Report of Analysis

Reference Material 8431a

Mixed Diet

Prepared by the United States Department of Agriculture

Distributed by the National Institute of Standards and Technology (formerly National Bureau of Standards)

This Reference Material (RM) is intended for use in calibrating instruments and evaluating the reliability of analytical methods for the determination of major, minor, and trace constituent elements, proximate content of ash, protein, fat, total sugar and starch in mixed diets and similar food and biological materials. RM 8431a was analyzed by a number of laboratories but not by NIST. Because NIST did not participate in the characterization, it is issued as a Reference Material rather than as an NIST Standard Reference Material (Certified Reference Material).

Material Application:

Together with other food-type RM's and Standard Reference Materials issued by NIST, RM 8431a is expected to be useful for evaluating the role of nutrient constituents in health and disease, establishing dietary requirements for nutrients, accumulating accurate base-line concentration data; and monitoring foods for nutrients, contaminants, and proximate constituents. RM 8431a consists of approximately 25 grams of freeze-dried material packaged in a plastic container.

Preparation:

This material is a replacement for RM 8431-Mixed Diet, supplies of which have been exhausted. The material for 8431a was collected at the Nutrient Composition Laboratory, United States Department of Agriculture, Beltsville, Maryland. A menu (Table I) of commonly consumed foods for 3 daily meals was selected from a human diet study being conducted in 1981 at the Beltsville Human Nutrition Research Center. Food was purchased in bulk, prepared for consumption and serving portions allocated to provide identical amounts of this daily menu for 40 subjects. Serving portions of two diets each were cut into bite size portions, mixed together in a large plastic box and immediately frozen for storage. Material from 12 of these boxes (24 daily diets of 3000 grams each, fresh weight) were then processed for RM 8431-Mixed Diet, as previously described(1). An additional 4 boxes of the material which had been long-term stored below -40 °C have now been processed for RM 8431a. Contents of separate boxes were individually blended after partial thawing, and then freeze-dried. The freeze-dried material from the four boxes was then mixed together and reblended. Care was taken throughout processing to avoid possible trace element contamination as the material was being developed primarily as a trace element Reference Material. All operations, except initial collection in a metabolic study diet kitchen, were carried out in clean room areas. Blending was carried out in a commercial food processor blender with a plastic bowl, using cutting blades specially fabricated of titanium. The final blended material was sieved through a 30 mesh polyethylene sieve. The material was then ^{60}Co gamma radiation sterilized in bulk at a dose of 3.3-3.9 Mrad to prevent bacterial growth. The material was then packaged in final units of 25 grams each in a Class 100 clean air hood. Preparation and packaging of the material were carried out at NCL, USDA, Beltsville, Md., under the direction of W.R. Wolf.

Gaithersburg, MD 20899
January 6, 1989

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

Recommended Procedures for Use:

RM 8431a should be kept refrigerated in its original container, tightly capped and not exposed to intense direct light or ultraviolet radiation.

Before each use, the contents of the bottle should be well mixed by gently shaking and rolling the container. The recommended minimum sample weight for this diet material is 250 milligrams (2). Moisture content should be determined on a separate sample for conversion of analytical results to a dry weight basis. The recommended drying method is to dry to constant weight in an air oven at 85 °C (16-20 hours). Recommended values reported in Table III represent total concentrations of elements in this Reference Material. Analytical methods and dissolution techniques should be capable of measuring total levels of elements for comparison with recommended values.

Chemical Analysis and Homogeneity Studies:

During packaging of RM 8431a, every tenth unit was retained as a control sample and homogeneity and analytical studies were conducted using these samples. Since the collected material for RM 8431a is a subbatch of that collected for the previous RM 8431 and the processing was as identical as possible to RM 8431, it was expected that constituent content of 8431a would be identical to that of RM 8431. In order to assure that this was correct, identical analyses of parallel samples of RM 8431 and RM 8431a were carried out for a number of elements by several techniques and for the carbohydrate fractions by one technique. Results of the elemental analysis comparison are shown in Table IIA as ratios of the results for RM 8431a to RM 8431. The results for similar comparative analysis of the individual carbohydrate fractions for the two materials are shown in Table IIB (3). The excellent agreement, within analytical error, of the two materials for both the inorganic elements and the organic constituents, confirms that RM 8431a is the same as RM 8431. Hence the extensive analytical information gathered for RM 8431 can be extended to recommended values for RM 8431a. As part of these comparison studies, one set of five of the control samples were analyzed for eight elements by line source AAS (Cu, Zn, Mn, Mg, Fe, and Ca) and flame emission (Na and K). Homogeneity of the material was evaluated from these analyses by computing the relative standard deviation (RSD) of all 5 analyses for each element. Since these RSD's reflect both analytical variability as well as sample heterogeneity, RSD less than or equal to 5% for this method were considered indicative of a homogeneous material. The RSD for Fe was slightly higher, but the method is known to have a higher analytical uncertainty and therefore still considered acceptable. The RSD's for all other elements were less than 4%. In addition, analyses of a number of elements by NAA showed excellent agreement for duplicate analysis of duplicate samples, further indicating sample homogeneity. Additional homogeneity checks for other constituents were not performed on RM 8431a.

Recommended Values:

Recommended values for 17 elements and estimates of uncertainties as given in Table III are taken from previously reported values for RM 8431 [2]. The method for computing final recommended values was to compute a mean value and standard deviation for each element from the mean value reported by each collaborator. No weighting was done based on increased confidence in a method or analyst and no data other than statistically identifiable outliers were excluded from the final compilation [2]. Uncertainties were computed as the 95% confidence interval by mulitplying the standard deviations by the appropriate Student "t" value and dividing by the $\sqrt{n}$. As a result, uncertainties for elements such as Ni, Co, As, and Cd are much larger than the actual computed standard deviations for the reported values because only 3 values were reported for these elements. Because the reported uncertainties in Table III include inter-laboratory and inter-method variations, analysts should expect smaller standard deviations within their respective laboratories using single analytical techniques. Table IV includes data for recommended values for proximate content of ash, protein, fat, total sugar and starch content [4]. Data for these values were analyzed in the same fashion as the elemental data in Table III.

Information Values:

Several investigators reported additional data on other nutrient constituents including individual sugars, phytate, and caloric content, which are included in Table V for information. One set of the individual sugar data representing two different methods of analysis has been previously published [3]. A confirming set of data on the individual sugars in close agreement with the previously published data, was obtained in the process of characterizing RM 8431. Data on fiber content of this material is listed in Table VI for information. As fiber content is dependent upon the methodology used for its measurement, no attempt is made to combine the data generated by

various methods. Each fiber content, generated by the identified method, is from an individual laboratory. These fiber values are not recommended values but are for information only, and are given in order to encourage further work in this area.

References:

1. Wolf, W.R. and Ihnat, M., Preparation of a Total Daily Diet Reference Material (TDD-1), in W.R. Wolf, Ed., Biological Reference Materials: Availability, Uses and Need for Validation of Nutrient Measurement., pp. 175-195, Wiley Interscience, N.Y., 1985.

2. Miller-Ihli, N.J., and Wolf, W.R., Characterization of a Diet Reference Material for 17 Elements. Analytical Chemistry, **58**, 3225-3230, 1986.

3. Li, B.W., Schuhman, P.J., and Wolf, W.R., Chromatographic Determinations of Sugars and Starch in Diet Composite Reference Material, J. Ag., Food Chemistry, **33**, 531-536, 1985.

4. Wolf, W.R., and Miller-Ihli, N.J., Characterization of a Mixed Diet Reference Material (NBS RM 8431), for Inorganic and Selected Organic Nutrients, Fresenius Zeitschrift fur Analytische Chemie 326, 702-704, 1987.

Cooperating Analysts:

RM 8431a was developed under the technical guidance of W.R. Wolf, USDA, Beltsville, MD. Analytical characterization of RM 8431, from which recommended values are used for RM 8431a, had been coordinated by N.J. Miller-Ihli, USDA, Beltsville, MD [2].

Comparative analytical data for RM 8431a and RM 8431 were supplied by:

Elemental Data:

W.C. Cunningham, FDA, Wash., DC by NAA.
N. Miller-Ihli,N.J., F.E. Green, USDA, Beltsville, MD by Flame AAS/Emission.
K. Patterson, C. Veillon, USDA, Beltsville, MD by SIDMS.
D.E. LaCrox, W. R. Wolf, USDA, Beltsville, MD by Flame AAS.

Carbohydrate Data:

B. Li, USDA, Beltsville, MD by Gas Chromatography.

The analytical expertise and elemental data for RM 8431 leading to recommended values in Table III was provided by the following collaborators and is gratefully acknowledged:

E. Greene, K. Patterson, S. Lewis, V. Iyengar, J. Morris, C. Veillon and O. Levander, USDA, Beltsville, MD.
R. Zeisler and S. Stone, NIST, Gaithersburg, MD.
J. Jones, K. Cook, M. Bueno, and A. Jones, FDA, Washington, DC.
C. Kuo, Northrop Services, Houston, Texas.
E. Offenbacher, St. Lukes/Roosevelt Hospital Center, New York, NY.
R. Parr, and R. Schelenz, International Atomic Energy Agency, Vienna, Austria.
D. Southgate and R. Faulks, Agriculture and Food Research Council, Norwich, England, U.K.
M. Stoeppler, Nuclear Research Center, Julich, Federal Republic of Germany.
J. Versieck and L. Vanballenberghe, University Hospital, Ghent, Belgium.

Thanks are also given to the following for supplying expertise and data on RM 8431 for ash, fat, protein, sugars and starch (Tables IV and V):

B. Li, C. Davis, H. Slover, USDA, Beltsville, MD.
E. Elkins and J. Dudek, National Food Processors Assoc. Washington, DC.
Hazelton Laboratories, Madison, WI.
D. Southgate and R. Faulks, Agriculture and Food Research Council, Norwich, England, U.K.

J. Hurst, Hershey Foods Corp., Hershey, PA.
C. Kuo, Northrop Services, Inc., Houston, TX.
E. Offenbacher, St. Lukes/Roosevelt Hospital Center, New York, NY.

Phytate data on RM 8431 (Table V) were generated by: R. Ellis, USDA, Beltsville, MD, (R. Ellis and E. Morris, Cereal Chemistry, (1983), 60, 121.)
Songsak Srianujata, Institute of Nutrition, Bangkok, Thailand.

Fiber data on RM 8431 (Table VI) were generated by:

D. Baker, USDA, Beltsville, MD, (Neutral Detergent Fiber, AACC Approved Methods: Method 32-20 (1978).
D. Southgate and R. Faulks, Agriculture and Food Research Council, Norwich, England, U.K. (Non-Starch Polysaccharides and Lignin, Englyst et. al., Analyst (1982), 107, p. 307). N. Asp. Chemical Center, University of Lund, Lund, Sweden, (Total Dietary Fiber, N. Asp et. al., J. Agric. Food Chem., (1983), 31 476).

Statistical support for RM 8431 was supplied by Larry Douglas, University of Maryland/USDA Statistical Consulting Analysis Services, Beltsville, MD.

Table I

Menu for Diet Reference Material 8431a[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 (1)

Table II

PARALLEL ANALYSES OF MIXED DIET REFERENCE MATERIALS

Ratio (8431a/8431)

A. Elements	INAA	AAS/FES	IDMS
Ca	0.987	0.981	
Na	0.957	0.960	
K	----	----	
Cl	0.988	----	
Mg	----	0.973	
Fe	0.976	0.952	
Zn	0.991	0.977	
Cu	----	0.964	
Se	0.991	----	0.987
Mn	----	0.991	
Al	1.028	----	
Sc	1.053	----	
Rb	1.006	----	
B. Carbohydrate Fractions			
Fructose	1.015		
Glucose	1.057		
Sucrose	1.016		
Lactose	1.020		
Maltose	0.946		
Total Sugar	1.026		
Starch	0.996		

Table III. Recommended Concentrations of Constituent Elements in Mixed Diet, Reference Material 8431a

Element	Recommended Value plus Estimate of Uncertainty[b]	Methods
K	0.790 ± 0.042%	3,7,9
P	0.332 ± 0.031%	1,7
Na	0.312 ± 0.016%	3,7,9
Ca	0.194 ± 0.014%	2,7,9
Mg	0.065 ± 0.004%	2,7,9
Fe	37.0 ± 2.6 μg/g	2,5,7,9
Zn	17.0 ± 0.6 μg/g	2,5,7,9,10
Mn	8.12 ± 0.31 μg/g	2,5,7,9
Al	4.39 ± 1.07 μg/g	5,7,9
Cu	3.36 ± 0.33 μg/g	2,5,7,9,10
As	0.924 ± 0.344 μg/g	4,9
Ni	0.644 ± 0.151 μg/g	5,7,10
Mo	0.288 ± 0.029 μg/g	5,7,9
Se	0.242 ± 0.030 μg/g	6,8,9
Cr	0.102 ± 0.006 μg/g	4,5,8,9
Cd	0.042 ± 0.011 μg/g	7,9,10
Co	0.038 ± 0.008 μg/g	7,9,10

[a] These values are obtained from RM 8431 as described in Reference [2].
[b] Analytical values are on a dry-weight basis. Uncertainties represent 95% confidence limits.

1. Colorimetry
2. Atomic Absorption Spectrometry-Flame
3. Atomic Emission Spectrometry-Flame
4. Electrothermal Atomic Absorption Spectrometry (Line Source)
5. Electrothermal Atomic Absorption Spectrometry (Continuum Source)
6. Fluorometry
7. Inductively Coupled Plasma Atomic Emission Spectrometry
8. Isotope Dilution Mass Spectrometry
9. Neutron Activation Analysis
10. Voltammetry

Table IV. Recommended Values for Proximate Constituents in Diet Reference Material 8431a[a]

Constituent	Value	Number of Laboratories Reporting Data
Ash	3.00 ± 0.09 %	7
Fat	9.5 ± 0.92 %	8
Protein	19.1 ± 0.62 %	5
Total Sugar	28.3 ± 1.7 %	3
Starch	24.6 ± 5.0 %	3

Due to changes during storage and handling, moisture correction should be determined on a separate subsample for each use of the material.

[a] These values are obtained from RM 8431 as described in Reference [4].

Table V. Data on Other Nutrient Constituents and Caloric Content*

Constituent/ Property	Value	Number of Laboratories Reporting Data
Fructose	(5.8%)	2
Glucose	(6.5%)	2
Sucrose	(11.1%	2
Lactose	(3.7%)	2
Maltose	(1.8%)	2
Phytate	(2.10 mg/g)	2
Caloric Content	(436 Cal/100 g)	2

* These values, from a limited number of laboratories are for information only and are included in this report to encourage use of this material for generating and reporting additional nutrient information on this material.[3].

Table VI. Fiber Content for Diet Reference Material 8431 by Different Methods

Method	Value*
Neutral Detergent	(1.83 %)
Non-Starch Polysaccharides + Lignin	(3.6 %)
Total Dietary Fiber	(5.3 %)

* Values for information only. These values reflect known method dependency of fiber measurements and are included to encourage further intercomparisons of these methodologies.

As more data become available, the recommended values may be revised and additional constituents added to the information list. Data, comments and inquiries from users of this material will be welcome and should be directed to:

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