; Beijing, China; <http://www.nrccrm.org.cn>); *NRC* National Research Council Canada (NRC; Ottawa, Ontario, Canada; <http://inms-ienm.nrc-cnrc.gc.ca/>); *NIES* National Institute for Environmental Studies in Japan (Ibaraki, Japan; <http://www.nies.go.jp/>)

food composition databases, or research studies on diet and food composition, including biodiversity and sample-to-sample variability, and the nutritional significance of particular nutrient concentrations in these contexts. Over

25 years ago, this concern was raised by Stewart [15], along with a criterion of quality needed for food composition data:

... a number of existing methods do not yield accurate results. If the nutrient is greater than 5% of the recommended daily allowance, pre standard serving, or daily intake, whichever is greater, the analytical value should be within 10% of the accurate value. Many methods fail this criterion.

It is difficult to determine how accuracy was being assessed in 1981, because, at that time, there were very few, if any, food-based CRMs for most nutrients. If this 10% criterion is used now, then it appears that major suppliers of CRMs, presumably employing the best possible methods to certify CRMs, give uncertainty limits of greater than 10% for concentrations of many nutrients in many materials. With these limits, we cannot assess the accuracy of any analytical measurement for these components within 10%. In certain contexts, better accuracy may be required;

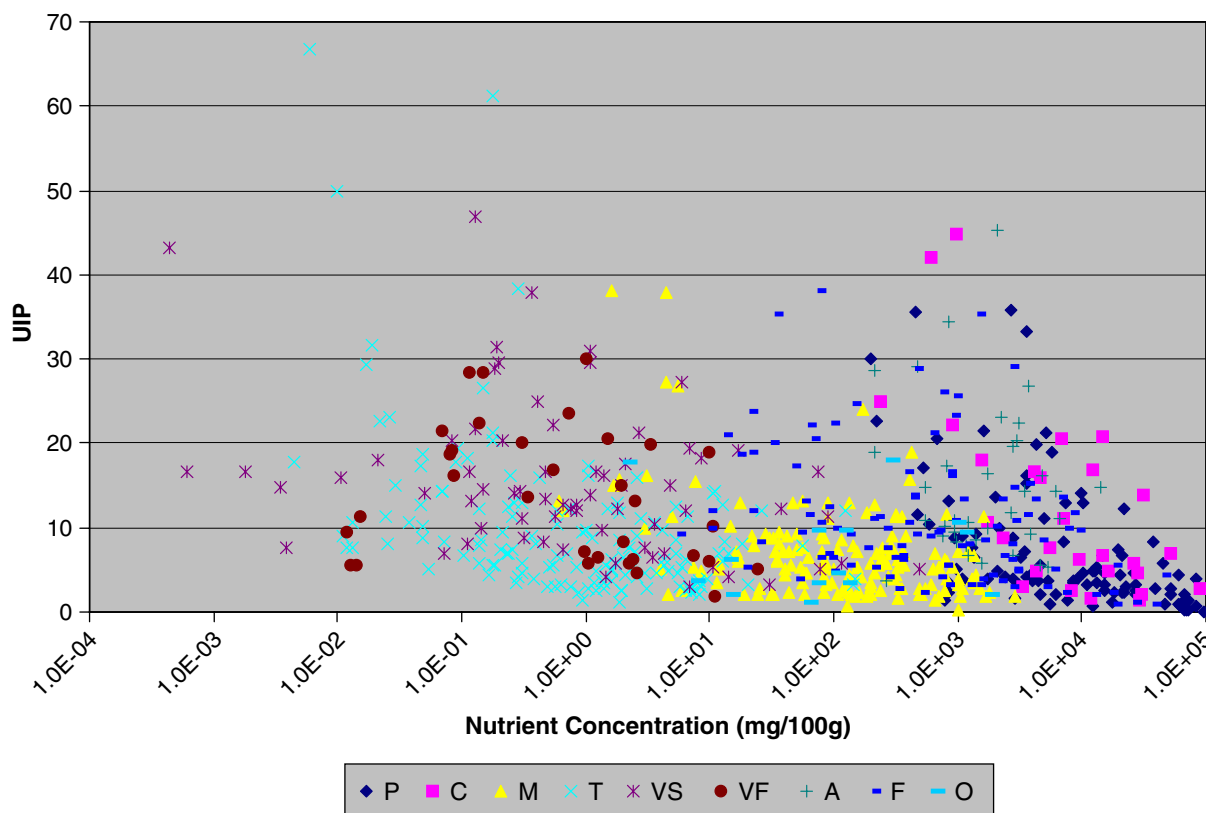


Fig. 3 Uncertainty interval percentages (UIPs) [$((\text{value}-\text{UI})/\text{value}) \times 100$] for certified and reference concentrations (in CRMs as a function of nutrient concentration (“value” and UI represent the assigned value and UI, respectively, on the certificate of analysis for

the certified reference material). P proximates; C carbohydrates; M minerals; T trace elements; VS water-soluble vitamins; VF fat-soluble vitamins; A amino acids; F fatty acids; O others (see Table 1 for the nutrients included in each group)

for example, analyses performed to validate differences in nutrient variables in experimental research diets through chemical analysis would generally require greater precision and accuracy than assays performed for food labeling purposes.

While models for evaluating the accuracy of a laboratory's nutrient measurements have been put forth [16–19], the precision requirement for CRM UIs has still not been significantly addressed. The precision (“measurement uncertainty”) of analytical methods and measurements, so-called “fitness for purpose,” has been discussed [20, 21]; however, when assigned values for CRMs are used to assess accuracy, the UI of the assigned value presents an inherent limit. Currently, CRM producers provide food-matrix materials with uncertainties of 10–20% of the assigned value, or higher in many cases. While this degree of uncertainty may be acceptable for assessing nutrients for food labeling or for nutrients present at nutritionally insignificant levels in a particular food, users of food composition data very definitely need to quantitatively assess the impact of the level of uncertainty in specific applications. Are the materials and assigned values presently available the best that can be produced, or can higher confidence in assigned values be accomplished? The viewpoint of most users is directed towards what additional CRM matrices are needed, not how precise the UIs must be. An analysis of the data for the CRMs analyzed will be presented in a separate publication as a basis to initiate this discussion. It is hoped that organizations supplying CRMs will begin to fill gaps in the CRMs for the matrices-nutrients that were identified during this study. A detailed analysis of a total of 2,629 CRM results for 26 CRMs, obtained over a 6.5-year period (1999–2006) from nine laboratories during routine analysis of samples for the NFNAP is currently underway.

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Disclaimer: Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology (NIST) or the United States Department of Agriculture (USDA), nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

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