

FAT ANALYSIS OF MILK POWDER

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Up to the present time the Association of Official Agricultural Chemists has not designated an official method for determining fat in milk powders. The Roese-Gottlieb method, however, is generally accepted as the best method available for this purpose. Probably because of the fact that this Association has not officially adopted a method for determining fat in milk powder, and because of the urgent need for a method which is entirely reliable, numerous modifications of existing methods have been proposed. Some of these modifications have involved an extraction from an acid solution instead of an alkaline solution as is used in the regular Roese-Gottlieb procedure; others have involved modifications of the Babcock test, whereby the method could be adopted for factory use.

Biesterfeldt and Evenson (1) advocate the use of weak acetic acid, claiming this reagent allows extraction of fatty acids which may be present and which are not extracted from an alkaline medium. They claim the Roese-Gottlieb method gives an average of 0.04 per cent too low for condensed milk, and further state that for milk powders, the results may be even lower.

Harding and Parker (2) claim that more fat is extracted from evaporated milk by the use of 25 per cent acetic acid, carbon tetrachloride and petroleum ether than by the reagents used in the regular Roese-Gottlieb method. Biesterfeldt and Evenson (1), however, think that the higher results obtained by this method are due to the use of rubber stoppers in contact with the reagents.

Mohs (3) recommends a preliminary treatment of milk powder with hydrochloric acid and subsequent extraction in the Soxhlet apparatus with ethyl ether. He states that all of the fat is not

¹Former Laboratory staff members participating in these comparative analyses were, A. H. Green and P. A. Bunce.

removed from milk powder by simple extraction with ethyl ether, and that there appears to be a decrease in fat from old powders due to absorption of the fat by the coagulated protein.

Phillipps (4) recommends extraction with trichlorethylene for rapid extraction of the fat.

Redmond (5) has recommended a modification of the Babcock test for factory use, the results from which are claimed to be from 0.1 per cent to 0.2 per cent higher than by any ether extraction method and 3 per cent higher than the regular Babcock method.

Roop (6) recommends the use of dilute sulphuric acid followed by digestion in the steam bath in the place of ammonia as used in the Roesse-Gottlieb method for fat in dried buttermilk.

It seems to be recognized that a simple ether extraction in the Soxhlet apparatus is distinctly inferior to the Roesse-Gottlieb method for liquid milk, evaporated, condensed or powdered milks. The majority of proposed modifications of methods have sought to improve the efficiency and reliability of the Roesse-Gottlieb method. These modifications as well as the simple Soxhlet extraction method have but little recognition at the present time for determining fat in milk powder, although the Soxhlet method is quite frequently used for this purpose.

The inadequacies of a simple ether extraction for determination of butter fat in milk products were no doubt widely recognized after the official adoption by the Association of Official Agricultural Chemists at the 32nd Annual Conference, November 15, 1915, of the Roesse-Gottlieb method for fat in milk and condensed milk, both sweetened and unsweetened. At the same time it was recommended by associate referee Hortvet (7), that a further study be made of this method in the analyses of ice cream, milk powders, malted milks and milk chocolates. This recommendation was, no doubt, stimulated by the relative accuracy and uniformity of results from evaporated and condensed milks. The results reported from a single sample of evaporated milk on which forty-five analyses were made by nineteen different analysts showed a maximum variation of 0.29 per cent. From two samples of sweetened condensed milk

analyzed by the same collaborators and involving twenty-five to twenty-seven determinations on each sample, the maximum variations were 0.21 per cent and 0.17 per cent.

At the Association meeting held November 20, 1917, Hortvet (8) reported results of collaborative work on analyses for fat in dried skimmed milk by the official Roese-Gottlieb method and by an acid extraction modification of the same. The results from the official method reported from a single sample on which twenty-five analyses were made by nine different analysts showed a maximum variation of 0.33 per cent on a powder containing 1.13 per cent fat as an average of all determinations. The results reported from the acid extraction modification showed a maximum variation of 0.65 per cent on the same sample. On the basis of these results it was recommended that further study be made of the Roese-Gottlieb method as applied to dried milk products containing a high as well as a low butter fat content.

COMPARISONS OF RESULTS FROM DIFFERENT METHODS

During our investigational and control work connected with desiccated milk, it has been desirable to make comparisons of different methods used for determining the butter fat content of this product. The results reported herein were obtained during a period extending over two years from dried milk made by the Just double roller process. The great majority of results, unless otherwise designated, were made with the Mojonnier apparatus which is considered a practical adaptation of the chemical principles of the Roese-Gottlieb method for routine factory analysis; those determinations made by this procedure will be hereinafter designated as the Roese-Gottlieb (Mojonnier) method.

VARIATIONS IN RESULTS OBTAINED BY THE ROESE-GOTTLIEB (MOJONNIER) METHOD

One of the first studies to be made in connection with this work on reliability of methods was for the purpose of ascertaining normal variations between duplicate determinations made by the Roese-Gottlieb (Mojonnier) method. The duplicate deter-

minations made by different analysts on samples of freshly made milk powder are shown in table 1. Since the two determinations on any one sample were made within short intervals, thereby eliminating the possibility of any discrepancy due to variable moisture content, the results are expressed on the original powder basis and not on the moisture-free basis. From the results shown in this table, the maximum variation between duplicate determinations by the Roese-Gottlieb (Mojonnier) method is 0.26 per cent; the minimum variation is zero; and the average variation is 0.099 per cent.

TABLE 1
Results of duplicate determinations for fat in milk powder, Roese-Gottlieb (Mojonnier) method.

SAMPLE NUMBER	DETERMINATION NUMBER 1, PER CENT FAT	DETERMINATION NUMBER 2, PER CENT FAT	DIFFERENCE, PER CENT FAT
1	23.93	24.19	0.26
2	24.47	24.51	0.04
3	26.55	26.55	0.00
4	31.12	31.12	0.00
5	28.12	28.02	0.10
6	25.68	25.47	0.21
7	27.21	27.39	0.17
8	27.07	26.95	0.12
9	28.33	28.29	0.04
10	30.09	30.05	0.04
11	26.21	26.32	0.11
12	19.75	19.84	0.09
13	53.51	53.65	0.14
14	55.00	54.93	0.07
Average.....			0.099

VARIATIONS IN RESULTS OBTAINED BY THE ROESE-GOTTLIEB
(MOJONNIER) METHOD FROM FRESH AND OLD MILK POWDER

In order to determine whether or not the ageing of milk powder introduced a factor which would tend to cause discrepancies in the results from the Roese-Gottlieb (Mojonnier) method, several samples of powder made from part skimmed milk and from whole milk were analyzed while fresh and after various storage periods;

moisture determinations were made on each sample at the time of analysis and the fat percentages converted to the moisture-

TABLE 2

Results from Roese-Gottlieb (Mojonnier) method on part skimmed milk powder while fresh and at age of two months

SAMPLE NUMBER	FRESH	AFTER TWO MONTHS
	<i>per cent fat</i>	<i>per cent fat</i>
1	12.52	12.23
2	13.07	12.73
3	11.37	11.24
4	11.82	11.91
5	11.28	11.23
6	12.47	12.55
7	12.70	12.49
8	12.24	12.54
9	12.58	12.42
10	12.18	12.25
Average.....	12.22	12.15

TABLE 3

Results from Roese-Gottlieb (Mojonnier) method on part skimmed milk powder while fresh and at age of three months

SAMPLE NUMBER	FRESH	AFTER THREE MONTHS
	<i>per cent fat</i>	<i>per cent fat</i>
1A	12.70	12.69
2A	12.16	12.39
3A	12.58	12.60
4A	12.39	12.47
5A	12.27	12.18
6A	12.29	12.35
7A	12.39	12.14
8A	12.46	12.65
9A	12.23	12.34
10A	12.63	12.56
Average.....	12.41	12.46

free basis. The results of these determinations are shown in tables 2 to 5 inclusive.

In tables 2 to 5 inclusive there is opportunity to compare results from two determinations on thirty-five different samples, one

determination in each case being made while the powder was still fresh and the other when the powder was from two to six months old. The maximum variation between two determinations on the same sample is 0.41 per cent; the minimum is 0.01 per cent; and the average is 0.17 per cent. The results of these

TABLE 4

Results from Roesse-Gottlieb (Mojonnier) method on part skimmed milk powder while fresh and at age of six months

SAMPLE NUMBER	FRESH	AFTER SIX MONTHS
	<i>per cent fat</i>	<i>per cent fat</i>
1B	12.82	13.08
2B	13.76	13.48
3B	12.33	12.18
4B	12.14	12.07
5B	13.23	12.84
Average.....	12.85	12.73

TABLE 5

Results from Roesse-Gottlieb (Mojonnier) method on whole milk powder while fresh and at age of four months

SAMPLE NUMBER	FRESH	AFTER FOUR MONTHS
	<i>per cent fat</i>	<i>per cent fat</i>
1	26.72	26.56
2	27.34	27.32
3	27.76	27.41
4	27.09	27.16
5	27.44	27.32
6	27.41	27.23
7	26.31	26.51
8	27.93	28.25
9	27.48	27.81
10	27.79	27.38
Average.....	27.32	27.29

determinations show wider variations than was found in the samples recorded in table 1. This is undoubtedly due, to some extent at any rate, to the fact that the results from these four series containing thirty-five samples are all expressed on the moisture-free basis, the wider variations being explained in part,

as the result of analytical discrepancies in the moisture determinations.

In considering the question of the extraction of less fat from old powder than from fresh, there were nineteen samples from which less fat was extracted after ageing; the average difference was 0.183 per cent. The remaining sixteen samples gave higher results after ageing; the average difference was 0.154 per cent. Although the average difference between fresh and old samples shows that 0.029 per cent more fat was extracted from the former, this is not considered as conclusive evidence that this method of analysis is less reliable for old powders.

ROESE-GOTTLIEB (MOJONNIER) AND SIMPLE ETHER EXTRACTION METHODS COMPARED

Although the simple ether extraction method with the Soxhlet apparatus is not generally recognized as an efficient method for the determination of fat in milk powder, it is, however, frequently used in commercial laboratories for the purpose of determining the fat content of different consignments of this and other food products carrying a guaranteed fat content. In order to obtain comparative data between the two methods, parallel determinations were made on powder from part skimmed milk and from whole milk when the powder was fresh and after a storage period of four months. In the ether extraction method the extraction was carried out for seven hours. These results which are shown in tables 6 and 7 are all expressed on the moisture-free basis.

From the four different comparisons between the Roese-Gottlieb (Mojonnier) and ether extraction methods which are possible in tables 6 and 7, there was on an average 0.22 per cent more fat extracted by the former method. Each group of parallel determinations shows a similar relationship between the two methods.

Again considering the variations in results between individual determinations on the same sample as shown by both of these methods, it was found that from the Roese-Gottlieb (Mojonnier) method there was a maximum variation of 0.49 per cent; a minimum of 0.01 per cent; and an average of 0.143 per cent. From the simple ether extraction method there was a maximum

variation of 1.29 per cent, a minimum of 0.05 per cent and an average of 0.369 per cent. In these comparisons as in those

TABLE 6

Rosse-Gottlieb (Mojonnier) and simple ether extraction methods compared on part skimmed powder, fresh and after four months

SAMPLE NUMBER	FRESH		AFTER FOUR MONTHS	
	Rosse-Gottlieb	Soxhlet	Rosse-Gottlieb	Soxhlet
	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>
1	12.46	12.01	12.49	12.21
2	12.24	12.34	12.64	12.18
3	12.45	12.36	12.94	12.62
4	12.68	12.13	12.60	13.42
5	12.27	12.19	12.18	11.73
6	12.29	11.99	12.35	11.88
7	12.39	11.96	12.45	12.25
8	12.46	11.88	12.65	12.96
9	12.23	12.06	12.34	12.11
10	12.63	12.33	12.56	12.27
Average.....	12.41	12.12	12.52	12.36

TABLE 7

Rosse-Gottlieb (Mojonnier) and simple ether extraction methods compared on whole milk powder, fresh and after four months

SAMPLE NUMBER	FRESH		AFTER FOUR MONTHS	
	Rosse-Gottlieb	Soxhlet	Rosse-Gottlieb	Soxhlet
	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>	<i>per cent fat</i>
1	26.98	26.33	26.92	26.42
2	26.81	26.75	26.56	26.45
3	27.30	27.09	27.32	27.45
4	27.41	27.43	27.28	27.97
5	27.06	27.21	27.16	26.75
6	27.51	27.39	27.32	26.91
7	27.14	27.49	27.23	26.96
8	26.77	25.73	26.51	26.11
9	26.92	26.28	26.77	26.20
10	27.37	27.29	27.38	27.14
Average.....	27.12	26.89	27.04	26.83

already recorded there is no conclusive evidence that less fat is extracted from old powder than from fresh, regardless of the method.

EXTRACTION FROM AN ACID MEDIUM

Since it has been claimed that more fat is extracted from an acid solution than from an alkaline solution, comparisons were made between one of these methods and the Roesse-Gottlieb (Mojonnier) method. The method used was the one recommended by Roop (6) for dried buttermilk in which powder is digested in dilute sulphuric acid (1:9) in the steam bath for thirty

TABLE 8
Roesse-Gottlieb (Mojonnier) and alkaline and acid extraction methods compared

SAMPLE NUMBER	ACID EXTRACTION	ALKALINE EXTRACTION
	<i>per cent fat</i>	<i>per cent fat</i>
1	27.21	27.85
2	27.47	26.72
3	27.08	26.40
4	26.10	25.73
5	26.51	26.37
6	26.15	25.56
7	26.15	25.46
8	25.49	25.26
9	25.69	25.75
10	26.29	25.70
11	27.46	25.91
12	27.01	26.26
13	26.18	25.57
14	25.96	25.71
15	25.98	25.33
16	25.75	26.05
17	24.52	25.09
18	25.51	26.14
19	26.12	26.01
Average.....	26.24	25.94

minutes prior to extraction. This method as applied to the Mojonnier manipulation differs only in the fact that in the latter method ammonium hydroxide is used to digest the casein, whereas digestion is accomplished by weak acid and heat in the former method. The results obtained from these two methods are shown in table 8. The figures show that out of 19 comparisons, 14 are higher from the acid extraction method, and that the average difference is 0.30 per cent higher from this latter method.

**ROESE-GOTTLIEB (MOJONNIER) AND REDMOND METHODS
COMPARED**

The results from the Redmond method, in which a given weight of powder is tested by the Babcock centrifugal method after a preliminary digestion with dilute sulphuric acid, have been compared with the Roese-Gottlieb (Mojonnier) method. From the results of these comparisons which are shown in table 9, it will be noted that the Redmond method does not compare in accuracy with the Roese-Gottlieb (Mojonnier) method. It would seem that this method can only be used as a rough control

TABLE 9
Roese-Gottlieb (Mojonnier) and Redmond methods compared.

SAMPLE NUMBER	REDMOND METHOD	ROESE-GOTTLIEB (MOJONNIER) METHOD
	<i>per cent fat</i>	<i>per cent fat</i>
1	33.30	28.77
2	28.08	28.55
3	28.08	27.40
4	28.08	27.24
5	27.36	26.85
6	56.88	55.41
7	55.62	54.02
8	56.39	54.08
9	55.98	52.81

method and even under these circumstances it appears to be of little value for close checking purposes. It is believed that the multiplication factor which must be used to obtain the final result is largely responsible for the discrepancies. The Redmond method results shown in table 9 are the averages of duplicate determinations. The maximum variation between duplicates of any one sample was 1.8 per cent.

SUMMARY

A review of the results recorded herein seem to warrant the following conclusions:

1. The normal variations in results from the same sample of milk powder analyzed by the Roesse-Gottlieb (Mojonnier) method should not be over 0.15 per cent when results are expressed on the original powder basis. If results are expressed on the moisture-free basis, the variation between duplicate samples may be increased due to discrepancies in the moisture determinations.

2. There is no positive evidence of less fat being extracted from old milk powder than fresh powder when both samples are expressed on the moisture-free basis. If results are expressed on the original powder basis, there is an apparent decrease in fat content of old powders due to the fact that they may have absorbed moisture.

3. The simple ether extraction method gives results from the double roller process powder which are on an average about 0.25 per cent lower than the Roesse-Gottlieb (Mojonnier) method. Duplicate results from the former method are also subject to wider variations than those from the latter method. From sixty-nine comparisons of duplicate determinations by the Roesse-Gottlieb (Mojonnier) method there is an average variation of 0.147 per cent; a maximum of 0.49 per cent and a minimum of zero. From the ether extraction method from twenty comparisons there is an average variation of 0.369 per cent; a maximum of 1.29 per cent; and a minimum of .05 per cent.

4. On the basis of results presented herein, the Redmond modification of the Babcock centrifugal method is very unreliable. The wide variations between the two methods is undoubtedly accounted for to some extent by the large multiplication factor required for converting the test bottle reading to percentage fat in the powder.

5. The modified Roesse-Gottlieb procedure from which extraction is made from an acid medium instead of an alkaline medium gave higher results in the majority of cases than did the regular extraction from an alkaline medium. The full significance of these higher results cannot be stated however, without further investigation as to the variability of numerous results from the same sample of powder.

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