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Variable Factors in the Determination of Fat and Moisture in Cheddar Cheese

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VARIABLE FACTORS IN THE DETERMINATION
OF FAT AND MOISTURE IN CHEDDAR CHEESE

by

William C. Winder

THESIS

submitted in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE

in

Dairy Manufacturing

in

THE GRADUATE SCHOOL

Utah State Agricultural College *

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I wish to acknowledge the help and cooperation of the eight laboratories participating in this research problem.

William C. Winder

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INTRODUCTION

The rapid growth of the dairy industry which began in the latter half of the nineteenth century necessitated the development of chemical tests for determining the composition of dairy products. Chemists devoted much study and labor in devising methods which would be both accurate and rapid. As a result of these researches a large number of tests were developed. Many of the methods possessed considerable merit and a few were so satisfactory that the rest did not come into general use. The basic principles of all the tests now in use were discovered between 1880 and 1890.

The Babcock method* for the determination of fat, first published in 1890, has gained world wide recognition. It was devised originally as a test for the fat in milk, but was soon adapted to other dairy products, including cheese.

Roese* published his method for the determination of fat in 1888. The principle was based upon the solubility of fats in ether.

*Babcock, S.M. a new method for the estimation of fat in milk especially adapted to creameries and cheese factories. Wis. Agr. Expt. Sta. Bul. 24. 1890.

*Roese, Bruno. Analysis of milk: fat determinations Zeitschrift fur angewandte chemie. Abst. in Jour. Chem.Soc., 54: 1135, 1888.

Gottlieb* in 1892, modified the Roesse method by changing the quantity of reagents used. The method is now known as the Roesse-Gottlieb method, and may be used for the determination of fat in all dairy products including cheese.

In 1917, J. J. Mojonnier* improved upon the Roesse-Gottlieb method by inventing a special ether extraction flask and apparatus which reduced the time of the test from three hours to thirty minutes. The Roesse-Gottlieb test using the Mojonnier ether extraction flask is the test now advocated by the Association of Official Agricultural Chemists (A.O.A.C.) (2) for the determination of fat in dairy products.

Along with the fat test, tests for total solids or moisture were developed. The moisture test and the fat test are the most important determinations made on dairy products. The earliest methods of total solids analysis involved the use of mathematical formulas based upon the butterfat test and the specific gravity. Of course, this method could not be applied to solid dairy products. For accurate determinations various gravimetric methods were soon devised. In all

*Gottlieb, E. Estimation of fat in milk. *Milkerei Zeitung* 1892, II Landw. Versuchs. - Stat. 40: 1. Abst. in *Jour. Chem. Soc.* 62: 549, 1893.

*Mojonnier T. and Troy, H.C. *The technical control of dairy products.* Chicago, 1st Ed. 1922.

cases a weighed quantity of the dairy product is dried to constant weight at about the temperature of boiling water. The better known methods include the A.O.A.C. method (2), the Mojonnier method (4), the Brabender method (3), and the method recommended by the American Dairy Science Association (7).

Today, the Babcock method for the fat analysis of cheese (1) (7) is used in the majority of laboratories because of the speed with which it is run. However, the method has very definite limitations in accuracy. An estimation must be made for a reading in the first decimal place. For more accurate work the Roese-Gottlieb method or its Mojonnier modification is used. Either method will check within 0.03% on duplicate samples. The mere fact that a laboratory is using the Roese-Gottlieb method, however, is no guarantee of accurate results. The operator of this test must use quantitative technique and must be absolutely sure of the purity of the reagents employed.

Difficulties are also met in the moisture or total solids analysis of cheese using the various techniques and pieces of apparatus available today. There are so many different types of drying ovens in use that it is difficult to find all of the techniques used. A brief survey shows that drying temperatures vary from 80° C to over 150° C, some using vacuum, some not. The size of sample varies from 0.5 gram to 10 grams and weighing procedures vary with the operator. By far the most common method used, utilizes an

electric oven at about 100° C without vacuum, and a sample of about two grams. This procedure would follow A.O.A.C. (2) if vacuum were used in the drying process. It is evident that here, there is a definite need for correlation of the various methods employed.

The need for accuracy in such tests is particularly felt today because of the more precise methods used in the manufacture of cheese and because of controls on composition imposed by federal and state authority.

The U.S.D.A. (6) has set a standard of 39% maximum moisture and a minimum of 50% fat on a dry matter basis for cheddar cheese. Forty-six of the forty-eight states have adopted similar regulations. To meet these requirements processors have developed more precise methods of manufacture based on the analysis of the finished product. Meeting these requirements is not the only concern of the manufacturer. The economics of the situation tells him that fat in excess of the requirements does not improve the cheese, but merely wastes the most expensive ingredient. Similarly, he knows that a lower moisture cheese is usually a higher quality cheese.

With these problems confronting him, the cheese maker relies heavily upon the analysis report of his own laboratory, of his buyer's laboratory and of state and government laboratories.

In the fall of 1946, following a series of reports from various cheese factories that there seemed to be a wide

discrepancy in the results of cheese analysis made by commercial, state and government laboratories, a series of preliminary tests were made by the Utah State Agricultural College dairy products laboratory with the cooperation of six laboratories in Utah, Idaho and California. The results of these tests tentatively confirmed the earlier report. It is evident that we need to know a great deal more about this problem.

This study was instituted as an attempt to determine the extent of discrepancy in cheddar cheese analysis by nine commercial, state and government laboratories, and the reason for it. A further goal was the improvement of testing procedure so that comparable results could be obtained from all laboratories.

REVIEW OF LITERATURE

This particular problem arises from an immediate need for more precise methods of analysis. The problem is current; a progressive industry is having growing pains which the chemist must relieve.

Only one reference and that unpublished, mentions this problem. Price (5), in the spring of 1947, announced that this was a national problem, not just a local one. The same difficulty has been met by the National Cheese Institute and research started to solve it. The attack has been upon entirely different lines, however, with the emphasis placed upon the sampling of the cheese. None of their work to date has been published.

The problem as met here, bypasses the sampling and concentrates on actual analytical procedure. Thus the work does not overlap but supplements that in progress.

PROCEDURE

In an effort to determine the variation in testing among various laboratories, a group of eight laboratories representing commercial, state and government interests in this area were asked to join in a cooperative testing program. Those included were the following:

Cache Valley Dairy Association

Smithfield, Utah

Brooklawn Creamery

Salt Lake City, Utah

Kraft Cheese Company

Pocatello, Idaho

Nelson-Ricks Creamery Company

Salt Lake City, Utah

U.S.D.A. Dairy Products Laboratory

San Francisco, California

State Chemist

Salt Lake City, Utah

Bacto-Chemical Dairy and Food Laboratory

Salt Lake City, Utah

Nelson-Ricks Creamery Company

Rexburg, Idaho

To protect the anonymity of the laboratories, this list is not in the same order as it appears later in the text.

Samples of cheddar cheese were prepared for analysis by grinding about four pounds of trimmed cheese in a small

power grinder. The ground cheese was then mixed thoroughly and placed in screw-cap sample jars as rapidly as possible to avoid loss of moisture due to evaporation. The jars were filled full to minimize evaporation in the jars and sealed with scotch tape. The jars were then dipped in wax to make them relatively airtight. After proper wrapping the sample jars were shipped by parcel post to the cooperating laboratories.

Nine jars were prepared on each sample of cheese, eight of which were shipped out. The ninth sample was kept at the U.S.A.C. laboratory under refrigeration for 48 hours to simulate delivery time to the other laboratories and was then analysed. Analysis by the eight cooperating laboratories was conducted using the procedure currently in use in that particular laboratory. Results were mailed to the U.S. A.C. dairy department and the results tabulated.

Analysis in the U.S.A.C. laboratory was conducted using various techniques to duplicate as nearly as possible the various methods used in commercial, state and government laboratories. Moisture analysis was made by the A.O.A.C. method (2), the Brabender method (3) using a drying time of fifty minutes at 120° C. and the Mojonnier method (3). Fat was analysed by the Mojonnier ether extraction method (3) and by the Babcock method (7). All samples were run in duplicate. Ten different samples were analysed by each laboratory and the results compared.

Upon completion of analysis of the ten samples and the data compilation, a complete report of the results of the analysis was sent to each participating laboratory. From these data, the laboratory operator could tell how his results deviated from the others. It was fairly obvious how accurately a laboratory was testing.

At this point the procedure of analysis recommended by the subcommittee for the analysis of cheese of the American Dairy Science Association was sent to each laboratory, not as a set pattern for testing, but as a reference for improved technique. Each laboratory also reported in detail the technique employed by them.

A new series of ten samples was then run by each laboratory in an effort to eliminate the discrepancies of the first series. These data were then compared with the first series and changes or improvements noted.

Procedures employed by each cooperating laboratory will be found in the appendix.

RESULTS

In analysing the data from the various methods of analysis employed in the U.S.A.C. laboratory, it was noted that the average difference between various methods did not exceed 0.3%. This figure was taken as a limit for the range within which the results from the cooperating laboratories should fall. Any test within 0.3% of the U.S.A.C. test was thus considered sufficiently accurate.

The complete results of the analysis of samples 1-10 appear in table 1. The results of the analysis of samples 11-20 appear in table 2. Where laboratories reported separate results of replicate tests, results are given as average. Inasmuch as no one of the three moisture tests employed at the U.S.A.C. laboratory could be considered superior to any other, an average of the three was taken. The Mojonnier fat test is generally considered more accurate than the Babcock, therefore the former was used as the U.S.A.C. average fat test.

It may be noted from tables 1 and 2 that the Brabender test in the majority of cases is the center between extremes of A.O.A.C. and Mojonnier. However, there is fairly good correlation among the three tests. It is a little surprising that the Babcock fat test comes as close to Mojonnier as it does inasmuch as an estimation in the first decimal place is required in reading the Babcock test.

TABLE 1.

AVERAGE RESULTS OF ANALYSIS OF CHEDDAR CHEESE -SAMPLES 1-10.

Sample Test	U.S.A.C.					COOPERATING LABORATORIES								
	AOAC	Bra- bender	Mojon- nier	Bab- cock	Mojon- nier	Aver- age	1	2	3	4	5	6	7	8
1 Fat				31.75	31.85	31.85	31.81	29.73	29.75	31.83	32.00	32.00	31.80	31.70
1 Moist.	36.33	36.40	36.48			36.42	36.40	37.14	36.35	35.86	36.53	36.30	36.60	36.40
2 Fat				31.50	31.20	31.20	31.76	29.87	30.00	31.00	32.00	30.00	31.30	*
2 Moist.	37.18	37.57	37.49			37.41	37.60	38.58	37.60	38.03	37.53	38.50	38.10	*
3 Fat				32.00	32.42	32.42	31.86	30.54	30.00	31.00	32.00	32.50	31.70	31.80
3 Moist.	35.46	35.55	35.71			35.57	35.65	36.65	36.10	36.60	35.20	35.60	36.00	35.30
4 Fat				31.50	31.34	31.34	31.56	30.96	30.25	32.00	31.00	32.00	31.00	*
4 Moist.	36.33	36.78	36.44			36.52	33.80	38.09	36.95	36.29	36.20	36.50	36.90	*
5 Fat				30.38	30.44	30.44	29.78	29.17	30.00	30.00	29.00	30.00	30.00	30.40
5 Moist.	39.65	39.77	39.59			39.67	37.25	40.05	40.10	39.18	39.80	39.80	40.00	39.60
6 Fat				32.50	32.55	32.55	31.96	30.11	30.50	32.00	29.50	32.50	32.50	32.00
6 Moist.	35.72	35.91	35.83			35.82	35.50	37.61	36.10	35.73	36.60	35.80	36.10	35.90
7 Fat				32.50	32.53	32.53	31.90	30.11	30.50	33.00	32.00	32.50	31.50	32.20
7 Moist.	35.00	35.40	35.82			35.47	35.20	36.68	36.20	34.80	34.80	35.50	35.80	35.70
8 Fat				32.30	32.46	32.46	31.67	30.71	*	34.00	32.00	32.00	31.70	31.80
8 Moist.	35.41	35.80	35.99			35.73	35.70	37.14	*	35.76	35.20	35.20	35.50	35.30
9 Fat				32.50	32.71	32.71	32.40	30.28	30.75	32.00	32.50	32.00	32.50	32.50
9 Moist.	34.95	35.28	35.27			35.17	34.80	36.55	35.30	35.83	34.60	34.90	34.70	34.90
10 Fat				33.00	33.06	33.06	32.08	30.28	31.75	33.25	32.00	32.00	32.50	32.60
10 Moist.	35.23	35.25	35.36			35.28	35.00	35.80	35.10	35.50	34.60	34.90	35.00	34.80

*Not reported

TABLE 2.

AVERAGE RESULTS OF ANALYSIS OF CHEDDAR CHEESE -SAMPLES 11-20.

Sample Test	AOAC	U.S.A.C.				COOPERATING LABORATORIES								
		Bra- bender	Mojon- nier	Bab- cock	Mojon- nier	Average	1	2	3	4	5	6	7	8
11 Fat				31.75	31.88	31.88	31.80	32.02	31.00	32.00	31.00	32.00	32.10	30.90
11 Moist.	36.35	36.40	36.06			36.27	36.10	36.51	35.90	37.36	36.20	35.80	36.30	36.30
12 Fat				32.30	32.22	32.22	32.22	32.78	31.50	32.25	32.00	32.00	32.10	32.80
12 Moist.	35.04	35.35	35.15			35.18	35.00	35.66	34.75	36.20	35.00	35.20	35.90	34.90
13 Fat				32.00	32.15	32.15	31.81	32.02	31.75	32.00	32.00	32.00	33.00	32.10
13 Moist.	35.36	35.27	35.06			35.23	35.20	35.49	35.50	35.80	34.40	35.20	35.40	35.20
14 Fat				32.00	31.87	31.87	31.71	31.98	31.50	32.00	32.00	32.00	31.20	32.80
14 Moist.	35.43	35.50	35.40			35.44	35.35	35.41	35.25	34.20	33.80	35.00	35.40	35.00
15 Fat				32.00	31.88	31.88	32.20	32.22	31.50	32.50	32.00	32.50	31.70	32.80
15 Moist.	35.11	35.24	35.08			35.14	35.25	35.20	34.95	34.27	34.20	35.30	35.20	35.40
16 Fat				32.00	32.47	32.47	31.78	31.43	31.50	32.00	32.00	32.00	32.40	32.30
16 Moist.	38.18	38.10	38.18			38.15	37.90	38.73	37.45	38.20	37.60	38.00	36.80	38.00
17 Fat				32.30	32.36	32.36	31.96	31.80	32.50	32.00	32.00	32.00	32.60	32.40
17 Moist.	35.25	35.39	35.15			35.28	35.15	35.34	34.75	38.73	34.40	35.00	35.40	35.30
18 Fat				32.50	32.32	32.32	32.02	31.60	32.75	31.00	32.00	32.00	32.70	32.20
18 Moist.	35.04	35.06	35.26			35.12	35.20	35.44	34.60	36.60	34.20	34.90	35.30	35.10
19 Fat				33.50	33.66	33.66	33.03	32.94	31.75	32.50	33.00	33.00	33.00	33.60
19 Moist.	37.21	37.22	37.34			37.29	37.60	37.46	35.85	38.00	37.00	37.20	37.00	37.60
20 Fat				33.30	33.45	33.45	33.85	33.32	31.00	32.50	32.50	33.00	32.60	33.20
20 Moist.	37.55	37.51	37.45			37.50	37.60	37.56	36.60	38.28	37.20	37.40	37.80	37.70

ANALYSIS OF DATA

To study the data from the cooperating laboratories more easily, a graph was drawn of the variation of each test from the U.S.A.C. test. Because either plus or minus variation from the U.S.A.C. test is still variation, the difference was plotted as positive variation. A line graph was then drawn showing the variation for each sample of cheese. Separate graphs were drawn for fat and for moisture. An allowed limit of 0.3%, as previously described is indicated on each graph.

Figures 1-8 are the graphs on fat analysis and figures 9-16 are the graphs on moisture analysis.

Figure 1 shows that the fat testing of laboratory 1 is generally quite good with a slight improvement in series 2 (samples 10-20). The method employed is Mojonnier ether extraction and the results indicate competent personnel.

Figure 2 is an interesting picture of what may happen with a change of procedure. Samples 1-10 were quite consistently out. After seeing the report of the first ten samples, this laboratory changed procedure (see appendix) with the resulting improvement in results on samples 11-15. This is another example of the value of ether extraction in fat analysis. The erratic results of samples 16-19 may possibly be explained by the fact that this laboratory was remodeling. The samples were held for two weeks before analysis. It is possible that during this period there was a slight oiling off of the samples with a resulting loss of fat.

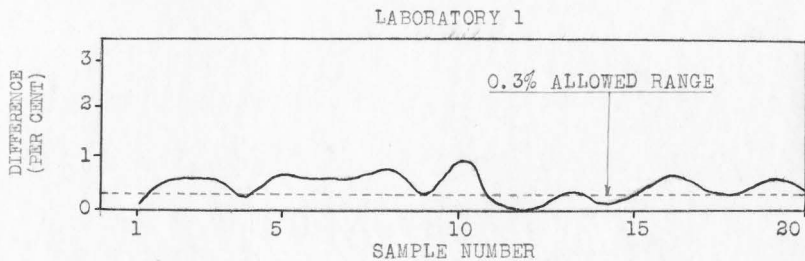


FIGURE 1. PER CENT VARIATION OF LABORATORY 1 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

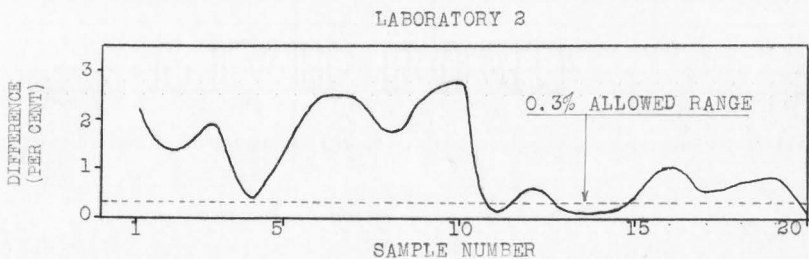


FIGURE 2. PER CENT VARIATION OF LABORATORY 2 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

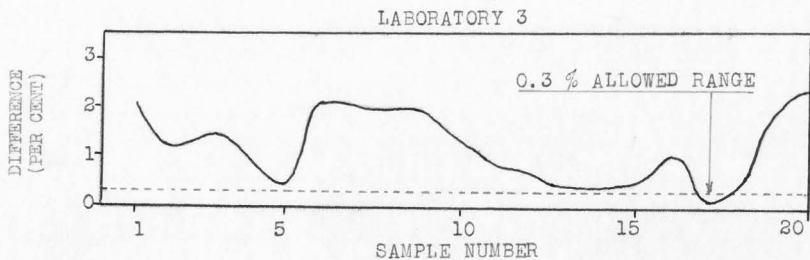


FIGURE 3. PER CENT VARIATION OF LABORATORY 3 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

Figure 3 shows that laboratory 3 is having considerable difficulty with fat testing. There was a marked improvement from samples 11 to 18 but 19 and 20 were out of range again. Although the Babcock method is employed here, the variation should not be greater than 0.3% if the prescribed method is followed closely. It is of paramount importance to weigh the cheese into the test bottle with dispatch because a five minute exposure to the air may cause as much as 0.3 per cent loss of moisture with a corresponding change in fat composition.

Figure 4 also shows an improvement in the second series with a trend away from normal on samples 18-20. The method here is Babcock and the comments on figure 3 apply here also.

Figure 5 indicates that laboratory 5 made a distinct improvement in series 2.

Figures 6, 7 and 8 show that these three laboratories are doing a fairly consistent job of testing with none of the variations exceptionally wide. Particularly noteworthy are samples 11-14 by laboratory 6 and 16-20 by laboratory 8.

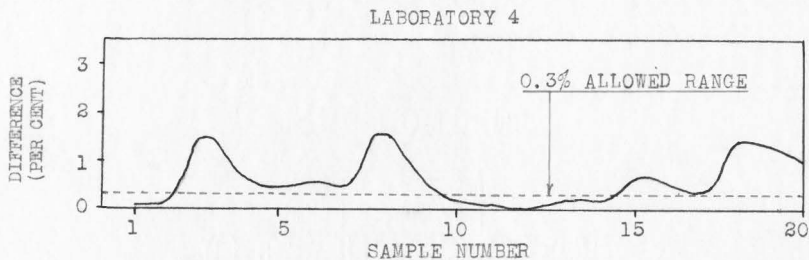


FIGURE 4. PER CENT VARIATION OF LABORATORY 4 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

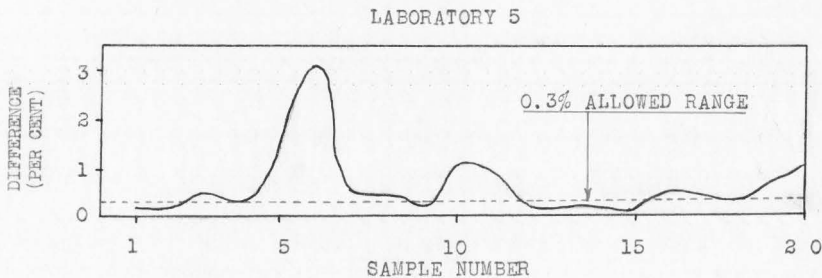


FIGURE 5. PER CENT VARIATION OF LABORATORY 5 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

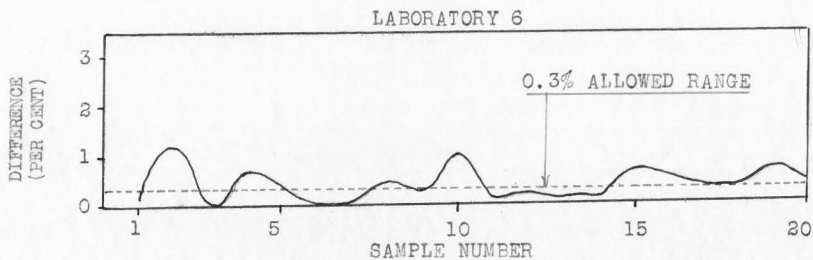


FIGURE 6. PER CENT VARIATION OF LABORATORY 6 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

LABORATORY 7

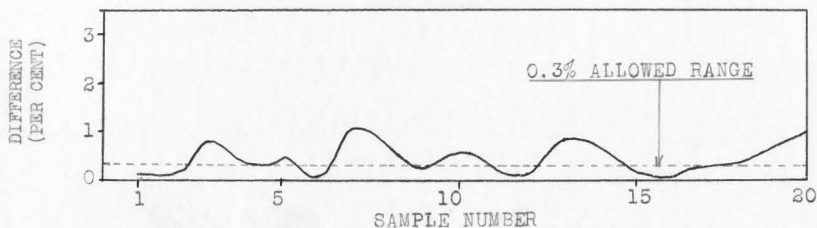


FIGURE 7. PER CENT VARIATION OF LABORATORY 7 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

LABORATORY 8

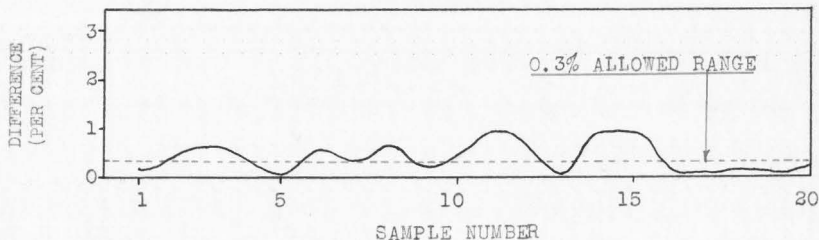


FIGURE 8. PER CENT VARIATION OF LABORATORY 8 FAT TEST FROM U.S.A.C. FAT TEST - SAMPLES 1-20.

Figure 9 shows that laboratory 1 is doing a good job in moisture testing. Samples 4 and 5 are explained by a faulty thermostat on the drying oven.

Figure 10 corresponds somewhat to figure 2 which represents the same laboratory. Without a change in procedure on the second series, the tests came abruptly into the proper range. There was a change of operator on the second series which may explain the improvement in moisture analysis without a change of procedure.

Figure 11 indicates that laboratory 3 was doing a good job on moisture analysis until sample 16. This trend away can probably be easily corrected.

Figure 12 shows quite erratic results of moisture analysis for laboratory 4 with the second series showing particular difficulty. One possible explanation for such difficulty may be the temperature of 150° C. employed in the drying process. Such a temperature is sufficient to cause decomposition of the cheese. It was found in charting drying curves on the Brabender tester in the U.S.A.C. laboratory that temperatures in excess of 130° C. caused decomposition of the cheese.

Figure 13 indicates that laboratory 5 does fairly well. After some variation, samples 19 and 20 came back into range. Difficulty here may be partially explained by the use of a torsion balance instead of an analytical balance. This applies as well to laboratory 4.

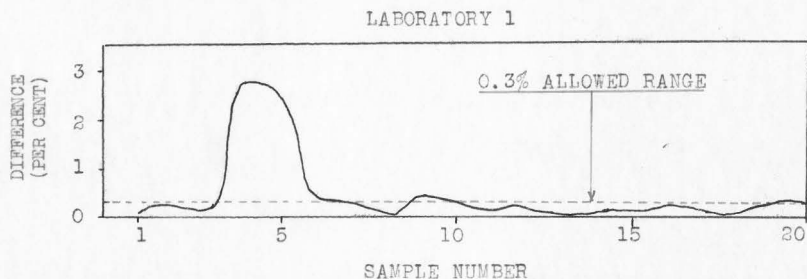


FIGURE 9. PER CENT VARIATION OF LABORATORY 1 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

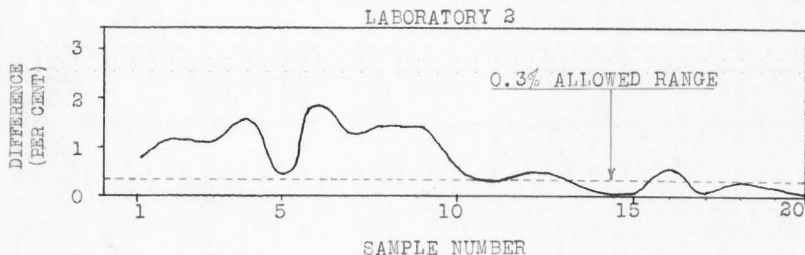


FIGURE 10. PER CENT VARIATION OF LABORATORY 2 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

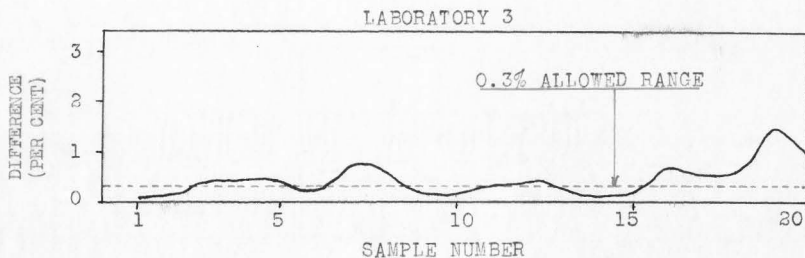


FIGURE 11. PER CENT VARIATION OF LABORATORY 3 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

LABORATORY 4

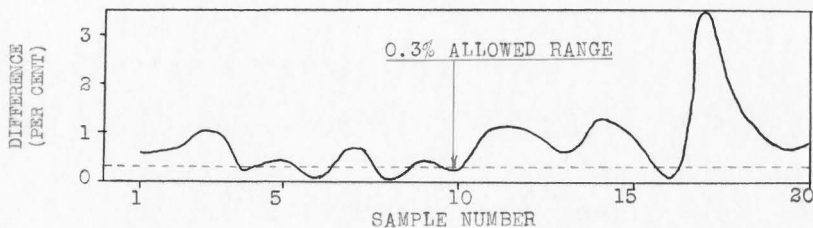


FIGURE 12. PER CENT VARIATION OF LABORATORY 4 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

LABORATORY 5

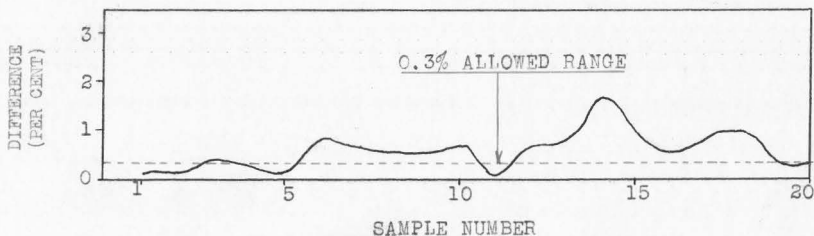


FIGURE 13. PER CENT VARIATION OF LABORATORY 5 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

LABORATORY 6

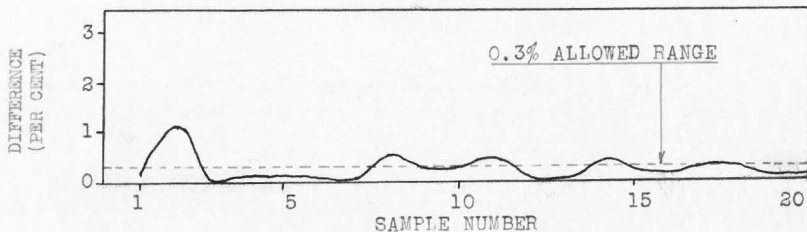


FIGURE 14. PERCENT VARIATION OF LABORATORY 6 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

Figure 14 shows very good testing methods for laboratory 6. This corresponds to good fat tests as well.

Figure 15 gives laboratory 7 a good record except for two samples. The results here may be considered particularly good for the equipment used. The drying oven thermostat allows too wide a range of temperature.

Figure 16 shows a remarkable series of moisture tests. It is particularly interesting because these people have had a great deal of experience in cheese analysis. Another interesting feature is the fact that this laboratory used the same procedure as laboratory 1 which also had very good results. It is evident that experience and training are big factors in accurate analytical work.

LABORATORY 7

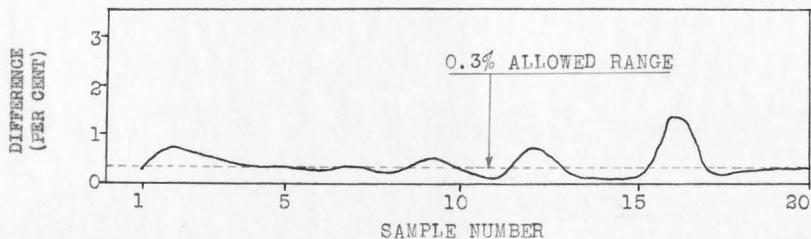


FIGURE 15. PER CENT VARIATION OF LABORATORY 7 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

LABORATORY 8

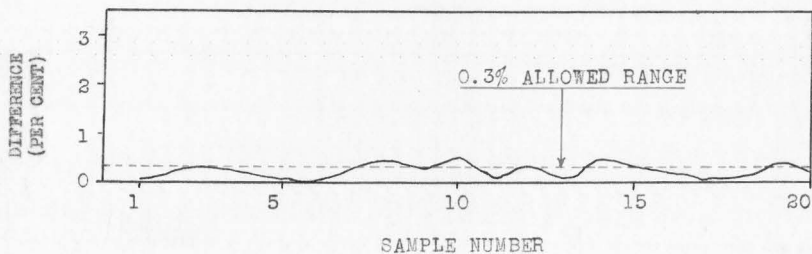


FIGURE 16. PER CENT VARIATION OF LABORATORY 8 MOISTURE TEST FROM U.S.A.C. MOISTURE TEST - SAMPLES 1-20.

CONCLUSIONS

In all cases where ether extraction was used for fat determination, results were good. This method is highly recommended.

Where the Babcock method or minor variations from it are used, speed of weighing and attention to temperatures and clean glassware are highly recommended.

Drying temperatures should not be less than the boiling point of water and not greater than 130° C. for practical purposes. These temperatures should be adapted to the device used.

For laboratories with a vacuum oven, either the A.O.A.C. moisture test or the method employed by laboratories 1 and 8 is recommended as standard procedure.

From the results of the study it is evident that much work needs to be done with the laboratories doing cheese analysis. Since here we have attacked only analytical procedures, the errors that sampling procedures may introduce give just cause to the cries of the industry.

A cooperative program of a series of a few samples run once or twice a year would do much to improve the results of the laboratories.

The program just completed indicates that some laboratories having difficulties can adapt and turn out consistently good results.

The study has reconfirmed the fact that analytical balances and well trained technicians are essential.

SUMMARY

1. Because preliminary work indicated that all laboratories did not agree in analysing the same piece of cheddar cheese, a cooperative testing program was instituted.

2. Eight commercial, state and government laboratories cooperated in testing twenty prepared samples of cheese for fat and moisture with the U.S.A.C. laboratory as control.

3. The first ten samples determined the degree of accuracy of the laboratory. The second ten samples were an attempt to improve results if improvement were needed.

4. In most cases an improvement was noted in the second series, but not in all cases. Some laboratories tested very well through both series.

5. In a study of results, both good and bad, it was evident that most laboratories can overcome testing difficulties if they have a control to test against.

6. Results of the program indicated that most important of all, to test consistently and accurately, well trained, experienced people should do the work.

7. As far as is economically possible, analytical balance should be used.

8. In all cases where ether extraction was used for fat determination, results were good. This method is highly recommended.

9. Where the Babcock method is used, or minor variations from it, speed of weighing and attention to temperatures and clean glassware are highly recommended.

10. Drying temperatures should not be less than the boiling point of water and not greater than 130° C. for practical purposes. These temperatures should be adapted to the device used.

11. For laboratories with a vacuum oven, either the A.O.A.C. moisture test or the method employed by laboratories 1 and 8 is recommended as standard procedure.

12. A cooperative program of a series of a few samples run once or twice a year would do much to keep the laboratories well correlated.

13. From the results of the study it is evident that much work needs to be done with the laboratories doing cheese analysis. Since here we have attacked only analytical procedures, the errors that sampling procedures may introduce give just cause to the cries of the industry.

APPENDIX

Following are the methods of moisture and fat analysis employed by the eight cooperating laboratories:

Laboratory 1:

"Butterfat analysis is made by the Mojonnier method. Moisture analysis is made by using a two gram sample, setting overnight at 100° C., drawing a vacuum of twenty-five inches or more for one hour and weighing to constant weight."

Laboratory 2: series 1.

"Weigh thirty grams of well-ground cheese into a 100 c.c. beaker. Transfer to Waring blender. Add 150 c.c. of 10% sodium citrate solution previously warmed to 150° F. Let stand for five minutes. Stir in blender for five minutes. Agitate blender occasionally to make sure all cheese is being agitated. Pour mixture into a 250 c.c. volumetric flask. Place flask in cold water bath to cut foam. Adjust to room temperature, dilute to mark and stir. Pipette 17.6 c.c. to Babcock milk test bottle. Add 17.5 c.c. cream test acid and proceed according to the usual Babcock method.

Calculation: $30:250 = x : 17.6$

$x = 2.112$ gms. in 17.6 c.c.

$$\frac{18}{2.112} = 8.53$$

8.53 x Fat reading on bottle = Fat in sample of cheese"

The following is the method we used for determining Moisture:

Weigh 2-3 g of prepared sample into round flat-bottomed metal dish, not less than 5 cm in diameter and provided with close-fitting slip-in cover. Place loosely cover-

ed dish on metal shelf (dish resting directly on shelf) in vacuum oven, kept at temp. of boiling H_2O . Dry to constant weight (ca 4 hours) under pressure not to exceed 100 MM (4") of Hg. Discontinue action of vacuum pump and carefully re-admit air into oven. Press cover tightly into dish, remove dish from oven, cool, and weigh. Express loss in weight as moisture.

You will recognize this method as the official AOAC test for Moisture in cheese."

Laboratory 2: series 2.

"The A.O.A.C. Vacuum Oven Method was used for moisture determinations and the A.O.A.C. Roese Gottlieb Method was used for the determination of fat."

Laboratory 3:

"Samples of cheese are weighed on a torsion balance. 5 gram samples used in Damrow moisture cups with inset vented cover, tared to uniform weight with sand. Dishes are usually placed in the oven at 12 noon or 1 p.m., temperature raised to $100^{\circ} C$ and, usually about 2 p.m., a vacuum of 25 inches pulled by water pump. Temperature is maintained all night, and the vacuum holds to about 6 inches by 8 a.m., although the water pump is turned off at 5 p.m. The dishes are cooled to room temperature in a dessicator and weighed back on the same torsion balance.

Our procedure on fat test is to weigh 9 grams of the grated sample into a 50% Babcock test bottle, add a small amount of hot water at $140^{\circ} F$, and warm thoroughly before

adding acid. Approximately 16 cc of normal strength sulphuric acid is used, and sample agitated in mechanical shaker for 20 minutes, or until all curd appears to be dissolved. Centrifuging and reading completed as in ordinary cream test."

Laboratory 4:

1. Balance Torsion cheese scales without weights with 8% cream test bottle.
 2. Remove sufficient cheese from cheese sample bottle onto a glass square, and as quickly as possible cut with paring knife into small pieces and place into test bottle, untouched by hands.
 3. Nine grams are thus weighed out.
 4. 17.5 c.c. water at 160° is then added to test bottle.
 5. 17.5 c.c. sulphuric acid added to test bottle.
 6. Hand shake and agitate.
 7. Test bottle put into mechanical shaker and agitated until cheese is fully dissolved.
 8. Centrifuge 5 minutes. Add hot water to test bottle neck. Centrifuge 3 minutes. Add hot water up the bottle neck. Centrifuge 3 minutes. Remove from centrifuge. Add red reader.
 9. Place in hot water bath, 130° -140° for at least 5 minutes.
 10. Read fat as in cream."
1. Balance Torsion cheese scales without weights.
 2. Add to balance 18 gram and 2 gram weights on one

- side. Add cheese sample cup and sufficient perfectly dried sand in cup to the other side until balance is obtained. All cups are prepared to and even balance.
3. Cheese removed from sample jar and quickly cut on glass square with paring knife into small pieces.
4. 10 grams are quickly weighed into cup.
5. Sample cheese in cup placed into Cenco-De Khotinsky Constant Temperature Oven without vacuum with thermostat set at 150° and baked for 15 hours.
6. Samples removed and placed in dessicator until cooled to room temperature.
7. Torsion balance is in balance without weights. 10 grams added to one side, sample on other and balanced with beam weights.
8. Direct reading from beams in %s."

Laboratory 5:

"We follow the Pennsylvania method of testing cheese for fat, which according to our interpretation is as follows: A representative sample is diced or cut into small pieces and then thoroughly mixed. Four and one-half grams of this sample are weighed into a nine-gram fifty per cent test bottle. Eight to ten ml. of water is added, and the aggregate heated to 150 - 180 degrees F. in a water bath, with intermittent shaking to facilitate dispersion of the sample. Reading is then made with dividers, in the same manner as is followed in reading a cream sample except that the results are multiplied by two."

"The procedure in running cheese moisture tests is a modification of several methods, which we have worked out over a period of recent years, and is as follows: The samples are diced or cut into small pieces in the sample bottle. (In our opinion this method of preparing the sample is preferable to using a food grinder in that the worm action of the grinder tends to press some of the moisture out of the sample.) Five grams of the sample is then weighed into a Damrow covered drying cup. The cup is placed in the drying oven and dried for seven hours at a steady temperature of 212 degrees F. (We use a damrow steam oven, equipped with a pressure control valve, which will maintain a cooking temperature of 212 degrees F.) Samples are removed from the drying oven and held in a desiccator for one hour, after which they are weighed back on a Torsion balance for final reading. The results are multiplied by two to arrive at the correct moisture content."

Laboratory 6.

"Fat is determined by the Babcock method. Moisture is determined by weighing the sample with a chainomatic balance in two inch cups. Drying is done in a triple-walled electric oven at 100° C. for 15 hours with no vacuum."

Laboratory 7.

"For the determination of moisture, we have been using the following method: Into an aluminum dish weigh 2 to 3 grams of the sample, drying this sample at the temperature of approximately 210° F. for 5 hours. We have a thermostatically controlled oven which holds the temperature with

an accuracy of approximately 15° F.

For the fat determination, we use the Babcock method. Into a regular 50% cream test bottle, weigh accurately 9 grams of the cheese sample. We are using a Torsion Balance. Add 9 ml of hot water, then 12 to 14 ml concentrated sulphuric acid. Shake until all of the cheese is emulsified. Centrifuge for 5 minutes in a regular cream test centrifuge, add hot water so that the upper end of the fat column almost reaches the top of the bottle, again centrifuge for 1 minute. Keep the bottle in a water bath at approximately 140° F. ± 2° F. for 5 minutes. Add a few drops of glymol letting it run down on the side of the bottle neck. Determine percentage of fat by measuring the fat column with calipers from the lowest point of the fat column to the point of separation between fat and glymol."

Laboratory 8.

"The moisture was determined by drying the samples over-night (15-16 Hours) in an oven held at the temperature of boiling water and pulling a vacuum on the samples for one hour before removing from the oven."

"The fat was determined by hydrolyzing the samples with NH_4OH and extracting with ether."

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