Characterization of a mixed diet reference material (NBS RM 8431) for inorganic elements and selected organic nutrients

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Summary. A mixed diet reference material was produced from a typical diet menu selected from a human study. It included foods for 3 meals consumed in a day. Recommended values for 17 elements and for ash, fat and protein are given. In addition information is provided for individual sugars, total sugar and starch, where recommended values cannot yet be given.

Introduction

The usefulness of reference materials (RM's) for accuracy transfer, method validation, technology transfer and quality control monitoring has been well established [1 – 3] and several agencies routinely produce RM's. The Nutrient Composition Laboratory (NCL) is involved in developing methods for the determination of a wide variety of nutrients in individual foods and diets for which previous research has shown that existing biological reference materials are not very useful [4]. Because of a recognized need for the essentiality of a mixed diet RM for method development and quality control both within the laboratory and in the overall measurement system for nutrient analysis and because no suitable material was available from any of the RM producers at that time, a mixed diet RM was produced at NCL in 1981.

This mixed diet material, comprised of commonly consumed foods, was selected because a good RM must provide similar matrix effects, analyte concentrations, and contain the same chemical form of the analyte as the real world samples for which it will serve as a control. A typical diet menu was selected from a human study being conducted at the Beltsville Human Nutrition Research Center (Table 1) which included foods for the 3 meals consumed in a day. A detailed description of the preparation of this material has been published [5]. Serving portions of the foods required for the full days menu were blended, freeze-dried and reblended, care being taken to avoid possible trace element contamination since this material was being developed primarily as an inorganic element RM. The final powdered material was sieved through a polyethylene sieve and a 30/60 mesh cut was retained and packaged in units of approximately 30 g each. The final packaged material was radiation sterialized using 5 – 7 megared of gamma radiation (Neutron Products, Dickerson, MD, USA) to avoid microorganism growth.

Table 1. Menu for Mixed Diet RM-8431 [a]

Breakfast	Weight (g)
Orange juice, frozen, unsweetened	384
Grapefruit segments, canned	160
Cereal, LIFE	44
Milk, whole	305
Muffins, English, with raisins, toasted	62
Jelly	27
Sugar	11
Lunch	
Chicken, breast, roasted	106
Noodles, egg, steamed	200
Carrots, cooked, without salt	194
Asparagus, canned, without salt	152
Egg yolk, cooked	6.3
Rolls, Brown 'n' Serve	65
Cookies, shortbread	69
Pear nectar, canned	312
Dinner	
Fish, haddock, baked	106
Lemon juice, bottled	6
Tomatoes, canned, stewed	151
Sugar	12
Potatoes, boiled, without salt	171
Parsley, flakes	0.4
Bread, rye	62
Carrots, shredded	35
Cucumbers, chopped	35
Brownies, with pecans and coconut	100
Milk, whole	305
Total	3080.7

[a] Reference [5]

A number of units of the material were then characterized with regard to homogeniety for 8 elements (Ca, Cu, Fe, K, Mg, Mn, Na, Zn) to assure suitable homogeniety for use as a RM. Units of this material were then distributed to a number of expert investigators for the final overall characterization for 17 elements for which final recommended values were computed. A detailed report of homogeneity and sample size studies, individual values reported by collaborators for the major, minor, and trace

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Table 2. Methods used in the characterization of Mixed Diet RM-8431[a]

Method	Elements
Atomic absorption spectrometry (AAS) – flame	Ca, Cu, Fe, Mg, Mn, Zn
Atomic emission spectrometry (AES) – flame	Na, K
Graphite furnace atomic absorption spectrometry (GFAAS)	As,Cr,Se
Continuum source GFAAS (SIMAAC)	Al, Cu, Cr, Fe, Mn, Mo, Ni, Zn
Inductively coupled plasma emission spectrometry (ICP-AES)	Al, Ca, Cd, Co, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, V, Zn
Colorimetry	P
Isotope dilution mass spectrometry (IDMS)	Cr, Se
Neutron activation analysis (NAA)	Al, As, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Se, V, Zn
Voltammetry	Cd, Co, Cu, Ni, Pb, Zn
Fluorometry	Se

[a] Reference [6]

elements, and a detailed description of the methods used to generate recommended values for 17 elements appear in a more extensive publication [6]. In addition valuable information on proximate content, starch and individual sugar data were generated by participating investigators and information values are included in this report.

This diet material, previously known as TDD-1D [6], has been accepted for distribution to the scientific community by the National Bureau of Standards, Gaithersburg, MD, USA as Reference Material 8431.

Results and discussion

The elements of interest in food and diet materials cover 6 orders of concentration. Those determined in this study fall into three categories: 1) Major (concentrations greater than 100 μg/g) including Mg, Ca, Na, K and P; 2) Minor (concentrations of 1 – 100 μg/g) including Mn, Cu, Al, Rb, Zn, and Fe; and 3) trace (concentrations less than 1 μg/g) including Cd, Co, Cr, Pb, Ni, Mo, As, and Se [6]. Although modern analytical methods permit individual laboratories to successfully determine several elements in different concentration ranges, most laboratories routinely determine only a few of the elements of interest. Characterization of Mixed Diet RM-8431 was carried out by the collaborators using one or more of the 10 different techniques listed in Table 2 [6].

Homogeneity studies were performed in the authors' laboratory on eight elements (Cu, Zn, Mn, Na, K, Mg, Fe, and Ca) using nine "control" samples which were systematically selected to be representative of the whole batch of diet material and were set aside during the packaging of the material. These elements were determined in each of three preparations of each of the nine "control" samples by line source atomic absorption spectrometry (AAL) and this experiment was done twice. The homogeneity was evaluated by looking at the RSD's ($n = 9$) for each element for each experiment. In most cases, the RSD's of better than 2.0% were considered indicative of a very homogeneous material since these RSD's include analytical variability as well as representating the homogeneity.

Table 3. Recommended values for Mixed Diet RM-8431[a]

Element	Concentration (dry weight basis)
K	0.790 ± 0.042%
P	0.332 ± 0.031%
Na	0.312 ± 0.016%
Ca	0.194 ± 0.014%
Mg	0.065 ± 0.004%
Fe	37.0 ± 2.6 μg/g
Zn	17.0 ± 0.6 μg/g
Mn	8.12 ± 0.31 μg/g
Al	4.39 ± 1.07 μg/g
Cu	3.36 ± 0.33 μg/g
As	924 ± 344 ng/g
Ni	644 ± 151 ng/g
Mo	288 ± 29 ng/g
Se	242 ± 30 ng/g
Cr	102 ± 6 ng/g
Cd	41.8 ± 11.2 ng/g
Co	37.6 ± 7.8 ng/g

[a] Reference [6]

Once the homogeneity of the material was established, studies were carried out to determine what sample sizes would provide representative analytical subsamples of the whole lot of material. Analysts are aware that a very small sample may not be representative and prone to contamination while a very large sample may not be completely digested during sample preparation, both producing erroneous results. The recommended minimum sample size for this diet material is 0.25 g.

Recommended values

Recommended values for 17 elements generated from the reported values submitted by the collaborators are shown in Table 3 [7]. These values were generated by computation of a mean value and an uncertainty for each element from all data reported, since not enough data were available for most elements to do a rigorous statistical evaluation. No weighting was done based on increased confidence in a method or analyst and no data other than statistically identifiable outliers were excluded [6] from the final compilation. The computed means for the various elements represent the average of the reported recommended values for these elements. The uncertainties represent the 95% confidence interval. As a result, uncertainties for elements such as Ni, Co, As, and Cd are much larger than the actual computed standard deviation for the reported values due to the fact that only three values were reported for these elements ($n = 3$). Obviously, a larger number of reported values would improve the confidence in the recommended mean values for these elements. The authors feel reasonably confident in the recommended mean values and corresponding uncertainties but realize that these are subject to scrutiny by the scientific community.

Table 4. Recommended values for proximate data for Mixed Diet RM-8431

Ash	3.0 ± 0.1%	Protein	19.5 ± 0.5%
Fat	9.5 ± 1.1%		

Table 5. Sugar and starch data for Mixed Diet RM-8431

Fructose	5.82%	Maltose	1.81%
Glucose	6.52%	Total Sugar	28.3 %
Sucrose	11.1 %	Starch	24.6 %
Lactose	3.66%		

Other nutrients

Several investigators also reported other nutrient data for this diet material including values for: proximates (ash, fat, protein, moisture) and total sugar, individual sugars and starch. Data for proximates were reported from 3 or more laboratories and recommended values representating means and standard deviations are listed in Table 4. The data in Table 5 for individual sugars are the mean values reported from two different laboratories and were in close agreement. These values are not extensive enough to be given as recommended values at this time. It is hoped that this material will help to initiate further research work and effort to allow production of much needed future reference materials with certified values for many of these components plus additional organic nutrients.

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