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REPORT 85.67 Pr.nr. 404.0070

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Verzendlijst: direkteur, sektorhoofden, direktie VKA, afd. AM (4x), Bibliotheek (2x), Projektbeheer, Projektleider (J.H. Slangen), circulatie, dhr. Katan.

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Report of the Eurofoods Interlaboratory trial 1985 on laboratory procedures as a source of discrepancies between food tables

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September, 1985.

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SUMMARY

The Eurofoods interlaboratory trial 1985 was set up to determine whether differences in laboratory procedures between countries form an important cause of discrepancies between nutrient values in different food tables and nutrient data banks. Twenty leading laboratories in Europe and the U.S.A. participated in the trial. Each received a well-homogenized dry sample of 100 g of egg powder, full-fat milk powder, whole rye meal, whole wheat meal, biscuits and french beans. Heterogeneity between samples of the same food was checked by analysis of nitrogen in 10 random samples of each food, and was found to be negligible (coefficient of variation 0.1-0.2%). Each laboratory was requested to perform analyses of dry weight by a prescribed vacuum stove method, and of protein, fat, available carbohydrates, total dietary fiber and ash by its own routine method. Analyses were made in duplicate, with two technicians each contributing one value. All results were later recalculated to dry weight to eliminate the effect of losses or gains in moisture.

- For dry weight, the coefficient of variation between laboratories ($CV_{between}$) ranged from 0.3-0.6%. Optional non-vacuum methods yielded results quite similar to those of the prescribed method.
- For protein the $CV_{between}$ ranged from 2.8% for egg to 6.4% for wheat and rye. Recalculation using uniform Kjeldahl factors reduced these CV's to 2.7, 4.7 and 5.2% respectively. Reproducibility within laboratories was occasionally poor.
- The $CV_{between}$ for total fat ranged from 5.4% for milk to 54.0% for french beans, the CV being higher when the absolute fat content of the food was lower. The reported fat content of egg powder ranged from 29 to 44 g/100 g dry weight. Part of the variability was clearly due to different laboratories using different methods for the same food, for instance acid hydrolyses versus solvent extraction. However, laboratories using ostensibly similar methods still reported widely diverging results.
- For available carbohydrates the $CV_{between}$ (excluding egg) ranged from 9% for biscuits to 27% for beans. Individual results for carbohydrate content of whole wheat meal ranged from 36 to 82 g/100 g dry weight.

Variability was somewhat reduced if differences in mode of expression (as g of polymeric starch versus as g of equivalent monosaccharides) were eliminated; the $CV_{between}$ now ranged from 7 to 23%. Effects of specific methods could not be identified because too many different methods were used.

- The $CV_{between}$ for total dietary fiber ranged from 23% for french beans to 84% for biscuits. A major part of this variability was due to the use of methods of different principle.
- Results for ash were reasonably consistent, with a $CV_{between}$ ranging from 3.3% for milk to 6.7% for egg.
- It is concluded that leading laboratories in different countries may produce widely different values for proximate constituents (macronutrients) in common foods. There is a need for better standardization of methods. As an initial step, reference materials of certified nutrient concentration should be produced and be made widely available.

1. INTRODUCTION

In order to improve the compatibility of nutrient data banks in Europe, Eurofoods had developed different activities (West, 1). The present trial was planned to determine the influence of differences in analytical and other procedures in laboratories that contribute to food tables, on the nutrient values in these tables.

Substantial information is available on the precision of specified analytical procedures, e.g. methods described by the Association of Official Analytical Chemists and the International Standards Organization. These data are collected by means of collaborative studies, in which all participating laboratories use the same accurately described method for the analysis of identical samples. However, it is a well-known fact that different laboratories actually use different methods to determine a certain nutrient in a certain food. Even if the same methodological principles are followed, subtle differences in procedure and in calibration materials could still cause large differences in outcome. Very few data are available about the influence of these differences. Therefore the Eurofoods subcommittee on laboratory analyses planned the present Eurofoods Interlaboratory trial 1985.

It was agreed to study only the major macronutrients protein, total fat, available carbohydrates, total dietary fiber and ash and to use products that can easily be homogenized and handled. Each participant was encouraged to apply his own methods of analysis and calculation as used routinely.

One American and 19 European laboratories that regularly contribute nutrient values to nutrient data banks were invited to participate in this study. All laboratories agreed to participate in the trial (see list of participants).

2. MATERIALS

The six foods described were selected for the trial.

1) Egg powder: commercially available spray dried whole egg powder.
2) Full-fat milk powder: commercially available spray dried full cream milk powder.
3) Whole rye meal: whole rye grains, donated by RIVRO-Institute (Wageningen, The Netherlands), were ground by RIKILT to pass a sieve of 1 mm openings.

4) Whole wheat meal: whole wheat grains, donated by TNO/IGMB (Wageningen, The Netherlands) were ground by RIKILT to pass a sieve of 1 mm openings.
5) Biscuits: Maria-biscuits (Koninklijke Verkade Fabrieken BV, Zaandam, The Netherlands), were broken and ground by RIKILT to pass a sieve of 1 mm openings.
6) French beans: freeze-dried french beans (Summer Season, Coöp. Condensfabriek "Friesland" w.a., Leeuwarden, The Netherlands), were ground by RIKILT to pass a sieve of 1 mm openings.

About 3 kg of each of these six foods were ground to pass a sieve of 0.5 mm openings. The foods were carefully homogenized by quartering and divided into samples of $\pm$ 100 g, using the sample divider of the Institute for Livestock Feeding and Nutrition Research at Lelystad, The Netherlands.
This sample divider consisted of a rotary tube system, rotating at a frequency of 100 min^{-1}. The samples were packed into airtight black plastic bottles with screwcaps. Prior to the distribution of the samples to the participants, sample homogeneity was tested by RIKILT as follows. Ten samples of each product were randomly chosen and each sample was analyzed for protein. To determine the analytical precision one sample of each product was also analyzed for protein ten times. All analyses of one foodstuff were carried out in rapid succession by one analyst on one day. The results (Table 1) show that there is no significant difference (F-test, 5%-level) between the standard deviation within the sample (analytical precision) and between the samples. The variation between samples was extremely small, and could largely be ascribed to analytical error rather than to true differences between different samples of one food. Samples can thus be regarded as homogeneous. Sample bottles , labelled with the name of the product, were vacuum-sealed in airtight plastic foil and were sent, together with instructions (Appendix 2) to the participants by the end of February 1985. No report of damaged samples was received. Analyses were performed during the months of March, April and May 1985.

Table 1. Homogeneity of the samples as judged by the variation in protein content within and between samples (bottles)

	Within sample			Between samples			
	n	mean protein (g/100 g)	s_{within}	n	mean protein (g/100 g)	$s_{between}$	$CV_{between}$
Egg powder	10	50.231	0.0595	10	50.240	0.0801	0.16%
Milk powder	10	27.406	0.0422	10	27.377	0.0408	0.15%
Rye meal	9	9.840	0.0384	10	9.870	0.0254	0.25%
Wheat meal	9	11.764	0.0230	10	11.766	0.0246	0.21%
Biscuits	9	7.974	0.0159	10	7.964	0.0143	0.11%
French beans	10	14.654	0.0212	10	14.642	0.0282	0.19%

n = number of determinations
s_{within} = standard deviation within one bottle
$s_{between}$ = standard deviation between bottles

3. STATISTICAL ANALYSIS AND PRESENTATION OF THE RESULTS

All results are calculated on dry matter as determined by each separate laboratory with the prescribed vacuum stove method (Appendix 1). Dry weight values of laboratories 6, 7, 17 and 18 are based on other dry weight methods. The results, calculated per 100 g dry weight are presented in Tables 3 to 22. For each sample and nutrient a graph with the two individual and the mean values per laboratory is given, Figures 1 to 8.

As all laboratories were asked to perform all analyses in duplicate with two technicians on different days, each providing one value, it was possible to calculate the variation within the laboratories. Statistical evaluation followed the principles of the International Standards Organization norm ISO 5725-1981 (2) to calculate the standard deviations and coefficients of variation of overall reproducibility (s, CV), within-laboratories variation (s_{within}, CV_{within}) and between laboratories variation ($s_{between}$, $CV_{between}$). Individual extreme values were detected by Dixon test (extreme mean values) and poor duplicates by Cochran test (extreme differences between duplicates). Moreover the Youden rank test was used to detect laboratories that reported high or low results for a certain nutriënt throughout all samples. Values that tend to be outlying with marginal significance (stragglers: $5\% > P \geq 1\%$) are marked in the tables with a single asterisk (*) and outliers ($P < 1\%$) are marked with a double asterisk (**).

Contrary to ISO 5725, outliers are not rejected, because ISO 5725 only applies to interlaboratory tests with one method. Rejecting an outlying laboratory could imply rejecting a method that gives the "true" value. Moreover the aim of this interlaboratory trial was to investigate the influence of different laboratory procedures. To get an impression of this influence, the 95% confidence limits for each nutrient and sample may be calculated.
If a laboratory performs an analysis in duplicate under the conditions of this trial and finds a value x, the "real" nutrient value is expected to lie between

$$x \pm 2\sqrt{s^2 - \frac{1}{2}(s_{within})^2}$$

This deviation from the mean is given in the tables as a percentage of the mean ("Confidence limits").

Horwitz (3) examined the results of more than 150 collaborative studies, organized by the Association of Official Analytical Chemists and found a general curve relating the reproducibility with the concentration of the analyte, as shown in Figure 0. It represents the reproducibility that can be obtained when all laboratories use the same rigidly defined standardized methods. Horwitz also derived an empirical equation that relates $CV_{between}$ to the concentration (C) of the analyte, expressed in negative powers of 10:

$$CV_{between} = 2\ (1-0{,}5\ \log C)$$

In the tables this value is given as "Achievable $CV_{between}$".

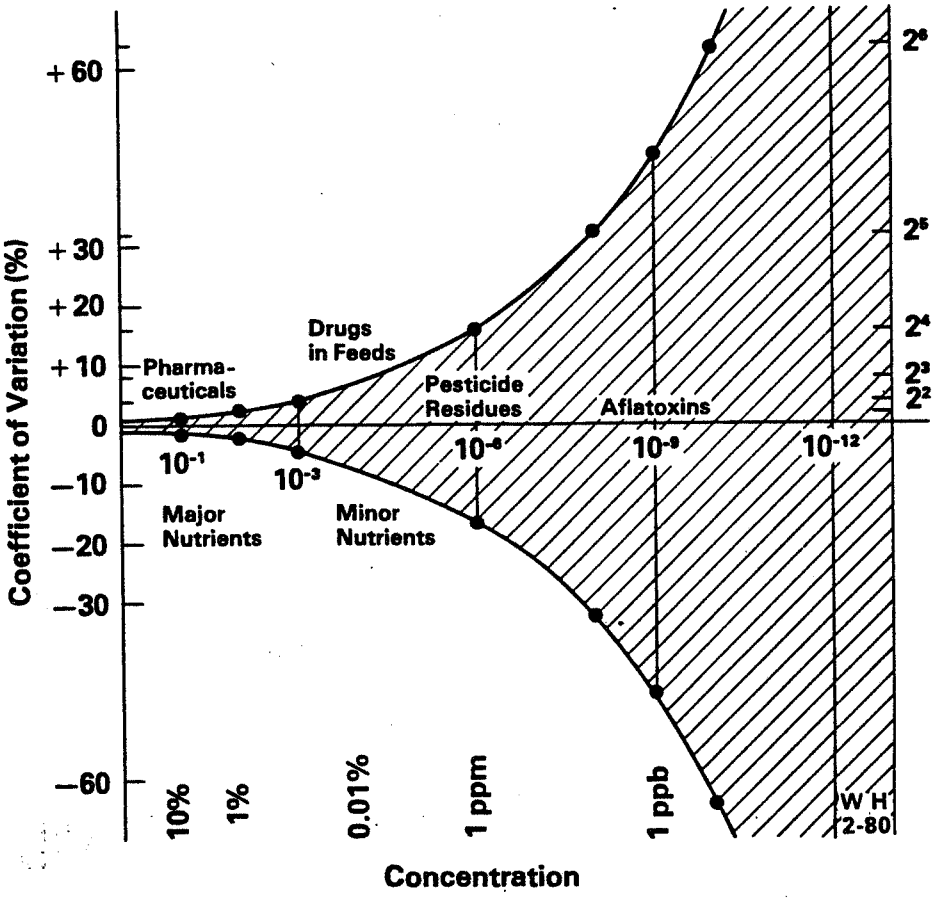

Figure 0. The general curve relating overall reproducibility (coefficient of variation) with concentration of the analyte (expressed as negative powers of 10).

4. RESULTS

In a number of cases results were missing because a laboratory was not used to perform this particular analysis. One laboratory did not had sufficient time to complete the analysis and reported the results after the statistical evaluation had taken place. It was not possible to make a new evaluation including the results of this laboratory. In other cases no explanation for the missing data was available.

Table 2. Number of laboratories not reporting an analysis, by nutrient and food. Twenty laboratories participated in the trial

Nutrient	Egg	Milk	Rye	Wheat	Biscuits	French beans
Dry weight, prescribed	5	5	4	4	4	6
Dry weight, optional	8	8	8	8	8	8
Protein	3	2	1	1	1	2
Total fat	2	2	1	1	1	3
Available carbohydrates	13	4	4	4	4	5
Total dietary fiber	16	13	6	6	6	6
Ash	2	2	1	1	1	2

4.1 Dry weight

The results of the dry weight determination by the prescribed vacuum stove method (Appendix 1) are given in Table 3 and Figure 1. Lab 20 showed outlying high values for rye, wheat and biscuits. The Cochran test showed poor duplicates for lab 14 (wheat) and lab 10 (french beans). As all laboratories used the same method, outlying results were deleted before calculations (Table 4). The results agree with the Horwitz equation (3), that predicts a $CV_{between}$ of 2%.

Thus packing and storage conditions of the samples proved to be adequate to protect against changes in moisture content, and the prescribed method gives reproducible results. The slightly high results of lab. 20 were still used to recalculate the other analyses of lab 20 to g/100 g dry matter, because the differences with the mean dry weight values of the other laboratories were quite small (Table 4), and besides the samples might actually have lost some moisture. Table 5 summarizes the reported results of the optional dry weight methods which some labs performed in addition to the prescribed method. The Youden rank test detects laboratory 6 and 18 as producing outlying high results. No outlying low results were detected.

The Dixon test also reveals laboratory 18 for three samples as outlying. The Cochran test detects that laboratory 1 gives a poor duplicate for wheat.

Optional dry weight methods used (Table 6) varied in duration and temperature of the drying process. Laboratories 1 and 6 applied vacuum. The results of these labs are somewhat higher but only laboratory 6 proved to be significantly higher. Comparing the mean results of the optional methods with the prescribed vacuum method, there are no significant differences, except for french beans (t-test, P=5%).

Values of CV_{within} and CV of the optional methods (Table 4) show that the different methods used did not have an important influence on the precision.

Conclusions

- The similarity in dry weight values reported by different laboratories using the prescribed method shows that little or no loss or gain of moisture had occured.
- The similarity in moisture content found with the various optional methods shows that prescribing a standard dry weight method may have been an unnecessary precaution in this trial.

Table 4. Summary of the results of the dry weight determinations

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Eurofoods trial method						
Number of labs.	15	15	15	14	15	14
		g dry weight/100 g product as received				
Mean	95.260	97.370	91.897	88.225	97.827	94.454
Range	94.6-96.5	96.9-98.6	91.3-93.0	86.7-89.2	97.4-98.5	93.3-95.3
CV	0.54%	0.47%	0.54%	0.65%	0.27%	0.57%
CV_{within}	0.18%	0.12%	0.13%	0.18%	0.08%	0.13%
$CV_{between}$	0.50%	0.45%	0.52%	0.61%	0.26%	0.55%
Confidence limits $\pm$	1.0%	0.9%	1.1%	1.3%	0.5%	1.2%

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Optional methods						
Number of labs.	12	12	12	12	12	12
			g dry weight/100 g product as received			
Mean	95.195	97.273	92.062	88.410	97.934	93.769
Range	94.7-96.6	96.9-98.0	91.4-94.0	87.7-90.5	97.1-99.0	91.9-96.0
CV	0.54%	0.35%	0.79%	0.87%	0.56%	1.16%
CV_{within}	0.23%	0.16%	0.40%	0.39%	0.38%	0.41%
$CV_{between}$	0.49%	0.32%	0.68%	0.77%	0.41%	1.1%
Confidence limits ±	1.0%	0.7%	1.5%	1.7%	1.0%	2.2%

4.2 Protein

Seventeen to 19 laboratories submitted values for protein (Table 7 and Figure 2). The results for protein show an overall coefficient of variation (CV) of 3-8% (Table 8). For a number of samples part of this variation is caused by the use of different Kjeldahl-factors (Table 9).

Table 9. Variation in Kjeldahl nitrogen-to-protein factors between laboratories

Product	Number of labs using Kjeldahl factor					Total
	5.70	5.83	6.25	6.38	6.68	
Egg			16		1	17
Milk			5	13		18
Rye	5	7	7			19
Wheat	6	7	6			19
Biscuits	5	2	12			19
French beans			18			18

To eliminate the effect of these differences all results were recalculated (Table 10 and Figure 3) using the following Kjeldahl-factors as recommended by FAO/WHO (4):

egg : 6.25
milk : 6.38
rye : 5.83
wheat : 5.83
biscuits : 6.25
french beans : 6.25.

These recalculated results indeed show some decrease in the variation between laboratories especially with rye and wheat (Table 8).
Inspection of the rank order of the laboratories shows that lab. 20 gives consistently high values throughout the range of foods, and lab 5 consistently low values. Dixon test also indicates that the results of lab. 20 are higher in most samples. The results of lab 5 were not detected as outlying by Dixon test. Within-laboratory variation examined with the Cochran test revealed poor duplicate values for lab. 18.
The methods used differ in choice of catalyst, and procedures for digestion, destillation and determination of the ammonia formed (Table 11). Most laboratories used $CuSO_4$ as a catalyst, some used selenium (lab. 1, 4, 5), others used mercury (lab. 7,8, 19). Combinations of $CuSO_4$ and selenium (lab. 10, 13, 18) and of $CuSO_4$ and TiO_2 (lab. 3, 11) were also used. The influence of the type of catalyst on the results for egg powder was investigated with the t-test. No significant differences (P=5%) were found, so we did not examine the other products. Digestion was performed in block digestors, but classical Kjeldahl flasks were also used. Destillation of the ammonia was generally performed by steam destillation. Receiver solutions consisted of boric acid or sulfuric acid. Ammonia was mostly determined by titrimetric methods, sometimes using automated equipment.
One laboratory used a colorimetric continuous flow method to determine the ammonia.
Only lab 19 made a correction for non-protein-nitrogen. This correction appears to have the greatest effect on values for milk powder and french beans, where the Dixon test shows outlying low protein values for lab 19. Note that these outlying values may be closer to the true protein contents than the mean of the other laboratories.
Precision data summarized in Table 8 lead to the following conclusions.
$CV_{between}$ is somewhat higher than predicted by the Horwitz-equation (achievable $CV_{between}$). However, this can be expected as this equation has been derived from collaborative studies using a uniform method. Therefore the differences in methods have only a small effect.
The CV_{within} is higher than the values claimed by several laboratories (< 1%) for their own methods. The influence of differences in Kjeldahl factors is modest.

Conclusions

- The discrepancies in protein values between laboratories are rather small, but are still higher than expected.
- Variability for protein in cereals would be decreased if all laboratories used the Kjeldahl factor of 5.83 recommanded by FAO/WHO.
- The difference between duplicate values is quite high in some laboatories.

Table 8. Summary of the results of the protein determination, and the CV achievable with uniform methods

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Results as reported						
Number of labs.	17	18	19	19	19	18
			g protein/100 g dry weight			
Mean	52.983	28.100	10.175	12.662	7.840	15.092
Range	49.7-56.9	25.7-32.8	9.2-11.8	11.1-14.3	7.2-9.5	11.7-15.8
CV	3.1%	6.0%	7.0%	6.7%	7.9%	6.3%
CV_{within}	1.4%	3.1%	2.9%	2.0%	4.8%	1.3%
$CV_{between}$	2.8%	5.2%	6.4%	6.4%	6.2%	6.2%
Confidence limits ±	6.0%	11.3%	13.5%	13.0%	14.2%	12.5%
Recalculated using uniform Kjeldahl-factors						
Number of labs.	17	18	19	19	19	18
			g protein/100 g dry weight			
Mean	52.774	28.251	9.981	12.485	8.094	15.092
Range	49.7-56.9	26.2-33.4	9.4-11.3	11.3-14.7	7.3-9.5	11.7-15.8
CV	3.0%	6.2%	5.5%	5.5%	7.1%	6.3%
CV_{within}	1.4%	3.1%	2.9%	2.0%	5.0%	1.3%
$CV_{between}$	2.7%	5.4%	4.7%	5.2%	5.0%	6.2%
Confidence limits ±	5.7%	11.7%	10.3%	10.7%	12.3%	12.5%
Achievable $CV_{between}$	2.2%	2.4%	2.8%	2.8%	2.8%	2.7%

4.3 Total fat

The reproducibility of the fat determination was rather poor, especially for products low in fat (Table 12). Thus, fat content reported for whole wheat meal ranged from 1.8 to 5.8 g/100 g dry weight. For egg powder the range was 29 to 44 g/100 g. For collaborative trials using carefully standardized uniform methods, an analte such as fat should give a $CV_{between}$ of only 2 to 4% (3); the CV's obtained where much higher (Table 12).

The Youden rank test applied to the results of the total fat determination (Table 13, Figure 4) reveals that lab. 3 gives high results in the six foods more often than can be expected by chance, whereas lab. 17 shows low results. However these deviations are not confirmed as outlying by the Dixon test. The Cochran test shows that lab. 3 gives poor duplicates in 3 samples.

Methods used are reviewed in Table 14. Acid hydrolysis followed by extraction with petroleum ether, or diethylether was applied by most of the participants. Two different procedures were used: methods according to Weibull-Stoldt (W.S.) and methods according to Schmid-Bondzynski-Ratzlaf (S.B.R.). With all samples S.B.R.-methods gave on average higher results than Weibull-methods. These differences are significant (P=5%) for wheat, rye, biscuits and french beans. Milk powder was analyzed mostly with Röse-Gottlieb methods. It appears that in milk powder S.B.R-methods give higher results than Röse-Gottlieb-methods, which in turn give higher results than Weibull-methods. However these differences did not prove significant (P=5%). A number of laboratories used extraction techniques with different solvents such as chloroform/methanol (lab. 1, 6, 7, 15) and dichloormethane/methanol (lab. 3), more or less similar to the Folch-method. The performance of these methods with the different products is not quite consistent. Thus labs 1, 3 and 6, using the Folch method, obtained a high value for fat in egg powder, but lab 15, also using Folch, reported a lower value than average. Labs 3, 7 and 15 reported high values for fat in french beans, wheat, rye and biscuits using the Folch method, but lab. 6, which also used Folch, consistently found lower-than-average fat contents in these products.

Conclusions

- The differences between laboratories in the fat content found in these foods are unacceptably high. Only part of this variability is due to differences in methods.
- Within-laboratory variations were relatively large for products low in fat.

Table 12. Summary of the results of the total fat determination, and the CV achievable with uniform methods

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Number of labs.	18	18	19	19	19	17
			g fat/100 g dry weight			
Mean	37.779	27.278	2.554	3.036	11.558	2.747
Range	29.4-44.2	24.5-30.0	1.6-4.5	1.8-5.8	9.9-15.4	1.2-5.8
CV	8.9%	5.7%	43.9%	42.4%	10.6%	59.8%
CV_{within}	2.0%	2.0%	24.5%	30.6%	2.7%	25.7%
$CV_{between}$	8.7%	5.4%	36.4%	29.3%	10.3%	54.0%
Confidence limits ±	17.7%	11.1%	80.7%	72.9%	20.9%	114%
Achievable $CV_{between}$	2.3%	2.4%	3.5%	3.4%	2.8%	3.5%

4.4 Available carbohydrates

Thirteen of the 20 laboratories performed carbohydrate analyses, and three more calculated carbohydrates by difference.Seven laboratories expressed their carbohydrate results as monosaccharides, four as polymeric starch and two as "carbohydrates". These results are given in Table 15 and Figure 5.

The effect of these different modes of expression was investigated by recalculating all data to monosaccharides (Table 16, Figure 6).

A factor of 1.11 was used to convert polysaccharides into monosaccharides. Lab. 5 expressed its results as carbohydrates, i.e. the sum of fructose, glucose, saccharose, maltose and starch. Because lab. 5 did not report mono- and disaccharides separately, the relative amount of each sugar was estimated from the data of lab. 1 before recalculating the data of lab. 5. Few results have been reported for egg, so they are not shown in the tables.

Evaluation of the results expressed as monosaccharides by the Youden rank test, shows that no extremely high or low values throughout the whole range of samples occur. However, the Dixon and Cochran tests indicate that lab. 3 gives an outlying low result and poor duplicates for milk.

Available carbohydrates were defined as follows: the sum of free sugars (mono-disaccharides and other oligosaccharides up to approximately 10 monosaccharides units) and starch. Methods used (Table 17) show many differences. Lab. 1, 3, 6 and 19 isolated sugars and starch by separate extraction and determined in each extract sugars and starch with various methods. Labs 11 and 5 only made a separate extraction and determination of the sugars. The other laboratories did not separate sugars and starch. Solubilization and hydrolysis of starch was done by various techniques. Sugars were determined by enzymatic, colorimetric, gas chromatographic and reductiometric techniques. All these procedures were combined in different combinations leading to the analytical methods shown in Table 17. Only labs 2 and 12 used entirely the same method and perhaps because of this their results differ little (except for french beans). The method used by lab. 8 is similar to that of labs 2 and 12 except for the solubilization of starch, and gives much lower results. This can be expected. The results of labs 5 and 6 were very similar, probably because their methods only showed little differences. Labs 10, 14 and 15 did not use an analytical method to determine the content of carbohydrates, but calculated this value by difference. As can be seen (Table 15) this leads to values close to the mean value, except for lab. 10 whose results are generally higher. These high results can be expected because lab. 10 determined crude fiber instead of total dietary fiber. As a result, certain fiber components were counted as carbohydrate.

Because CV_{within} was much smaller than the total CV (Table 18) it is obvious that the differences in analytical methods have an important influence on the results. With rigid standardization (3), theoretical CVs of 2.1 to 2.3% should be possible (Table 18). Expressing the results as monosaccharides (equal units) and omitting results calculated by difference, improves the precision slightly.

Conclusions

- The reproducibility of the available carbohydrate determination between laboratories was very poor.
- Only a small part of the variability is due to different modes of expression, e.g. starch as monosaccharides versus starch as polymer weight.
- Differences in methodology probably explain some but not all of the variability.
- Calculation of carbohydrates by difference causes no major bias, except when crude instead of total dietary fiber is used.

Table 18. Summary of the results for available carbohydrates, and the CV achievable with uniform methods

	Milk	Rye	Wheat	Biscuits	Frenchbeans
Original results					
Number of labs.	16	16	16	16	15
			g/100 g dry weight		
Mean	34.724	69.645	69.294	75.239	42.398
Range	14.9-44.4	38.4-94.0	35.7-82.1	63.4-89.3	28.3-67.5
CV	20.1%	20.8%	18.0%	9.9%	27.4%
CV_{within}	4.8%	4.2%	4.5%	3.3%	3.1%
$CV_{between}$	19.4%	20.4%	17.5%	9.3%	27.3%
Confidence limits ±	39.5%	41.2%	35.5%	19.3%	54.7%
Expressed as monosaccharides and "by difference" values eliminated					
Number of labs.	14	13	13	13	12
			g/100 g dry weight		
Mean	35.317	71.745	71.535	78.168	41.990
Range	14.9-44.4	42.6-94.0	39.7-82.1	70.4-89.3	31.5-67.5
CV	20.1%	19.0%	16.4%	7.7%	22.8%
CV_{within}	4.8%	4.4%	4.8%	3.4%	3.3%
$CV_{between}$	19.5%	18.5%	15.6%	6.9%	22.6%
Confidence limits ±	39.6%	37.5%	32.0%	14.7%	45.4%
Achievable $CV_{between}$	2.3%	2.1%	2.1%	2.1%	2.3%

4.5 Total dietary fiber

Fourteen laboratories reported values for total dietary fiber. Two more had determined crude fiber; these values were not used in the statistical analysis, because crude fiber is a small and variable part of total dietary fiber.

Only a few laboratories reported results for egg and milk (Table 19, Figure 7). Results reported as not detectable are represented as 0.000.

The Youden rank test applied to biscuits, french beans, rye and wheat shows that lab. 7 and 16 give outlying high results throughout these products. No outlying low results could be detected. The Dixon test revealed one outlying high result: lab. 7, biscuits. Lab. 3 generally reported the highest differences between duplicates, but only in one sample (rye) this was regarded as outlying (Cochran test).

The candidate AOAC-method described by Prosky et al (5), used by 5 laboratories, and the related method described by Asp, used by two laboratories (Table 20), resulted in values that agreed well. Labs 3, 11 and 19 used the Englyst method or a modification of it. Although the Youden rank test did not yield low outliers, labs 3, 6, 8, 11 and 19 did tend to report lower dietary fiber values than the trial mean. Prosky et al. (5) also found that the Englyst method gave lower values than the AOAC-method, and pointed out that dietary fiber as determined by the Englyst method does not include lignin. The low values of lab. 6 can be explained, because this laboratory used the neutral detergent fiber method, which determines only the water-insoluble fiber components.

Comparing CV_{within} and total CV (Table 21) it is clear that there exists a strong influence of the different analytical procedures on the results of the determination of total dietary fiber. In an interlaboratory study (5) recently organized to test the candidate AOAC method, a CV for whole wheat of 11% was found, as opposed to 27% in our trial where a variety of methods was used.

Conclusions

- There was a large variability in dietary fiber values as reported by different laboratories. This was probably due to well-known differences between methods.

Table 21. Summary of the results for total dietary fiber

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Number of labs.	4	7	14	14	1 4	14
			g/100 g dry weight			
Mean	0.361	0.278	15.427	13.109	3.116	27.610
Range	0-0.8	0-0.8	10.0-22.0	8.7-19.8	0.7-10.9	15.6-35.8
CV	117%	130%	26.3%	26.9%	84.3%	23.8%
CV_{within}	22.6%	15.6%	9.5%	5.1%	7.1%	6.8%
$CV_{between}$	115%	129%	24.6%	26.4%	84.0%	22.8%
Confidence limits $\pm$	233%	260%	50.9%	53.3%	168%	46.5%

4.6 Ash

Eighteen or 19 laboratories submitted values for ash.

Results for ash are summarized in Table 22 and Figure 8. The Youden rank test only detects lab. 10 as giving an outlying high result throughout the range of foods. The Dixon test indicates two outlying high results for laboratory 2 and one outlying high result for laboratory 4. The Cochran test indicates poor duplicates for lab. 20.

Methods used (Table 23) show various pre-ashing procedures, ashing times and temperatures. Lab. 10 uses a very different method: this laboratory determines the sulphate ash and converts it to the ash content. This laboratory generally produced high results. Apart from this, precision data (Table 24) suggest that there is little influence of the different analytical procedures on the results for ash. The $CV_{between}$ agrees with the achievable $CV_{between}$ obtained when methods were rigidly standardized (3).

Conclusions

- The results for ash agreed rather well between laboratories, although outliers did occur.

Table 24. Summary of the results for ash, and achievable CV with uniform methods

	Egg	Milk	Rye	Wheat	Biscuits	Frenchbeans
Number of labs.	18	18	19	19	19	18
			g/100 g dry weight			
Mean	4.652	6.014	1.795	1.781	1.665	6.634
Range	4.3-5.8	5.7-6.7	1.6-2.0	1.6-2.1	1.5-1.9	5.9-7.8
CV	6.9%	3.4%	5.7%	5.5%	7.2%	6.1%
CV_{within}	1.8%	0.7%	3.0%	2.1%	4.4%	1.6%
$CV_{between}$	6.7%	3.3%	4.9%	5.1%	5.7%	5.9%
Confidence limits $\pm$	13.7%	6.7%	10.7%	10.6	13.0%	12.0%
Achievable $CV_{between}$	3.2	3.1	3.7	3.7%	3.7%	3.0%

5. DISCUSSION

5.1 Results of this trial

The aim of this trial was to determine whether laboratory procedures could be a serious cause of discrepancies between different nutrient data banks in Europe. The trial has shown that this may indeed be the case. Prominent laboratories in various countries produced widely different values for the concentration of fat, carbohydrate and fiber, and to a lesser extent also of protein, in everyday foods. Thus the reported fat content of the egg powder ranged from 29 to 44 g/100 g and that of the biscuits from 10 to 15 g/100 g.

The situation for available carbohydrates was even worse, with reported values for e.g. the carbohydrate content of whole wheat meal ranging from 36 to 82 g/100 g dry weight.

It should be noted that several sources of error that occur commonly in routine analyses of foods had already been reduced or eliminated beforehand in this trial. Thus the foods were supplied as stable, well-ground powders of uniform particle size, easy to store, handle and sample. Also the samples had been carefully packaged and clearly marked and identified. Thus, the analyst could find out if his values were more or less correct simply by consulting a food table (Still, an interchange did occur in one laboratory.

As a result e.g. a value for the protein content of egg powder of 8.1 g/100 g was reported. This incident points out how vulnerable laboratories are to administrative or labelling errors.) Last but not least, the Eurofoods trial samples may have been analyzed with more than usual care and attention.
Because of all this, values produced in daily routine analyses of unknown samples will probably show an even larger variation between and within laboratories than the values reported here.

5.2 Causes of variability

As for the causes of those discrepancies, differences in methods probably play an important role. Thus Elkins (6) reported a much lower interlaboratory variability for protein and fat in the cooperative study of the Committee of Canning Industry Chemists. A main difference with the present study was that the participants in the Canning Industry study all used the same methods, as defined and described by the Association of Official Analytical Chemists, AOAC (7).
Horwitz (3) analyzed more than 150 collaborative studies (participants using exactly the same methods) organized by the AOAC, and was able to derive an empirical equation that relates the between-laboratory variation to the concentration of the analyte, independent of the nature of the analyte or the analytical technique. This calculated achievable $CV_{between}$ showed much lower values than the real $CV_{between}$ in the present trial, for fat, available carbohydrates and total dietary fiber. So method effects were clearly visible in the present trial in the results for fat, available carbohydrates and fiber. However, differences in methodological principles are not the full explanation of the variability in results, as laboratories using similar methods sometimes still reported widely diverging results. The cause for this is unknown.
Variability caused by differences in Kjeldahl nitrogen-to-protein conversion factors was present but small. Still, more widespread use of standard Kjeldahl factors is to be recommended. There was a clear effect of differences in conventions for expressing carbohydrate content, i.e. as polymeric starch versus as the weight of the monosaccharides produced from starch by hydrolysis. Although the variability caused by these different modes of expression was small compared with total variability, better standardization is again desirable.

5.3 Consequences for food table users

The consequences of this analytical variability for users of nutrient data banks depends on the particular application of nutrient data that is made. Individual dietary recalls are subject to large errors in the recollection of amount and identity of foods consumed and to large day-to-day variability within one subject or patient. As a result, errors in food analyses, even of the size reported here are less important for such purposes.

They do become influential in other applications, e.g. in deciding which individual foodstuffs are allowed for a patient on a certain prescribed diet, and in estimation of group mean intakes, where individual errors tend to cancel out. Such group means are typically used in epidemiological studies where diet and disease prevalence are compared between countries. For such studies, better standardization of food analysis procedures is required.

5.4 Possible remedies

The trial has brought to light two types of variability.

Firstly, within-laboratory variation was rather large for certain laboratories when analyzing certain nutrients and products. Such variability could be monitored and controled by doing more duplicate or triplicate analyses, but such multiple analyses are usually performed by one technician within one batch and do not give information on fluctuations between days or differences between technicians. A better method is to use a large batch of a homogeneous, stable foodstuff, such a as a nasogastric tube feed and analyze it in two- or fourfold alongside with each run of unknown samples. The results can be evaluated with standard quality control statistics. This will detect fluctuations within and between days in the concentration of a certain nutrient. However, it will not tell whether the concentration itself is correct (accuracy).

Such differences in level are responsible for most of the variability between laboratories observed in this trial. They can be detected by regular interlaboratory trials or by using external reference materials with a certified concentration of the nutrient of interest. This trial has shown that the production of such reference materials should have a high priority.

6. REFERENCES

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7. DATA FOR INDIVIDUAL LABORATORIES

7.1 Figures

Figure 1. Results of individual laboratories for DRY WEIGHT by the prescribed vacuum stove method.

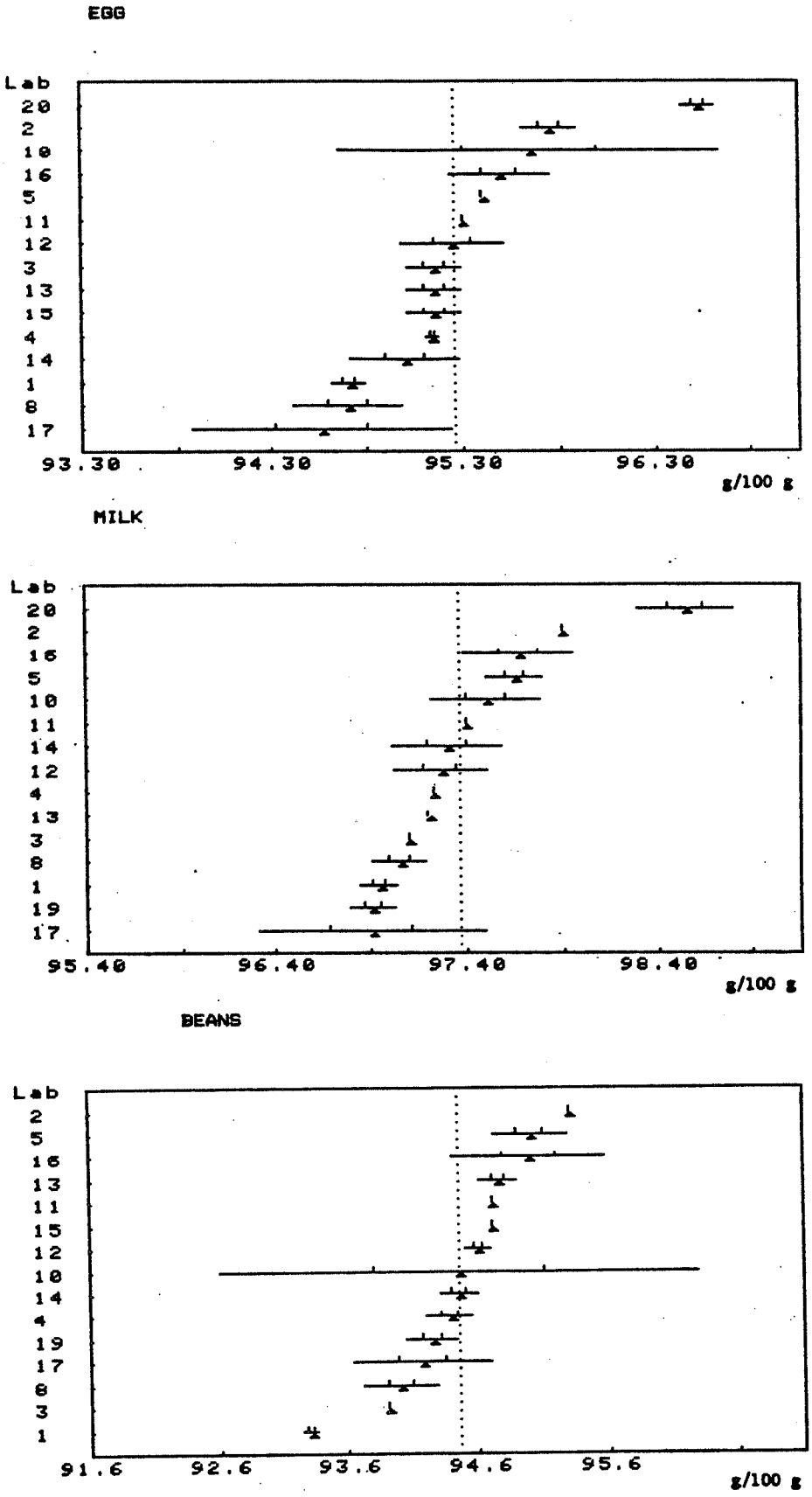


Figure 1 cont. Results of individual laboratories for DRY WEIGHT by the prescribed vacuum stove method.

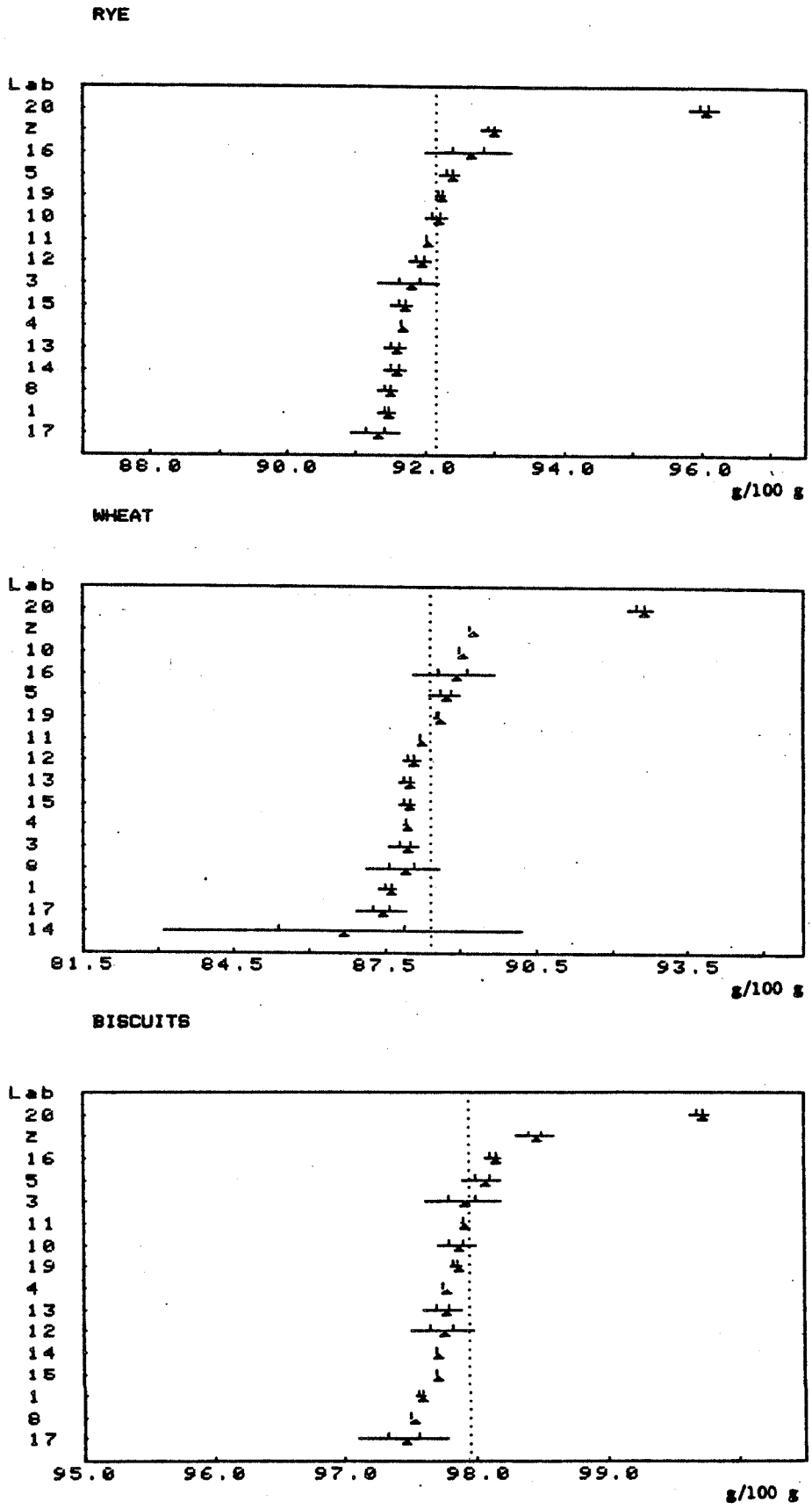


Figure 2. Results of individual laboratories for PROTEIN as reported.

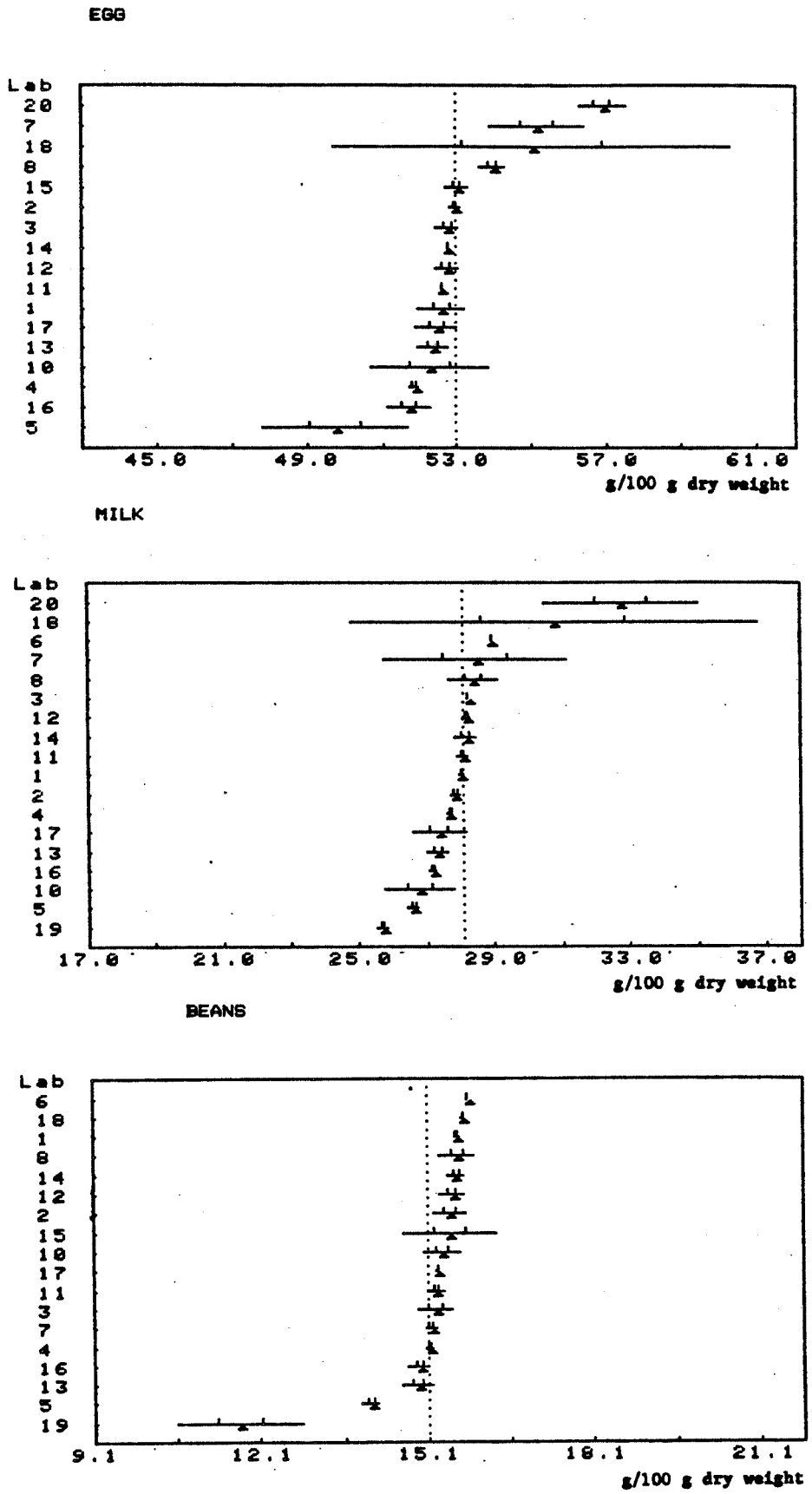


Figure 2 cont. Results of individual laboratories for PROTEIN as reported.

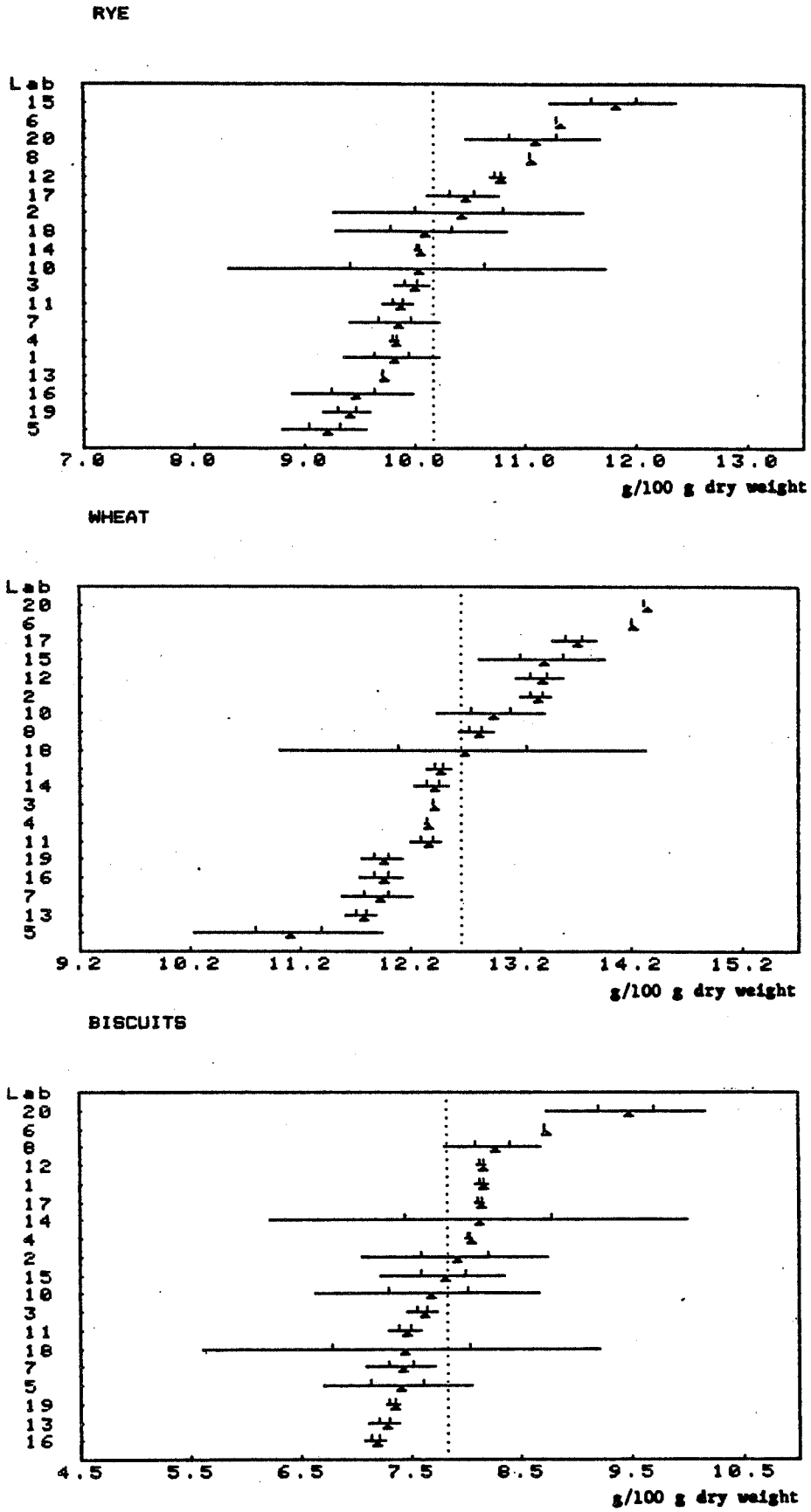


Figure 3. Results of individual laboratories for PROTEIN recalculated using uniform Kjeldahl factors.

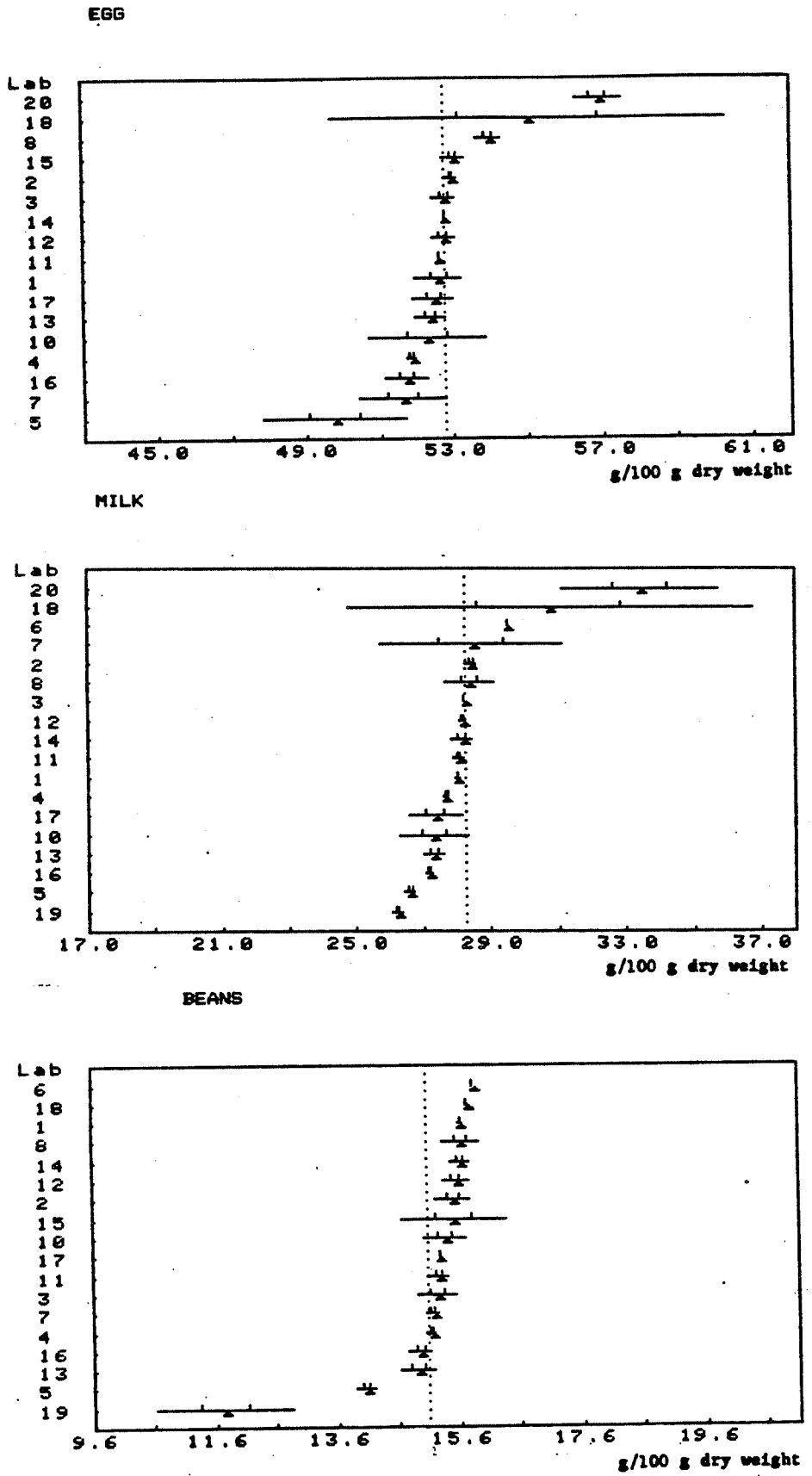


Figure 3 cont. Results of individual laboratories for PROTEIN recalculated using uniform Kjeldahl factors.

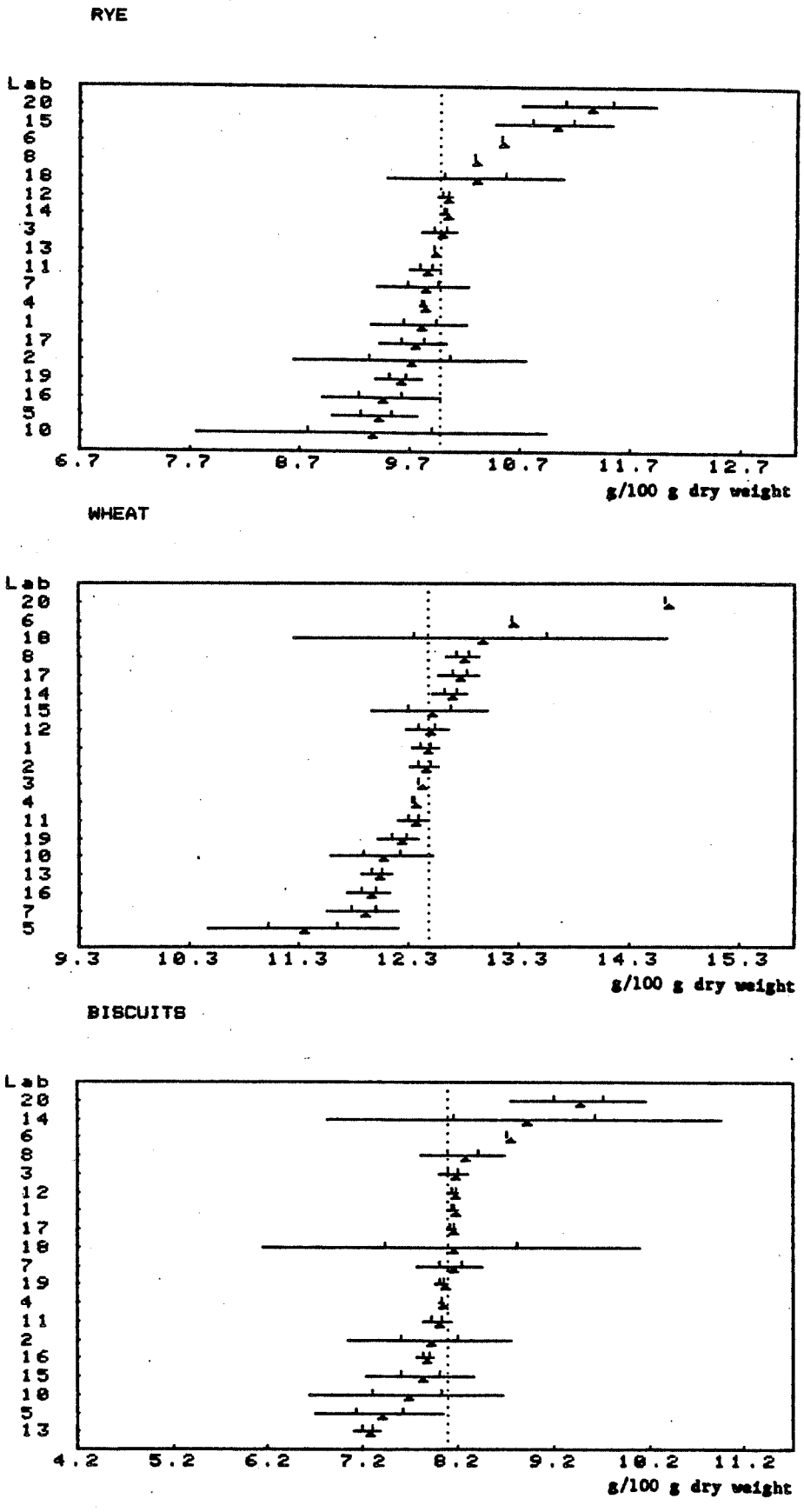


Figure 4. Results of individual laboratories for FAT.

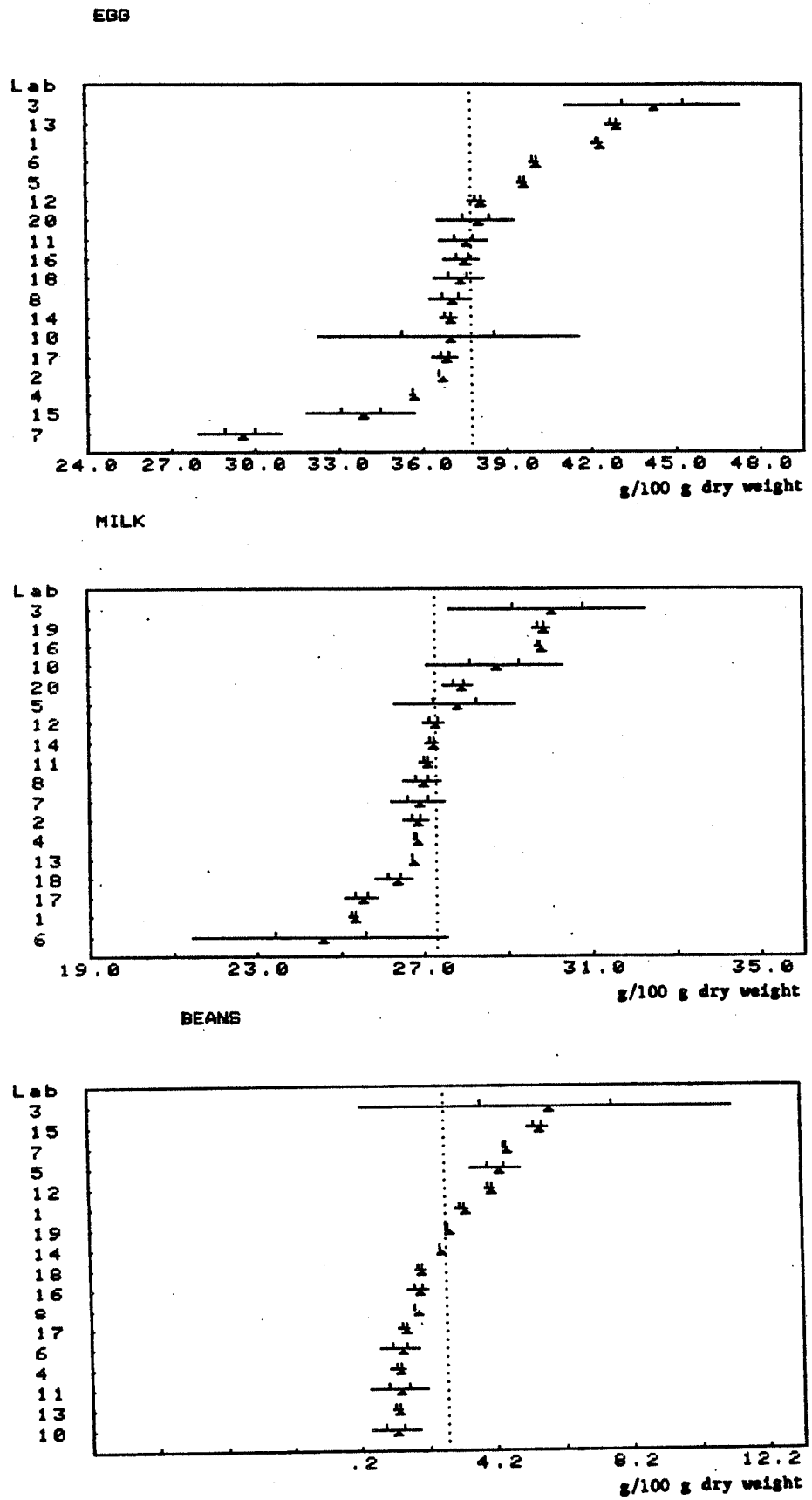


Figure 4 cont. Results of individual laboratories for FAT.

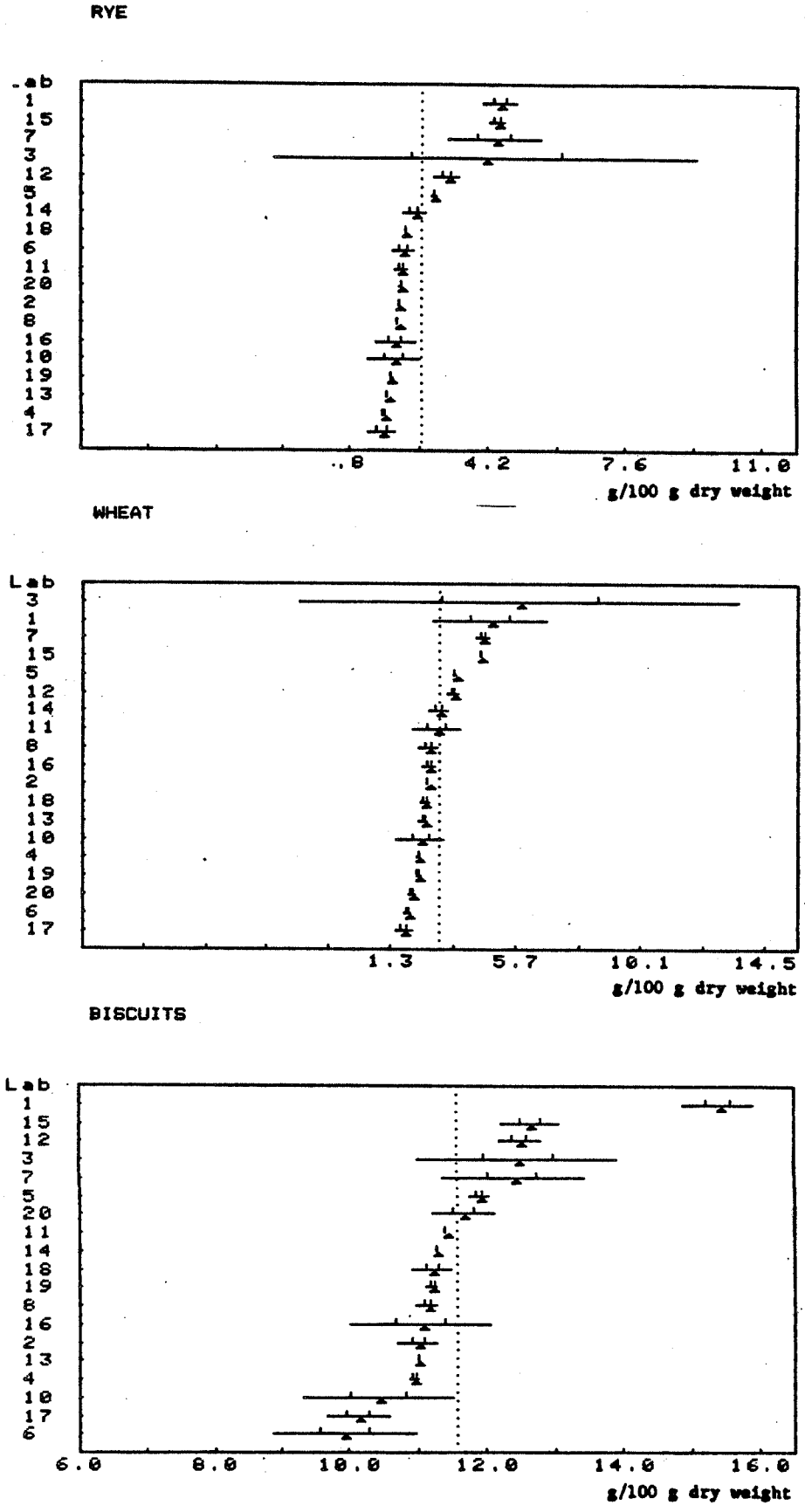


Figure 5. Results of individual laboratories for AVAILABLE CARBOHYDRATES as reported.

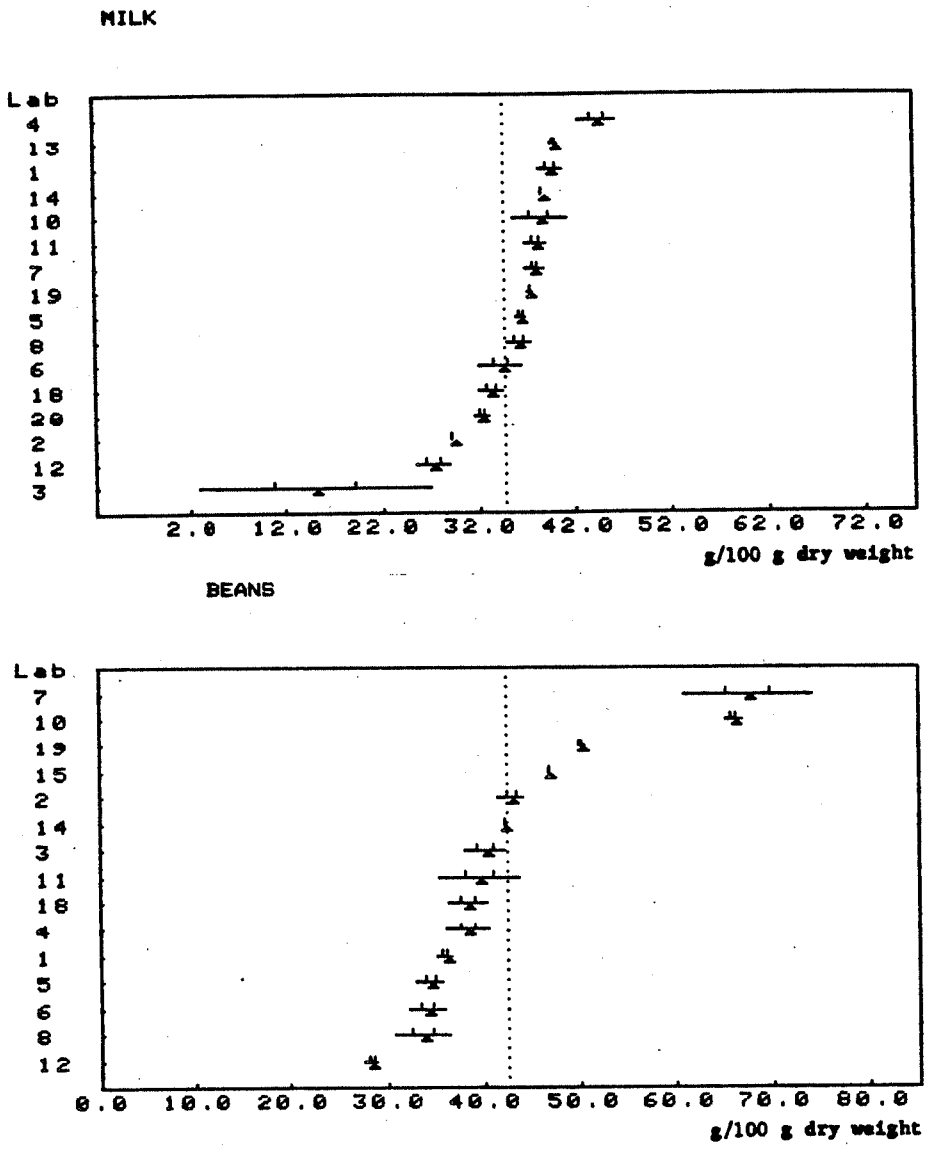


Figure 5 cont. Results of individual laboratories for AVAILABLE CARBOHYDRATES as reported.

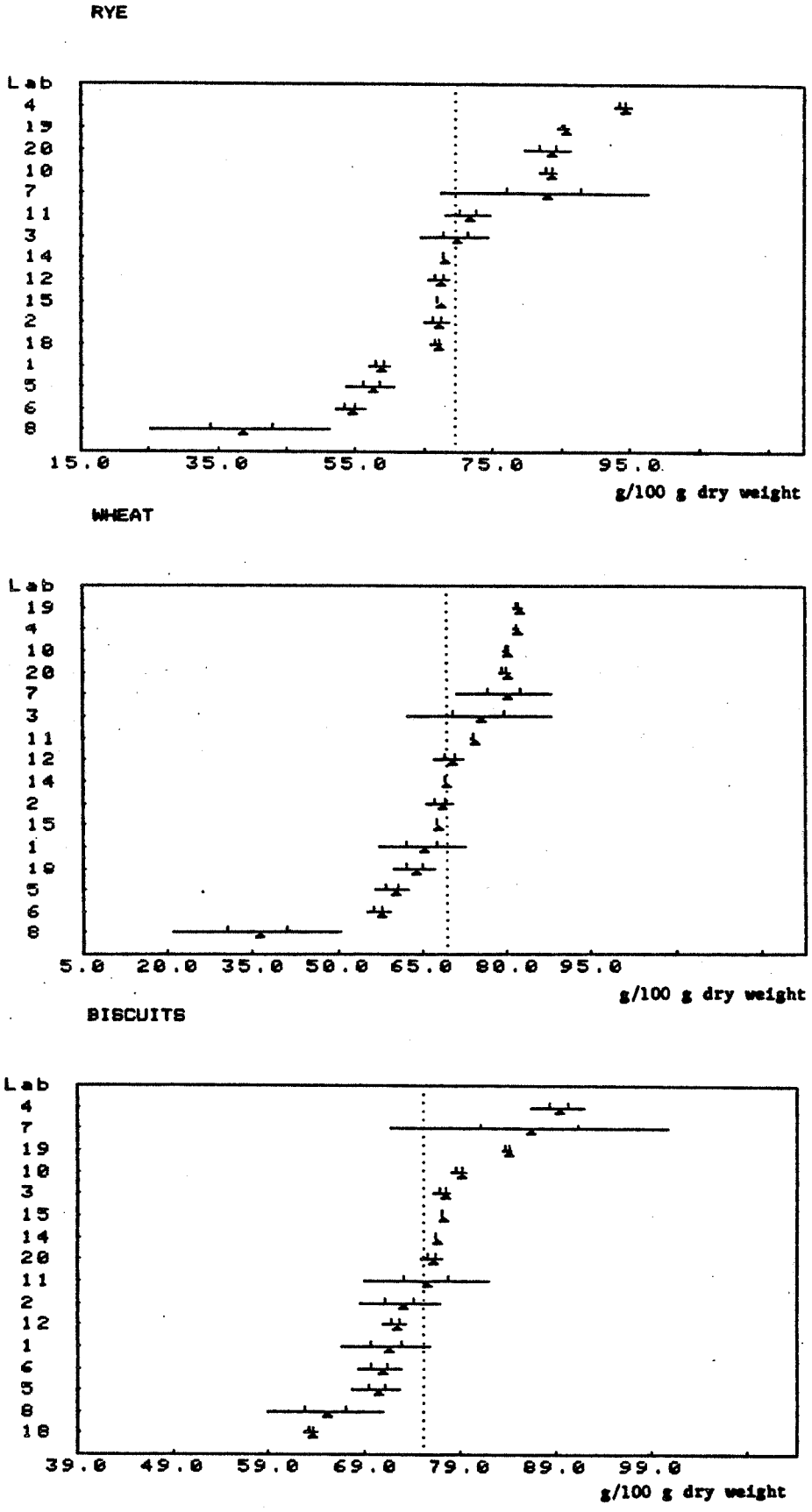


Figure 6. Results of individual laboratories for AVAILABLE CARBOHYDRATES recalculated to monosaccharides and "by difference" methods eliminated.

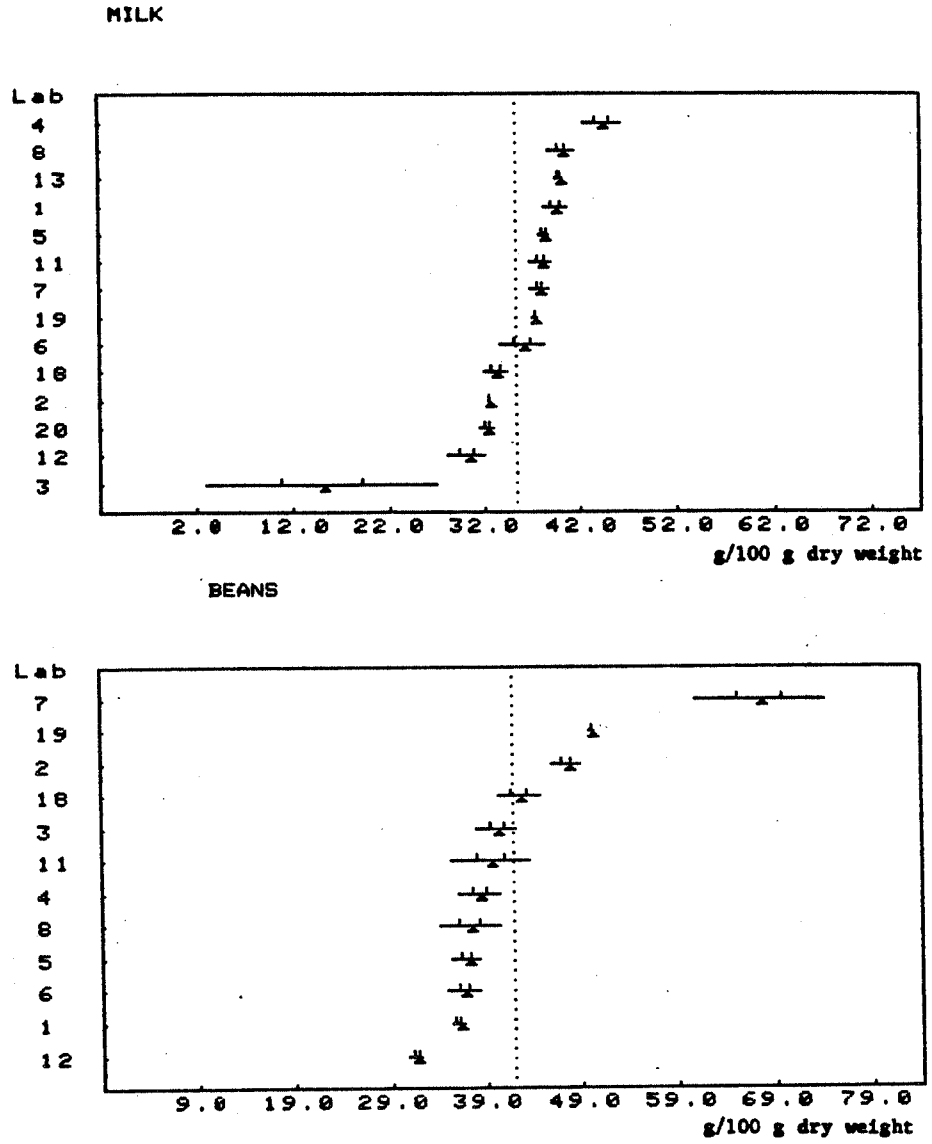


Figure 6 cont. Results of individual laboratories for AVAILABLE CARBOHYDRATES recalculated to monosaccharides and "by difference" methods eliminated.

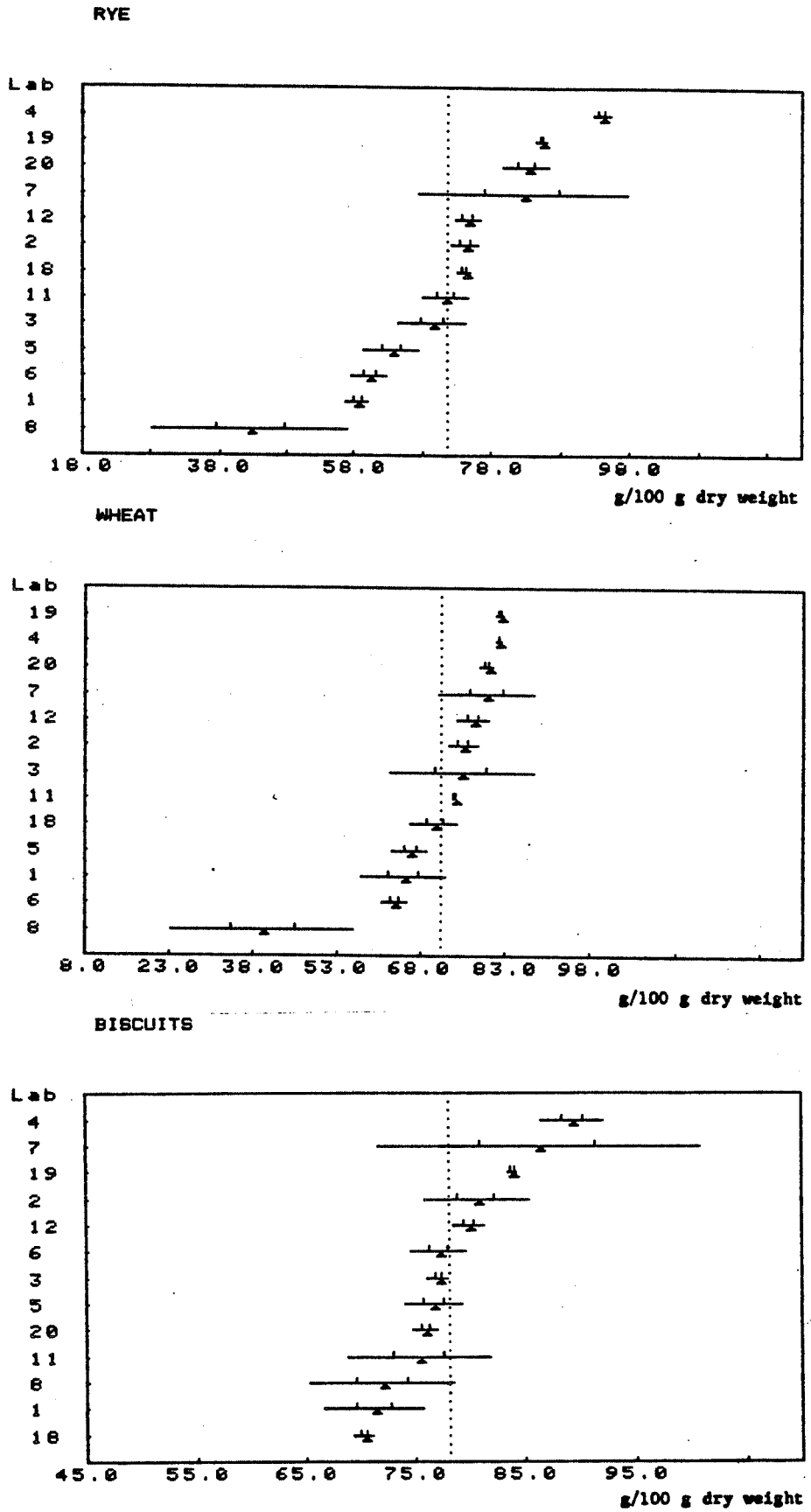


Figure 7. Results of individual laboratories for TOTAL DIETARY FIBER.

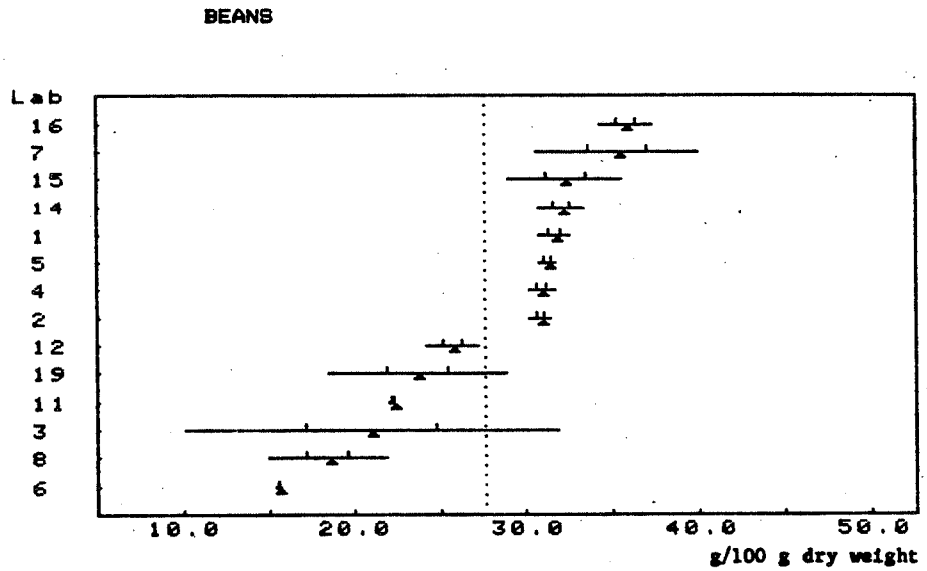


Figure 7 cont. Results of individual laboratories for TOTAL DIETARY FIBER.

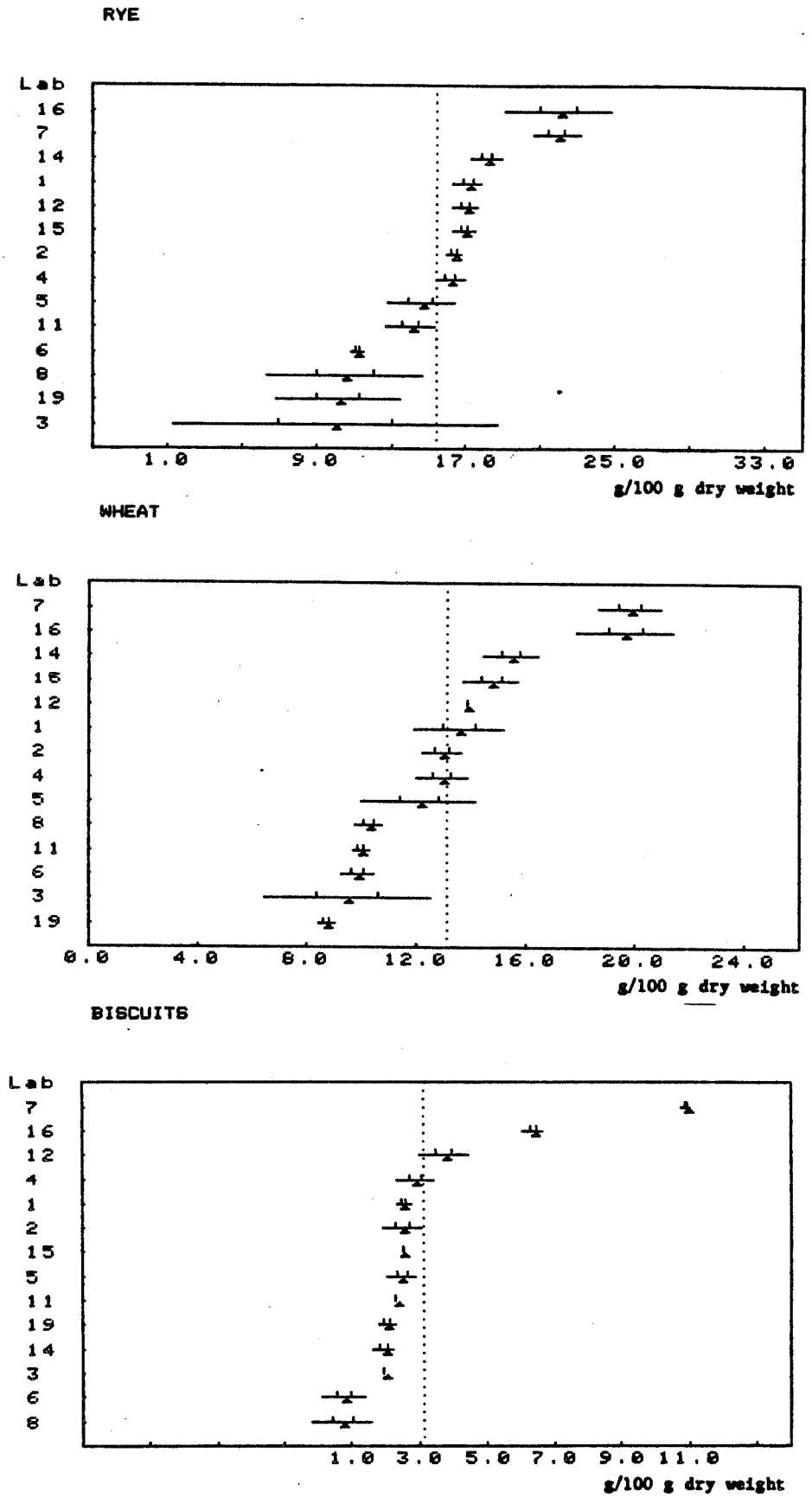


Figure 8. Results of individual laboratories for ASH.

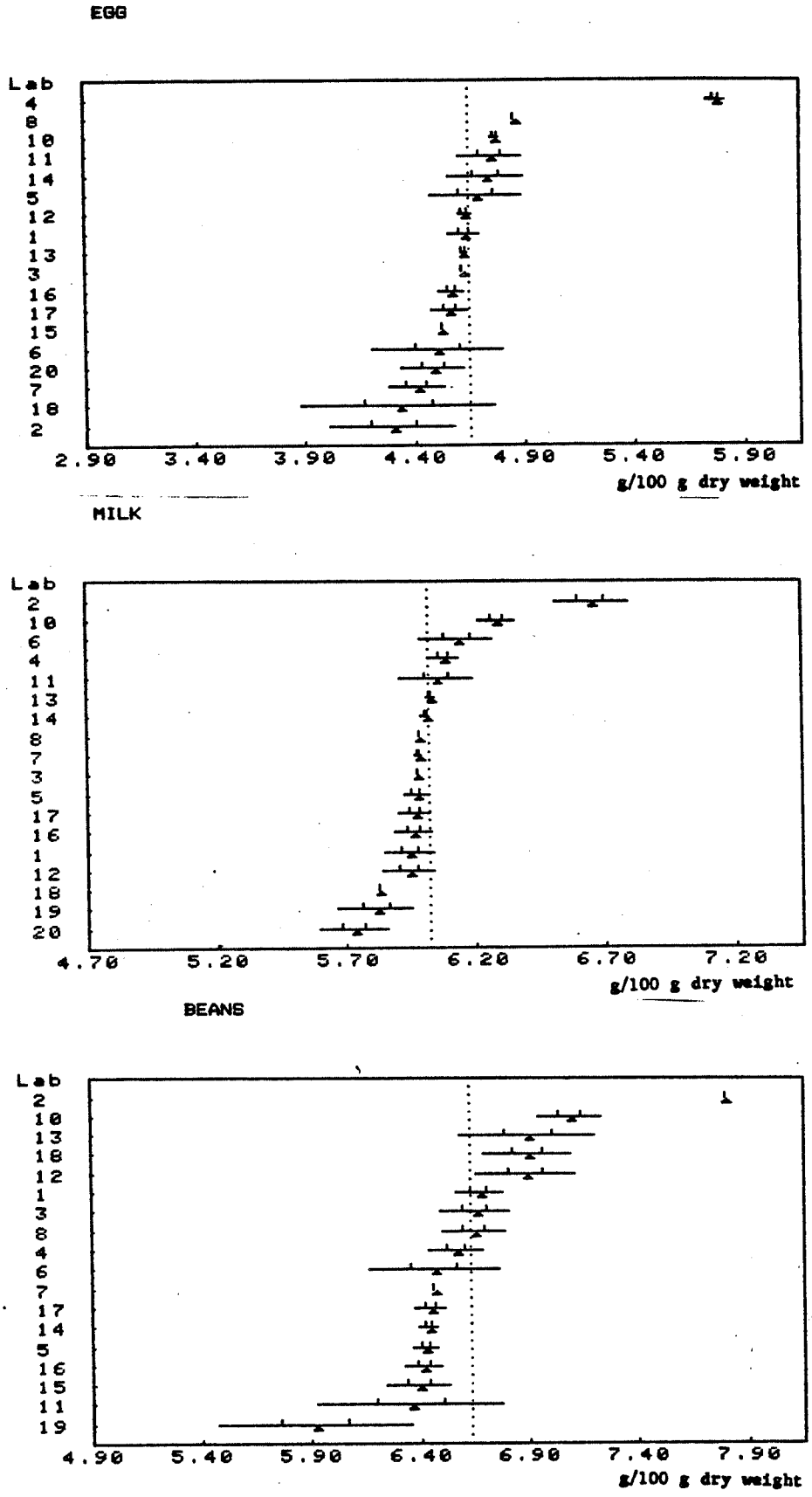
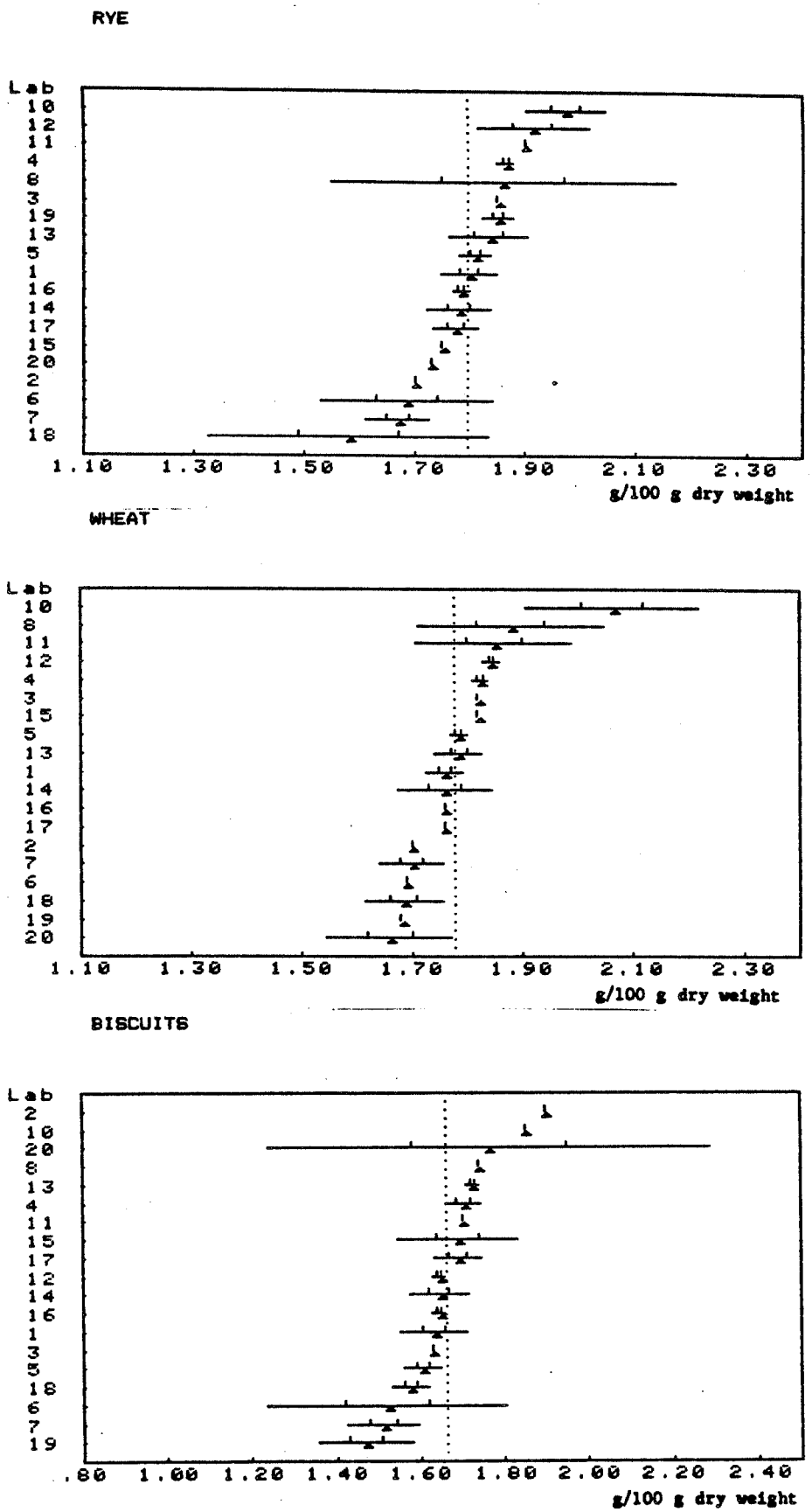


Figure 8 cont. Results of individual laboratories for ASH.



7.2 Tables with individual data

Table 3. Results of individual laboratories for DRY WEIGHT (g/100 g) by the prescribed vacuum stove method.

Stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	94.736	94.674	94.705	0.062
2	95.800	95.700	95.750	0.100
3	95.100	95.200	95.150	0.100
4	95.150	95.130	95.140	0.020
5	95.400	95.400	95.400	0.000
8	94.800	94.600	94.700	0.200
10	95.300	96.000	95.650	0.700*
11	95.300		95.300	
12	95.150	95.340	95.245	0.190
13	95.200	95.100	95.150	0.100
14	94.900	95.100	95.000	0.200
15	95.200	95.100	95.150	0.100
16	95.400	95.580	95.490	0.180
17	94.800	94.320	94.560	0.480
20	96.560	96.500	96.530	0.060

MEAN of the results of 15 labs : 95.260

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	96.977	96.910	96.944	0.067
2	97.900	97.900	97.900	0.000
3	97.100	97.100	97.100	0.000
4	97.230	97.230	97.230	0.000
5	97.600	97.700	97.650	0.100
8	97.000	97.100	97.050	0.100
10	97.400	97.600	97.500	0.200
11	97.400		97.400	
12	97.350	97.180	97.265	0.170
13	97.200	97.200	97.200	0.000
14	97.200	97.400	97.300	0.200
16	97.570	97.770	97.670	0.200
17	97.110	96.690	96.900	0.420
19	96.950	96.870	96.910	0.080
20	98.640	98.460	98.550	0.180

MEAN of the results of 15 labs : 97.370

Table 3 cont. Results of individual laboratories for DRY WEIGHT (g/100 g) by the prescribed vacuum stove method.

Stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	93.281	93.323	93.302	0.042
2	95.300	95.300	95.300	0.000
3	93.900	93.900	93.900	0.000
4	94.440	94.320	94.380	0.120
5	95.100	94.900	95.000	0.200
8	93.900	94.100	94.000	0.200
10	93.800	95.100	94.450	1.300**
11	94.700		94.700	
12	94.560	94.630	94.595	0.070
13	94.700	94.800	94.750	0.100
14	94.400	94.500	94.450	0.100
15	94.700	94.700	94.700	0.000
16	94.780	95.190	94.985	0.410
17	94.350	93.980	94.165	0.370
19	94.180	94.320	94.250	0.140

MEAN of the results of 15 labs : 94.454

Biscuits

Lab nr	Results		Mean	Difference
1	97.582	97.560	97.571	0.022
2	98.400	98.500	98.450	0.100
3	97.800	98.000	97.900	0.200
4	97.750	97.750	97.750	0.000
5	98.100	98.000	98.050	0.100
8	97.500		97.500	0.000
10	97.900	97.800	97.850	0.100
11	97.900		97.900	
12	97.660	97.830	97.745	0.170
13	97.700	97.800	97.750	0.100
14	97.700	97.700	97.700	0.000
15	97.700	97.700	97.700	0.000
16	98.110	98.150	98.130	0.040
17	97.560	97.320	97.440	0.240
19	97.860	97.830	97.845	0.030
20	99.740	99.690	99.715**	0.050

MEAN of the results of 16 labs : 97.953

Table 3 cont. Results of individual laboratories for DRY WEIGHT (g/100 g) by the prescribed vacuum stove method.

Stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	91.474	91.391	91.433	0.083
2	92.900	93.000	92.950	0.100
3	91.600	91.900	91.750	0.300
4	91.640	91.650	91.645	0.010
5	92.400	92.300	92.350	0.100
8	91.400	91.500	91.450	0.100
10	92.200	92.100	92.150	0.100
11	92.000		92.000	
12	91.860	91.970	91.915	0.110
13	91.600	91.500	91.550	0.100
14	91.600	91.500	91.550	0.100
15	91.700	91.600	91.650	0.100
16	92.400	92.840	92.620	0.440
17	91.400	91.150	91.275	0.250
19	92.190	92.240	92.215	0.050
20	96.110	95.970	96.040**	0.140

MEAN of the results of 16 labs : 92.164

Whole wheat meal

Lab nr	Results		Mean	Difference
1	87.622	87.508	87.565	0.114
2	89.200	89.200	89.200	0.000
3	87.800	88.000	87.900	0.200
4	87.910	87.940	87.925	0.030
5	88.800	88.600	88.700	0.200
8	87.600	88.100	87.850	0.500
10	89.000	89.000	89.000	0.000
11	88.200		88.200	
12	87.980	88.100	88.040	0.120
13	88.000	87.900	87.950	0.100
14	87.900	85.400	86.650	2.500**
15	88.000	87.900	87.950	0.100
16	88.590	89.150	88.870	0.560
17	87.610	87.270	87.440	0.340
19	88.530	88.560	88.545	0.030
20	92.650	92.470	92.560**	0.180

MEAN of the results of 16 labs : 88.403

Table 5. Results of individual laboratories for DRY WEIGHT (g/100 g) by optional methods.

Stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	95.187	95.768	95.478	0.581
3	94.800	94.800	94.800	0.000
6	95.460		95.460	
7	94.960	95.310	95.135	0.350
8	95.100	95.000	95.050	0.100
10	94.300	95.000	94.650	0.700
12	95.040	95.220	95.130	0.180
13	95.000	94.900	94.950	0.100
14	95.000	95.200	95.100	0.200
16	95.020	95.050	95.035	0.030
17	95.240	95.040	95.140	0.200
18	96.570	96.530	96.550**	0.040

MEAN of the results of 12 labs : 95.195

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	97.321	97.829	97.575	0.508
3	96.900	96.900	96.900	0.000
6	97.820	97.570	97.695	0.250
7	97.180	97.280	97.230	0.100
8	97.200	97.200	97.200	0.000
10	97.100	97.100	97.100	0.000
12	97.270	97.350	97.310	0.080
13	97.000	96.800	96.900	0.200
14	97.200	97.300	97.250	0.100
16	97.090	96.750	96.920	0.340
17	97.370	97.120	97.245	0.250
18	97.950	97.950	97.950	0.000

MEAN of the results of 12 labs : 97.273

Table 5 cont. Results of individual laboratories for DRY WEIGHT (g/100 g) by optional methods.

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	94.447	95.120	94.784	0.673
3	91.800	91.900	91.850	0.100
6	95.860	96.040	95.950	0.180
7	93.330	93.300	93.315	0.030
8	94.100	93.800	93.950	0.300
10	93.600	92.300	92.950	1.300
12	93.900	93.650	93.775	0.250
13	93.900	93.700	93.800	0.200
14	94.500	94.900	94.700	0.400
16	93.300	92.500	92.900	0.800
17	93.850	93.190	93.520	0.660
18	93.750	93.710	93.730	0.040

MEAN of the results of 12 labs : 93.769

Biscuits

Lab nr	Results		Mean	Difference
1	97.774	98.486	98.130	0.712
3	97.800	97.800	97.800	0.000
6	98.140	99.000	98.570	0.860
7	97.860	98.070	97.965	0.210
8	97.900	97.700	97.800	0.200
10	97.700	97.900	97.800	0.200
12	97.810	97.940	97.875	0.130
13	97.700	97.800	97.750	0.100
14	97.700	96.400	97.050	1.300
16	97.770	97.520	97.645	0.250
17	97.980	97.690	97.835	0.290
18	99.060	98.910	98.985	0.150

MEAN of the results of 12 labs : 97.934

Table 5 cont. Results of individual laboratories for DRY WEIGHT (g/100 g) by optional methods.

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	91.771	93.047	92.409	1.276
3	91.800	91.800	91.800	0.000
6	92.030	92.150	92.090	0.120
7	91.900	92.370	92.135	0.470
8	92.100	91.900	92.000	0.200
10	91.100	92.000	91.550	0.900
12	92.010	92.130	92.070	0.120
13	91.300	91.400	91.350	0.100
14	91.500	91.600	91.550	0.100
16	91.970	91.590	91.780	0.380
17	92.280	91.760	92.020	0.520
18	93.910	94.070	93.990**	0.160

MEAN of the results of 12 labs : 92.062

Whole wheat meal

Lab nr	Results		Mean	Difference
1	87.946	89.483	88.715	1.537**
3	88.000	88.000	88.000	0.000
6	88.600	88.900	88.750	0.300
7	88.390	88.650	88.520	0.260
8	88.400	88.100	88.250	0.300
10	88.000	88.200	88.100	0.200
12	88.180	88.350	88.265	0.170
13	87.700	87.700	87.700	0.000
14	87.800	88.000	87.900	0.200
16	88.250	87.910	88.080	0.340
17	88.260	88.040	88.150	0.220
18	90.410	90.580	90.495**	0.170

MEAN of the results of 12 labs : 88.410

Table 6. Dry weight optional methods

lab	Temperature °C	Drying time hrs	Vacuum	Remarks
1	70	> 18	< 10 kPa	
6	60	3.5-4	0,5 mm Hg	
3	103	18	no	
7	103	4.5	no	
8	103	4	no	
10	105	3	no	repeat until constant weight
12	102	3	no	repeat until constant weight
13	103	3	no	repeat until constant weight for egg, milk, beans, biscuits
13	130	1.5	no	for rye, wheat
14	?	?	?	
16	105	7, 2 and 1	no	
17	103	4	no	repeat until constant weigth
18	105	overnight	no	

Table 7. Results of individual laboratories for PROTEIN (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	52.807	52.363	52.585	0.444
2	53.000	52.900	52.950	0.100
3	52.650	52.860	52.755	0.210
4	51.880	51.810	51.845	0.070
5	50.420	49.060	49.740	1.360
7	55.580	54.710	55.145	0.870
8	54.070	53.850	53.960	0.220
10	52.820	51.710	52.265	1.110
11	52.600	52.600	52.600	0.000
12	52.620	52.830	52.725	0.210
13	52.200	52.500	52.350	0.300
14	52.740	52.740	52.740	0.000
15	53.100	52.900	53.000	0.200
16	51.900	51.510	51.705	0.390
17	52.260	52.640	52.450	0.380
18	56.870	53.150	55.010	3.720**
20	56.660	57.100	56.880	0.440

MEAN of the results of 17 labs : 52.983

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	27.999	28.047	28.023	0.048
2	27.900	27.800	27.850	0.100
3	28.220	28.220	28.220	0.000
4	27.700	27.640	27.670	0.060
5	26.520	26.630	26.575	0.110
6	28.910		28.910	0.000
7	29.410	27.500	28.455	1.910
8	28.650	28.130	28.390	0.520
10	26.390	27.110	26.750	0.720
11	28.100	28.000	28.050	0.100
12	28.150	28.200	28.175	0.050
13	27.200	27.400	27.300	0.200
14	28.260	28.050	28.155	0.210
16	27.170	27.110	27.140	0.060
17	27.080	27.630	27.355	0.550
18	32.870	28.640	30.755	4.230**
19	25.720	25.620	25.670	0.100
20	33.550	31.960	32.755**	1.590

MEAN of the results of 18 labs : 28.100

Table 7 cont. Results of individual laboratories for PROTEIN (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	15.649	15.627	15.638	0.022
2	15.600	15.400	15.500	0.200
3	15.120	15.340	15.230	0.220
4	15.140	15.100	15.120	0.040
5	14.000	14.110	14.055	0.110
6	15.840		15.840	
7	15.120	15.190	15.155	0.070
8	15.740	15.530	15.635	0.210
10	15.240	15.480	15.360	0.240
11	15.300	15.200	15.250	0.100
12	15.470	15.620	15.545	0.150
13	14.800	15.000	14.900	0.200
14	15.670	15.560	15.615	0.110
15	15.800	15.200	15.500	0.600
16	14.870	15.000	14.935	0.130
17	15.280	15.270	15.275	0.010
18	15.740	15.750	15.745	0.010
19	12.120	11.330	11.725**	0.790*

MEAN of the results of 18 labs : 15.092

Biscuits

Lab nr	Results		Mean	Difference
1	8.130	8.168	8.149	0.038
2	8.200	7.600	7.900	0.600
3	7.560	7.660	7.610	0.100
4	8.040	8.020	8.030	0.020
5	7.140	7.620	7.380	0.480
6	8.720		8.720	
7	7.520	7.300	7.410	0.220
8	8.410	8.100	8.255	0.310
10	8.020	7.300	7.660	0.720
11	7.500	7.400	7.450	0.100
12	8.140	8.170	8.155	0.030
13	7.300	7.200	7.250	0.100
14	8.780	7.450	8.115	1.330
15	8.000	7.600	7.800	0.400
16	7.200	7.140	7.170	0.060
17	8.150	8.120	8.135	0.030
18	8.050	6.780	7.415	1.270
19	7.350	7.310	7.330	0.040
20	9.710	9.210	9.460*	0.500

MEAN of the results of 19 labs : 7.840

Table 7 cont. Results of individual laboratories for PROTEIN (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	9.641	9.950	9.796	0.309
2	10.800	10.000	10.400	0.800
3	9.920	10.030	9.975	0.110
4	9.830	9.810	9.820	0.020
5	9.050	9.320	9.185	0.270
6	11.290		11.290	
7	9.970	9.680	9.825	0.290
8	11.040	11.040	11.040	0.000
10	10.630	9.420	10.025	1.210*
11	9.900	9.800	9.850	0.100
12	10.730	10.780	10.755	0.050
13	9.700	9.700	9.700	0.000
14	10.020	10.040	10.030	0.020
15	12.000	11.600	11.800	0.400
16	9.630	9.250	9.440	0.380
17	10.550	10.320	10.435	0.230
18	10.340	9.790	10.065	0.550
19	9.460	9.310	9.385	0.150
20	10.860	11.280	11.070	0.420

MEAN of the results of 19 labs : 10.175

Whole wheat meal

Lab nr	Results		Mean	Difference
1	12.507	12.425	12.466	0.082
2	13.300	13.400	13.350	0.100
3	12.400	12.400	12.400	0.000
4	12.360	12.350	12.355	0.010
5	10.790	11.390	11.090	0.600
6	14.200		14.200	
7	12.010	11.780	11.895	0.230
8	12.860	12.750	12.805	0.110
10	12.760	13.110	12.935	0.350
11	12.400	12.300	12.350	0.100
12	13.300	13.450	13.375	0.150
13	11.800	11.700	11.750	0.100
14	12.460	12.350	12.405	0.110
15	13.200	13.600	13.400	0.400
16	12.010	11.870	11.940	0.140
17	13.760	13.620	13.690	0.140
18	13.260	12.090	12.675	1.170**
19	11.880	12.010	11.945	0.130
20	14.320	14.320	14.320	0.000

MEAN of the results of 19 labs : 12.662

Table 10. Results of individual laboratories for PROTEIN (g/100 g dry weight) recalculated using uniform Kjeldahl factors.

stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	52.807	52.363	52.585	0.444
2	53.000	52.900	52.950	0.100
3	52.650	52.860	52.755	0.210
4	51.880	51.810	51.845	0.070
5	50.420	49.060	49.740	1.360
7	52.002	51.188	51.595	0.814
8	54.070	53.850	53.960	0.220
10	52.820	51.710	52.265	1.110
11	52.600	52.600	52.600	0.000
12	52.620	52.830	52.725	0.210
13	52.200	52.500	52.350	0.300
14	52.740	52.740	52.740	0.000
15	53.100	52.900	53.000	0.200
16	51.900	51.510	51.705	0.390
17	52.260	52.640	52.450	0.380
18	56.870	53.150	55.010	3.720**
20	56.660	57.100	56.880*	0.440

MEAN of the results of 17 labs : 52.774

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	27.999	28.047	28.023	0.048
2	28.480	28.378	28.429	0.102
3	28.220	28.220	28.220	0.000
4	27.700	27.640	27.670	0.060
5	26.520	26.630	26.575	0.110
6	29.511		29.511	0.000
7	29.410	27.500	28.455	1.910
8	28.650	28.130	28.390	0.520
10	26.939	27.674	27.307	0.735
11	28.100	28.000	28.050	0.100
12	28.150	28.200	28.175	0.050
13	27.200	27.400	27.300	0.200
14	28.260	28.050	28.155	0.210
16	27.170	27.110	27.140	0.060
17	27.080	27.630	27.355	0.550
18	32.870	28.640	30.755	4.230**
19	26.255	26.153	26.204	0.102
20	34.248	32.625	33.437**	1.623

MEAN of the results of 18 labs : 28.251

Table 10 cont. Results of individual laboratories for PROTEIN (g/100 g dry weight) recalculated using uniform Kjeldahl factors.

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	15.649	15.627	15.638	0.022
2	15.600	15.400	15.500	0.200
3	15.120	15.340	15.230	0.220
4	15.140	15.100	15.120	0.040
5	14.000	14.110	14.055	0.110
6	15.840		15.840	
7	15.120	15.190	15.155	0.070
8	15.740	15.530	15.635	0.210
10	15.240	15.480	15.360	0.240
11	15.300	15.200	15.250	0.100
12	15.470	15.620	15.545	0.150
13	14.800	15.000	14.900	0.200
14	15.670	15.560	15.615	0.110
15	15.800	15.200	15.500	0.600
16	14.870	15.000	14.935	0.130
17	15.280	15.270	15.275	0.010
18	15.740	15.750	15.745	0.010
19	12.120	11.330	11.725**	0.790*

MEAN of the results of 18 labs : 15.092

Biscuits

Lab nr	Results		Mean	Difference
1	8.130	8.168	8.149	0.038
2	8.200	7.600	7.900	0.600
3	8.105	8.212	8.159	0.107
4	8.040	8.020	8.030	0.020
5	7.140	7.620	7.380	0.480
6	8.720		8.720	
7	8.246	8.004	8.125	0.242
8	8.410	8.100	8.255	0.310
10	8.020	7.300	7.660	0.720
11	8.040	7.933	7.987	0.107
12	8.140	8.170	8.155	0.030
13	7.300	7.200	7.250	0.100
14	9.627	8.169	8.898	1.458
15	8.000	7.600	7.800	0.400
16	7.895	7.829	7.862	0.066
17	8.150	8.120	8.135	0.030
18	8.827	7.434	8.131	1.393
19	8.059	8.015	8.037	0.044
20	9.710	9.210	9.460	0.500

MEAN of the results of 19 labs : 8.094

Table 10 cont. Results of individual laboratories for PROTEIN (g/100 g dry weight) recalculated using uniform Kjeldahl factors.

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	9.641	9.950	9.796	0.309
2	10.074	9.328	9.701	0.746
3	9.920	10.030	9.975	0.110
4	9.830	9.810	9.820	0.020
5	9.256	9.533	9.395	0.277
6	10.531		10.531	0.000
7	9.970	9.680	9.825	0.290
8	10.298	10.298	10.298	
10	9.916	8.787	9.352	1.129*
11	9.900	9.800	9.850	0.100
12	10.009	10.056	10.033	0.047
13	9.921	9.921	9.921	0.000
14	10.020	10.040	10.030	0.020
15	11.194	10.821	11.008	0.373
16	9.630	9.250	9.440	0.380
17	9.841	9.627	9.734	0.214
18	10.576	10.013	10.295	0.563
19	9.676	9.522	9.599	0.154
20	11.108	11.537	11.323	0.429

MEAN of the results of 19 labs : 9.981

Whole wheat meal

Lab nr	Results		Mean	Difference
1	12.507	12.425	12.466	0.082
2	12.406	12.500	12.453	0.094
3	12.400	12.400	12.400	0.000
4	12.360	12.350	12.355	0.010
5	11.036	11.650	11.343	0.614
6	13.246		13.246	
7	12.010	11.780	11.895	0.230
8	12.860	12.750	12.805	0.110
10	11.903	12.229	12.066	0.326
11	12.400	12.300	12.350	0.100
12	12.406	12.546	12.476	0.140
13	12.069	11.967	12.018	0.102
14	12.744	12.632	12.688	0.112
15	12.313	12.686	12.500	0.373
16	12.010	11.870	11.940	0.140
17	12.835	12.705	12.770	0.130
18	13.562	12.366	12.964	1.196**
19	12.151	12.284	12.218	0.133
20	14.647	14.647	14.647**	0.000

MEAN of the results of 19 labs : 12.485

Table 11. Methods protein

Lab	Catalyst/Salt	Digestion	Destillation/Receiver solution	Measurement
2	$CuSO_4.5H_2O$	sample + H_2SO_4 conc.	destillation in to boric acid	titration
12	11 g $CuSO_4.5H_2O/K_2SO_4$ (1:10)	1 g sample H_2SO_4 conc. block digestor 400°C, ± 2 hrs (digest. until clear, then continue for 1 hr)	steam destillation into boric acid	automated titration
14	15 g $CuSO_4.5H_2O/K_2SO_4$ (1:15)	> 1 g sample + 20 ml H_2SO_4 conc./g dry matter block digestor, digest until clear, then continue for 4 hrs	steam destillation into boric acid	automated titration
15	$CuSO_4/K_2SO_4$ (1:3)	samples + H_2SO_4 conc./H_2O (1:1) + H_2O_2 (30%)	steam destillation into H_2SO_4	titration
16	$CuSO_4.5H_2O/K_2SO_4$ (1:25)	sample + 10 ml H_2SO_4 conc. electric oven 5 hrs (until clear)	steam destillation into 4% boric acid	titration
17	$CuSO_4.5H_2O/K_2SO_4$ (1:10)	0.5-20 g sample + 25 ml H_2SO_4 conc. digest until clear, then continue for 1 1/2 hrs	destillation into 4% boric acid	titration
20	10.5 g $CuSO_4.5H_2O/K_2SO_4$ (1:30)	0.5-1.5 g sample + 25 ml H_2SO_4 conc.	destillation into H_2SO_4	titration
10	1 g $Se/CuSO_4/K_2SO_4$ (1:10:100)	sample + 20 ml H_2SO_4 conc.	destillation into 5% boric acid	titration
13	2.5 g $Se/CuSO_4.5H_2O/K_2SO_4$ (1:10:40)	0.3-2.5 g sample + 25 ml H_2SO_4 conc. digest until clear, then continue for 30 min.	steam destillation into HCl	titration

Table 11 cont. Methods protein

Lab	Catalyst/Salt	Digestion	Destillation/Receiver solution	Measurement
18	$SeSO_2/CuSO_4$	2-5 g sample + H_2SO_4 conc.	steam destillation into 2% boric acid	titration
3	7.4 g $TiO_2/CuSO_4.5H_2O/K_2SO_4$ (1:1:33)	1 g sample + 20 ml H_2SO_4 conc., digest until clear, then continue for 45 min.	steam destillation into 2% boric acid	automated titration
11	7.4 g $TiO_2/CuSO_4.5H_2O/K_2SO_4$ (1:1:33)	sample + 20 ml H_2SO_4 conc. + 10 ml H_2O_2 block digestor 435°C, 40 min.	steam destillation into 4% boric acid	automated titration
1	7 g Se/K_2SO_4 (1:1000)	0.5-1.0 g sample + 12 ml H_2SO_4 conc., block digestor 400-420°C, 60 min.	steam destillation into 1% boric acid	automated titration
4	1.5 g Se/K_2SO_4 (1:200)	300 mg sample + 3 ml H_2SO_4/H_3PO_4 (95:5) + 2.5 ml H_2O_2	steam destillation into 1% boric acid	automated titration
5	Se/K_2SO_4	sample + H_2SO_4 conc. + H_2O_2 block digestor 420°C, 30 min.	colorimetric determination of ammonium, using hypochlorite and salicylate - automated determination (continuous flow)	
7	0.7 g HgO, 15 g K_2SO_4	1 g + 25 ml H_2SO_4 conc.	destillation into H_2SO_4	titration
8	HgO/K_2SO_4	H_2SO_4 conc./H_2O_2 automated equipment (Kjel Foss)		
6	HgO/K_2SO_4	H_2SO_4 conc./H_2O_2 automated equipment (Kjel Foss)		
19	Hg/Na_2SO_4	> 1.2 g sample (lyophylized) + 20 ml H_2SO_4 conc.	AOAC, 11th ed. 1970, 858	
		determination NPN: precipitate with TCA (10%), determine protein		

Table 13. Results of individual laboratories for FAT (g/100 g dry weight).

stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	42.266	42.156	42.211	0.110
2	36.600	36.600	36.600	0.000
3	45.300	43.090	44.195	2.210
4	35.610	35.660	35.635	0.050
5	39.620	39.520	39.570	0.100
6	40.040	39.930	39.985	0.110
7	29.950	28.910	29.430	1.040
8	36.750	37.280	37.015	0.530
10	35.300	38.550	36.925	3.250*
11	37.800	37.200	37.500	0.600
12	37.870	38.090	37.980	0.220
13	42.900	42.700	42.800	0.200
14	36.840	37.050	36.945	0.210
15	33.110	34.470	33.790	1.360
16	37.650	37.220	37.435	0.430
17	36.960	36.650	36.805	0.310
18	37.600	36.980	37.290	0.620
20	38.390	37.430	37.910	0.960

MEAN of the results of 18 labs : 37.779

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	25.268	25.350	25.309	0.082
2	26.900	26.700	26.800	0.200
3	30.790	29.150	29.970	1.640
4	26.780	26.800	26.790	0.020
5	28.260	27.240	27.750	1.020
6	25.580	23.420	24.500	2.160*
7	27.090	26.640	26.865	0.450
8	26.790	27.100	26.945	0.310
10	28.130	29.270	28.700	1.140
11	27.000	27.100	27.050	0.100
12	27.160	27.330	27.245	0.170
13	26.700	26.700	26.700	0.000
14	27.130	27.240	27.185	0.110
16	29.720	29.780	29.750	0.060
17	25.370	25.630	25.500	0.260
18	26.440	26.140	26.290	0.300
19	29.730	29.870	29.800	0.140
20	27.970	27.730	27.850	0.240

MEAN of the results of 18 labs : 27.278

Table 13 cont. Results of individual laboratories for FAT (g/100 g dry weight). stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	3.178	3.305	3.241	0.127
3	7.670	3.830	5.750	3.840 **
4	1.380	1.230	1.305	0.150
5	3.990	4.510	4.250	0.520
6	1.560	1.160	1.360	0.400
7	4.560	4.510	4.535	0.050
8	1.810		1.810	
10	0.920	1.430	1.175	0.510
11	1.600	1.000	1.300	0.600
12	4.010	4.110	4.060	0.100
13	1.300	1.200	1.250	0.100
14	2.560	2.570	2.565	0.010
15	5.600	5.390	5.495	0.210
16	1.810	2.010	1.910	0.200
17	1.430	1.530	1.480	0.100
18	2.030	1.920	1.975	0.110
19	2.780	2.760	2.770	0.020

MEAN of the results of 17 labs : 2.747

Biscuits

Lab nr	Results		Mean	Difference
1	15.229	15.586	15.408 **	0.357
2	11.100	10.900	11.000	0.200
3	12.970	11.950	12.460	1.020
4	10.920	10.960	10.940	0.040
5	11.830	11.930	11.880	0.100
6	10.300	9.560	9.930	0.740
7	12.750	12.020	12.385	0.730
8	11.080	11.180	11.130	0.100
10	10.030	10.810	10.420	0.780
11	11.400	11.400	11.400	0.000
12	12.390	12.600	12.495	0.210
13	11.000	11.000	11.000	0.000
14	11.260	11.260	11.260	0.000
15	12.790	12.490	12.640	0.300
16	11.400	10.680	11.040	0.720
17	10.290	9.970	10.130	0.320
18	11.310	11.110	11.210	0.200
19	11.170	11.230	11.200	0.060
20	11.510	11.820	11.665	0.310

MEAN of the results of 19 labs : 11.558

Table 13 cont. Results of individual laboratories for FAT (g/100 g dry weight).

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	4.625	4.351	4.488	0.274
2	2.000	2.000	2.000	0.000
3	5.990	2.290	4.140	3.700 **
4	1.630	1.600	1.615	0.030
5	2.870	2.850	2.860	0.020
6	2.010	2.190	2.100	0.180
7	4.760	3.950	4.355	0.810
8	1.970	1.970	1.970	0.000
10	1.640	2.090	1.865	0.450
11	2.100	2.000	2.050	0.100
12	3.070	3.290	3.180	0.220
13	1.700	1.700	1.700	0.000
14	2.270	2.470	2.370	0.200
15	4.470	4.360	4.415	0.110
16	1.730	2.070	1.900	0.340
17	1.440	1.680	1.560	0.240
18	2.130	2.130	2.130	0.000
19	1.810	1.790	1.800	0.020
20	2.040	2.030	2.035	0.010

MEAN of the results of 19 labs : 2.554

Whole wheat meal

Lab nr	Results		Mean	Difference
1	5.471	4.068	4.770	1.403
2	2.600	2.600	2.600	0.000
3	8.530	3.070	5.800	5.460 **
4	2.250	2.260	2.255	0.010
5	3.540	3.510	3.525	0.030
6	1.930	1.870	1.900	0.060
7	4.580	4.440	4.510	0.140
8	2.500	2.730	2.615	0.230
10	2.040	2.610	2.325	0.570
11	3.200	2.600	2.900	0.600
12	3.410	3.530	3.470	0.120
13	2.400	2.500	2.450	0.100
14	3.080	2.840	2.960	0.240
15	4.430	4.430	4.430	0.000
16	2.540	2.690	2.615	0.150
17	1.670	1.870	1.770	0.200
18	2.540	2.430	2.485	0.110
19	2.230	2.280	2.255	0.050
20	2.090	2.000	2.045	0.090

MEAN of the results of 19 labs : 3.036

Table 14. Methods total fat

Weibull-Stoldt methods

Lab	Hydrolysis	Drying residue	Extraction	Drying fat	Products
2	4 M HCl 1 hr 100°C	1 hr, 103 ± 2°C	petroleum ether, 4 hrs	1 hr, 103 ± 2°C	all
4	4 M HCl 20 min 100°C	2-4 hrs, 105°C	petroleum ether, 3 hrs	105°C until constant weight	all
8	4 M HCl	dry	petroleum ether		all
11	4 M HCl 1 hr 100°C	100°C	petroleum ether, 4 hrs	100°C constant weight	wheat, rye, biscuits, butter beans
13	4 M HCl 20 min 100°C	2-4 hrs, 103 ± 1°C	petroleum ether, 20 hrs	1 hr, 103 ± 1°C, const. weight	wheat, rye, biscuits, butter beans
16	4 M HCl 15 min 80-90°C 20 min 100°C	dry	diethyl ether, 3 hrs		wheat, rye, biscuits, butter beans
17	3 M HCl 1 hr 100°C	1 1/2 hrs 100 ± 3°C	petroleum ether, 6 hrs	1 1/2 hrs, 100 ± 3°C	all
18	5.5 M HCl	dry	diethyl ether		all
20	HCl	dry	petroleum ether		all

Table 14 cont. Methods total fat

Schmid-Bondzynski-Ratzlaf methods

Lab	Hydrolysis	Extraction	Drying fat	Products
1	sample + 2 ml ethanol + 10 ml 8.3 M HCl 30 min 70°C	ethanol + diethylether (DEE), petroleumether (PE) (1:1) report 2 x with DEE, PE (1:1)	dry	rye, wheat, biscuits
5	7.7 M HCl, 1 hr 80°C	DEE, PE (1:1)	dry	rye, wheat, biscuits, butter beans
	7.7 M HCl, 30 min 100°C	DEE, PE (1:1)	dry	egg
	7.7 M HCl, 1 hr 100°C	DEE, PE (1:1)	dry	milk
11	8.6 M HCl 30 min 100°C	DEE, PE (1:1) repeat 2 x with DEE, PE (1:1)	100°C until constant weight	egg
12	8.6 M HCl ..., 100°C	ethanol + DEE, PE (1:1) repeat 3 x with DEE, PE (1:1)	100°C until constant weight	egg, wheat rye, biscuits, butter beans
14	HCl	ethanol, DEE, PE	dry	all
16	7.7 M HCl 30 min 100°C	water + DEE, PE (1:1) repeat 3 x with DEE, PE (1:1)		egg
19	sample + 2.7 ml 0.8 M HCL + 13 ml ethanol room temp., invert 4x	water + DEE, PE (1:1) repeat 2 x with ethanol + DEE, PE (1:1)	60°C vacuum	milk, rye, wheat, biscuits, butter beans

Table 14 cont. Methods total fat

Röse-Gottlieb methods

Lab	Hydrolysis	Extraction	Drying fat	Products
1	10 ml sample diss. in water + 1.2 ml ammonia 25%, 15 min 60-70°C	ethanol + diethylether (DEE), petroleum (PE) (1:1) repeat 2 x with DEE, PE (1:1)	dry	milk
7	10 ml sample diss. in water + 2 ml ammonia 25%, 10 min 60-70°C	ethanol + DEE, PE (1:1) repeat 2 x with DEE, PE (1:1)	101 ± 1°C, 1 hr until constant weight. Fat dissolved in PE, discarded, residue dried at 101°C and weighed	egg, milk BS. 1741-1963
10	10 ml sample diss. in water (80°C) + 2 ml ammonia 25%	ethanol + DEE, PE (1:1)	105°C until constant weight	milk
11	10 ml sample diss. in water + 2 ml ammonia 25%	ethanol + DEE, PE (1:1) repeat 2 x with ethanol, DEE, PE (1:1)	102°C until constant weight fat dissolved in PE, discarded, residue dried 102°C and weighed)	milk
12	10 ml sample diss. in water (65°C) + 2 ml ammonia 25%	ethanol + DEE, PE (1:1) repeat 2 x with ethanol, DEE, PE (1:1)	100-105°C, 6 min continue until constant weight	milk
13	10 ml sample diss. in water + 2 ml ammonia 25%, 15 min waterbath (temp.?)	ethanol + DEE, PE (1:1) repeat 1 x with DEE, PE (1:1)	103 ± 1°C, 1 hr, until constant weight	milk
16	10 ml sample diss. in water + 2 ml ammonia 25%, 15 min, 60-70°C	ethanol + DEE, PE (1:1) repeat 2 x		milk

Table 14 cont. Methods total fat

Folch, Folch-like methods

Lab	Extraction	Purification	Products
1	sample + water (1:1) extract in polytron homogenizer with chloroform/methanol (2:1)	filter extract, add water, centrifuge, wash upperphase 3 x with chloroform/methanol/0,73% NaCl (3:48:47) evaporate solvent, dry residue, redissolve in chloroform filter, evaporate, dry	egg, butter beans
6	sample extract in homogenizer with chloroform/methanol (2:1)	filter extract, shake with 0.58% NaCl, allow to seperate, evaporate solvent, dry 1 hrs 100°C until constant weight	all
7	Soxhlet extraction with chloroform/methanol (2:1), 6 hrs	shake extract with 0.88% KCl, allow to separate, evaporate solvent, dry 1 hr 101 $\pm$ 1°C, repeat until constant weight	rye, wheat, biscuits, butter beans
15	sample + glass pearls + chloroform/methanol (1:2) shake mechanically for 20 min, centrifuge, extract residue for 10 min with chloroform/methanol (1:1), centrifuge, extract residue overnight with chloroform/methanol (1:1)	filter extract, evaporate solvent, dry 30 min 100°C	egg, rye, wheat, biscuits, butter beans
3	Soxhlet extraction with dichloormethane/methanol (9:1), 1 1/2 hrs	evaporate solvent, add Na_2SO_4, dry room temp., extract with petroleumether, evaporate solvent, dry room temp.	all

Table 14 cont. Methods total fat

Other methods

Lab	Extraction	Drying fat	Products
10	Soxhlet extration petroleumether/diethylether (1:1), 8 hrs	105°C, until constant weight	rye, wheat, biscuits, beans
	sample + $CaSO_4$, Soxhlet extraction petroleumether/ diethylether (1:1), 16 hrs	105°C, until constant weight	egg
13	sample + sand, dry at 103°C, 30 min, Soxhlet extraction, ethanol/ benzene (1:1), 45 min	103°C, 1 hr, repeat until constant weight	egg

Table 15. Results of individual laboratories for AVAILABLE CARBOHYDRATES (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	39.108	39.954	39.531	0.846
2	29.200	29.200	29.200	0.000
3	19.150	10.710	14.930**	8.440 **
4	43.690	45.100	44.395	1.410
5	36.559	36.252	36.406	0.307
6	35.000	33.500	34.250	1.500
7	37.500	38.190	37.845	0.690
8	35.755	36.579	36.167	0.824
10	39.354	37.415	38.385	1.939
11	38.400	37.600	38.000	0.800
12	27.790	26.536	27.163	1.254
13	40.000	39.800	39.900	0.200
14	38.597		38.597	
18	33.691	32.770	33.231	0.921
19	37.230	37.360	37.295	0.130
20	32.490	31.970	32.230	0.520

MEAN of the results of 16 labs : 34.724

Table 15 cont. Results of individual laboratories for AIVAILABLE CARBOHYDRATES (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	36.210	35.720	35.965	0.490
2	43.300	42.400	42.850	0.900
3	39.400	40.890	40.145	1.490
4	39.080	37.520	38.300	1.560
5	33.895	34.842	34.369	0.947
6	34.800	33.500	34.150	1.300
7	65.190	69.890	67.540	4.700
8	32.553	34.574	33.564	2.021
10	65.664	66.278	65.971	0.614
11	38.000	40.900	39.450	2.900
12	28.130	28.543	28.337	0.413
14	42.165		42.165	
15	46.700		46.700	
18	39.048	37.661	38.355	1.387
19	50.050	50.230	50.140	0.180

MEAN of the results of 15 labs : 42.398

Biscuits

Lab nr	Results		Mean	Difference
1	69.641	72.866	71.254	3.225
2	74.200	71.200	72.700	3.000
3	76.810	77.430	77.120	0.620
4	88.330	90.250	89.290	1.920
5	69.352	71.086	70.219	1.734
6	71.300	69.700	70.500	1.600
7	91.340	81.060	86.200	10.280**
8	62.769	66.974	64.872	4.205
10	79.152	78.661	78.907	0.491
11	73.100	77.700	75.400	4.600
12	71.646	72.536	72.091	0.890
14	76.372		76.372	
15	77.060		77.060	
18	63.646	63.141	63.394	0.505
19	84.090	83.790	83.940	0.300
20	75.600	76.360	75.980	0.760

MEAN of the results of 16 labs : 75.239

Table 15 cont. Results of individual laboratories for AIVAILABLE CARBOHYDRATES (g/100 g dry weight) as reported.

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	58.937	57.826	58.382	1.111
2	67.500	66.200	66.850	1.300
3	71.170	67.680	69.425	3.490
4	93.530	94.390	93.960	0.860
5	58.365	55.983	57.174	2.382
6	55.000	53.500	54.250	1.500
7	77.110	87.770	82.440	10.660
8	33.789	42.974	38.382	9.185
10	82.615	83.494	83.055	0.879
11	70.100	72.400	71.250	2.300
12	66.540	67.682	67.111	1.142
14	67.745		67.745	
15	66.880		66.880	
18	67.028	66.496	66.762	0.532
19	85.500	84.980	85.240	0.520
20	84.236	81.924	83.080	2.312

MEAN of the results of 16 labs : 69.645

Whole wheat meal

Lab nr	Results		Mean	Difference
1	62.271	67.630	64.951	5.359
2	68.900	67.300	68.100	1.600
3	79.640	70.650	75.145	8.990
4	81.670	81.870	81.770	0.200
5	60.654	58.625	59.640	2.029
6	57.800	56.400	57.100	1.400
7	76.680	82.560	79.620	5.880
8	30.507	40.979	35.743	10.472
10	80.135	79.798	79.967	0.337
11	74.100	74.000	74.050	0.100
12	68.993	70.768	69.881	1.775
14	68.852		68.852	
15	67.420		67.420	
18	62.213	64.865	63.539	2.652
19	82.270	81.840	82.055	0.430
20	80.090	79.350	79.720	0.740

MEAN of the results of 16 labs : 69.294

Table 16. Results of individual laboratories for AVAILABLE CARBOHYDRATES (g/100 g dry weight) recalculated to monosaccharides and "by difference" methods eliminated.

stragglers are indicated by*, outliers by**

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	39.108	39.954	39.531	0.846
2	32.410	32.410	32.410	0.000
3	19.150	10.710	14.930**	8.440**
4	43.690	45.100	44.395	1.410
5	38.480	38.160	38.320	0.320
6	36.840	35.260	36.050	1.580
7	37.500	38.190	37.845	0.690
8	39.680	40.600	40.140	0.920
11	38.400	37.600	38.000	0.800
12	30.850	29.460	30.155	1.390
13	40.000	39.800	39.900	0.200
18	33.690	32.770	33.230	0.920
19	37.230	37.360	37.295	0.130
20	32.490	31.970	32.230	0.520

MEAN of the results of 14 labs : 35.317

Table 16 cont. Results of individual laboratories for AVAILABLE CARBOHYDRATES (g/100 g dry weight) recalculated to monosaccharides and "by difference" methods eliminated.

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	36.210	35.720	35.965	0.490
2	48.060	47.060	47.560	1.000
3	39.400	40.890	40.145	1.490
4	39.080	37.520	38.300	1.560
5	36.400	37.420	36.910	1.020
6	37.170	36.010	36.590	1.160
7	65.190	69.890	67.540*	4.700
8	36.130	38.380	37.255	2.250
11	38.000	40.900	39.450	2.900
12	31.220	31.680	31.450	0.460
18	43.340	41.800	42.570	1.540
19	50.050	50.230	50.140	0.180

MEAN of the results of 12 labs : 41.990

Biscuits

Lab nr	Results		Mean	Difference
1	69.641	72.866	71.254	3.225
2	82.360	79.030	80.695	3.330
3	76.810	77.430	77.120	0.620
4	88.330	90.250	89.290	1.920
5	75.760	77.610	76.685	1.850
6	78.090	76.350	77.220	1.740
7	91.340	81.060	86.200	10.280*
8	69.670	74.340	72.005	4.670
11	73.100	77.700	75.400	4.600
12	79.530	80.510	80.020	0.980
18	70.650	70.090	70.370	0.560
19	84.090	83.790	83.940	0.300
20	75.600	76.360	75.980	0.760

MEAN of the results of 13 labs : 78.168

Table 16 cont. Results of individual laboratories for AVAILABLE CARBOHYDRATES (g/100 g dry weight) recalculated to monosaccharides and "by difference" methods eliminated.

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	58.937	57.826	58.382	1.111
2	74.930	73.480	74.205	1.450
3	71.170	67.680	69.425	3.490
4	93.530	94.390	93.960	0.860
5	64.780	61.940	63.360	2.840
6	61.010	59.260	60.135	1.750
7	77.110	87.770	82.440	10.660
8	37.510	47.700	42.605	10.190
11	70.100	72.400	71.250	2.300
12	73.860	75.130	74.495	1.270
18	74.400	73.810	74.105	0.590
19	85.500	84.980	85.240	0.520
20	84.240	81.920	83.080	2.320

MEAN of the results of 13 labs : 71.745

Whole wheat meal

Lab nr	Results		Mean	Difference
1	62.271	67.630	64.951	5.359
2	76.480	74.700	75.590	1.780
3	79.640	70.650	75.145	8.990
4	81.670	81.870	81.770	0.200
5	67.230	64.980	66.105	2.250
6	64.160	62.540	63.350	1.620
7	76.680	82.560	79.620	5.880
8	33.860	45.490	39.675*	11.630
11	74.100	74.000	74.050	0.100
12	76.420	78.380	77.400	1.960
18	69.060	72.000	70.530	2.940
19	82.270	81.840	82.055	0.430
20	80.090	79.350	79.720	0.740

MEAN of the results of 13 labs : 71.535

Table 17. Available Carbohydrates methods

Lab	Extraction Sugars	Extraction Starch	Solubilization Starch	Hydrolysis Starch	Determination Sugars
1	H_2O	ins. 80% methanol/ diethylether	DMSO	amyloglucosidase	enzym.
11	H_2O	no	autoclave	amyloglucosidase	enzym.
19	85% methanol	ins. 85% methanol	10 min, 100°C	amyloglucosidase	color. anthrone
3	80% ethanol	ins. 80% ethanol	1 hr, 100°C	α-amylase/pullulanase	color. anthrone
6	80% methanol	ins. 80% methanol	autoclave	amyloglucosidase	GLC (TMS)
5	80% ethanol lactose:H_2O	no	DMSO	amyloglucosidase	GLC (TMS) starch enzym. enzym. (latose)
2, 12	no	no	autoclave	pancreas (α-amylase)	Luff/Schoorl, reductiometric Osborne, Voogt, The Analysis of Nutrients in Foods (1978); Academic Press London-New York
8	no	no	no	pancreas	Luff/Schoorl
18	?	?	2.5% HCl, 3 hrs temp. ?		Luff/Schoorl
4	no	no	perchlor. acid,	20 min, 25°C	color. orcine
20	no	no	perchlor. acid,	16 hrs 25°C	color. anthrone
7	no lactose:H_2O 40°C	no	1 hr 100°C		color. phenol/H_2SO_4 chlooramine-T(lactose)
13	lactose:H_2O				grav. Munson/Walker

Table 17 cont. Available Carbohydrates methods

Calculations by difference

lab 10 available carbohydrates = 100 - (moisture + protein + fat + crude fiber + ash)

lab 14 available carbohydrates = dry weight - (protein + fat + ash + dietary fiber)

lab 15 available carbohydrates = dry weight - (protein + fat + dietary fiber)

Table 19. Results of individual laboratories for TOTAL DIETARY FIBER (g/100 g dry weight).

stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
3	0.840		0.840	
4	0.110	0.100	0.105	0.010
5	0.840	0.640	0.740	0.200**
8	0.000	0.000	0.000	0.000

MEAN of the results of 4 labs : 0.361

Full-fat milk powder

Lab nr	Results		Mean	Difference
3	0.100		0.100	
4	0.790	0.740	0.765	0.050
5	0.670	0.650	0.660	0.020
6	0.000		0.000	
8	0.110	0.000	0.055	0.110
14	0.000		0.000	
19	0.000	0.000	0.000	0.000

MEAN of the results of 7 labs : 0.278

Table 19 cont. Results of individual laboratories for TOTAL DIETARY FIBER (g/100 g dry weight).

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	31.938	31.346	31.642	0.592
2	30.600	31.000	30.800	0.400
3	24.810	17.150	20.980	7.660*
4	30.640	31.160	30.900	0.520
5	31.370	31.050	31.210	0.320
6	15.630	15.530	15.580	0.100
7	33.630	36.940	35.285	3.310
8	19.680	17.230	18.455	2.450
11	22.200	22.300	22.250	0.100
12	25.160	26.220	25.690	1.060
14	32.500	31.550	32.025	0.950
15	33.400	31.100	32.250	2.300
16	35.210	36.280	35.745	1.070
19	25.530	21.920	23.725	3.610

MEAN of the results of 14 labs : 27.610

Biscuits

Lab nr	Results		Mean	Difference
1	2.459	2.609	2.534	0.150
2	2.700	2.300	2.500	0.400
3	1.940	1.940	1.940	0.000
4	3.070	2.700	2.885	0.370
5	2.630	2.320	2.475	0.310
6	0.980	0.550	0.765	0.430
7	10.970	10.880	10.925**	0.090
8	1.030	0.410	0.720	0.620
11	2.300	2.300	2.300	0.000
12	3.990	3.480	3.735	0.510
14	2.050	1.840	1.945	0.210
15	2.500	2.500	2.500	0.000
16	6.270	6.480	6.375	0.210
19	1.940	2.110	2.025	0.170

MEAN of the results of 14 labs : 3.116

Table 19 cont. Results of individual laboratories for TOTAL DIETARY FIBER (g/100 g dry weight).

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	16.805	17.339	17.072	0.534
2	16.500	16.200	16.350	0.300
3	6.980	13.080	10.030	6.100**
4	16.440	15.910	16.175	0.530
5	13.970	15.270	14.620	1.300
6	11.290	11.080	11.185	0.210
7	22.290	21.440	21.865	0.850
8	12.030	9.080	10.555	2.950
11	14.500	13.600	14.050	0.900
12	16.750	17.190	16.970	0.440
14	18.350	17.800	18.075	0.550
15	16.700	17.100	16.900	0.400
16	22.930	20.960	21.945	1.970
19	9.010	11.350	10.180	2.340

MEAN of the results of 14 labs : 15.427

Whole wheat meal

Lab nr	Results		Mean	Difference
1	12.983	14.134	13.559	1.151
2	13.200	12.700	12.950	0.500
3	8.420	10.580	9.500	2.160
4	13.250	12.590	12.920	0.660
5	11.390	12.850	12.120	1.460
6	9.650	10.080	9.865	0.430
7	20.240	19.440	19.840	0.800
8	10.470	10.130	10.300	0.340
11	9.900	10.100	10.000	0.200
12	13.860	13.860	13.860	0.000
14	15.810	15.120	15.465	0.690
15	14.400	15.100	14.750	0.700
16	20.280	19.030	19.655	1.250
19	8.630	8.850	8.740	0.220

MEAN of the results of 14 labs : 13.109

Table 20. Total dietary fiber methods

Lab	Enzymatic digestion	Determination	Reference
1,2, 12,14 15	gelatinize 15 min, 100°C with Thermamyl, digest 60 min, 60°C with protease, and 30 min, 60°C with amyloglucosidase	precipitate with 96% ethanol 60°C, filter, wash with 78%, 95% ethanol and aceton, dry overnight, weigh correct for ash and protein	L. Prosky et al., J.Assoc. Off. Anal. Chem., (1984), 67, (6), 1044
4,5	gelatinize 30 min, 100°C with Thermamyl, digest 60 min, 40°C with pepsine and 60 min 40°C with pancreatine	see method Prosky	N.-G. Asp et al., J.Agric. Food Chem., (1983), 31, 476
16	gelatinize 15 min, 100°C with rohalase, digest 60 min, 40°C with pepsine and 60 min 40°C with pancreatine	precipitate with ethanol, filter dry overnight, weigh, correct for ash only	
7	extract sample 3 x with boiling 80% ethanol, suspend residue in water, gelatinize 1 hr, 120°C, digest 20 hrs, 37°C with pepsine and 18 hrs, 37°C with pancreatine and amyloglucosidase	centrifuge, precipitate with absolute ethanol, centrifuge, transfer precipitates to crucible, wash with 80% ethanol, aceton, diethylether, dry overnight weigh, correct for ash	T.F. Schweizer et al., J. Sci., F. Agric., (1979), 30, 613
19	suspend sample in water, gelatinize 1 hr, 100°C, digest 16 hrs, 42°C with α-amylase, pullulanase	centrifuge, wash precipitate 2 x with 80% ethanol, wash residue with aceton, dry hyrolyse residue with 12 M H_2SO_4 1 hr, 35°C, 2 hrs 100°C, 1 M H_2SO_4 determine, uronic acids (colorimetric) and sugars (GLC) determine non-cellulosic polysaccharides as described, except for hydrolysis with H_2SO_4 (2 hrs, 100°C, 1 M H_2SO_4)	H. Englyst et al., Analyst (1982), 107, 307

Table 20 cont. Total dietary fiber methods

Lab	Enzymatic digestion	Determination	Reference
3	see method Englyst lab 19, except:	lignin is determined gravimetrically in residue after hydrolysing with H_2SO_4	
11	gelatinize sample by heating with acetate buffer digest overnight with α-amylase, pullulanase	precipitate with 80% ethanol, dry, digest with 12 M H_2SO_4, determine sugars with GLC, uronic acids colorimetrically	H. Englyst et al., Analyst, (1984), 109, 937
8	suspend sample in water, digest 18 hrs, 40°C with pepsine, and 1 hr, 40°C with pancreatine	centrifuge, filter, wash residue with water (3x), aceton (3x), dry overnight	E. Hellendoorn, J. Sci. F. Agric. (1975), 26, 1461
6	boil 1 hr in neutral-detergent solution, filter, digest overnight, 37°C with α-amylase	wash, dry 3 hrs 110°C	AACC Approved Methods: Method 32-20 (1978)

Table 22. Results of individual laboratories for ASH (g/100 dry weight).

stragglers are indicated by*, outliers by**

Egg powder

Lab nr	Results		Mean	Difference
1	4.606	4.653	4.630	0.047
2	4.200	4.400	4.300	0.200
3	4.620	4.620	4.620	0.000
4	5.760	5.790	5.775 **	0.030
5	4.610	4.760	4.685	0.150
6	4.610	4.400	4.505	0.210
7	4.360	4.450	4.405	0.090
8	4.860	4.860	4.860	0.000
10	4.767	4.778	4.773	0.011
11	4.800	4.700	4.750	0.100
12	4.620	4.640	4.630	0.020
13	4.620	4.630	4.625	0.010
14	4.790	4.670	4.730	0.120
15	4.520	4.520	4.520	0.000
16	4.550	4.590	4.570	0.040
17	4.530	4.590	4.560	0.060
18	4.170	4.480	4.325	0.310
20	4.530	4.430	4.480	0.100

MEAN of the results of 18 labs : 4.652

Full-fat milk powder

Lab nr	Results		Mean	Difference
1	5.973	5.906	5.940	0.067
2	6.700	6.600	6.650 **	0.100
3	5.970	5.970	5.970	0.000
4	6.060	6.100	6.080	0.040
5	5.950	5.980	5.965	0.030
6	6.180	6.080	6.130	0.100
7	5.970	5.980	5.975	0.010
8	5.980	5.980	5.980	0.000
10	6.260	6.310	6.285	0.050
11	6.100	6.000	6.050	0.100
12	5.970	5.900	5.935	0.070
13	6.030	6.020	6.025	0.010
14	6.000	6.010	6.005	0.010
16	5.930	5.980	5.955	0.050
17	5.940	5.980	5.960	0.040
18	5.820	5.820	5.820	0.000
19	5.860	5.760	5.810	0.100
20	5.770	5.680	5.725	0.090

MEAN of the results of 18 labs : 6.014

Table 22 cont. Results of individual laboratories for ASH (g/100 dry weight).

stragglers are indicated by*, outliers by**

French beans

Lab nr	Results		Mean	Difference
1	6.636	6.712	6.674	0.076
2	7.800	7.800	7.800 **	0.000
3	6.710	6.600	6.655	0.110
4	6.610	6.520	6.565	0.090
5	6.400	6.440	6.420	0.040
6	6.570	6.360	6.465	0.210
7	6.460	6.460	6.460	0.000
8	6.700	6.600	6.650	0.100
10	7.040	7.140	7.090	0.100
11	6.500	6.200	6.350	0.300
12	6.810	6.970	6.890	0.160
13	7.010	6.790	6.900	0.220
14	6.420	6.450	6.435	0.030
15	6.340	6.440	6.390	0.100
16	6.440	6.380	6.410	0.060
17	6.420	6.470	6.445	0.050
18	6.830	6.970	6.900	0.140
19	5.760	6.070	5.915	0.310

MEAN of the results of 18 labs : 6.634

Biscuits

Lab nr	Results		Mean	Difference
1	1.604	1.660	1.632	0.056
2	1.900	1.900	1.900	0.000
3	1.630	1.630	1.630	0.000
4	1.690	1.720	1.705	0.030
5	1.620	1.590	1.605	0.030
6	1.620	1.420	1.520	0.200
7	1.480	1.540	1.510	0.060
8	1.740	1.740	1.740	0.000
10	1.850	1.850	1.850	0.000
11	1.700	1.700	1.700	0.000
12	1.640	1.650	1.645	0.010
13	1.720	1.730	1.725	0.010
14	1.620	1.670	1.645	0.050
15	1.640	1.740	1.690	0.100
16	1.650	1.640	1.645	0.010
17	1.670	1.710	1.690	0.040
18	1.590	1.560	1.575	0.030
19	1.510	1.430	1.470	0.080
20	1.950	1.580	1.765	0.370 **

MEAN of the results of 19 labs : 1.665

Table 22 cont. Results of individual laboratories for ASH (g/100 dry weight).

stragglers are indicated by*, outliers by**

Whole rye meal

Lab nr	Results		Mean	Difference
1	1.781	1.817	1.799	0.036
2	1.700	1.700	1.700	0.000
3	1.850	1.850	1.850	0.000
4	1.870	1.860	1.865	0.010
5	1.800	1.820	1.810	0.020
6	1.740	1.630	1.685	0.110
7	1.690	1.650	1.670	0.040
8	1.970	1.750	1.860	0.220*
10	2.000	1.950	1.975	0.050
11	1.900	1.900	1.900	0.000
12	1.880	1.950	1.915	0.070
13	1.860	1.810	1.835	0.050
14	1.760	1.800	1.780	0.040
15	1.750	1.750	1.750	0.000
16	1.790	1.780	1.785	0.010
17	1.760	1.790	1.775	0.030
18	1.670	1.490	1.580	0.180
19	1.860	1.840	1.850	0.020
20	1.730	1.730	1.730	0.000

MEAN of the results of 19 labs : 1.795

Whole wheat meal

Lab nr	Results		Mean	Difference
1	1.748	1.772	1.760	0.024
2	1.700	1.700	1.700	0.000
3	1.820	1.820	1.820	0.000
4	1.830	1.820	1.825	0.010
5	1.780	1.790	1.785	0.010
6	1.690	1.690	1.690	0.000
7	1.720	1.680	1.700	0.040
8	1.940	1.820	1.880	0.120
10	2.010	2.120	2.065*	0.110
11	1.900	1.800	1.850	0.100
12	1.840	1.850	1.845	0.010
13	1.800	1.770	1.785	0.030
14	1.730	1.790	1.760	0.060
15	1.820	1.820	1.820	0.000
16	1.760	1.760	1.760	0.000
17	1.760	1.760	1.760	0.000
18	1.660	1.710	1.685	0.050
19	1.680	1.680	1.680	0.000
20	1.700	1.620	1.660	0.080

MEAN of the results of 19 labs : 1.781

Table 23. Ashing methods

Lab	Pre-ashing	Temp. muffle furnace °C	Time in muffle furnace	Remarks
1	hot plate	525	> 5 hrs	continue until grey ash
3	hot plate	525	48 hrs	
7	muffle furnace 300°C	540	overnight	sample is dried before 102°C, 4 1/2 hrs
8	flame	550		
13	sand bath 400°c	525 ± 25		continue until light grey ash
16	hot plate	550	7 + 2 + 1 hrs	
4	muffle furnace, slowly rise of temp.	550	overnight	
5	15 hrs at 80°C 3 hrs at 350°C	550	15 hrs	after ashing, ash is wetted with water and procedure is repeated
6	rise of temp. 25°-500°C in 3 hrs	500	overnight	
11	rise of temp. 25°-550°C in 4 hrs	550	16 hrs	
10	flame	600	3-4 hrs	after pre-ashing sample is washed with hot water, continue until white ash

Table 23 cont. Ashing methods

Lab	Pre-ashing	Temp. muffle furnace °C	Time in muffle furnace	Remarks
15	dried sample + glycerol/ethanol (1:1) pre-ash on flame	500 - 600	overnight	
17	sample + methanol/ water (1:1) pre-ash on flame	550 ± 5		continue until light grey ash
12	no	550	1 + 1/2 hrs	egg, rye, wheat, biscuits,
	no	525	1 + 1/2 hrs	beans
	no	500	1 1/2 hrs	milk
14	no	525	16-20 hrs	repeat 4 hrs until constant weight
18	no	480 ± 20	overnight	
19	no	530	2 hrs	
20	?	500	?	
2	destruction by boiling with H_2SO_4, dry, raise temp., pre-ash	800		8/9 calculation factor to convert sulphate ash into ash content

8. APPENDICES

Appendix 1

Prescribed vacuum stove method

EUROFOODS Interlaboratory trial 1985

Determination of the dry weight content (EUROFOODS trial method)

Principle

The loss in weight of a sample dried under vacuum at 70°C is determined.

Reagents and apparatus

1. Vacuum oven:
   With thermostat (± 2°C), pressure < 13 kPa (10 cm Hg) and containing at least 300 g pre-dried CaO (2).
2. Calcium oxide (drying agent):
   Heat at least 16 hours at 800°C and store in an air-tight container.
3. Metal dish:
   Diameter ca. 55 mm, height ca. 20 mm with an inverted slip-in cover fitting tightly on the inside.
4. Air-tight desiccator:
   With a suitable drying agent, e.g. silica gel (blue!).

Procedure

Dry a metal dish and cover (3) at 103°C for > 30 min., cool in a desiccator (4) and weigh dish and cover (3) to the nearest 0.1 mg. Weigh ca. 3 g well mixed sample to the nearest 0.1 mg into the dish. Place dish and loosened cover into the oven (1) at 70°C, adjust the pressure to < 13 kPa. Heat for 16 ± 1/2 hours after the oven has reached the temperature of 70°C. Admit dry air into the oven (1) and bring to atmospheric pressure. Tighten immediately the cover on the dish and transfer to the desiccator (4). Weigh to the nearest 0.1 mg soon after reaching room temperature.

Calculation

Determine the loss in weight of the sample and divide by the original weight of the sample. Multiply by 100% to obtain the % moisture. Substract from 100 to obtain the % age dry weight.

Appendix 2

Instructions to participants

Date: February 22, 1985.

EUROFOODS Interlaboratory trial on laboratory procedures as a source of discrepancies between nutrient values in different food tables

INSTRUCTIONS AND REPORT FORMS

- In order to determine the variation within the laboratory it is necessary that two different technicians perform the analyses on different days (with the same method). They must perform independent analyses with, as far as possible, different standard solutions, reagents, calibrations and so on. We suggest a period of one week between the two analyses. Each technician should only contribute one value (no average of duplicates).
- Store the samples in a refrigerator. Before analysis, allow to equilibrate at room temperature, because cold samples will attract water from the air.
- All samples have been ground by RIKILT to pass a sieve of 0,5 mm openings.
- Please do not determine the dry weight content of the samples using your routine method, but do use the methode described below. This will make it possible to compare the results for the different laboratories on a dry weight basis.
- Report your results on the attached report forms.
- Also indicate to which nutrient data bank you contribute values on a regular basis.

Example of Report form

EUROFOODS Interlaboratory trial 1985

(Please fill in with typewriter; keep copies of all forms)

Results - Protein

Laboratory: .....

Sample	Analyst 1	Date	Analyst 2	Date	Kjeldahl factor
Egg powder					
Full-fat milk powder					
Rye meal					
Whole wheat meal					
Biscuits					
French beans					

Report the values in the way you are used to do for your nutrient databank.
Indicate below exactly which units you have used:

Preferred unit

☐ gram protein/100 gram dry weight, Eurofoods dry weight method

Alternatives

☐ gram protein/100 gram dry weight, other dry weight method (which?)

☐ gram protein/100 gram product as received

☐ other: .....