

Edited By
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Components related to BCR CRM **BCR CRM** BCR CRM BCR CRM BCR CRM BCR CRM BCR CRM nutritional labeling 380 whole 381 rye 382 wheat 383 haricot 384 lyoph 184 bovine 185 bovine milk flour flour beans pork muscle muscle liver powder Total carbohydrate (90) Avail. (89) Avail. (79) Avail. weight, % CHO CHO CHO Sugars weight, % 36.3 lactose Dietary fiber weight, % 8.22 3.25 11.91 Kjeldahl N weight, % 4.50 1.25 2.12 1.05 13.69  $86.6^{b}$  $63.1^{b}$ 19 5 Sodium, mg/kg (3920)2820 (2000)(2100)

(1750)

(1510)

7750

2850

15530

230

(16600)

(150)

79

(11200)

(131)

214

Table 3. Certified reference materials for nutritional labeling<sup>a</sup>

(12450)

(9450)

Potassium, mg/kg

Calcium, mg/kg

Vitamin A, mg/kg Vitamin C, mg/kg

Iron, mg/kg

[From Ikins, W., DeVries, J., Wolf, W.R., Oles, P., Carpenter, D., Fraley, N., Ngeh-Ngwainbi, J. (1993) The Referee, AOAC International, 17(7), pp. 1,6,7]

# A Food Matrix Organizational System Applied to Collaborative Studies Purpose

2850

220

Currently, no systematic procedure exists for validating analytical methods as applicable to all foods. Clearly, such a systematic procedure is needed. AOAC Associate Referees must assure the methods they validate and recommend are, or are not, applicable to all foods, and AOAC Official Method Committees must judge the applicability range of the methods. Further, such a system should be designed to minimize the effort required for collaborative studies while maximizing the value of the resulting data to AOAC Official Method users.

The Subcommittee on Food Definition of the AOAC Task Force on Methods for Nutrient Labeling Analyses investigated systematic approaches to validate analytical methods as applicable to all foods and food products. This Subcommittee report describes a system that better reflects how foods vary on a chemical basis rather than on a commodity basis, thereby reducing the number of samples that would represent all foods to a manageable number. The Subcommittee recommended this system for studies applicable to all food.

#### Discussion

The prospect of coordinating a collaborative study involving 40 or more different foods may discourage many researchers from fully exploring the scope of applicability of their method. As a result, researchers may limit the scope of the study to a few food groups to reduce the analytical burden on the participating laboratories.

<sup>&</sup>quot; Distributed by the Community Bureau of Reference (BCR), Brussels, Belgium

<sup>&</sup>quot; Total protein.

Many of the 40 or more foods selected to represent all foods for a collaborative study may be very similar to one another on a dry basis, and may behave chemically, and thus analytically, in a very similar way. In any analytical procedure, water can be added or subtracted to suit the requirements of the method. Ash, in general, does not have a great impact on the performance of analytical methods for organic material in foods. Thus, the behavior of a given food in an analytical method is primarily determined by the relative proportions of protein, fat, and carbohydrate.

Figure 1 depicts a scheme by which all foods can be organized according to their relative levels of these three major classes of food components. The points of the triangle represent 100% of either protein, fat, and carbohydrate (moisture and ash are excluded for the reasons cited above). The triangle is divided into nine sectors that serve to group foods according to their basic chemical makeup. Table 1 contains a listing of some of the foods found in the U.S. Department of Agriculture Handbook No. 8 that might be useful for a collaborative study. The foods have been categorized into each of the nine sectors of the triangle. Careful selection of two foods or food products from each

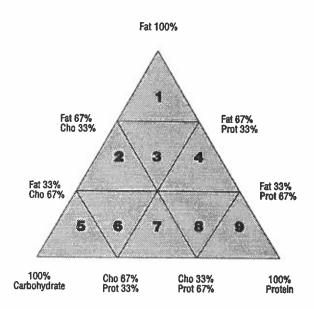


Figure 1. Schematic layout of food matrixes suggested for a collaborative study based on protein, fat, and carbohydrate content, excluding moisture and ash.

sector will cover the entire range of carbohydrate, protein, and fat, as well as other food attributes.

If a diagram such as Figure 1 were to be used to select samples for a collaborative study, two samples from a sector could be selected to account for variation in the type of protein, fat, or carbohydrate that may have an impact on the performance of the method. Examples of these variations within carbohydrates are high fiber foods vs high sugar foods. Other variations include fats containing significant amounts of short chain fatty acids vs those containing predominantly long chain fatty acids, or foods containing more hydrophilic proteins as opposed to those containing predominantly hydrophobic proteins. In addition, two foods may be selected within a sector that vary according to the extent of processing each has undergone.

## Example

Figure 2 illustrates how this system can be used with the Youden concept for statistical evaluation of an analyte of interest. For the purposes of this example, the U.S. Department of Agriculture Handbook No. 8 was used to select representative foods that fell within each of the nine sectors. Samples containing similar levels of the analyte of interest will be ultimately paired for statistical purposes. The actual percent of protein, fat, and carbohydrate used to construct Figure 2 are shown in Table 2.

When conducting a collaborative study to validate a method to be used on all foods or a particular subset of matrixes, a system such as that depicted in Figure 1 can be used to select foods for the validation study. In doing so, samples that are truly chemically different from one another can be used to test the limitations of a method. If a method fails for food matrixes located within certain sectors, those factors that cause the method to be ineffective can be more clearly understood and delineated.

#### Recommendation

The AOAC Task Force on Methods for Nutrient Labeling Analyses recommends the four AOAC food committees and the Official Methods Board adopt this systematic approach for validating methods for all foods. Adoption of this approach will minimize the efforts required for collaborative studies while maximizing the value of the resulting method to its users.

Comments from AOAC members, volunteers, and method users are cordially invited. Please contact Nancy Palmer, AOAC Technical Director, at the AOAC offices.

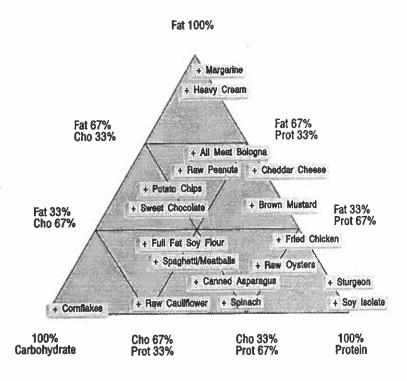


Figure 2. Schematic layout of food matrixes to be used in a collaborative study based on protein, fat, and carbohydrate content, excluding moisture and ash.

Table 1. Selected example foods from U.S. Department of Agriculture Handbook No. 8 and their corresponding sectors in the triangle<sup>a</sup>

111 1110 1110			<del>_</del> _	
Sector	ITEM	PROT%	FAT%	CHO'S%
1		0-33%	67-100%	0-33%
	Cooking oil	0	100	0
	Margarine	1	99	0
	Cream/heavy whipping	5	88	. 7
	Avocados/California	9	67	24
	Bacon	11	88	1
2		0-33%	33-67%	33-67%
	Potato chips	6	42	53
	Chocolate/sweet	5	36	59

Table 1. Selected example foods from U.S. Department of Agriculture Handbook No. 8 and their corresponding sectors in the triangle"

Dried almonds 20 59 21 Raw peanuts 28 52 20 All-meat bologna 33 57 9 Brown mustard 34 36 30 Blue cheese 40 56 4 Cheddar cheese 42 54 4 Hamburger 46 54 0 Raw eggs 51 45 4 Caviar 60 33 37 0	Sector	ITEM	PROT%	FAT%	CHO'S%
Raw peanuts  All-meat bologna  33 57 9  33-67% 33-67% 0.33%  Brown mustard  34 36 30  Blue cheese 40 56 4  Cheddar cheese 42 54 4  Hamburger 46 54 0  Raw eggs 51 45 4  Caviar 60 33 7  Canned turkey 63 37 0	3		0-33%	33-67%	
Raw peanuts     28     52     20       All-meat bologna     33     57     9       33-67%     33-67%     0-33%       Brown mustard     34     36     30       Blue cheese     40     56     4       Cheddar cheese     42     54     4       Hamburger     46     54     0       Raw eggs     51     45     4       Caviar     60     33     7       Canned turkey     63     37     0       0-33%     0-33%     67-100%       Bananas     5     1     94       Rice bran     6     1     92       Cornflakes     8     0     91       Noodles     14     5     81       Canned lima beans     23     1     76       Frozen biscuit dough     9     18     74       Raw cauliflower     33     2     64       0-33%     0-33%     33-67%       Plain cocoa     19     27     54       Spaghetti with meatballs     24     20     56       Whole milk yogurt     27     30     43       Wheat germ     33     13     54       Baby food-chicken/vegetables		Dried almonds	20	59	21
Brown mustard  Brown mustard  Blue cheese  40  56  4  Cheddar cheese  42  54  Hamburger  46  54  Caviar  60  33  7  Canned turkey  63  37  0-33%  67-100%  Bananas  5  1  94  Rice bran  6  1  92  Cornflakes  8  0  91  Noodles  14  5  81  Canned lima beans  23  1  76  Frozen biscuit dough  Raw cauliflower  33  2  64  Spaghetti with meatballs  24  20  56  Whole milk yogurt  27  30  43  Wheat germ  33  13  54  Baby food-chicken/vegetables  39  24  38		Raw peanuts	28	52	20
Brown mustard 34 36 30  Blue cheese 40 56 4  Cheddar cheese 42 54 4  Hamburger 46 54 0  Raw eggs 51 45 45  Caviar 60 33 7  Canned turkey 63 37 0  Bananas 5 1 94  Rice bran 6 1 92  Cornflakes 8 0 91  Noodles 14 5 81  Canned lima beans 23 1 76  Frozen biscuit dough 9 18 74  Raw cauliflower 33 2 64  Spaghetti with meatballs 24 20 56  Whole milk yogurt 27 30 43  Wheat germ 33 13 54  Baby food-chicken/vegetables 39 24 38		All-meat bologna	33	57	9
Blue cheese 40 56 4 Cheddar cheese 42 54 4 Hamburger 46 54 0 Raw eggs 51 45 4 Caviar 60 33 7 Canned turkey 63 37 0 -33% 0-33% 67-100% Bananas 5 1 94 Rice bran 6 1 92 Cornflakes 8 0 91 Noodles 14 5 81 Canned lima beans 23 1 76 Frozen biscuit dough 9 18 74 Raw cauliflower 33 2 64 Spaghetti with meatballs 24 20 56 Whole milk yogurt 27 30 43 Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38	4		33-67%	33-67%	0-33%
Cheddar cheese		Brown mustard	34	36	30
Hamburger 46 54 0 Raw eggs 51 45 4 Caviar 60 33 7 Canned turkey 63 37 0 0-33% 0-33% 67-100% Bananas 5 1 94 Rice bran 6 1 92 Cornflakes 8 0 91 Noodles 14 5 81 Canned lima beans 23 1 76 Frozen biscuit dough 9 18 74 Raw cauliflower 33 2 64 Spaghetti with meatballs 24 20 56 Whole milk yogurt 27 30 43 Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38		Blue cheese	40	56	4
Raw eggs       51       45       4         Caviar       60       33       7         Canned turkey       63       37       0         Do-33%       0-33%       67-100%         Bananas       5       1       94         Rice bran       6       1       92         Cornflakes       8       0       91         Noodles       14       5       81         Canned lima beans       23       1       76         Frozen biscuit dough       9       18       74         Raw cauliflower       33       2       64         0-33%       0-33%       0-33%       33-67%         Plain cocoa       19       27       54         Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38		Cheddar cheese	42	54	4
Caviar       60       33       7         Canned turkey       63       37       0         Dananas       0-33%       0-33%       67-100%         Bananas       5       1       94         Rice bran       6       1       92         Cornflakes       8       0       91         Noodles       14       5       81         Canned lima beans       23       1       76         Frozen biscuit dough       9       18       74         Raw cauliflower       33       2       64         0-33%       0-33%       33-67%         Plain cocoa       19       27       54         Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38		Hamburger	46	54	
Canned turkey 63 37 0  0-33% 0-33% 67-100%  Bananas 5 1 94  Rice bran 6 1 92  Cornflakes 8 0 91  Noodles 14 5 81  Canned lima beans 23 1 76  Frozen biscuit dough 9 18 74  Raw cauliflower 33 2 64  Plain cocoa 19 27 54  Spaghetti with meatballs 24 20 56  Whole milk yogurt 27 30 43  Wheat germ 33 13 54  Baby food-chicken/vegetables 39 24 38		Raw eggs	51	45	4
0-33%   0-33%   67-100%	4	Caviar	60	33	7
Bananas 5 1 94 Rice bran 6 1 92 Cornflakes 8 0 91 Noodles 14 5 81 Canned lima beans 23 1 76 Frozen biscuit dough 9 18 74 Raw cauliflower 33 2 64  Plain cocoa 19 27 54 Spaghetti with meatballs 24 20 56 Whole milk yogurt 27 30 43 Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38		Canned turkey	63	37	0
Rice bran       6       1       92         Cornflakes       8       0       91         Noodles       14       5       81         Canned lima beans       23       1       76         Frozen biscuit dough       9       18       74         Raw cauliflower       33       2       64         0-33%       0-33%       33-67%         Plain cocoa       19       27       54         Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38	5		0-33%	0-33%	67-100%
Cornflakes 8 0 91  Noodles 14 5 81  Canned lima beans 23 1 76  Frozen biscuit dough 9 18 74  Raw cauliflower 33 2 64		Bananas	5	<sup>3</sup> 21 <b>1</b>	94
Noodles		Rice bran	6	1	92
Canned lima beans 23 1 76  Frozen biscuit dough 9 18 74  Raw cauliflower 33 2 64  0-33% 0-33% 33-67%  Plain cocoa 19 27 54  Spaghetti with meatballs 24 20 56  Whole milk yogurt 27 30 43  Wheat germ 33 13 54  Baby food-chicken/vegetables 39 24 38		Cornflakes	8	0	91
Frozen biscuit dough 9 18 74  Raw cauliflower 33 2 64  0-33% 0-33% 33-67%  Plain cocoa 19 27 54  Spaghetti with meatballs 24 20 56  Whole milk yogurt 27 30 43  Wheat germ 33 13 54  Baby food-chicken/vegetables 39 24 38		Noodles	14	5	81
Raw cauliflower       33       2       64         0-33%       0-33%       33-67%         Plain cocoa       19       27       54         Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38		Canned lima beans	23	I	76
0-33% 0-33% 33-67%  Plain cocoa 19 27 54  Spaghetti with meatballs 24 20 56  Whole milk yogurt 27 30 43  Wheat germ 33 13 54  Baby food-chicken/vegetables 39 24 38		Frozen biscuit dough	9	18	74
Plain cocoa       19       27       54         Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38		Raw cauliflower	33	2	64
Spaghetti with meatballs 24 20 56 Whole milk yogurt 27 30 43 Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38			0-33%	0-33%	33-67%
Spaghetti with meatballs       24       20       56         Whole milk yogurt       27       30       43         Wheat germ       33       13       54         Baby food-chicken/vegetables       39       24       38		Plain cocoa	19	27	
Whole milk yogurt 27 30 43 Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38		Spaghetti with meatballs	24	20	
Wheat germ 33 13 54 Baby food-chicken/vegetables 39 24 38		Whole milk yogurt	27	30	
Conned orners are		Wheat germ	33	13	
Conned conserve		Baby food-chicken/vegetables	39	24	38
		Canned asparagus	39	6	

Table 1. Selected example foods from U.S. Department of Agriculture Handbook No. 8 and their corresponding sectors in the triangle<sup>a</sup>

Sector	ITEM	PROT%	FAT%	CHO'S%
7		33-67%	0-33%	33-67%
	Skim milk	41	1	58
	Spinach	41	4	55
	Full-fat soy flour	42	23	35
	Defatted peanut flour	54	10	36
8		33-67%	0-33%	0-33%
	Fried chicken (dark meat)	64	29	7
	Raw oysters	62	13	25
9		67-100%	0-33%	0-33%
	Raw beef liver	69	13	18
	Soy protein isolate	83	0	17
	Cottage cheese	66	20	14
	Dried chipped beef	84	16	0
	Raw catfish	85	15	0
	Sturgeon	91	10	0
	Canned tuna in water	97	3	0
	Gelatin	100	0	0

<sup>&</sup>quot;Percent of protein, fat, and carbohydrate, excluding moisture and ash, are normalized to 100%.

Table 2. Selected example foods from the USDA Handbook No. 8 and their corresponding sectors in the triangle<sup>a</sup>

ITEM	SECTOR	PROT%	FAT%	CHO's %
Margarine	1	1	99	0
Cream/heavy whipping	1	5	88	7
Potato chips	2	6	42	53
Chocolate/sweet	2	5	36	59
Raw peanuts	3	28	52	20
All-meat bologna	3	33	57	9
Brown mustard	4	34	36	30

Table 2. Selected example foods from the USDA Handbook No. 8 and their corresponding sectors in the triangle

ITEM	SECTOR	PROT%	FAT%	CHO's %
Cheddar cheese	4	42	54	4
Cornflakes	5	8	0	91
Raw cauliflower	5	33	2	64
Spaghetti with meatballs	6	24	20	56
Canned asparagus	6	39	6	55
Spinach	7	41	4	55
Full-fat soy flour	7	42	23	35
Fried chicken (dark meat)	8	64	29	7
Raw oysters	8	62	13	25
Soy protein isolate	9	83	0	17
Sturgeon	9	91	10	0

<sup>&</sup>quot;Percent of protein, fat, and carbohydrate, excluding moisture and ash, are normalized to 100%.

[Excerpted and revised from Craft, N., & Boyer, K. (1993) The Referee, AOAC International, 17(5), pp. 6-8]

## Preparation of In-House Quality Assurance Control Materials for Food Analysis A. Purpose

To provide guidelines for the preparation and use of affordable, well-characterized, stable foods or food-like materials for use as quality assurance control materials (CM) in nutritional analyses of foods.

#### **B.** Desirable Properties

Macro- and/or micronutrients may be added if no source can be found to mimic major food classes or matrix types, but spiking should be avoided where natural sources are available. The addition of nutrients does not necessarily mimic the form and compartmentalization of a real food matrix. Lyophilized materials may be used to improve storage and stability only if comparability to the original matrix has been demonstrated.

Stability.—Candidate CMs should be stable at normal laboratory room temperatures, or well-defined instructions should be determined for keeping the material stable over time with respect to macro- and micro-nutrient content. It is always better to err on the side of conservatism when packaging and storing CMs (i.e., if the influence of exposure to light on an analyte is unknown, then protect the CM from light).

### C. Sources and Selection of Matrixes for Characterization

Major data bases.—Major data bases such as the U.S. Department of Agriculture Handbook 8, can be searched for foods that provide the best combination of nutrients and macro components (fat, carbohydrate, protein, and moisture) to represent broad classes of foods. This information may be used to locate food items within the "Food Triangle" which is being considered by AOAC INTERNATIONAL as a means of identifying a small number