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Collaborative Study of Precision Characteristics of the AOAC Method for Crude Fat in Meat and Meat Products

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A collaborative study of determination of fat by AOAC method 24.005(a) or (b) was conducted to gain more knowledge regarding its characteristics of precision. Twelve analysts in meat and food industry laboratories each performed 4 determinations on 7 samples containing 3.4–48% fat. The following characteristics of precision of the method were established: Variation between duplicate determinations was 0.3% fat; within-laboratory repeatability on separate days was 0.3% fat for samples containing up to 27% fat and 0.7% for samples containing 48% fat; variation among laboratories, including variation due to laboratory-sample interaction, was 0.4% fat; and reproducibility, which includes variations of determinations on a sample by different analysts using different sets of equipment in different laboratories, was 0.6% fat. This information is especially useful for comparative evaluations of alternative methods of fat determination.

The official AOAC method of analysis for crude fat in meat and meat products is performed by ether extraction following the use of either of 2 drying methods, **24.005(a)** or **(b)** (1). The status of this method is established on the basis of evaluations by Windham (2, 3), Philbeck (4), and Pettinati *et al.* (5), and by extensive successful use. Other, more rapid alternative methods proposed should yield results equivalent to those obtained by this standard analysis in critical studies of their accuracy and precision. The accuracy of the official method is established on a de facto basis; for comparative purposes, data on its precision are incomplete. The collaborative study reported here provides such data in the form of 5 components of precision: (*1*) repeatability between duplicates (the deviation between paired determinations by an analyst on a sample at essentially the same time); (*2*) repeatability on different days of duplicate determinations (also defined as within-laboratory replication, or random error; it is the deviation between independent determinations performed on one day and repeated on any other day by the same analyst on separate portions of the same sample); (*3*) laboratory-sample interaction (the variation among determinations on sets of samples obtained by different laboratories, caused by differences in sample handling, homogeneity, treatment, spoilage, and time element, for instance); (*4*) variation among laboratories (the deviation of determinations on a sample performed by different analysts in different laboratories); and (*5*) reproducibility (consisting of the sum of the variances resulting from the above components *2*, *3*, and *4* and reflecting variations in determinations performed on a sample by different analysts using different sets of equipment in different laboratories). These criteria provide insight regarding the precision of methods for purposes of standardization and predict the expected agreement between analyses under practical conditions.

Collaborative Study

Each of 12 collaborators used equipment and supplies for the determinations independent of the others. Each was asked to use an experienced analyst, but not the laboratory's most experienced analyst. Seven samples, including fresh meat and emulsified meat products representative of the different products encountered in regulatory and quality control work, were ground, mixed, and handled according to AOAC method **24.001** (1) and distributed frozen in plastic bags to the collaborators as follows: 3 beef (about 10, 20, and 25% fat), 2 pork (about 3.5 and 48% fat), 1 frankfurter (about 27% fat), and 1 bologna (about 22% fat). The beef and pork samples were prepared by mixing quantities of commercial lean and fatty tissues purchased from local packers. The frankfurter and bologna samples were prepared from quantities of commercial lots purchased from local

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processors. The collaborators were requested to store the samples frozen until analyses were to be performed; then thaw the sample required, transfer it to a vessel and thoroughly remix it, return the unused portion of the sample to its original plastic bag or to a similar one, and refrigerate but not refreeze it until ready for the second analysis. Collaborators were requested to perform duplicate analyses on a sample, or samples, on one day and repeat determinations in duplicate within less than 1 week.

METHOD

Use AOAC method **24.005(a)** or **(b)** with the precaution that drying the extracted fat from samples containing 30% fat or more be continued for 30-min intervals at 100°C to constant weight.

Results and Discussion

The collaborative determinations were statistically treated following the procedures of Youden and Steiner (6) and the American Society for Testing and Materials (ASTM) (7, 8). The data were subjected to a number of outlier tests to statistically indicate suspect determinations or laboratories before components of precision were calculated. Following calculation of standard deviation between duplicate determinations, analyses of variance were performed using 2 equally advantageous formats. First, repeatability, between-laboratory variation, and reproducibility were calculated by applying to the collaborative determinations, one sample at a time, an analysis of variance designed to yield results relative to each sample, a design which permitted determination of poolable variances. Pooled results from this analysis were then compared with those obtained by a second analysis of variance from which results were obtained already pooled when the entire block of collaborative data involving all 7 samples was taken. The latter analysis also resolved the laboratory-sample interaction component separated from the between-laboratory variation, which was not possible with the former. Once the overall components of precision and overall mean were calculated, the values were used as a basis for comparison of the individual laboratory variations and means. Finally, the determinations of crude fat by the AOAC standard were compared with determinations of total fat in the same samples by another method to indicate the usefulness of the standard as a reference method.

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Outlying Data

Table 1 lists the individual determinations of the collaborative replicate analyses. From the average of each laboratory's 4 determinations, the first of a number of outlier tests was performed on the data by ranking. The score for Collaborator 5 was well below the ranking score limits specified for a study involving 12 laboratories and 7 materials, and indicated the presence of a pronounced systematic error in the laboratory's results. This error was also conspicuous by inspection of 2-sample plots of the laboratory averages.

Other outlier tests were applied to differences between duplicates, between days, and among laboratory averages. Results of these tests confirmed the observations made in the tests described above and specifically indicated the outliers present in the 3 categories of original and averaged collaborative determinations (Table 2). In the subsequent calculation of components of precision, it was an objective to exclude only the indicated outliers instead of all data from a particular laboratory. For this reason, only the 10 duplicate determinations listed in the day 1 and day 2 columns were excluded from the data in calculating the precision between duplicates. However, it was also an objective to arrive at precision estimates representative of a consensus of the laboratories. For this reason, all data from Collaborator 5 were excluded from the analyses of variance, but calculations were made both with and without outliers in data from Collaborator 9, as will be indicated.

Standard Deviation between Duplicate Determinations

Sample means, variances, standard deviations, and pooled results of duplicates are shown in Table 3. Sample variances were pooled by weighting for degrees of freedom after testing for homogeneity of variance by Bartlett's chi-square test (9) and by the $F_{max.}$-test (10). By both tests, homogeneity was indicated after excluding the higher variance of Sample P-2. With and without Sample P-2, the standard deviation was 0.28 and 0.26%, respectively, which, in both cases, was rounded off to 0.3% fat.

Table 1. Collaborative fat analyses by official AOAC method[a] for meat and meat product samples (% fat)

	Beef—1		Beef—2		Beef—3		Pork—1		Pork—2		Frankfurter		Bologna	
Coll.	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2	Day 1	Day 2
1[b]	11.39	10.86	20.12	19.97	25.14	25.57	3.84	3.66	48.86	48.98	27.42	27.58	22.56	22.70
	11.19	10.90	20.42	20.47	25.24	25.62	3.58	3.67	49.00	48.69	27.13	27.25	22.53	22.92
2	11.14	11.46	20.67	20.34	24.31	25.08	3.75	3.59	47.83	48.57	27.32	26.96	22.10	21.72
	11.07	10.95	20.47	20.16	24.03	25.09	3.46	3.75	47.86	47.99	26.80	27.00	21.41	21.70
3	10.86	11.14	20.24	20.29	25.09	25.27	3.40	3.65	48.38	48.80	27.19	27.61	21.90	22.05
	10.92	10.96	20.82	20.44	25.11	25.43	3.80	3.53	48.28	48.55	27.08	27.41	22.10	22.68
4	10.61	11.44	20.50	20.36	24.94	24.65	3.70	3.75	48.39	48.55	27.34	28.16	22.62	22.25
	10.73	11.27	20.77	20.11	24.94	24.74	4.04	3.73	48.38	48.09	27.21	27.60	22.74	22.16
5	26.04	20.69	16.37	37.58	41.82	32.58	8.78	7.89	59.02	48.47	32.06	31.13	25.88	27.81
	25.58	22.91	15.50	24.02	39.65	30.97	11.74	4.74	82.20	48.12	32.03	30.57	29.93	27.57
6	10.36	10.68	21.15	20.72	26.21	26.36	3.98	3.60	49.27	48.65	27.69	28.48	23.40	23.21
	10.95	10.67	19.99	21.27	26.18	26.30	3.84	3.70	49.66	49.07	27.86	27.57	24.00	22.88
7	10.80	10.90	19.10	19.40	24.90	25.20	3.30	3.40	48.90	47.50	27.20	26.90	22.20	22.40
	11.20	10.90	19.30	19.50	24.40	25.10	3.50	3.30	48.70	47.90	27.50	27.20	21.70	22.30
8	10.59	10.18	18.94	19.51	24.23	24.37	3.62	3.49	48.11	48.39	27.26	27.22	22.20	22.24
	10.70	10.83	19.16	19.60	24.43	24.26	3.49	3.91	48.53	48.10	27.25	27.53	22.53	22.11
9	10.65	11.12	19.06	18.92	24.95	24.33	1.90	2.26	48.77	49.99	27.25	23.96	21.81	22.84
	9.80	10.81	19.53	19.60	25.05	25.89	1.29	2.53	48.10	52.21	27.15	26.05	21.42	24.19
10	10.86	10.21	21.70	20.59	26.34	26.12	3.94	3.91	48.75	49.11	26.50	27.76	22.24	22.77
	10.24	10.43	21.78	20.75	26.35	26.36	3.82	3.73	48.84	49.49	26.93	27.37	22.59	22.93
11	12.23	11.08	20.00	20.25	24.00	24.86	2.00	2.67	48.00	48.59	28.07	28.02	22.92	23.13
	11.49	10.99	20.00	20.07	24.00	26.04	2.00	2.98	48.00	48.73	28.11	27.77	23.28	23.30
12	10.08	10.49	20.57	20.10	25.28	25.59	3.63	3.60	48.81	48.35	27.82	27.19	22.61	22.42
	10.03	10.76	20.87	20.24	25.19	25.65	3.62	3.70	48.68	47.70	27.45	27.51	22.22	22.33

[a] 10 collaborators performed fat determinations, using **24.005(a)** (1). Collaborators 3 and 10 used **24.005(b)** (1).
[b] Associate Referee.

Precision of duplicate determinations was relatively constant for different fat contents when expressed in terms of standard deviation (absolute) rather than coefficient of variation (relative) (Fig. 1). The relative variation (coefficient of variation = 100 standard deviation/mean fat %) decreased smoothly from about 5% for low fat content to about 0.8% for about 25% fat and then appeared to remain at 0.8% for fat content up to about 48%.

Table 2. Laboratories with data rejected by outlier tests

	Lab. No.			
	Between dupl.			
Sample	Day 1	Day 2	Between days	Among labs.
B-1	none	5	5	5
B-2	none	5	5	5
B-3	5	5,9	5	5
P-1	5	5	5	5
P-2	5	none	5	5
Fr	none	9	9	5
Bol	5	none	9	5

Based on the tests of homogeneity of variance and the graphical treatment of coefficient of variation, the maximum range for duplicate values acceptable at a 95% confidence level (Table 3) was calculated to be 0.7% fat for a sample containing up to 27.8% fat and 1.2% for a sample containing 48.6% fat.

Analysis of Variance from Single Sample Data Sets

Collaborative duplicate determinations were averaged so that the day averages were used to calculate the 3 sums of squares for each sample (Table 4). It can be visualized that the between-laboratories sum of squares is the same as would be derived from $\bar{X} - \bar{\bar{X}}$ differences, that for between-days from $X - \bar{X}$ differences, and that for total from $X - \bar{\bar{X}}$, where X represents a laboratory's single day average, $\bar{X}$ is a laboratory's 2-day average, and $\bar{\bar{X}}$ is the overall average of all laboratories. A between-laboratories mean square value listed in the table estimated the compound variance of within-laboratory var-

Table 3. Statistics of precision of duplicate determinations on samples individually and pooled

Sample	Sample mean, fat %	No. of dupl. pairs[a]	Variance	Std dev., ± % fat	Max. range for dupl.,[b] 95% conf. level, ± % fat
B-1	11.49	23	0.0744	0.27	0.80
B-2	19.99	23	0.0929	0.30	0.89
B-3	25.19	21	0.0455	0.21	0.63
P-1	3.40	22	0.0304	0.17	0.51
P-2	48.63	23	0.1578	0.40	1.16
Fr	27.77	23	0.0640	0.25	0.74
Bol	22.74	23	0.0943	0.31	0.90
All samples	22.74	158	0.0807	0.28	0.79
All samples except P-2	18.43	135	0.0675	0.26	0.73

[a] The 10 duplicate pairs of suspect data listed in the day 1 and day 2 columns of Table 2 were excluded.
[b] Calculated by multiplying standard deviation by a factor (7, p. 520) which consisted of $(\sqrt{2})$ $(t_{0.05})$ for the corresponding degrees of freedom.

iations and twice (for days) the actual between-laboratory component. The F ratios shown in the table were used to test the significance of the mean squares for between laboratories and between days. The F-test ($P = 0.05$) indicated the presence of a significant difference between the laboratory means of 4 of the samples. It would not have been unusual if the test on all 7 had indicated significance, because between-laboratory variations are generally larger than within-laboratory variations. This test for significant difference was confirmed by the F ratios of the components of variance for 3 of the same 4 samples (Table 5). The table also shows that F ratios were not significant for 3 of the samples, indicating that means of determinations differed about the same between and within laboratories. For one of the samples, P-2, the F ratio was significant when the variances were tested inversely and indicated that at the 48% fat level repeatability was a greater source of variation than reproducibility in determining fat content.

Pooled values of the within-laboratory, between-laboratory, and summed (within- and between-laboratory) sample variances (Table 5) were obtained by weighting for degrees of freedom and testing for homogeneity of variance by Bartlett's chi-square test and $F_{max.}$-test. Within-laboratory sample variances were indicated to be homogeneous for 6 of the samples after excluding the higher variance of the high fat sample, P-2. Between-laboratory sample variances were indicated to be homogeneous in 2 subgroups,

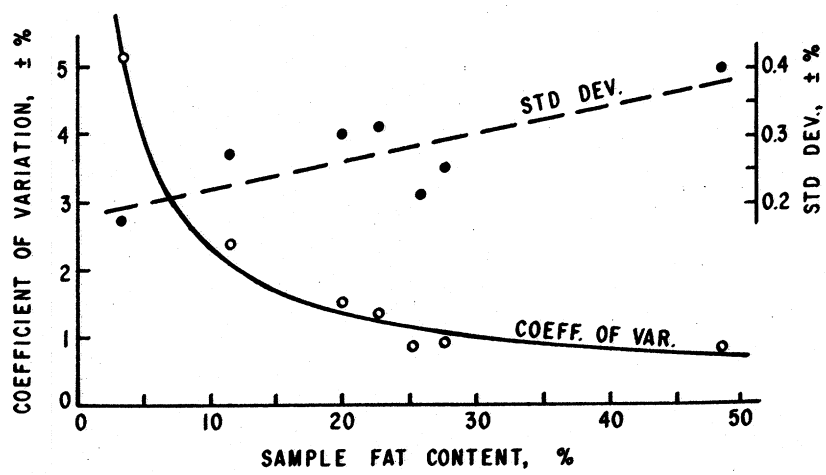

FIG. 1—Relative and absolute deviation between duplicates of collaborative fat determinations.

Table 4. Analysis of variance of averages of duplicates on individual samples on different days

Source of variance	Sum of squares	Degrees of freedom	Mean square	*F* ratio
		Sample B-1		
Between labs.	2.3146	10	0.2315	2.21
Between days (within labs.)	1.1538	11	0.1049	
Total	3.4684	21		
		Sample B-2		
Between labs.	8.0023	10	0.8002	7.75[a]
Between days (within labs.)	1.1363	11	0.1033	
Total	9.1386	21		
		Sample B-3		
Between labs.	7.9894	10	0.7989	4.79[a]
Between days (within labs.)	1.8364	11	0.1669	
Total	9.8258	21		
		Sample P-1		
Between labs.	7.4640	10	0.7464	11.37[a]
Between days (within labs.)	0.7224	11	0.0657	
Total	8.1864	21		
		Sample P-2		
Between labs.	5.2949	10	0.5295	1.14
Between days (within labs.)	5.0935	11	0.4630	
Total	10.3884	21		
		Sample Fr[b]		
Between labs.	1.8064	9	0.2007	2.62
Between days (within labs.)	0.7663	10	0.0766	
Total	2.5727	19		
		Sample Bol[b]		
Between labs.	4.2446	9	0.4716	7.56[a]
Between days (within labs.)	0.6237	10	0.0624	
Total	4.8683	19		

[a] Exceeded tabular *F* ratio (P = 0.05), indicating variations between means obtained by laboratories differed significantly compared with variations within laboratory means or variances.
[b] Data from Collaborator 9 were excluded.

divided randomly rather than according to fat content or meat type: a lower variance group composed of Samples B-1, P-2, and Fr, and a higher variance group composed of Samples B-2, B-3, P-1, and Bol. Summed sample variances were indicated to be homogeneous for 6 of the samples after excluding the lower variance of Sample Fr. The $F_{max.}$-test confirmed the chi-square tests except for the summed variance group, in which case homogeneity was indicated without excluding Sample Fr.

The components of precision which summarize the statistics of this analysis of variance (Table 6) are expressed in terms of standard deviation and relative deviation. Collaborative means of fat content shown in the first column differ in most cases from those given in Table 3 as a result of having excluded different outliers from the different statistical treatments. From the pooled results it was concluded that the repeatability of the method on meat and meat products was 0.4% for fat contents up to 48.6% and, more specifically, 0.3% for fat contents up to 27% and 0.7% for fat contents of about 48%; reproducibility of the method was 0.6% for meat samples containing up to 48.6% fat and 0.45% for processed products such as frankfurter and bologna containing 22–27% fat. The maximum

Table 5. Estimates of precision from analysis of variance on individual samples

Sample	Variance: s^2_a (within-lab.)	Variance: s^2_b (between-lab.)	Variance: Sum, $s^2_a + s^2_b$	F ratio: s^2_b/s^2_a	F ratio: s^2_a/s^2_b
B-1	0.1049	0.0633	0.1682	—	1.66
B-2	0.1033	0.3485	0.4518	3.37[a]	—
B-3	0.1669	0.3160	0.4829	1.89	—
P-1	0.0657	0.3404	0.4060	5.18[a]	—
P-2	0.4630	0.0332	0.4963	—	13.95[b]
Fr	0.0766	0.0620	0.1387	—	1.22
Bol	0.0624	0.2046	0.2670	3.28[a]	—
All samples	0.1512	0.1972	0.3484	1.30	—
Homogeneous variance	0.0975[c]	0.0525[d]	0.3805[e]	—	1.86
	—	0.3047[f]	—	3.13[a]	—

[a] Same indication of significance as was stated in footnote *b*, Table 4.
[b] Exceeded tabular *F* ratio (P = 0.05) indicating variations within laboratory means differed significantly cc pared with variations between laboratory means or variations.
[c] Pooled value for 6 samples, excluding highest variance (P-2).
[d] Pooled value for 3 samples with low variance: B-1, P-2, and Fr.
[e] Pooled value for 6 samples, excluding lowest variance (Fr).
[f] Pooled value for 4 samples with higher variance: B-2, B-3, P-1, and Fr.

Table 6. Summary of statistics of analytical variations on samples individually and pooled

Sample	Sample mean, fat %	Std dev., ± % fat: Repeat-ability, s_a	Std dev., ± % fat: Be-tween-lab., s_b	Std dev., ± % fat: Repro-duci-bility, s_{a+b}	Rel. dev., %: CV_a	Rel. dev., %: CV_b	Rel. dev., %: CV_{a+b}	Max. range fo detns,[a] 95% conf. level, ± %: Repeat-ability	Max. range fo detns,[a] 95% conf. level, ± %: Repr duci bilit
B-1	10.84	0.32	0.25	0.41	2.99	2.32	3.78	0.94	1.20
B-2	20.18	0.32	0.59	0.67	1.59	2.93	3.33	0.94	1.96
B-3	25.19	0.41	0.56	0.69	1.62	2.23	2.76	1.21	2.03
P-1	3.40	0.26	0.58	0.64	7.53	17.15	18.73	0.76	1.88
P-2	48.64	0.68	0.18	0.70	1.40	0.37	1.45	1.99	2.05
Fr	27.43	0.28	0.25	0.37	1.01	0.90	1.36	0.82	1.08
Bol	22.50	0.25	0.45	0.52	1.11	2.01	2.30	0.73	1.52
All samples	22.60	0.39	0.44	0.59	1.73	1.95	2.61	1.10	1.66
Homogeneous groups	—	0.31[b]	0.23[c]	0.62[d]	1.70	0.79	2.85	0.87	1.75
	—	—	0.55[e]	—	—	3.09	—	—	—

[a] Calculated as noted in footnote *b*, Table 3.
[b] Calculated from pooled variance of 6 samples with mean fat content of 18.26%, excluding P-2.
[c] Calculated from pooled variance of Samples B-1, P-2, and Fr with mean fat content of 28.97%.
[d] Calculated from pooled variance of 6 samples with mean fat content of 21.79%, excluding Fr.
[e] Calculated from pooled variance of Samples B-2, B-3, P-1, and Bol with mean fat content of 17.82%.

ranges for determinations with the method acceptable at a 95% confidence level are shown in the last 2 columns of the table.

Analysis of Variance of Data Sets of All Samples Combined into One Block

Collaborative duplicate determinations were averaged and, from the day averages, 5 sums of squares (Table 7) were calculated. Results in the table are arranged in 2 sections that either included or excluded determinations from Collaborator 9, since the format of this analysis of variance required a complete block of data wi out gaps that would have occurred if several o liers had been eliminated. The objective was express the consensus of method variations the average of results of the 11- and 10-labo tory treatments. *F* ratios shown in the table w significant (P = 0.05) when tested and indicat from the between-laboratories term, that a c sistent laboratory bias existed and, from interaction term, that between-laboratory va tion was greater than within-laboratory va tion. Components of variance calculated fr

Table 7. Analysis of variance between laboratories, samples, and days

Source of variance	Results from 11 labs. Sum of squares	Degrees of freedom	Mean square	*F* ratio	Results from 10 labs.[a] Sum of squares	Degrees of freedom	Mean square	*F* ratio
Between labs.	11.9178	10	1.1918	2.52[b]	9.6367	9	1.0708	3.14[b]
Between samples	63.7568	6	—	—	65.6269	6	—	—
Lab.-sample interaction	28.4151	60	0.4736	2.35[c]	18.4057	54	0.3408	3.32[c]
Between days (within labs.)	15.5354	77	0.2018	—	7.1939	70	0.1027	—
Total	119.6250	153	—	—	100.8633	139	—	—

[a] Calculations exclude data from Collaborator 9.
[b] Calculated by dividing between-laboratory mean square by that of laboratory-sample interaction; value exceeded tabular value (P = 0.05) and indicated that between-laboratory variations were significantly greater than interaction, e.g., variations caused by different ways of handling samples.
[c] Calculated by dividing laboratory-sample interaction mean square by that of between-days; value exceeded tabular value (P = 0.05) and indicated that random differences within laboratories and samples were significantly greater than between days (replication).

the mean squares of this table, the standard deviations calculated from the variances, and the average standard deviations are summarized in Table 8. The averaged values cited in the bottom row of the table compared favorably with the standard deviations from pooled variances obtained in the preceding analysis of variance of individual samples: 0.39% fat within-laboratory was identical to its counterpart; from the sum of the laboratory-sample interaction and the between-laboratory variances, a compound between-laboratory standard deviation of 0.43% from the 11-laboratory treatment and 0.41% from the 10-laboratory treatment yielded an average 0.42% which closely approximated its counterpart, 0.44%; 0.57% for the total variation, reproducibility, closely approximated its counterpart, 0.59%.

Table 8. Estimates of precision from analysis of variance between laboratories, samples, and days

Var. anal. data block	Component of precision: Within-lab.	Lab.-sample interaction	Between-lab.	Total of components
	Variance			
11 labs.	0.2018	0.1359	0.0513	0.3890
10 labs.	0.1027	0.1191	0.0521	0.2739
	Standard Deviation, ±% Fat			
11 labs.	0.45	0.37	0.23	0.62
10 labs.	0.32	0.35	0.23	0.52
Av. of 11- and 10- lab. data	0.39	0.36	0.23	0.57

Analytical Variations within Single Laboratories

Except for calculations to indicate outlier data early in the statistical treatment, the treatments of duplicate determinations and analysis of variance were concerned with within-sample variations. A rudimentary treatment of the collaborative results on a single-laboratory basis was also performed for comparison with the overall results. The calculated standard deviations and means are shown in Table 9. For standard deviation between duplicate determinations, 7 laboratories demonstrated a precision within 0.23% fat and 8 were within the interlaboratory precision of 0.28% (Table 3). For standard deviation between days, 6 laboratories demonstrated a precision within 0.30% fat and 8 were within the interlaboratory precision of 0.39% (Tables 6 and 8). For standard deviation of either individual determinations or average of duplicates each day, both relative to sample means listed in Table 3, 5 laboratories were within 0.6% fat which value corresponds to the interlaboratory reproducibility precision of 0.6% (Tables 6 and 8). The difference between each laboratory's mean for all samples and the overall interlaboratory mean (22.74%, Table 3) indicated that 11 of the laboratories determined fat within a range of agreement of +0.39 to −0.55% fat which is within 0.6%, the reproducibility determined for the method.

Comparison of Total and Crude Fat Determinations

One of the participants in this study submitted an extra set of data consisting of deter-

Table 9. Estimates of variations of precision and means within each laboratory

Lab.	Std dev., ± % fat: Between dupl.	Between day avs	Between individual detns and overall sample mean	Between day av. and overall sample mean	Fat, %: Lab. mean of all sample detns	Mean diff., lab. and overall
1	0.17	0.18	0.35	0.33	22.76	+0.01
2	0.24	0.29	0.67	0.66	22.38	−0.37
3	0.20	0.19	0.44	0.42	22.61	−0.14
4	0.18	0.30	0.45	0.44	22.63	−0.11
5	5.24	7.71	10.68	10.18	30.06	+7.31
6	0.36	0.28	0.77	0.74	23.13	+0.39
7	0.20	0.36	0.57	0.57	22.31	−0.44
8	0.21	0.16	0.66	0.65	22.24	−0.50
9	0.76	1.09	1.42	1.33	22.19	−0.55
10	0.21	0.41	0.89	0.89	22.94	+0.20
11	0.29	0.53	0.64	0.62	22.59	−0.15
12	0.19	0.31	0.59	0.59	22.59	−0.16
Av.[a]	0.23	0.30	0.60	0.59	22.58	−0.17

[a] The 4 averages of standard deviations were calculated from the above column values from 10 laboratories, excluding Collaborators 5 and 9. The average of laboratory means was calculated from the above column values from 11 laboratories, excluding Collaborator 5. The mean differences were calculated relative to the overall sample mean shown in Table 3, 22.74% fat.

minations of total fat in the distributed samples. The determinations provided a demonstration of the use of paired analyses with the AOAC method as a comparison standard in evaluating results with another method and, additionally, in this case, the comparison served to confirm the significant difference established elsewhere between crude and total fat determinations. The collaborator performed British Standard **4401**, Part 4, Method A, which requires acid digestion and drying the digest residue prior to solvent extraction and gravimetric determination of fat in the extract. Results with this method were compared with crude fat determinations by both the collaborator and all collaborators (Table 10). From the 2 comparisons, the overall mean difference indicated total fat was 0.4% (1.9% relative) higher than crude fat, the standard deviation of the results, between methods, was of the same order, 0.5 and 0.6% fat, and the calculated t value indicated that total fat determined by British standard method and crude fat by AOAC method were significantly ($P = 0.05$) different.

Based on the results of this collaborative study, which indicate the expected agreement between analyses under practical conditions, it is suggested that the precision characteristics determined for AOAC method **24.005(a)** and **(b)** for crude fat in meat and meat products be used as a basis for comparing alternative methods of determining fat content in meat and meat products when a method which may merit standardization is evaluated for accuracy and precision.

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Table 10. Comparison of crude fat determinations by AOAC method 24.005(a) or (b) and total fat determinations by British Standard method 4401-4-A for the collaborative samples

		Mean fat,[a] %				
		24.005(a) or (b)		4401-4-A,	Diff.	
Sample	Day	Coll. 12	11 colls	Coll. 12	3−1[b]	3−2[c]
B-1	1	10.06	10.82	11.31	1.25	0.49
	2	10.63	10.87	11.13	0.50	0.26
B-2	1	20.72	20.24	21.19	0.47	0.95
	2	20.17	20.12	19.88	−0.29	−0.24
B-3	1	25.24	25.02	25.71	0.47	0.69
	2	25.62	25.36	26.08	0.46	0.72
P-1	1	3.63	3.52[d]	4.02	0.39	0.50
	2	3.65	3.57[d]	3.95	0.30	0.38
P-2	1	48.75	48.55	47.96	−0.79	−0.59
	2	48.03	48.73	47.94	−0.09	−0.79
Fr	1	27.64	27.35	28.53	0.89	1.18
	2	27.35	27.28	27.92	0.57	0.64
Bol	1	22.42	22.42	23.02	0.60	0.60
	2	22.38	22.60	23.60	1.22	1.00
Overall mean	—	22.59	22.60	23.01	0.42	0.41
Std dev.	—	—	—	—	0.54	0.58
t value	—	—	—	—	2.92[e]	2.66[e]

[a] Determinations are means of duplicates.
[b] Differences were calculated from means in columns 3 and 1.
[c] Differences were calculated from means in columns 3 and 2.
[d] Mean was calculated from results from 10 collaborators (data from Coll. 9 were excepted).
[e] This value exceeds tabular value where $t = 2.16$ for $P = 0.05$ and $df = 13$, indicating a significant difference in fat determined by British Standard and AOAC methods.

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