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The Connective Tissue Content of Female Bovine Forequarter Cuts

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Data on the composition, particularly the connective tissue content, of trimmed bovine brisket, chuck and blade, clod and neck muscle are presented.

Public Analysts and Authorised Officers serving the Northern English and Welsh Counties of Cheshire, Clwyd, Greater Manchester and Lancashire have co-operated in a survey of the composition of four female bovine forequarter cuts —brisket, chuck and blade, clod and neck muscle.

The same group took part in earlier surveys of bovine cuts[1,2] including the equivalent male cuts of brisket, chuck and blade, clod and neck muscle[2].

For the purpose of this survey a cow was defined as

"A female bovine animal which has borne at least one calf."

The Meat and Livestock Commission define "cull cow" as an animal no longer used for milking and ready for slaughter (age usually in the range 5–10 years)[3]. In practice the animals covered by the two definitions are similar.

This survey was particularly aimed at establishing data on the connective tissue content of the lean part of the cuts although other useful data are provided. Connective tissue is defined as in a previous paper[4] i.e.

$$\text{Wet fat-free connective tissue} = \text{hydroxyproline} \times 37.$$

Sampling and Sub-sampling

All samples of the four cuts were taken from cows as defined. Sub-sampling methods were designed so that "Lean Meat" was prepared for analysis. The aim was to produce, by trimming, lean meat close to the consumer understanding, but perhaps biased a little in favour of the manufacturer, in leaving the epimysium intact.

Approximately 5-lb samples of the four cuts were submitted to laboratories. The samples comprised lean meat with attached fatty tissue. Fatty tissue was trimmed to produce lean meat. To ensure consistency in sub-sampling the four laboratories had previously held a joint demonstration of trimming procedures.

Sub-samples were passed through mincers using progressively smaller cutting grilles (at least twice) followed by homogenisation in a chopper/blender in

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TABLE I

COLLABORATIVE SURVEY OF NITROGEN CONTENTS OF CIRCULATED MEAT-BASED MATERIALS

	Lab 1	Lab 2	Lab 3
Mean nitrogen content found (per cent.)			
First material	1·80	1·84	1·82
Second material	4·96	5·06	4·87
Intra-laboratory standard deviation*			
First material	0·02	0·01	0·03
Second material	0·03	0·01	0·04
Difference from overall mean (as percentage of overall mean)			
First material	−1·3	1·2	0·1
Second material	0	1·9	−1·9

* Based on at least two analyses by each of two analysts.

TABLE II

COLLABORATIVE SURVEY
HYDROXYPROLINE CONTENTS OF CIRCULATED MEAT-BASED MATERIALS

	Lab 1	Lab 2	Lab 3
Mean hydroxyproline content found (per cent.)			
First material	0·13	0·13	0·12
Second material	0·66	0·66	0·61
Intra-Laboratory Standard Deviation*			
First material	0·01	0·01	0·01
Second material	0·01	0·02	0·03
Difference from overall mean (as percentage of overall mean)			
First material	5·7	8·1	−6·5
Second material	3·1	4·1	−3·8

* Based on at least two analyses by each of two analysts.

preparation for analysis. Ninety-one samples of the bovine cuts from home produced cows were analysed.

Methods of Analysis and Quality Assurance

Each laboratory used its normal methods for determination of water, ash, fat (acid digestion procedures), nitrogen and hydroxyproline. There were slight procedural differences between laboratories except for hydroxyproline for which all laboratories used the BS 4405, Part II method[5]. As might be expected in a cow beef study, homogenisation of samples prior to analysis presented greater difficulties than in bullock beef studies because of the relatively greater toughness of the meat cuts. In a total of eight samples, homogenisation proved unusually difficult and subsequent analytical data failed to meet expected precision levels. The results for these samples were rejected.

During the survey, each laboratory was required to adopt a minimum within-laboratory quality control rate of 20 per cent. in which one in five

TABLE III

THE COMPOSITION OF FOUR FEMALE BOVINE CUTS

Cut	No. of samples		Water *per cent.*	Fat *per cent.*	Nitrogen *per cent.*	Hydroxyproline *per cent.*	Wet fat-free connective tissue* *per cent.*	Nitrogen on fat-free meat *per cent.*
Brisket	26	Range	69·0–76·9	2·3–22	2·86–3·6	0·1–0·4	3·7–14·8	3·24–3·92
		Mean	71·1	7·3	3·3	0·2	7·4	3·59
		Standard deviation	3·8	4·8	0·19	0·07	2·6	0·14
Chuck and blade	18	Range	63·9–74·9	1·8–16·9	2·91–3·62	0·15–0·4	5·6–14·8	3·33–3·80
		Mean	70·9	8·0	3·2	0·2	7·4	3·58
		Standard deviation	3·7	4·7	0·2	0·08	3·0	0·14
Clod	22	Range	69·2–76·2	1·8–10·7	3·1–3·61	0·16–0·51	5·9–18·9	3·41–3·76
		Mean	73·1	5·2	3·40	0·3	11·1	3·59
		Standard deviation	2·1	2·6	0·1	0·10	3·8	0·13
Neck	25	Range	62·8–75·1	1·8–17·2	3·04–3·83	0·16–0·47	6·0–17·4	3·29–3·98
		Mean	72·5	5·5	3·39	0·32	11·9	3·63
		Standard deviation	2·8	3·5	0·32	0·08	3·1	0·17

* Wet fat-free connective tissue = Hydroxyproline × 37.

TABLE IV

CONNECTIVE TISSUE CONTENTS OF MALE AND FEMALE BOVINE CUTS

	Male			Female		
Cut	No. of samples	Mean connective tissue content *per cent.*	Standard deviation	No. of samples	Mean connective tissue content *per cent.*	Standard deviation
Brisket	32	9·1	3·1	26	8·7	2·6
Chuck and blade	30	8·9	2·8	18	8·7	3·0
Clod	27	9·8	3·4	22	11·7	3·8
Neck	26	12·6	2·7	25	11·9	3·1

determinations were to be replicated, twice the minimum rate recommended by the Association of Public Analysts[6]. Laboratories were advised to continue their normal recovery quality control procedures for nitrogen determination (dl-alanine and nicotinic acid are used by some laboratories for internal quality control).

The acceptability of internal quality control data was assessed by individual laboratories by their usual procedures.

To complement internal quality assurance procedures the co-ordinating laboratory circulated at different times during the course of the survey, four homogeneous meat-based materials for the determination of hydroxyproline and nitrogen respectively. Laboratories were required to have the materials analysed in duplicate by each analyst concerned in the survey. In practice, each Laboratory had no more than two analysts concerned in the work.

The results of these inter-laboratory exercises are shown in Tables I and II.

The nitrogen data shown in Table I are similar to those found in earlier surveys[1,2] and in the opinion of the authors indicate acceptable between-laboratory variance.

The hydroxyproline data shown in Table II are also similar to those found in the earlier surveys and, furthermore, absolute reproducibilities obtained fell within the expected range at each hydroxyproline level[7].

Table III summarises the analytical data for each of the bovine cuts. Wet fat-free connective tissue content has been calculated as indicated earlier and connective tissue levels for each group of samples are shown.

Discussion

The trimming of the cuts of brisket; clod; chuck and blade; and neck was designed to avoid removal of epimysium and hence could be argued to produce meat which was a little higher in fat and connective tissue content than if trimmed by the consumer. It is suspected that the cautious trimming procedure may have left excess fat on certain of the trimmed sub-sample. Nevertheless data from those samples have been included in the evaluation of the composition of lean meat. Table III clearly indicates that the "lean" portions of the four cuts would have connective tissue contents (as defined) on average of around 10 per cent. Furthermore none of the 91 samples of the cuts have a connective tissue content above 20 per cent.

The connective tissue contents of the female cuts are compared with those of the male cuts from the previous survey[2] in Table IV.

Table IV shows the similarity between the connective tissue contents of male and female brisket, chuck and blade and neck cuts. The difference between the male and female clod is not statistically significant ($P = 0{\cdot}95$).

Overall the survey has increased the knowledge of the distribution of connective tissue in male and female animals. The earlier recommendation[1] that an allowance be made by the official analyst for a 10–20 per cent. connective tissue content in the lean meat of beef products is seen from the data provided by these surveys to be reasonable.

Table III also shows nitrogen levels in the lean meat expressed on the fat-free meat content. The mean for each cut was found to be around 3·6 per cent. This is consistent with similar data from earlier surveys[1,2] and not inconsistent with the Analytical Methods Committee recommendation for an average nitrogen factor of 3·55 "as the best compromise for general use"[8].

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An Evaluation and Comparison of the CEM Meat Analysis System with Official Standard Methods for the Determination of Moisture and Fat in Meat and Meat Products

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The CEM meat analysis system is an automated instrumental method for rapid measurement of the moisture and fat content of meat products. The system utilises microwave drying for the measurement of moisture combined with enclosed solvent extraction for fat determination. In the evaluation presented here, samples of raw meats and sausages, with fat contents ranging from 0·3 to 43 per cent. and moisture contents ranging from 43 to 78 per cent., were analysed by the CEM system and the results compared with reference analyses, in which moisture content was determined by drying to a constant weight under vacuum and fat content by extraction with diethyl ether in a Soxhlet apparatus. For moisture, the CEM system gave results which were in good agreement with those obtained by the reference method, but it consistently under-estimated fat content in raw meat and sausages by values of 0·37 and 0·98 per cent. respectively.

Within the meat industry there is an increasing desire for more rapid analytical methods, since the standard laboratory procedures as recommended by the British Standard Institute (BS) or the Association of Official Analytical Chemists (AOAC), for the measurement of moisture and fat in meat and meat products are time consuming. The classical BS method[1] for the determination of moisture in such foods involves mixing the sample with sand and ethanol, evaporating the solvent on a water bath and then drying to a constant weight at 103 ± 2°C in an air oven. Alternative approaches are direct drying in an air oven at 100–102°C for 16–18 h[2] or in a vacuum oven at 100°C for 4 h[3]. For the determination of fat, the method involves continuous extraction of a dry sample for 8 h with petroleum spirit or diethyl ether, followed by evaporation of the solvent and weighing of the extracted fat[4].

More rapid methods for the measurement of these components are available and they have been extensively reviewed and evaluated[5]. Most rapid instrumental methods developed for multicomponent analyis of meat and meat products employ the principles of infrared spectrometry. For example, a method based on infrared transmission has been tested in a collaborative study by Bjarno[6], in which the fat, moisture and protein content of meat or meat products can be determined in a few minutes. Instruments working in the near infrared region can carry out these determinations within a few seconds on a prepared sample[7].

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One significant disadvantage of all infrared instruments is that they depend upon careful calibration against standard reference methods: thus a range of products may require a different calibration for each product type and hence any change in formulation may result in the need for recalibration. This will require additional time-consuming reference analysis.

The recently developed CEM Meat Analysis System (CEM Corporation, Indian Trail, North Carolina) should not require calibration in this way as it relies upon direct analytical methods: the determination of moisture is by microwave drying and fat is determined gravimetrically by enclosed solvent extraction. Bostian *et al.*[8] carried out a collaborative study comparing the CEM with official AOAC procedures. A correction factor of 0·55 needed to be added to the figure for percentage of moisture content for cooked sausage, pre-blends/emulsions and cured/cooked meats. Additive correction factors for fat of 0·4 for raw meats, pre-blends, emulsions, cured/cooked meats, and 0·8 for cooked sausages were also presented in the methods section but justification of their use was not given. Perusal of their results also suggested that the extraction of fat was poor at low fat levels.

This paper presents the results of an in-house evaluation intended to detect and quantity sources of bias in the CEM method by comparison with standard laboratory procedures involving the analysis of a range of meat and meat products.

Equipment

The CEM system is designed to perform rapid moisture and fat determinations on a wide range of foodstuffs, and consists of two instruments. The first is the automatic volatility computer (AVC 80) which is a moisture/solids analyser with a microwave drying system linked to an electronic balance and a microprocessor digital computer. The second instrument is the fat extraction unit consisting of an automatic extraction system (AES) and solvent recovery system. The electronic balance in the AVC 80 weighs to an accuracy of 1 mg, and the whole unit is controlled by the microcomputer which monitors and controls the microwave drying system, accepts and processes data from the balance and computes the percentage weight loss on drying. The initial weight, final weight and percentage moisture/solids are displayed and printed.

Materials

The CEM was evaluated by analysis of 116 samples selected to give a wide range of moisture and fat contents, thus enabling a thorough examination of the instrument's performance. Raw meats (beef and pork) and meat products (sausages) were analysed and the results compared with analyses of the same materials by standard procedures. For beef there were samples of commercial minced beef, comprising a selection of dissected muscles and forequarter joints, including neck, flank, skirt and shin. For pork, the samples were dissected longissimus dorsi muscle, spare rib, shoulder, belly and head meat. The sausages were all commercial brands chosen at random. All samples were reduced to smooth homogeneous mixtures by chopping in a double-knifed electric food processor.

Methods

All determinations were carried out in duplicate. Reference analyses were performed on all samples using the laboratory's normal operational procedures which are based on modifications of the AOAC[3] and BS[4] methods. Moisture content was determined by freeze drying followed by drying under vacuum at 100°C to a constant weight, and fat content by continuous extraction of a freeze dried sample with diethyl ether in a Soxhlet apparatus.

The CEM analysis is carried out following the prescribed two-stage procedure:

(i) The sample (about 5g) is spread thinly and sandwiched between two rectangular glass fibre pads and presented for microwave drying. The weight loss on drying is detected by the electronic balance, calculated as moisture content and displayed.

(ii) The pads containing the dried sample are removed from the AVC 80 and placed in the fat extraction unit. A round filter pad is inserted in the lid of the unit. On commencement of the extraction cycle 300 ml of methylene chloride enter the chamber and the sample is ground and agitated to extract the lipid. The dry, defatted residue is collected automatically on the filter pad, redried in the AVC 80 and the weight loss due to extraction is calculated as percentage fat content. The fat content is displayed and printed with the moisture content. A typical analysis time is about 6 minutes.

Statistical Method

In comparing the performance of the CEM with the standard analytical methods two types of bias may be defined: constant and proportional bias. These may be tested by simple linear regression of CEM results on those obtained by the standard method. This gives an equation of the form:

$$\text{CEM} = a + b.\ \text{Standard} + e$$

If there was no bias in the CEM results then the intercept a would be zero and the slope b would be unity; e denotes a random error. A non-zero intercept indicates constant bias, and a non-unit slope indicates proportional bias[9]. For example a unit slope and intercept of 5 would indicate that the CEM results are 5 units greater than the standard results; a zero intercept and slope of 0.7 would indicate that the CEM results are 0.7 times the magnitude of the standard result.

Results and Discussion

The mean values and summary statistics for moisture and fat content determined by the CEM system and by standard methods are given in Tables I and II. The repeatabilities are defined as the between-replicate standard deviations. These data are presented separately for each meat type and sausages. Also presented are overall values for meats which exclude sausages since they are a processed product. From the summary statistics, there appears to be no difference between the methods in terms of range and repeatability, with the exception of the moisture of pork meat, which, when determined by the

TABLE I

MEAN VALUES AND SUMMARY STATISTICS FOR MOISTURE CONTENT DETERMINED BY STANDARD METHOD AND CEM SYSTEM

	Number of samples	Mean	sd	Minimum	Maximum	Repeat-ability
Pork meat						
CEM	6	57·64	9·89	43·55	68·85	0·53
Standard	6	57·82	9·92	43·59	69·35	1·34
Pork LD						
CEM	36	75·01	0·71	73·75	76·29	0·18
Standard	36	75·08	0·68	73·91	76·70	0·15
Beef meat						
CEM	18	66·39	5·89	53·67	74·96	0·61
Standard	18	66·50	5·71	54·20	74·67	0·51
Beef muscle						
CEM	19	75·83	1·05	73·89	77·84	0·17
Standard	19	75·90	1·12	74·01	78·35	0·16
Beef LD						
CEM	6	76·31	0·47	75·76	77·01	0·07
Standard	6	76·29	0·46	75·77	77·07	0·16
Sausages						
CEM	31	53·46	5·43	45·07	62·89	0·42
Standard	31	53·10	5·48	44·02	63·19	0·40
Overall[a]						
CEM	85	72·23	6·57	43·55	77·84	0·38
Standard	85	72·31	6·52	43·59	78·35	0·49

[a] Excluding sausages.

standard method has a spuriously high repeatability. Informal comparison of the difference in mean values for moisture with repeatabilities reveals no overall differences, but similar comparison of fat contents reveals that the CEM consistently gives a lower value than the standard method, with some differences being large relative to repeatability. The previous comparisons are not designed to detect proportional bias, which can occur in two closely related ways. The simplest is to consider whether the bias is constant in the range of a particular product. The individual regressions presented in Tables III and IV provide a test for this. Since for moisture content, Table III, neither the intercepts nor the slopes differ significantly from the expected values of zero and 1.0 there is neither constant nor proportional bias for individual meat products. The large intercepts relative to standard errors, for beef muscle and beef LD (3.89 and 1.07 respectively) are a reflection of high variability in the samples. For fat content, (Table IV), the slopes obtained for each meat type are not significantly different from 1·0, indicating that there is no proportional bias. However, there is evidence of constant bias for fat content of pork meats and sausages, but not for beef meats.

TABLE II

MEAN VALUES AND SUMMARY STATISTICS FOR FAT CONTENT DETERMINED BY STANDARD METHOD AND CEM SYSTEM

	Number of samples	Mean	s.d.	Minimum	Maximum	Repeat-ability
Pork meat						
CEM	6	25·17	10·24	11·72	42·76	0·97
Standard	6	25·84	11·90	12·39	42·53	0·92
Pork LD						
CEM	36	0·66	0·46	0·15	2·42	0·09
Standard	36	1·06	0·47	0·38	2·79	0·06
Beef meat						
CEM	18	12·66	6·76	3·31	25·77	0·67
Standard	18	12·94	6·64	3·91	26·28	0·57
Beef muscle						
CEM	19	1·86	0·96	0·65	4·75	0·19
Standard	19	2·24	0·98	1·08	4·82	0·07
Beef LD						
CEM	6	0·64	0·24	0·38	1·00	0·11
Standard	6	0·78	0·21	0·44	0·97	0·03
Sausages						
CEM	31	20·16	7·29	6·33	33·78	0·51
Standard	31	21·14	7·28	7·06	34·10	0·58
Overall[a]						
CEM	85	5·20	8·47	0·15	42·76	0·46
Standard	85	5·57	8·45	0·38	42·53	0·46

[a] Excluding sausages.

TABLE III

REGRESSION OF MOISTURE CONTENT DETERMINED BY CEM SYSTEM ON STANDARD METHOD

	Intercept	SE[a]	Slope	SE[a]
Pork meat	0·08	1·17	0·995	0·020
Pork LD	0·67	4·32	0·990	0·058
Beef meat	−1.94	1·38	1·028	0·021
Beef muscle	6·56	3·89	0·913	0·051
Beef LD	1·90	1·07	0·976	0·140
Sausages	1·19	1·03	0·984	0·019
Overall[b]	−0·60	0·38	1·007	0·005

[a] SE = Standard Error of Regression Coefficient.
[b] Excluding sausages.

The individual regressions test bias over a limited range of moisture and fat contents for the individual products, but do not test bias over the whole range. In particular they do not test whether different meat types possess equal bias. This is provided by the overall regressions, which exclude sausages, in Table III and IV. This confirms the earlier result of no bias for moisture content. For fat

TABLE IV

REGRESSION OF FAT CONTENT DETERMINED BY CEM SYSTEM ON STANDARD METHOD

	Intercept	SE[a]	Slope	SE[a]
Pork meat	−1·39	0·42	1·028	0·015
Pork LD	−0·33	0·06	0·934	0·052
Beef meat	−0·49	0·25	1·016	0·017
Beef muscle	−0·25	0·16	0·942	0·065
Beef LD	−0·18	0·22	1·041	0·277
Sausages	−0·91	0·31	0·997	0·017
Overall[b]	−0·38	0·04	1·001	0·004

[a] SE = Standard Error of Regression Coefficient.
[a] Excluding sausages.

content the overall regression supports the absence of proportional bias and confirms the existence of constant bias.

Inspection of the individual regressions for fat content suggests that constant bias is present for pork meat (and sausages), but not for beef meats. Other analyses, not presented, confirmed the absence of proportional bias for fresh meats and tested the difference in constant bias between beef and pork meats. This was found to be not significant. Sausages were confirmed to have a significantly different bias from raw meats. Similar analysis supported the absence of bias for moisture content of all meats.

The lower fat content obtained using the CEM system when compared with the standard method may arise from differences in efficiency of fat extraction. A second extraction of the "fat free dry residue" recovered from the system results in a small weight reduction which would seem to indicate further removal of extractable material by the solvent. Furthermore, when determining moisture content in samples containing carbohydrate based meat extenders, there may be a risk of charring during the microwave drying cycle which could cause further decrease in dry weight and therefore a higher apparent moisture content for that sample.

Conclusion

This evaluation has shown that, overall, for the measurement of moisture content the CEM system gave equivalent results to the standard method. For fat content in raw meats and sausages, the respective percentage values are 0·37 and 0·98 low when compared with reference analysis. For these values to be equivalent to those obtained by reference analysis, adjustment by the addition of a correction factor would be necessary.

In a previous evaluation of this system, other workers[8] reported the need for correction factors for various categories of meats and meat products. The present results suggest that for fat content a single correction factor can be applied to all raw meats, but a different factor is required for sausages (processed meats). Moisture content required no correction.

However, apart from those limitations the CEM system does provide an effective rapid screening procedure for the determination of moisture and fat content in meat and meat products. Where high levels of accuracy are required the standard method remains the most reliable.

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A Collaborative Trial of a Method for the Determination of Lead in Street Dust

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Three European Community Bureau of Reference certified reference materials were analysed by 14 laboratories using a prescribed method. The results of these analyses are presented together with their statistical evaluation.

In response to a request from the Association of Public Analysts' Environment Sub-Committee, thirteen Public Analysts' Laboratories agreed to participate with the co-ordinating laboratory in a Collaborative Trial for the Determination of Lead. The method tested had been designed by the co-ordinating laboratory (Lancashire County Analyst's Department) for the measurement of lead in street dust.

To provide reliable materials for use in the trial, three European Community Bureau of Reference certified reference materials were chosen for analysis. These materials, though not directly comparable to street dust, did offer some similarity in matrix. The materials chosen were:

Code number	*Name of material*
BCR 142	Light sandy soil
BCR 143	Sewage sludge amended soil
BCR 144	Sewage sludge of domestic origin

In total 14 samples of each material were purchased, each laboratory analysing one sample of each material. Each sample was accompanied by common certification details from the Community Bureau of Reference.

Sample Preparation and Drying

Each sample of reference material is issued with documentation stipulating the method of sample preparation prior to analysis[1,2,3]. The certification documents also stipulate a method of drying the sample to constant weight over phosphorus pentoxide in order to record results of analysis with respect to sample dry weight[1,2,3].

However, following communication with the Reference Bureau[4], it was stated that heating the material to 105 ± 2 °C for a period of 20 h was a satisfactory method for determining the dry matter content of the reference materials.

Method of Analysis

The method of analysis is reproduced in the appendix.

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Results

TABLE I

LEAD CONTENT OF BCR 142:(A LIGHT SANDY SOIL) INDIVIDUAL RESULTS OF COLLABORATIVE TRIAL

Laboratory	Replicate determinations (*mg/kg* on dry weight)		Mean value*
1	67·0	104·0	85·5
2	41·8	40·1	41·0
3	39·5	40·6	40·1
4	38·9	40·0	39·5
5	37·2	36·4	36·8
6	36·2	35·7	36·0
7	35·6	35·7	35·7
8	34·4	35·5	35·0
9	33·9	34·8	34·4
10	31·7	24·5	28·1
11	27·5	27·3	27·4
12	25·5	27·0	26·3
13	25·9	25·4	25·7
14	24·1	24·4	24·3

* Mean values reported to first decimal place, whereas calculated mean used for statistical evaluation of data.

TABLE II

LEAD CONTENT OF BCR:143:(A SEWAGE SLUDGE AMENDED SOIL) INDIVIDUAL RESULTS OF COLLABORATIVE TRIAL

Laboratory	Replicate determinations (*mg/kg* on dry weight)		Mean value*
1	1548	1621	1585
2	1350	1366	1358
3	1384	1339	1362
4	1334	1354	1344
5	1322	1318	1320
6	1279	1288	1284
7	1267	1288	1278
8	1366	1371	1369
9	1362	1355	1359
10	1387	1382	1385
11	1370	1373	1372
12	1304	1303	1304
13	1207	1187	1197
14	1245	1295	1270

* Mean value reported to nearest whole number, whereas calculated mean used for statistical evaluation of data.

TABLE III

LEAD CONTENT OF BCR 144 : (A SEWAGE SLUDGE OF DOMESTIC ORIGIN) INDIVIDUAL RESULTS OF COLLABORATIVE TRIAL

Laboratory*	Replicate values (*mg/kg* on dry weight)		Mean value
1	590	524	557
2	524	514	519
3	505	515	510
4	557	548	553
5	446	446	446
6	480	491	486
7	478	479	479
8	525	526	526
9	539	540	540
10	512	514	513
11	507	507	507
12	478	480	479
13	472	452	462
14	474	476	475

* Mean values reported to the nearest whole number, whereas calculated mean used for statistical evaluation purposes.

Analysis of Data

The data in Tables I, II and III were examined according to BS 5497 (Part I) to determine repeatability and reproducibility. "Stragglers" and "Outliers" were identified using Dixon's and Cochran's tests[5]. The following were identified:

TABLE IV

"OUTLIERS"* AND "STRAGGLERS" IDENTIFIED BY DIXON'S AND COCHRAN'S TESTS IN A COLLABORATIVE TRIAL OF BCR REFERENCE MATERIALS 142, 143, AND 144

BCR code for sample	Laboratory number	Dixon's test		Cochran's test	
		Outlier†	Straggler	Outlier†	Straggler
142	1	Yes	No	Yes	No
	10	No	No	Yes	No
143	1	Yes	No	No	No
144	1	No	No	Yes	No

* The data associated with the outlier values identified above were excluded from subsequent calculation of repeatability and reproducibility values.

† Outliers for Dixon's test noted at the $P = 0{\cdot}99$ level.

Calculation of Repeatability and Reproducibility

Calculation of these values after the exclusion of the outlier results given in Table IV gave the following:

TABLE V

LEAD CONTENT OF BCR REFERENCE MATERIALS: REPEATABILITIES AND REPRODUCIBILITIES IN COLLABORATIVE TRIAL

Re material	Certified level of lead	Repeatability (r) *mg/kg*	r as percentage of certified level	Reproducibility (R) *mg/kg*	R as certified level
BCR 142	37·84	2·1	5·5	17·6	46·5
BCR 143	1332·7	47·4	3·6	161·4	12·1
BCR 144	495	16·7	3·4	91·8	18·5

BCRS Assessment Criteria

In addition to the preceding evaluation of the data, the certification documents supplied with the reference materials stipulate methods of assessing the analytical results. The criterion for satisfactory precision of the data is that the repeatability, calculated as the standard error of the mean of the results, is smaller than the standard deviation (specified by BCRS) of the certified means for the reference material.

The standard error of the mean is given by $Si/\sqrt{ni}$—the standard error of the mean of the ith set, ni being the number of replicate measurements of the set.

The criterion for satisfactory bias of the data is that the mean of the results lie within the range

$$\text{certified value} \pm 2s$$

where s is the standard deviation of the distribution of the mean values given in the BCRS certification documents.

Table VI gives the values for the above criteria for the respective BCR reference materials.

TABLE VI

"ACCEPTABILITY" CRITERIA FOR BCR REFERENCE MATERIALS

BCR reference	Precision requirement (*mg/kg*)	Bias requirement mg/kg
142	3·646	30·55 to 45·15
143	65·27	1200 to 1464
144	30·9	433·2 to 556·8

Utilising these criteria of precision and bias, the following results (Table VII) would not meet the respective specification.

TABLE VII

DATA OUTSIDE THE SPECIFICATION CALCULATED FROM BCR DOCUMENTATION

BCR reference	Results from laboratory number
142	1, 10, 11, 12, 13, 14
143	1
144	1

The Table VII data indicate that at the low lead content of BCRS material 142 (i.e. 37·84 mg/kg) the precision of the results is poor. This is readily seen in a plot of reproducibility calculated on the trial results against certified lead level for each of the reference materials, which shows that the optimum reproducibility (i.e. in the range 15% to 25%) of the lead content is achieved at lead concentration above 150 mg/kg.

It is therefore recommended that the method is most suitable for use at lead concentrations of 150 mg/kg or greater.

Conclusion

A method for the determination of lead in street dust has been collaboratively tested using certified reference materials whose matrix has some semblance to street dust. The repeatability and reproducibility of results produced are acceptable and indicate that the optimum performance of the method is achieved at lead concentrations above 150 mg/kg.

References

1. "BCR 142: The Certification of the Contents of Cadmium, Copper, Mercury, Nickel, Lead and Zinc in a Light Sandy Soil." Community Bureau of Reference, CBC, Brussels, Belgium, 1983.
2. "BCR 143: The Certification of the Contents of Cadmium, Copper, Mercury, Nickel, Lead and Zinc in a Sewage Sludge Amended Soil." Community Bureau of Reference, CBC, Brussels, Belgium, 1983.
3. "BCR 144: The Certification of the Contents (Mass Fractions) of Cadmium, Cobalt, Copper, Manganese, Mercury, Nickel, Lead and Zinc in a Sewage Sludge of Domestic Origin. Community Bureau of Reference," CBC, Brussels, Belgium, 1983.
4. Personal communication, Community Bureau of Reference.
5. British Standards Institution "Precision of Test Methods Part 1 Guide for the Determination of Repeatability and Reproducibility for a Standard Test Method." B.S. 5497: Part 1, London 1979.

Appendix: Method of Analysis for the Determination of Lead in Street Dust

Transfer the samples as received from site into a tared dish (using a fine jet of water if necessary).

Dry to constant weight at 105 ± 2° C.

Record the weight of the dried sample.

Sieve the dried sample through a 1 mm plastic sieve and weigh the fine fraction.

Quarter the fine fraction until a portion weighing 1–2 g (weighed accurately) is obtained.

To this portion, contained in a 250 ml beaker, add 10 ml of concentrated nitric acid and evaporate to dryness.

Extract the residue with 20 ml of concentrated nitric acid and 30 ml of water by boiling for 10 min.

Filter the extract into a 200 ml volumetric flask and make up to volume.

Measure the lead concentration by atomic absorption spectrophotometry at 283·3 nm using standards prepared in 10% nitric acid.

Calculate the lead content on the dried weight of the original material.

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The Determination of Fat Content and Egg-yolk Content of Mayonnaise: Collaborative Trial

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The results of a collaborative trial, involving 20 participants, of methods for the determination of the total fat and egg-yolk contents of mayonnaise are reported. The results indicate that the methods are acceptable and so can be recommended for inclusion in the Codex European Regional Standard for Mayonnaise. The recommended precision values to be included in the methods are: 1·1 and 2·0 g/100 g for repeatability and reproducibility respectively of the fat content method, and 0·6 and 0·7 g/100 g for the repeatability and reproducibility respectively of the egg-yolk content method.

The proposed draft Codex European Regional Standard describes mayonnaise as a condiment sauce obtained by emulsifying edible vegetable oil in an aqueous phase consisting of vinegar, the oil-in-water emulsion being produced by the hen's egg yolk. Mayonnaise may also contain certain optional ingredients. The minimum content of vegetable oil shall be 77 per cent. and the technically pure egg yolk (defined as containing not more than 20 per cent. of egg white) shall be 6 per cent., related to the total product.

The above, taken from the Report of the Fifteenth Session of the Codex Co-ordinating Committee for Europe meeting in Thun, Switzerland 16–20 June 1986[1], was amended at the Sixteenth Session, held in Vienna, Austria, 27 June–1 July 1988, the 77 per cent. vegetable oil provision being replaced by a minimum total fat content of 78·5 per cent.

It is a requirement of the Codex Alimentarius Commission that methods of analysis be prescribed to determine and enforce such compositional standards. At the Fourteenth (1984) Session of the Codex Co-ordinating Committee for Europe of the Codex Alimentarius Commission, the Comite des Industries des Mayonnaises et Sauces Condimentaires de la Communaute Économique Européenne (CIMSCEE) undertook to draft a section on methods of analysis for possible inclusion in the mayonnaise Standard[2]. The methods were drafted[3] and discussed at the Fifteenth Session of the Co-ordinating Committee for Europe; they were recommended for adoption at that Session[4].

The methods were referred to Codex Committee for Methods of Analysis and Sampling (CCMAS) for endorsement and were considered at the Fifteenth Session of CCMAS (November 1986) but were not endorsed[5] mainly because of

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lack of documentation at that meeting. CIMSCEE wished for the methods to be re-considered at the next Session of CCMAS and would prepare a report to aid such consideration. Included in such a report would be the results of any collaborative trials carried out to validate the recommended methods.

Collaborative trials had been carried out previously under the auspices of CIMSCEE to validate a fat content method and under the auspices of the European Commission to validate an egg-yolk content method; however such trials had not been designed satisfactorily. In the light of this the U.K. Ministry of Agriculture, Fisheries and Food (MAFF) decided to examine the methods to assess their suitability for inclusion in any Codex Standard on mayonnaise.

The following methods were examined:

Fat content—Method 1/20 of the Bundesverband der Deutschen Feinkostindustrie[6];

Egg-yolk content—Quinoline Molybdate Method of the BENELUX organisation[7].

Collaborative Trial Organisation, Samples, Methods and Results

Participants

Twenty laboratories agreed to participate in the collaborative trial (19 U.K. public analyst laboratories and the Laboratory of the Government Chemist).

Samples

Four small quantities of mayonnaise were prepared at H. J. Heinz Co. Ltd., Hayes; the recipes for each sample are given in Table I. The egg yolks used to prepare the samples were carefully separated by hand from 100 medium-sized commercial fresh eggs, obtained from a number of different batches, and then blended with a domestic electric mixer. The egg yolk was stored in a tightly lidded container in a refrigerator overnight. From the mean duplicate determinations of the water content, obtained by vacuum oven drying at 70°C, i.e. 51·1 per cent., and calculating on the basis of the recognised average compositional figures for pure egg yolk containing 48·5 per cent. of water and egg white 87·5 per cent., the prepared egg yolk contained 7 per cent. of white of egg.

Each batch was made by adding the appropriate quantities of the ingredients other than the vegetable oil to the bowl of a planetary mixer (Hobart Manufacturing Co. Ltd.) and stirring to dissolve the salt and sugar. The vegetable oil was then added in a thin stream from a tap funnel with continuous rapid mixing of the bowl contents during a period of approximately 8 min. The resulting mayonnaise was then subject to a high shear mixing (Moritz Chemical Engineering Co. Ltd.) for 3 min to ensure effective emulsification, and filled into 200-g jars fitted with lids.

Assuming that pure egg yolk contains 31 per cent. of fat, the composition of each mayonnaise may be calculated to have a percentage of total fat and pure egg yolk, as in Table II:

TABLE II

CALCULATED COMPOSITION OF MAYONNAISE SAMPLES

Sample	Total Fat *g per 100 g*	Pure Egg Yolk *g per 100 g*
1	78·7	5·6
2	75·5	4·7
3	76·6	5·1
4	77·9	6·0

Sample 4 was used as the pre-trial sample, to be analysed in duplicate. All samples were sent to participants in sealed 200-g glass jars. Samples 1 to 3, the trial samples proper, were sub-divided and sent to participants as blind duplicates.

Methods of Analysis Collaboratively Tested

Participants were asked to familiarise themselves with the methods by using a practice (pre-trial) sample. The methods used by participants in the trial for the analyses of the trial sample proper are given in the Appendix.

Results

Each participant was asked to analyse each sample once only and to report the single result as a percentage by weight (g/100 g) on the sample as received. The results obtained by participants are given in Tables III–V.

Statistical Analysis of the Results

The results obtained were statistically analysed according to procedures outlined by the British Standards Institution[8]. Significant differences between pairs of individual results were identified using Cochran's test and the extremes of magnitude of pairs of results were identified by Dixon's test. Outlying results are marked in the tables of results.

The values of the means, repeatabilities and reproducibilities, each as defined according to the British Standards Institution procedure[8], were calculated and these are also given in the tables.

Comments on the Results Obtained

The values obtained for precision parameters, and the reproducibility especially, may be considered to be somewhat higher than initially expected; however they are acceptable by comparison with similar determinations for other commodities. It is frequently observed that the determination of fat at high levels in a commodity exhibits variability similar to that given by this trial.

The values for the content of fat determined are in the same relative order as the "expected" values though the absolute values are somewhat higher.

In this determination a substantial quantity of sample is taken and because of the percentage of fat in the samples, there is a significant fat residue weighed as the gravimetric end-point. It is possible that solvent is trapped in this residue thus marginally enhancing the determined fat values.

The values for the egg contents determined in the trial are very similar to the "expected" values. This is as anticipated because the eggs used to prepare the samples were obtained from a number of different sources and the average factor was used in the method of analysis.

The precision values observed were marginally better for the pre-trial sample than for the trial samples proper, thus indicating some analyst bias.

The results reported by laboratory 6 (Table V) were abnormally high and subsequently rejected by Dixon's outlier test. The source of these high results were later traced back to the batch of magnesium acetate (4.7) used as the ashing aid (6.2.4) which was found to contain 0·019 per cent. of phosphorus, highlighting the need to specify a low phosphorus and/or phosphate content for this and other reagents.

Conclusions

The methods tested in the trial were found to be satisfactory with respect to precision and recovery. They may therefore be recommended to the Codex Coordinating Committee for Europe for inclusion in the draft European Regional Standard on Mayonnaise for the determination of egg-yolk and total fat contents in mayonnaise. Some consideration should be given by that Committee as to whether the standard for vegetable oil should be expressed as "vegetable oil" or as "total fat".

Further work may be carried out to ascertain whether the entrapment of extraction solvent occurs in the fat residue during the gravimetic end-point determination.

It is recommended that the accepted values of repeatability and reproducibility for the two methods be those given in Table VII.

TABLE VII

RECOMMENDED VALUES OF REPEATABILITY AND REPRODUCIBILITY FOR THE ANALYTICAL METHODS FOR MAYONNAISE

Determination	Repeatability (r) *g/100 g*	Reproducibility (R) *g/100 g*
Fat	1·1	2·0
Egg yolk	0·6	0·7

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Appendix

METHOD 1: DETERMINATION OF TOTAL FAT IN MAYONNAISE

1. SCOPE

The method permits the determination of the total fat content in mayonnaise and other emulsified sauces.

2. DEFINITION

The fat content: the content of fat as determined by the method specified.

3. PRINCIPLE

The well-mixed sample is digested with hydrochloric acid and the resulting liquid filtered through two moistened pleated filter papers. The residue remaining on the filter papers is dried and extracted for 4 h with petroleum ether

or n-hexane. The solvent is distilled off and the residual fat is dried at 103 ± 2°C under atmospheric pressure, cooled and weighed.

The fat content is calculated from the weight obtained.

4. REAGENTS

All reagents should be of recognised analytical grade unless specified otherwise.

4.1 *Indicator paper.*

4.2 *Petroleum ether*, boiling range 40–60°C, or n-hexane.

4.3 *Hydrochloric acid*, approximately 4 N.

4.4 *Silver nitrate* solution, 0·1 N.

4.5 *Water*, distilled or demineralised.

4.6 *Cotton wool*, defatted.

5. APPARATUS

5.1 Ceramic wire gauze (for Bunsen burner and tripod).

5.2 Beakers, 600 ml, tall form.

5.3 Desiccator containing silica gel or other suitable drying agent.

5.4 Soxhlet extraction apparatus—siphon capacity about 100 ml with ground glass joints and 250 ml flat-bottomed flask.

5.5 Extraction thimbles, defatted (e.g. Schleicher & Schull No. 603 or Macherey & Nagel No. 645F).

5.6 Double pleated filter papers 150–200 mm diameter with average pore diameter 5 μm maximum (e.g. Schleicher & Schull No. 597½ and No. 595½ or Macherey & Nagel No. 616¼ or 615¼).

5.7 Glass rod.

5.8 Glass funnel 100 mm diameter minimum.

5.9 Sand or water bath, with suitable means of controlled heating.

5.10 Anti-bumping granules.

5.11 Watch-glass cover, 100 mm diameter.

5.12 Drying oven, electrically heated and thermostatically controlled at 103 ± 2°C.

6. PROCEDURE

6.1 *Sample Preparation and Storage*

Take the contents of an entire package or several packages to provide a sub-sample of at least 200 g. Store in a tightly closed container at 2–6°C in the dark to prevent any alteration. Allow the sample to reach uniform room temperature before analysis, stirring if necessary.

6.2 *Procedure for Fat Determination*

6.2.1 Dry a flat-bottomed extraction flask, containing an anti-bumping granule, in the oven for 1 h at 103 ± 2°C, cool in a desiccator to room temperature, and weigh (designate as weight *A*).

6.2.2 Weigh 3–5 ± 0·001 g of the well-mixed sample (depending upon the weight of fat expected, which should not exceed 3 g) into a 600 ml beaker (5.2) (designate weight of sample as *C*).

6.2.3 Add 150 ml of 4 N hydrochloric acid to the beaker and stir with a glass rod. Add a few anti-bumping granules, cover the beaker with a watch-glass, and heat to boiling. Keep the contents boiling gently on a low heat for 1 h, stirring frequently.

6.2.4 Add 150 ml of hot water to the beaker. Place the fluted filter papers in the funnel and moisten thoroughly with hot water. Filter the hot digested liquid quickly, and wash the beaker, watch glass cover and glass rod three times with hot water, passing each successive washing through the filter papers.

Test the washings for absence of acidity, using indicator paper, or for absence of chloride, using 0·1 N silver nitrate solution. Continue washing the filters until the filtrate is free of acid.

6.2.5 Place the funnel containing the filter papers in the beaker with the watch-glass and glass rod, and dry in the oven for 1 h.

6.2.6 Transfer the dry filter papers to an extraction thimble. Remove any traces of fat present in the beaker with a piece of cotton wool damped with extraction solvent (4.2), and add this to the extraction thimble.

Place the thimble in the extraction apparatus, add solvent to the extraction flask, and assemble the extractor. Rinse the beaker, watch-glass cover and glass rod with solvent and add the rinsings to the extraction apparatus. Heat the extraction flask on a sand or water bath, and allow the extraction to proceed continuously for 4 h.

6.2.7 Remove the bulk of the solvent by distillation, and any traces of solvent remaining with a gentle stream of air. Dry the flask in a horizontal position in the oven for 1 h at 103 ± 2°C, cool in the desiccator and weigh to the nearest mg.

Repeat the drying, cooling and weighing process until successive weights differ by no more than 0·1 mg (designate per cent as weight *B*).

7. EXPRESSION OF RESULTS

7.1 *Calculation*

The total fat content, in g/100 g, is calculated according to the following:

$$\text{Fat content (g/100 g)} = \frac{(B - A) \times 100}{C}$$

where: A = weight of empty flask and granule in g (6.2.1)
B = weight of flask with extracted fat after drying (6.2.7)
C = weight of sample taken (6.2.2)

If the difference between the results from two determinations does not exceed 0·5 per cent. of the fat content, the mean value is taken as the fat content. If this condition is not fulfilled, two further determinations are carried out. The value then taken for the fat content is the mean of four determinations.

The result is given to one place of decimals, the second place being subject to rounding.

METHOD 2: DETERMINATION OF EGG YOLK IN MAYONNAISE

1. SCOPE

The method permits the determination of egg-yolk content in mayonnaise and emulsified sauces.

2. DEFINITION

The egg-yolk content: the content of egg yolk as determined by the method specified.

3. PRINCIPLE

The phospholipids are extracted together with fat using a mixture of chloroform and ethanol. After ashing, the phosphate content is determined gravimetrically as the quinoline phospho-molybdate.

4. REAGENTS

All reagents should be of recognised analytical grade unless specified otherwise.

4.1 *Ethanol* 96% by volume.

4.2 *Chloroform.*

4.3 *Chloroform–ethanol mixture*, 3 : 2 by volume.

4.4 *Acetone.*

4.5 *Sulphuric acid* (density 1·84 g/cm^3).

4.6 *Nitric Acid* (density 1·40 g/cm^3).

4.7 *Magnesium acetate*, Mg $(CH_3COO)_2.4H_2O$ (amendment: "low in phosphorus").

4.8 *Quinoline Molybdate Solution*

4.8.1 Dissolve 70 g of sodium molybdate $Na_2MoO_4.2H_2O$ in 150 ml of distilled water.

4.8.2 Dissolve 60 g of citric acid in 150 ml of distilled water and add 85 ml of nitric acid.

4.8.3 Slowly pour solution (4.8.1) into solution (4.8.2) stirring constantly.

4.8.4 To 100 ml of distilled water, carefully add 35 ml of nitric acid and 5 ml of freshly distilled quinoline. Pour this solution into solution (4.8.3) stirring

continuously. Allow to stand for 24 h at room temperature. If a precipitate forms, remove it by filtration. Add 280 ml of acetone and then dilute to 1 litre with water. Keep the molybdate reagent (4.8) in a well closed plastic container in a dark place.

5. APPARATUS

5.1 Electrical hot plate with magnetic stirrer.

5.2 Erlenmeyer flask, 300 ml with reflux condenser.

5.3 Pleated filter 15 cm diameter.

5.4 Volumetric flask 250 ml.

5.5 Platinum dish, approximately 130 ml capacity.

5.6 Sintered glass crucible G4.

5.7 Muffle furnace, maintained at 800°C.

5.8 Water bath.

5.9 Desiccator.

5.10 Erlenmeyer flask, 250 ml.

5.11 Watch-glass.

5.12 Glass rod.

5.13 Filter paper, ashless.

5.14 Hotplate, electrical.

5.15 Büchner flask.

5.16 Drying oven, electrically heated and thermostatically controlled at 260 ± 20°C.

6. PROCEDURE

6.1 *Sample Preparation and Storage*

Take the contents of an entire package or several packages to provide a sub-sample of at least 200 g. Store in a tightly closed container at 2–6°C in the dark to prevent any alteration. Allow the sample to reach uniform room temperature before analysis, stirring if necessary.

6.2 *Separation of Phospholipids*

6.2.1 Weigh 12–13 ± 0·01 g of sample into a 300 ml Erlenmeyer flask (5.2).

6.2.2 Add 100 ml of chloroform and 75 ml of ethanol to the flask and mix thoroughly using the magnetic stirrer until a homogeneous suspension is obtained. Heat for 1 h under reflux with continuous stirring.

6.2.3 Allow the flask to cool and stand overnight. Filter the contents of the flask through a pleated filter paper, previously moistened with chloroform–ethanol

mixture (4.3), into a 250 ml volumetric flask. Rinse the Erlenmeyer flask and filter with more chloroform–ethanol solvent, and add to the volumetric flask, finally diluting with the same solvent to 250 ml.

6.2.4 Pipette 100 ml of the solution (6.2.3) into a platinum dish, cover with an ashless filter paper and evaporate off the solvent cautiously over a water bath to dryness.

Add 3·5 g of magnesium acetate to the dish. Cut the filter paper into pieces and cover the contents of the dish. Cover the dish with another ashless filter paper.

Calcine the residue gently over a flame and then in a muffle furnace at 800°C until a white powder is obtained (*ca.* 1 h).

6.2.5 Dissolve the ash (6.2.4) carefully in 15 ml of nitric acid (by allowing the acid to flow along a glass rod) and transfer to a 250 ml Erlenmeyer flask. Rinse the dish several times with water adding the rinsings to the flask. Dilute the flask contents to 50 ml and allow to cool to room temperature.

6.2.6 Add 50 ml of quinoline molybdate reagent (4.8) to the flask with continual stirring. Cover the flask with a watch-glass and boil on the hotplate for 1 min.

Allow the flask to cool to room temperature, stirring 2–3 times.

6.2.7 Heat a sintered glass filter crucible (5.6) at 260 ± 20°C for 30 min, cool in a desiccator and weigh to the nearest mg.

6.2.8 Transfer the precipitate (6.2.6) to the sintered glass filter crucible with gentle suction and wash five times with 20 ml volumes of water.

6.2.9 Dry the crucible and contents at 260 ± 20°C in the drying oven for 1 h, cool in a desiccator and weigh to the nearest mg.

7. EXPRESSION OF RESULTS

7.1 *Calculation*

7.1.1 Calculate the lipid phosphoric acid (lecithin) content (expressed as P_2O_5 g/100 g) from:

$$\text{lecithin } P_2O_5 \text{ (g/100 g)} = \frac{2{\cdot}5 \times \text{weight precipitate} \times 0{\cdot}03207 \times 100}{\text{weight of sample}}$$

7.1.2 Calculate the egg-yolk content (expressed as g/100 g) from the relationship: egg-yolk content (g/100 g) = 102 × lecithin P_2O_5 content (g/100 g) (7.1.1).

TABLE I

FORMULATIONS OF SAMPLES OF MAYONNAISE USED IN THE TRIAL

Code	Vegetable oil *per cent.*	Egg yolk *per cent.*	Salt *per cent.*	Sugar *per cent.*	12% acetic acid spirit vinegar *per cent.*	Water *per cent.*
1	77	6	1·5	3·5	5	7
2	74	5	1·5	3·5	5	11
3	75	5.5	1·5	3·5	5	9.5
4	76	6.5	1.5	3.5	5	7.5

TABLE III

TOTAL FAT AND EGG-YOLK CONTENTS OF MAYONNAISE COLLABORATIVE TRIAL—PRE-TRIAL RESULTS

Laboratory	Fat content *g/100 g*	Egg-yolk content *g/100 g*
1	78·3, 78·6	5·91, 5·54
2	78·5, 78·5	5·92, 5·98
3	79·5, 79·2	6·34, 6·40
4	78·8, 78·6	5·90, 5·92
5	79·3, 79·5	5·49, 5·95
6	79·1, 79·1	5·87, 5·83
7	78.9, 78·7	5·64, 5·80
8	78·7, 78·8	6·01, 6·01
9	78·7, 79·0	5·96, 6·08
10	78·6, 78·4	6·00, 5·80
11	76.4,[c] 78·1[c]	5·94, 5·61
12	79·3, 78·8	8·66,[c] 7·33[c]
13	78·7, 78·3	6·01, 6·12
14	77·9, 78·1	6·10, 6·10
15	78·3, 78·5	5·75, 5·76
16	78·8, 78·5	5·90, 5·90
17	75·6,[c] 78·6[c]	5·80, 6·00
18	78·6, 78·5	5·98, 6·49
19	79·1, 79·2	5·90, 5·90
20	79·9, 80·4	5·80, 5·20
Mean ($\bar{x}$)	78·8	5·90
Repeatability (r)	0·54	0·51
Reproducibility (R)	1·43	0·67
"Expected" value*	77·9	6·0

* Calculated values obtained from Table II.

[a] Sample broken in transit. "Equivalent" result not used in calculation of mean, repeatability or reproducibility.

[b] Sample broken in transit, sample 3 analysed in duplicate.

[c] Results rejected by Cochran's test, $P \leq 0{\cdot}05$. Values not used in calculation of mean, repeatability or reproducibility.

[d] Results rejected by Dixon's test, $P \leq 0{\cdot}05$. Values not used in calculation of mean, repeatability or reproducibility.

TABLE IV

TOTAL FAT CONTENT OF MAYONNAISE: COLLABORATIVE TRIAL

Laboratory	Fat content *g/100 g*		
	Sample code 1, 3	Sample code 4, 6	Sample code 2, 5
1	76·3, 76·4	77·2, 76·8	79·2, 79·7
2	76·8, 76·7	77·4, 77·8	79·7, 79·5
3	—[a], 76·3[a]	76·8, 77·3	79·4, 79·9
4	76·7, 76·7	76·9, 78·3	79·5, 79·5
5	76·9, 77·4	77·9, 77·9	80·2, 80·4
6	77·7, 77·8	78·3, 78·2	80·7, 80·6
7	76·0, 76·4	77·3, 77·4	79·1, 79·5
8	76·6, 76·6	77·6, 76·9	79·7, 79·5
9	76·9, 77·9	77·8, 77·7	79·8, 80·3
10	76·0, 75·5	76·7, 76·5	78·0, 79·1
11	76·6,[c] 71·5[c]	75·6, 76·9	78·4, 77·8
12	75·2,[c] 78·2[c]	77·8, 77·7	79·7, 77·7
13	77·9, 77·6	78·1, 78·0	79·2, 79·8
14	76·2, 75·8	74·1,[d] 76·0[d]	77·8, 76·4
15	76·1,[b] 76·4	76·7, 76·4	79·2, 79·2
16	76·0, 76·1	77·0, 77·1	79·3, 79·4
17	80·2,[c] 75·0[c]	77·2, 76·2	77·8, 76·1
18	75·9, 76·8	76·9, 76·9	79·3, 79·4
19	76·9, 77·5	78·9, 78·9	80·7, 80·0
20	75·9, 76·9	77·2, 77·2	80·4, 79·7
Mean ($\bar{x}$)	76·6	77·4	79·3
Repeatability (r)	1·03	1·12	1·60
Reproducibility (R)	1·89	2·03	2·99
"Expected" Value*	75·5	76·6	78·7

* Calculated value obtained from Table II.
For key, see Table III.

TABLE V

TOTAL EGG-YOLK CONTENT OF MAYONNAISE: COLLABORATIVE TRIAL

Laboratory	Egg yolk content *g/100 g*		
	Sample code 1, 3	Sample code 4, 6	Sample code 2, 5
1	4·56, 4·99	5·92, 4·92	5·83,[c] 5·29[c]
2	4·45, 4·53	4·94, 4·97	5·36, 5·42
3	—,[a] 4·84[a]	5·40, 5·06	6·27,[d] 6·05[d]
4	4·60, 4·87	5·06, 5·02	5·33, 5·48
5	4·35, 3·84	4·87, 4·96	5·25, 5·34
6†	7·88,[d] 8·08[d]	8·54,[d] 8·68[d]	8·88,[d] 9·14[d]
7	4·67, 4·57	5·03, 5·04	5·57, 5·56
8	5·62, 4·71	5·12, 5·19	5·62, 5·72
9	4·43, 4·68	5·10, 5·59	5·45, 5·26
10	4·70, 4·60	4·90, 4·90	5·50, 5·60
11	4·29, 4·40	4·39, 4·91	5·40, 5·38
12	5·17, 4·56	4·88, 5·84	5·46, 5·31
13	4·64, 4·15	5·16, 4·87	5·29,[d] 4·84[d]
14	4·60, 4·20	5·10, 5·20	5·40, 5·60
15	4·70,[b] 4·64	5·02, 5·25	5·58, 5·66
16	4·70, 4.60	4·90, 5·10	5·50, 5·60
17	4·80, 4·40	5·30, 5·10	5·60, 5·30
18	4·59, 4·81	4·80, 5·05	5·30, 5·60
19	4·60, 4·50	4·80, 5·00	5·40, 5·40
20	4·30, 4·30	4·80, 5·10	5·40[c], 4·30[c]
Mean ($\bar{x}$)	4·6	5·1	5·5
Repeatability (r)	0·74	0·79	0·31
Reproducibility (R)	0·85	0·78	0·37
"Expected" value*	4·7	5·1	5·6

* Calculated value obtained from Table II.

† High results due to use of magnesium acetate (4·7) found to contain 0·0191 per cent. phosphorus; "adjusted" results are: 1,3 (4·80, 4·87); 4,6 (5·33, 5·42) and 2,5 (5·71, 5·93) g/100 g.

For key, see Table III.