SAMPLING · PRESERVING · TESTING MILK
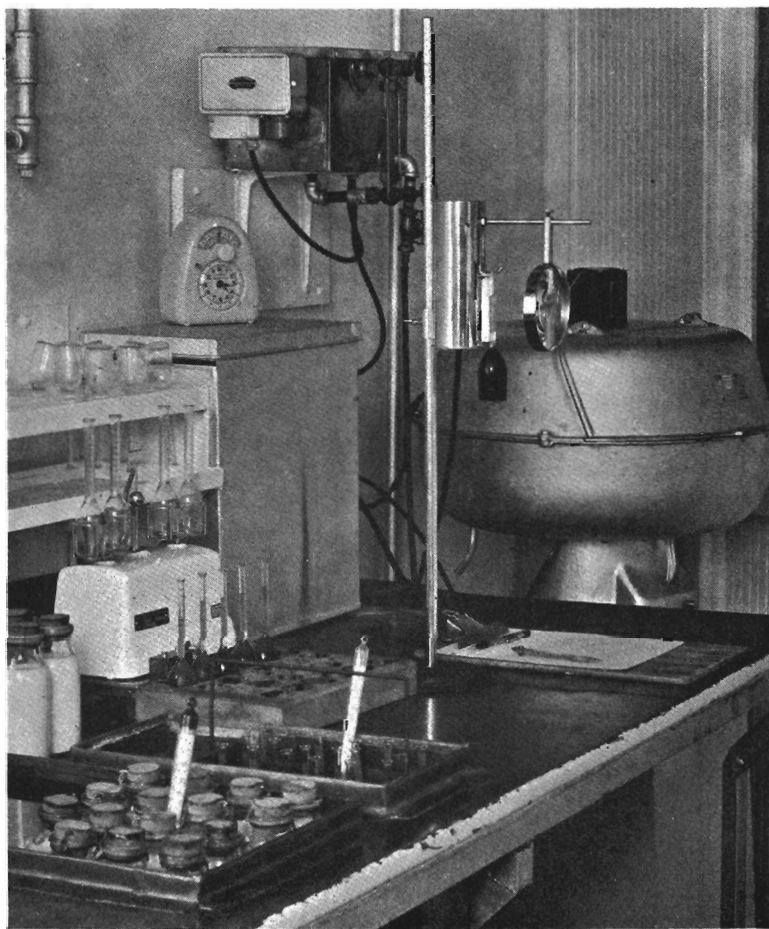
G. H. Wilster
R. P. Robichaux

PREFACE

B ECAUSE of the development of problems concerning methods of sampling, preserving, and testing milk the Oregon Milk Control Board, in pursuance of the Act that created the board, requested the Oregon Agricultural Experiment Station to investigate the subject.

The study was undertaken in April 1939 by the Experiment Station with funds provided by the Milk Control Board.

This bulletin gives a complete report of the significant findings obtained during the investigation.

On the basis of the data obtained and observations made, a complete set of directions for sampling, preserving, and testing milk has been prepared and is presented in this bulletin. The Experiment Station believes that if these directions are accurately followed, milk can be marketed without discrimination against the buyer or the seller.

Wm A Schoenfeld

Director

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SUMMARY AND CONCLUSIONS

A. DAILY VERSUS COMPOSITE TESTING OF MILK

During the period May 1939 to April 1940, 2,064 tests for fat in milk were made by the Babcock method in accordance with a standardized procedure and 1,032 tests for fat were made by the Mojonnier method.

1. When composite samples of milk were tested by the Babcock method the results showed small decreases in the fat percentages as compared with the average fat percentages obtained from the testing of the milk daily.

2. A statistical analysis of the results failed to show that the decreases obtained with the composite method of testing had much significance. The trend observed, however, was in one direction: the fat tests with the composite method were generally lower than with the daily method.

3. Testing all the samples by the Mojonnier method also gave small decreases for the composite method of testing.

B. MISCELLANEOUS STUDIES, INCLUDING SAMPLING MILK AT MILK PLANTS, STORING COMPOSITE SAMPLES, TESTING PROCEDURES, ETC.

This involved making more than 2,000 individual tests on milk.

1. When milk was received at milk plants, the agitation to which the milk was subjected when it was dumped into the weighing vats from the cans, not previously stirred, was not sufficient to obtain a representative sample.

2. A mixing and sampling device constructed at the Experiment Station proved to be an aid in obtaining a representative sample of milk deliveries at milk plants.

3. With composite samples of milk the failure to mix daily the contents of the bottles or allowing the bottles to remain at room temperature long enough each day to increase the temperature of the milk before the samples were returned to the refrigerator caused a greater decrease in the average test than was the case when the composites were mixed daily and returned to the refrigerator promptly after the daily additions of milk. The refrigerator temperature was kept at from 35° to 40° F.

4. When composite samples were kept at room temperature (approximately 70° F.) greater decreases from the average daily test were obtained than when the samples were stored in a refrigerator.

5. Formalin did not prove superior to mercuric chloride for preserving composite samples of milk when sterilized bottles were used, regardless of the time and temperature at which the samples were held.

6. The addition of saponin to composite samples of milk did not prove of any benefit in arresting decreases, in the fat percentages of composite samples.

7. No significant difference was obtained in the fat tests from preserved milk that had been stored in rubber-stoppered sample bottles for periods of 7 and 14 days at 40° F. and from preserved milk of the same lot measured into 8-per-cent test bottles, tightly stoppered, and then stored for 7 and 14 days at 40° F.

8. Varying the temperature of the milk at the time of withdrawing the 17.6 cc. charge of milk used for the Babcock test by only a small amount, such as 12° F. from 68° F., did not cause any measurable change in the final results. Measuring at 100° F. instead of at 68° F. decreased the average fat reading 0.05. Measuring the milk at a temperature of 120° F. as compared with measuring at 55° F. caused a difference in the test of 0.08. The lower test was obtained from the milk measured at 120° F. The generally recognized variation permitted is from 60° to 70° F.

9. Legally standard Babcock 8-per-cent milk-test bottles were found to vary as much as 0.015 cc. above and 0.018 cc. below the correct capacity of 1.600 cc. of the graduated portion of the neck. Variations of the graduated portion of the neck from 1.585 cc. to 1.615 cc. were found to cause measurable differences in the fat reading.

10. Standardization of the strength, temperature, and amount of acid used for the Babcock test was found to be necessary, if dependable, uniform, and accurate results were to be obtained.

11. A speed of 200 revolutions per minute below the specified speed of a 20-inch diameter centrifuge gave fat tests that averaged 0.112 per cent less than those obtained at the correct speed. A speed of 200 revolutions per minute above the specified speed for a 20-inch diameter centrifuge gave an average increase of only 0.009 in the readings.

12. Measuring the fat columns immediately when bottles were removed from the centrifuge, the interior of which was maintained at from 175° to 180° F., resulted in tests that were 0.046 per cent higher, on an average, than tests after the bottles and contents had been maintained in the water bath at a temperature of 138° F. for not less than 5 minutes.

13. When tests were being measured, a higher temperature than normal gave a higher fat percentage, and a lower temperature gave a lower fat percentage.

14. The length of time the test bottles and contents remained in the water bath at 138° F. after the first 5 minutes had no effect on the fat readings.

15. The use of a reading lamp, equipped with a magnifying glass, and needle-pointed calipers when measuring the fat columns from 1,044 samples of milk tested in duplicate and measured by the same operator gave 646 tests that showed no variation between the duplicates, 380 that showed a variation of 0.05, and 18 that showed a variation of 0.10. No variation greater than 0.10 occurred.

16. The duplicate tests from 834 of the 1,044 samples were measured by a second person. The individual readings obtained by the two persons agreed exactly or within 0.05 per cent in the majority of the readings. A variation of 0.1 per cent occurred with 27 of the tests; the second reader obtained a reading 0.1 per cent higher than the first reader 17 times and a reading 0.1 per cent lower 10 times.

C. DIRECTIONS FOR SAMPLING, PRESERVING, AND TESTING MILK FOR FAT.

On the basis of the results obtained and observations made in this study, directions for the sampling, preserving, and testing of milk have been prepared. If these directions are accurately followed, errors in testing will be reduced to a minimum.

In the determination of the fat content of milk received at milk plants it is impossible to reach absolute perfection. Small variations due to slight inaccuracies in the glassware used and normal small errors by the operator are bound to occur. The determination should be made with such accuracy and integrity that duplicate determinations will not vary by more than 0.1 in the fat percentage.

If the fat tests are carefully and honestly made, small variations, over a period of time, will give an average that closely approaches the true value. Under these circumstances neither the seller nor the buyer of the milk will be favored.

Sampling, Preserving, and Testing Milk

By

G. H. Wilster, Professor of Dairy Manufacturing, and
R. P. Robichaux, Research Assistant in Dairy Manufacturing*

Purpose and Plan of Study

OF the approximately 100 billion pounds of milk produced in the United States in 1937, 31 billion pounds were skimmed for sale as butterfat and 39 billion pounds were sold as milk to market milk plants and different manufacturing plants. Of the 1⅓ billion pounds of milk produced in Oregon during 1937, 522 million pounds were skimmed for sale as butterfat and 501 million pounds were sold to different dairy plants.

The Babcock test is universally used in the United States for determining the amount of fat in milk and cream. It is recognized as one of two official methods by the Association of Official Agricultural Chemists.† The methods of this association are commonly recognized as official by law-enforcing agencies in the different states.

The Oregon law requires that "Every milk products plant shall at all times employ a licensed tester, who shall sample and test all milk or cream purchased or received by such plant and who shall be responsible for the operation of the Babcock test of such milk products plant. Every milk products plant shall take accurate samples of any milk or cream received. It shall be unlawful for any milk products plant or any agent or employee or tester thereof to underweigh, undertest, or overtest, or incorrectly weigh, measure, or test any milk or cream received or purchased, or to fraudulently manipulate any weight, test, or measure of any milk or cream, or to make any false entry thereof as to weight or test or measure thereof upon any statement, record, invoice or milk or cream test sheet."‡

The present Oregon law governing the purchase of milk and cream furthermore provides that composite samples of milk must not be made up over a period longer than 15 days or twice a month, and that the glassware and other equipment used in testing and also the procedure of testing be in accordance with certain specific standards.

The State Department of Agriculture is vested with authority to enforce the law that governs the purchase of milk and cream in accordance with the Babcock test.

In 1938 the Central Testing Laboratory at Portland was established. This laboratory has been testing most of the market milk sold in Portland. It is operated on a cost basis by the State Department of Agriculture.

The Oregon Milk Control Board needed further scientific data regarding sampling and testing milk. It made available to the Oregon Agricultural Experiment Station necessary funds for conducting an investigation on this subject.

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* Resigned.
† Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists, 4th ed. 1935. Published by the Association of Official Agricultural Chemists, Washington, D. C.
‡ Section 41-718 Oregon Code 1935 Supplement.

Because of the difference in results obtained by using various methods and procedures in sampling and testing and because of the variations obtained by different operators of the Babcock test, the immediate problem was to find out whether the conflicting results were due to faulty equipment and procedures or to inaccuracies in the method.

The investigation as outlined by the Oregon Agricultural Experiment Station involved, (1) a study of the variations between different methods of sampling, preserving, and testing milk for fat during various seasons in accordance with a standardized procedure, and (2) a study to determine the error in the results obtained in the Babcock test when unstandardized practices were used. The purpose of this was to try to solve the problem whereby the sampling, preserving, and testing of milk for fat could be more uniformly accomplished by different operators in dairy plants.

PRELIMINARY INVESTIGATION AND PREPARATION

Before proceeding with the investigation proper, it was deemed necessary to obtain the best type of equipment for sampling and testing, and also necessary to adapt methods and procedures in accordance with the official specifica-
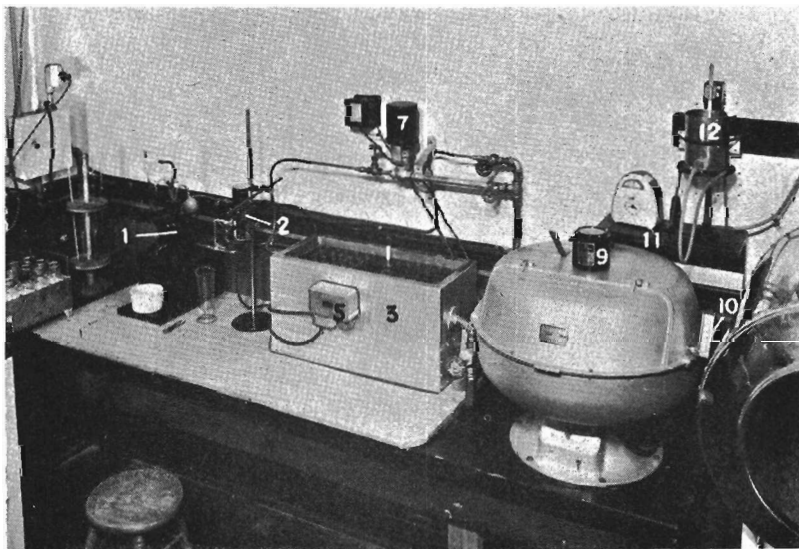
Figure 1. Babcock testing equipment used in the investigation.
1. Sulphuric acid container and burette.
2. Reading lamp for measuring fat columns.
3. Combined water-tempering bath for milk samples (68° F.) and final tests (138° F.).
4. Automatic control (at right bottom of tank) (for 68° F.).
5. Automatic control (for 138° F.).
6. Electric heating element (for 138° F.) (at left bottom of tank).
7. Magnetic water valve (for 68° F.).
8. Centrifuge, electrically operated, equipped with an electric heater.
9. Speed indicator.
10. Indicating thermometer.
11. Interval timer.
12. Hot water container.

tions in order to develop a high degree of accuracy in the investigational work. This preliminary work extended over a period of approximately 1 month. Because certain pieces of equipment and apparatus could not be purchased, it was necessary to design and construct them at Oregon State College. This was done in order that a considerable number of samples could be tested accurately and efficiently.

METHODS AND PROCEDURES USED FOR THE PART OF THE INVESTIGATION THAT INVOLVED DAILY AND COMPOSITE TESTING

In order to study the seasonal variations, if any, it was decided to divide the observations into a number of periods, or units. A unit meant the collection of six samples of milk daily for daily and composite testing; five samples were obtained at a commercial market-milk plant in the city of Corvallis and one sample was obtained in the Experiment Station dairy products laboratory. These were collected over a period of 15 days for each unit.

When sampling the milk, about 1 pint representing the well-mixed milk delivered by each producer was mixed thoroughly by pouring from one container to another until six pourings had been completed. A ¾-ounce portion of this sample was then poured into each of three rubber-stoppered sample bottles, to which one No. 1 mercuric chloride (corrosive sublimate) tablet had been added. The average weight of a tablet was 0.4 gram. The tablets were reported by the manufacturer to contain 68 per cent mercuric chloride. Two of the bottles were of the 8-ounce size for 5- and 7-day composites and one bottle
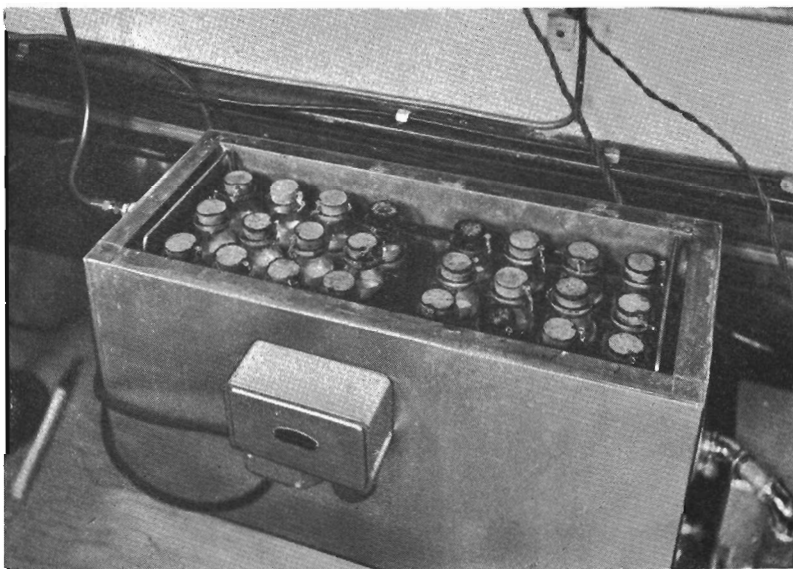

Figure 2. Water-tempering bath used for milk samples (68° F.).

was of the 16-ounce size for the 15-day composite. Another sample (about 4 ounces) was placed in a rubber-stoppered bottle for the daily test.

A total of 10 units extending over a period of 10 months was completed. This covered the four seasons of the year from spring 1939 to spring 1940. In the first two units, 5-, 7-, and 15-day composite samples were used. In the remaining eight units, only the 7- and 15-day composite methods of testing were compared with the daily method.
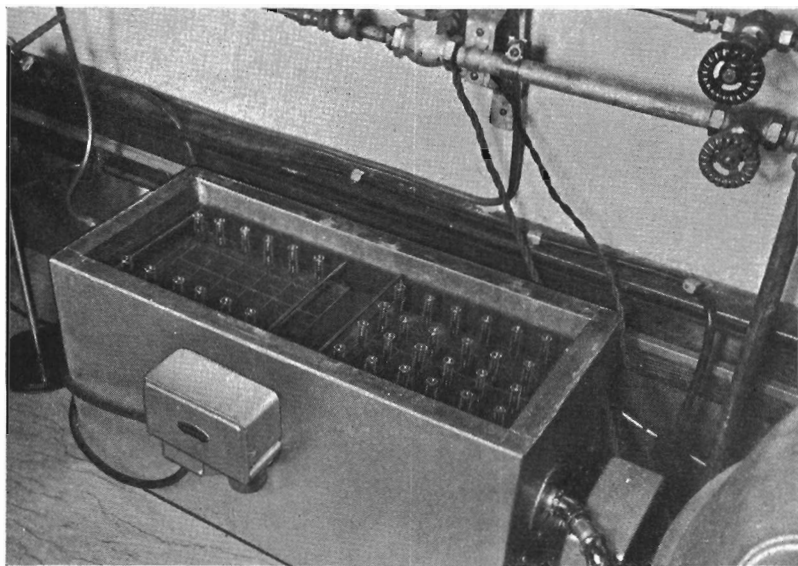
Figure 3. Water-tempering bath used for test bottles (138° F.).

The composite samples were stored in the refrigerators in the two different establishments at a temperature of from 35° to 40° F. The samples were mixed with a rotary motion each day after the addition of the daily portion until all the cream had been removed from the sides of the bottles. The samples were then tilted to mix completely the contents of the bottles. The composite samples were returned to the refrigerator as soon as possible after the addition of the daily portions.

Both the fresh and composite samples were handled and tested as follows:

The samples were placed in a water bath maintained automatically at a temperature of 68° F. by means of an electric regulating device. They were kept in this water bath for approximately ½ hour.

The standard Babcock milk-measuring pipettes were used. They were graduated to hold 17.6 cc. The pipettes were guaranteed by the manufacturers to be within ±0.01 cc. of the correct capacity.

Standard 8-per-cent Babcock test bottles were used. No test bottle was used that had a variation in the total volume of the graduated portion of the bottle greater than 0.005 cc. as determined by the use of mercury delivered by means of a specially constructed burette.

The sulphuric acid used was tested for its specific gravity at 60° F. by means of an acid hydrometer. The acid was always standardized and checked by means of a hydrometer so that it had a specific gravity of 1.825 or 1.830. The acid purchased usually had a specific gravity of 1.835 to 1.840. Dilution of the acid was usually done by adding slowly 2 liters of acid to about 40 cc. cold distilled water. The standardized acid was then cooled to the proper temperature in a water bath before being used.
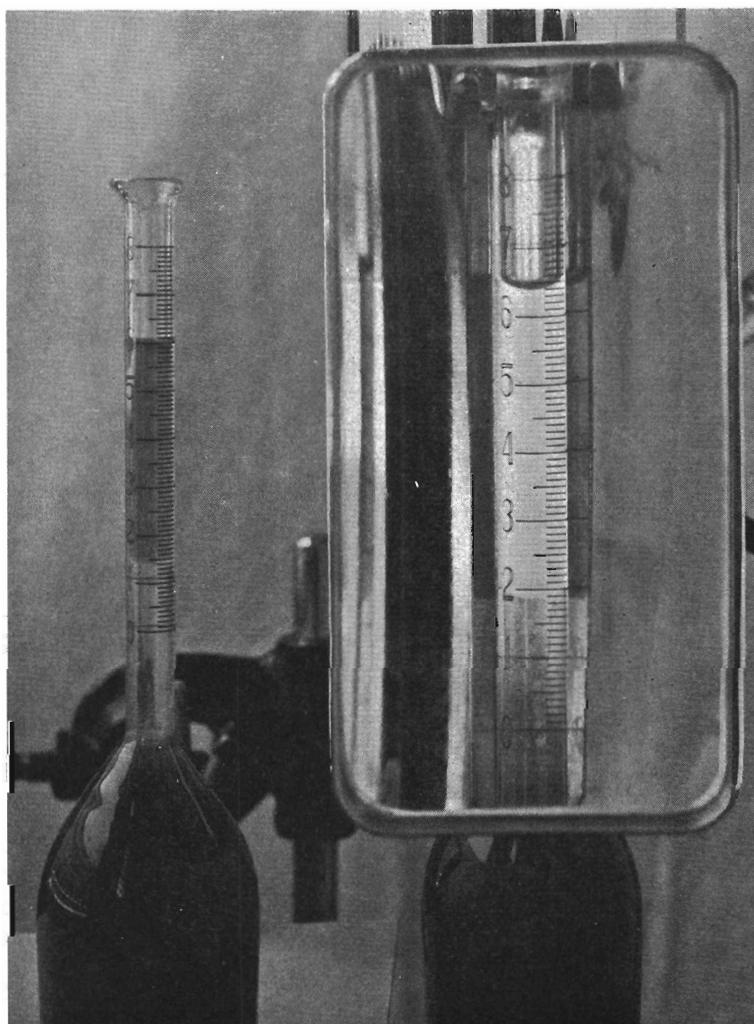

Figure 4. Showing the advantage of using a lamp and magnifying glass for measuring fat columns. The test bottle is attached at the top to the lamp. The complete length of the fat column is illuminated.

The acid when added to the milk was of a temperature of from 60° to 70° F. The milk in the test bottles was brought to a uniform temperature before the acid was added by placing the rack containing the test bottles in a bath of cold water for about 5 minutes.

An electrically driven centrifuge equipped with a speedometer was used. The air in the centrifuge was maintained during the centrifuging at a temperature of from 135° to 140° F. by means of an electric heater.

Distilled water was added to the test bottles after the first and second centrifuging periods. The water was of a temperature of from 140° to 150° F.

The test bottles and contents were centrifuged at the proper speed for periods of 5 minutes, 2 minutes, and 1 minute. A time-alarm clock was used for determining the centrifuging periods.

After the centrifuging, the bottles were placed in a water-tempering bath with the part of the neck that contained the fat column submerged. The temperature of the water was 138° F. This was automatically maintained by means of an electric heating unit. The bottles were kept in this bath for not less than 5 minutes prior to measuring the fat.

All determinations from each sample of milk were made in duplicate and the fat columns were nearly always measured by two different persons. The average of the four readings was accepted as the result for each determination. No difficulty was experienced with charred or curdy fat columns. Differences greater than 0.1 per cent between duplicates did not occur.

A specially constructed reading lamp equipped with a magnifying glass was used when measuring the fat column. The fat column was measured to the nearest 0.05 per cent. (See Figure 4.)

The two test bottles used when testing each daily sample were nearly always used when testing the composite sample that represented the daily additions.

Needle-pointed calipers were used for measuring the fat column. The fat was measured from the lowest extreme point of the column to the extreme top of the meniscus on the surface.

The composite samples of milk were handled and tested identically with the daily fresh samples except when it was impossible to obtain a good mixture of the cream with the underlying milk. The samples were then heated to a temperature of 100° F., mixed thoroughly, and subsequently cooled to a temperature of 68° F. before measuring the necessary amount.

SURVEY OF SAMPLING AND TESTING PROCEDURES IN PORTLAND MILK PLANTS

In addition to the preliminary testing work described above, a survey was made of the methods of sampling, preparing composite samples, and storing samples, and of the types of milk-weighing vats used. A total of 20 different market-milk plants was visited. The survey was made jointly with a representative of the Portland Central Testing Laboratory.

The methods of obtaining the daily sample portion from the milk in the weighing vat and mixing this with the milk from previous shipments in the sample bottle varied between the different plants.

In all the plants, the milk in each can was stirred by means of a suitable stirrer in accordance with the rules of the Testing Laboratory. In several plants, the milk was stirred not only before but also after it had been dumped

into the weighing vat. In one plant, a mechanical stirrer was used for mixing the milk in the weighing vat. After the milk had been placed in the weighing vats, a ¾-ounce sample was taken by means of a flat-bottomed ¾-ounce dipper. When the weighing vat was too small to hold all of the milk in the shipment, the lot of milk was divided and a ¾-ounce sample was taken from each vat. These portions were mixed together and a ¾-ounce portion was taken of this mixture to be added to the composite sample bottle. The standard ¾-ounce dipper as required by the Portland Central Testing Laboratory was used in all plants. The method of securing an aliquot portion of the milk in each delivery by means of a proportional sampler was not used in any of the plants visited.

The weighing vats used were not of uniform construction. Some had square bottoms and others had rounded bottoms. Most of the vats were equipped for gravity drainage but in two plants a suction pump was used for pumping the milk out of the vat. It was observed that when pumps were used a considerable amount of foam remained in the weighing vat. Little or no foam remained when the vats were emptied by gravity through an outlet valve located in the bottom of the vat. Most of the vats were suspended on regular hanging devices with a dial scale arrangement but others were placed on platform beam scales of different types.

The ideal method of adding the daily portion to the sample bottle is to rotate each bottle gently after the addition of each portion until all of the cream has been removed from the side of the bottle, and then tipping the bottle to reincorporate any moisture that had accumulated through condensation on the side of the bottle and stopper and then return the sample to the cooler before it has been heated to any considerable extent. In some plants the bottles were hardly handled at all in order to mix the samples. Some bottles were merely shaken a little without tipping to remove the material from the side of the bottle and the stopper. Some, however, were shaken vigorously after the addition of the daily portion. Upon examination of samples kept at the different plants, it was found that a number of them showed a churned condition. Some had fat and cream adhering to the inside upper surface of the bottle, while milk was spilled on the outside of some bottles. Most of the samples, however, were found to be in a good condition.

In most cases all of the composite samples were removed from the plant refrigerator early during the morning, in some instances a considerable time before the milk was received. The sample bottles were left out of the refrigerator at room temperature on the receiving platform until the last sample had been taken. Occasionally, this required a total period of 5 hours or more.

ACCURACY OF MILK-TEST BOTTLES

Another preliminary investigation was a determination of the accuracy of the graduated portion of the standard milk-test bottle. The Oregon law requires this to be graduated from 0 to 8 per cent, with the smallest divisions representing 0.1 per cent fat.

For this work, the Experiment Station designed and had constructed a burette. This burette was checked for accuracy by the U. S. Bureau of Standards, Washington, D. C. Redistilled mercury was used in calibration.

The following results were obtained:

1. Number of bottles checked 356
2. Number of bottles found to hold 1.600 cc. mercury at 20° C. ± 0.005 cc. tolerance (equivalent of 0.025 per cent fat) 264
3. Number of bottles with 0.005 cc. tolerance found to hold 1.600 cc. or more (43.9 per cent of 264) 116
4. Number of bottles with 0.005 cc. tolerance found to hold 1.599 cc. and under (56.1 per cent of 264) 148
5. Number of bottles found to hold more than 1.605 cc.   37
6. Number of bottles found to hold under 1.595 cc.   55
7. Number of bottles found to be within the state tolerance of ± 0.02 cc. (equivalent of 0.1 per cent fat)   356

Results from the Investigation

DAILY VERSUS COMPOSITE TESTING OF MILK

COMPARISON OF THE RESULTS OBTAINED WHEN MILK SAMPLES WERE TESTED DAILY AND WHEN 5-DAY, 7-DAY, AND 15-DAY COMPOSITE SAMPLES WERE TESTED

A total of 2,064 individual determinations was made, using the Babcock method of testing. All the samples of milk were tested in accordance with a standardized procedure decided upon and already described, after the preliminary month of observations and testing. The methods used were in harmony with those of the Association of Official Agricultural Chemists* and also with the methods and procedures outlined in the Oregon state law except as indicated.† All samples were tested in duplicate and the fat column in each bottle was generally measured by two different men. The average of the four readings was taken as the final test. All measurements were made to the nearest 0.05 per cent fat.

Space does not permit the inclusion of a complete tabulation of all the individual tests obtained. The complete record of this is on file at the Oregon Agricultural Experiment Station. In Table 1 are given in summarized form the average fat percentages obtained in the investigational work. Since the measurements were made to the nearest 0.05 per cent, three decimals are

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* See reference on page 7.
† Oregon Code 1930, Oregon Code 1935 supplement, and Chapter 116, Oregon Laws, 1939.
    Section 41-711 Oregon Code 1930 specifies that the pipette used should deliver 17.6 cc. water at 20° C. This is apparently a misprint. The common pipette used is graduated to hold 17.6 cc. This latter type of pipette was therefore used.
    The speeds given by the Association of Official Agricultural Chemists are slightly at variance with those given in Section 41-713, Oregon Code, 1930. The latter appears to be in error for the 24-inch-diameter centrifuge wheel and does not give the speed for the 22-inch wheel. The speed of the centrifuge used was that specified by the Association of Official Agricultural Chemists. In a letter to the senior author under date of November 25, 1940, Mr. A. W. Metzger, Chief of the Division of Foods and Dairies, State Department of Agriculture, writes as follows regarding this matter: "The difference between the Oregon law and the method as recommended by the Association of Official Agricultural Chemists has no doubt crept into the Oregon law through typographical errors. It is the intention of the Department of Agriculture to bring this matter to the attention of the Committee on Foods and Dairies at the 1941 legislative session and ask that the law be made to conform to the methods as prescribed by the Association of Official Agricultural Chemists."

used in giving the average results. Although this third decimal is not very significant, it does assist in showing the trend and was therefore used for this purpose.

It is evident from the data presented that regardless of whether the 5-, 7-, or 15-day composite method of sampling and testing was used the difference

Table 1. AVERAGE DAILY AND COMPOSITE TESTS. STANDARDIZED PROCEDURE. 2,064 INDI-VIDUAL DETERMINATIONS BABCOCK METHOD

Number of daily samples	Babcock average daily test	Babcock average composite test	Decrease in percentage due to composite testing
	Per cent	Per cent	
5-day composites 60	4.774	4.758	0.016
7-day composites 420	4.534	4.513	.021
15-day composites 900	4.548	4.521	.027

between the average daily tests and the composite test representing the same milk was quite consistent. Slightly lower results were obtained with the composite method of testing than with the daily method. The decrease in the average percentage of fat as compared to the average daily tests amounted to 0.016 for the 5-day composites, 0.021 for the 7-day composites, and 0.027 for the 15-day composites.

The data obtained were subjected to statistical analysis. The statistical treatment failed to demonstrate a significant difference between the different means.

In Table 2 are shown the average daily and the average composite tests in the 10 different units. These tests were summarized in Table 1.

Table 2. A COMPARISON OF THE AVERAGE DAILY TESTS IN THE 10 UNITS WITH THE AVERAGE 7-DAY AND 15-DAY COMPOSITE TESTS. 900 SAMPLES MARKET MILK USED.

Babcock method

Testing unit	Average daily test	Average 7-day composite test	7-day composite lower than daily	Average daily test	Average 15-day composite test	15-day composite lower than daily
	Per cent	Per cent		Per cent	Per cent	
1	4.776	4.747	0.029	4.837	4.825	0.012
2	4.858	4.843	.015	4.859	4.838	.021
3	4.669	4.642	.027	4.577	4.573	.004
4	4.553	4.533	.020	4.585	4.540	.045
5	4.570	4.535	.035	4.563	4.548	.015
6	4.618	4.615	.003	4.640	4.643	+.003
7	4.380	4.363	.017	4.487	4.455	.032
8	4.632	4.597	.035	4.643	4.580	.063
9	4.233	4.198	.035	4.188	4.130	.058
10	4.047	4.060	+.013	4.097	4.073	.024

The average 7-day composite test was lower than the average daily test in nine units, and it was higher in one unit. When it was higher, the difference amounted to 0.013, and when it was lower the difference amounted to from 0.003 to 0.035.

The average 15-day composite test was lower than the average daily test in nine units and it was higher in one unit. When it was higher the difference amounted to 0.003 and when it was lower the difference amounted to from 0.004 to 0.063.

The greatest decreases in the 15-day composite test occurred during the two winter months December and January (Units 8 and 9).

The data obtained in the study confirm the recently reported findings at the Illinois Agricultural Experiment Station by Tracy and Tuckey,[*] who found slight decreases in the fat percentages on 7-day composites as compared with the results from daily testing. The differences amounted to 0.061 per cent for the first period of the winter series, 0.026 per cent for the second period of the winter series, and 0.020 per cent for the summer series. The milk from 425 producers was used for the two winter series and from 50 producers for the summer series.

At the Michigan Agricultural Experiment Station Lucas[†] compared the daily and bimonthly composite methods of testing over a period of 5½ months. The milk used in the investigation was sold by 21 producers to the College Dairy Department. The daily average per cent fat for all samples was 0.11 higher than the average for all composite samples, while for individual patrons the differences in the percentage ranged from 0.03 to 0.16. In every case the average daily test was higher than the average of the composite tests.

FREQUENCY DISTRIBUTION OF THE DIFFERENCES BE-TWEEN THE AVERAGE DAILY AND THE 7-DAY AND THE 15-DAY COMPOSITE SAMPLES (Oregon Agricultural Experiment Station).

A frequency distribution of the differences between the average daily and the 7-day and 15-day composite samples is given in Tables 3 and 4.

Table 3. FREQUENCY DISTRIBUTION OF THE DIFFERENCES BETWEEN THE AVERAGE DAILY AND THE 7-DAY COMPOSITE TESTS.
60 comparisons—Babcock method
(Units 1 to 10 inclusive)

Average composite test lower than daily test	Frequency
Per cent fat	Number of times
0.17	1
.13	1
.10	2
.08	2
.07	1
.06	2
.05	1
.04	5
.03	11
.02	10
.01	4
.00	4
+.01	4
+.02	6
+.03	2
+.04	2
+.06	1
+.10	1
Total	60

Average (mean) difference, in per cent, 0.021.
Per cent times composites lower than average daily 66.7
Per cent times composites higher than average daily 26.7
Per cent times composites the same as average daily 6.6

* Tracy, P. H., and Tuckey, S. L. Accuracy of Methods of Sampling Milk Deliveries at Milk Plants. Univ. of Ill. Agri. Exp. Sta. Bulletin 459, 1939.
† Lucas, P. S. Factors Involved in Accuracy of Testing Milk Samples. Michigan Agri. Exp. Sta. Tech. Bul. 158, 1938.

Table 4. FREQUENCY DISTRIBUTION OF THE DIFFERENCE BETWEEN THE AVERAGE DAILY
AND THE 15-DAY COMPOSITE TESTS.

60 comparisons—Babcock method

(Units 1 to 10 inclusive)

Average composite test lower than daily test*	Frequency
Per cent fat	Number of times
0.14	1
.13	1
.10	3
.09	1
.08	3
.07	3
.06	2
.05	3
.04	7
.03	3
.02	8
.01	5
.00	6
+.01	5
+.02	3
+.03	1
+.04	5
Total	60

* The average (mean) difference in the per cent fat from the frequency table is 0.028. The actual difference from the data is 0.027. The difference is due to the rounding off of the numbers in forming the frequency distribution.

Per cent times composite test lower than average daily test 66.7
Per cent times composite test higher than average daily test 23.3
Per cent times composite test the same as average daily test 10.0

In 60 comparisons, the average 7-day composite test was lower than the average daily test in 40, it was the same in 4, and it was higher in 16 comparisons. When the composite tests were lower than the daily tests, the differences ranged from 0.01 to 0.17, and when they were higher, the differences ranged from 0.01 to 0.10.

With 60 comparisons, when the daily testing was compared with the 15-day composite testing, the average composite test was lower than the average daily test 40 times, it was the same 6 times, and it was higher 14 times. When the tests of the composites were lower, the differences ranged from 0.01 to 0.14, and when they were higher the differences ranged from 0.01 to 0.04.

DETERMINATION OF THE FAT CONTENT OF THE MILK USED IN THE INVESTIGATION BY THE MOJONNIER METHOD

As the Mojonnier method of testing milk for fat is well known by the industry and by laboratories, all samples were also tested by this method. The Mojonnier method is a modification of the Roese-Gottlieb method.* The purpose of this comparison was not to determine whether the results obtained by the Babcock method of testing were higher or lower than with the ether

* See Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists for a description of the official Roese-Gottlieb method.

extraction method, but to determine by means of an entirely different method whether the differences, if any, as obtained by the Babcock method between the testing of individual daily samples and composite samples of milk were similar in both methods of testing. All the daily samples and all the composite samples tested by the Babcock method were, accordingly, also tested by the Mojonnier ether extraction method. Only single determinations were made on each sample. The milk charge for each determination was weighed on an analytical balance.

In Table 5 are shown the average fat contents obtained when the samples were tested by the Mojonnier method.

Table 5. AVERAGE DAILY AND COMPOSITE TESTS.
1,032 individual determinations.
Mojonnier method

Number of daily samples	Mojonnier average daily test	Mojonnier average composite test	Decrease in percentage due to composite testing
	Per cent	Per cent	
5-day composites 60	4.697	4.674	0.023
7-day composites 420	4.460	4.439	.021
15-day composites 900	4.472	4.417	.055

The data show that there was approximately the same decrease for the 5- and 7-day composites with this method of testing as was obtained with the Babcock method. With the 15-day composite method, however, there was a greater difference shown with the Mojonnier than with the Babcock method. Whereas with the Babcock method the average 15-day composite test was 0.027 below the average daily test, with the Mojonnier method it was 0.055 below the average daily test.

The larger decrease for the 15-day composite tests, using the Mojonnier method, does not appear to have been caused by sampling difficulties, since the sampling and preparation of the samples for the Mojonnier determination were done as carefully and also at the same time and under the same conditions as for the Babcock method.

While it was not the purpose of the investigation to check the results obtained by the Babcock method of testing by means of the Mojonnier method, it is observed, however, that the results obtained by the Mojonnier method of testing were lower, on an average, than the results obtained on the same samples of milk by the Babcock method. With the fresh daily samples the difference between the average fat percentage was 0.077 for the first 60 samples, 0.074 for the next 420 samples, and 0.076 for the 900 daily samples. Similar differences have been reported by other investigators. The average per cent fat of the composite samples when tested by the Mojonnier method was 0.084 lower for the 5-day composites, 0.074 lower for the 7-day composites, and 0.104 lower for the 15-day composites than was obtained by the Babcock method.

MISCELLANEOUS STUDIES, INCLUDING SAMPLING MILK AT MILK PLANTS, STORING COMPOSITE SAMPLES, TESTING PROCEDURES, ETC.

The purpose of this phase of the investigation was to determine to what extent different methods and procedures in sampling milk, storing the composite samples, and testing the milk for fat affected the results obtained. It had been suggested to the Experiment Station by the members of the industry and also by members of the State Department of Agriculture that these methods and procedures be thoroughly investigated. The results from this work should be valuable as a guide to the formulation of rules for sampling, storing composite samples, and testing milk in different milk plants with the view of unifying the methods and practices and thus avoiding discrepancies.

1. SAMPLING AND STORING COMPOSITE SAMPLES

(A). **Milk-receiving vats.** The rules made by the Central Testing Laboratory at Portland for weighing and sampling of milk call for the stirring of each can of milk thoroughly by means of a rolled- or smooth-edge perforated stirring rod before the milk is poured into the weighing vat. They require also that the milk be poured into the weighing vat within 2 or 3 minutes after it has been stirred. Immediately after the milk from each lot has been placed in the weighing vat, a ¾-ounce sample of the milk should be transferred to the sample bottle. The dipper used must first be filled with milk and emptied in each lot of milk to be sampled before the sample is obtained and transferred to the sample bottle.

In order to investigate whether or not it is necessary to stir the milk in the vat after the milk has been poured into the weighing vat, samples were taken in seven Portland milk plants that used the several different types of weighing vats. From 6 to 12 lots of milk received at each plant were sampled and tested. The milk was not stirred in the can prior to dumping. None of the vats was equipped with a mechanical stirrer. The milk was sampled by means of a dipper from the part of the vat from which the sample was usually withdrawn immediately after the milk had been placed in the vat without stirring in the vat. The milk in the vat was then thoroughly stirred by means of a stirring rod and another sample was taken by means of a dipper. All determinations in the laboratory were made in duplicate and the fat column in each bottle was measured by two different persons. The results obtained are shown in Table 6.

It will be noted that in six of the plants the average test after stirring the milk in the vat resulted in a lower test and in one it resulted in a higher test. In the six plants where there was a decrease, as a result of stirring, the decreases in per cent fat ranged from 0.008 to 0.075. In the plant where there was an increase this amounted to 0.070.

The data showed that when milk was delivered to milk plants and not previously stirred when it was sampled from the milk-weighing vats, differences in the test of the milk occurred. Some form of stirring device is apparently necessary for stirring the milk in the vat before a sample is obtained.

Table 6. A COMPARISON OF TESTS OF MILK OBTAINED FROM WEIGHING VATS IN PORTLAND MILK PLANTS BEFORE AND AFTER STIRRING THE MILK IN THE VATS.

Milk plant	Number of lots tested	Capacity of weighing vat	Before stirring in vat (average test)	After stirring in vat (average test)	Test after stirring lower than test before stirring
		Gallons	Per cent	Per cent	
A	10	80	4.950	4.875	0.075
B	6	50	4.617	4.575	.042
C	6	50	4.433	4.400	.033
D	6	60	4.675	4.666	.009
E	12	65	4.783	4.758	.025
F	12	118	4.504	4.496	.008
G	10	100	4.410	4.480	+.070

Frequency distribution

Milk plant	Number lots milk	Times test higher before stirring	Times test lower before stirring	Times test no different before and after stirring
A	10	6	1	3
B	6	3	....	3
C	6	2	3	1
D	6	2	2	2
E	12	6	4	2
F	12	5	4	3
G	10	2	7	1
Total	62	26	21	15

Maximum differences in tests of stirred and unstirred milk

Milk plant	Number lots milk	Maximum difference in test of individual lots
		Per cent
A	10	0.30
B	6	.15
C	6	.30
D	6	.10
E	12	.30
F	12	.25
G	10	.25

The results obtained at plant G did not compare with those obtained at the other six plants. The weighing vat used in this plant was rectangular in shape and had the dimensions 52 inches long, 32 inches wide, and 20 inches deep. The milk entered the center of the vat, whereas the sample was taken from one end of the vat. This may account for the higher average test that was obtained after the milk had been stirred. At any rate, the results obtained with this vat show the necessity for a thorough stirring of the milk before sampling it.

In a plant in Illinois where the milk-weighing vat was of such a construction that little mixing took place when the milk was poured into it Tracey

and Tuckey* found that with 72 lots of milk the average test of the milk taken from the front of the vat before the milk was stirred was 4.40 per cent and from the rear of the vat it was 4.60 per cent, a difference of 0.20 per cent. The average test of the milk after it had been stirred with a stirring rod was 4.51 per cent. Variations in the test of the milk obtained from the front and rear of the vat ranged from 0 to 1.0 per cent fat in the 72 lots of milk.

(B). **Sampling devices.** Two different sampling devices for obtaining a sample of milk that would be representative of each shipment of milk were studied.

(1) *The sampling tray.* The construction and use of the sampling tray are shown in Figure 5. The tray was constructed at the Oregon Agricultural Experiment Station.† The tray was located on top of the milk-weighing vat. When the milk was poured into this tray, it traveled to the end of the tray, after which, because of the curved shape of the rear end of the tray, a portion of it returned to the front, effecting a mixing. The mixed milk passed out through two portholes located at the front of the tray. A perforated pipe that extended to the outside was located directly under these ports for the purpose of collecting a proportionate amount of the milk as it passed to the milk-weighing vat. This milk was used for the ¾-ounce portion necessary for the composite sample.

In order to determine whether or not this method of sampling was accurate, six comparisons were made using milk that was not stirred in the cans before it was poured into the sampling tray, and two comparisons were made when the milk was stirred in the cans before it was poured into the sampling tray. The results of two of these eight comparisons are shown in Table 7.

The average test for the six comparisons when the milk was not stirred before dumping was 5.05 per cent when the sampling tray was used as compared with a test of the heated milk in the pasteurizing vat of 5.08 per cent, a difference of only 0.03 in the percentage. In the two comparisons where the milk was stirred before dumping, there was no difference between the fat test obtained when the sampling tray was used and that of the test of the milk in the pasteurizing vat after it had been heated.

On the basis of the results obtained with the sampling tray, it would seem that such a device may be of considerable advantage in milk plants for taking representative samples of the milk delivered. Perhaps some modification of the construction of the tray may be desirable.

(2) *The proportionate sampler.* The following comparison was made to study the effect on the average fat test when a proportionate sampler‡ and a dipper sampler were used for sampling milk from a herd of cows. Two samples of milk were taken from the complete milking of each of 20 cows of the Agricultural Experiment Station dairy herd, which is composed of Holsteins, Ayrshires, and Jerseys. One sample was taken by means of a ¾-ounce dipper, using an equal amount of the milk of each cow regardless of weight of milk. The other sample was taken with the proportionate sampler by adjusting the dial on the sampler before sampling in accordance with the weight of the

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* Tracey, P. H., and Tuckey, S. L. Accuracy of Methods of Sampling Milk Deliveries at Milk Plants. Illinois Agr. Exp. Sta. Bul. 459, 1939.
† After sketch supplied by F. S. Board.
‡ Loaned to the Experiment Station by the Braun-Knecht-Heimann Company of San Francisco, California.
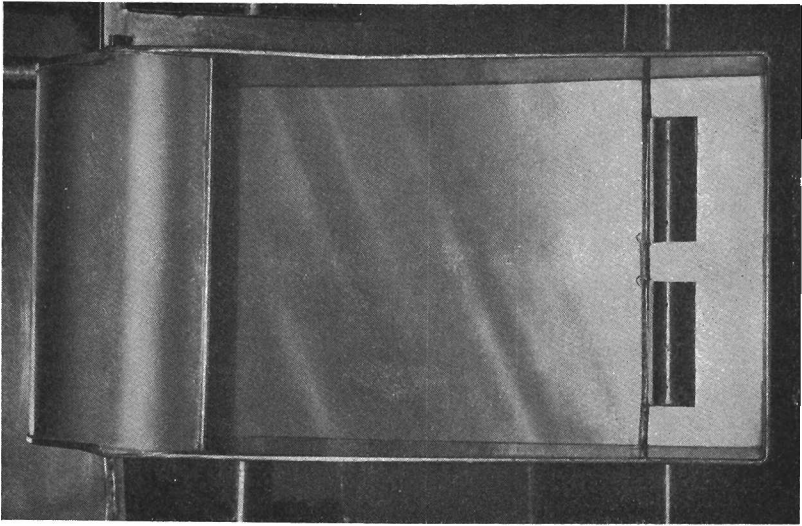
Figure 5.   *Top*: Construction of the sampling tray.   *Bottom*: Using the sampling tray.

Table 7. THE EFFICIENCY OF THE SAMPLING TRAY IN MIXING MILK.

Sample number	Duplicate test		Average test	Weight of milk	Amount of fat (test × weight divided by 100)
	1	2			
	Per cent fat	Per cent fat	Per cent fat	Pounds	
Milk not stirred in cans before dumping					
1	5.60	5.60	5.60	380	21.28
2	4.85	4.85	4.85	172	8.34
3	4.60	4.55	4.57	108	4.93
4	4.90	4.90	4.90	308	15.09
5	5.35	5.35	5.35	167	8.93
Total				1,135	58.57 pounds fat
					$\dfrac{58.57 \times 100}{1135} = 5.16\%$ fat
Sampling tray composite	5.10	5.15	5.12	....	5.12% fat
Pasteurizing vat test before heating	5.15	5.15	5.15	....	5.15% fat
Pasteurizing vat test after heating	5.20	5.15	5.17	....	5.17% fat
Milk stirred in cans before dumping					
1	4.60	4.65	4.62	347	16.03
2	4.80	4.80	4.80	122	5.85
3	5.15	5.15	5.15	123	6.33
4	4.20	4.20	4.20	278	11.67
5	5.05	5.05	5.05	346	17.47
6	4.90	4.95	4.92	172	8.46
Total				1,388	65.81 pounds fat
					$\dfrac{65.81 \times 100}{1388} = 4.74\%$ fat
Sampling tray composite	4.80	4.80	4.80	....	4.80% fat
Pasteurizing vat test before heating	4.75	4.75	4.75	....	4.75% fat
Pasteurizing vat test after heating	4.80	4.80	4.80	....	4.80% fat

Table 8. SAMPLING MILK FROM A HERD OF COWS.*

(All breeds)

Number of comparison	Proportionate sample. Average of 20 samples each comparison	Equal amounts with ¾-ounce dipper samples	Difference
	Per cent fat	Per cent fat	
1	4.25	4.20	0.05
2	4.00	4.25	.25
3	4.15	4.45	.30
4	4.20	4.60	.40
5	4.20	4.40	.20
6	4.25	4.30	.05

* No attempt was made to select the same cows for each comparison.

milk produced by each cow so that a proportionate sample would be taken from the milk in accordance with the total pounds of milk produced.

The results obtained are shown in Table 8.

The milk used in the experiment varied in weight, for each cow, from 8 to 35 pounds. The individual tests of the milk from the cows varied from 2.6 to 6.6 per cent.

The data obtained show the importance of obtaining an aliquot sample when there is a considerable variation between the weights and the per cent of fat present in the milk produced by the cows. Although such variations would not be expected in the milk received from each producer at a milk plant from day to day, the experiment was conducted to show the inaccuracies that are likely to occur in the event there is a considerable difference from day to day.

Table 9. THE EFFECT OF DIFFERENT METHODS OF HANDLING AND STORING COMPOSITE
MILK SAMPLES ON THE FINAL FAT TEST.

Mercuric chloride preservative used
7-day composite samples
Babcock method

Method used in handling the samples	Average test	Lower than fresh daily test
	Per cent fat	
Tested fresh	4.30	......
Handled according to standardized procedure. Stored at 40° F.		
Sample 1	4.28	0.02
Sample 2	4.25	.05
Not sufficiently stirred daily to reincorporate cream and moisture. Remained at room temperature 4 hours daily before returning to 40° F. storage		
Sample 1	4.20	.10
Sample 2	4.20	.10
Shaken vigorously up and down. Remained out at room temperature 4 hours daily before returning to 40° F. storage		
Sample 1	4.30	.00
Sample 2	4.30	.00
Only slightly stirred daily. Kept at room temperature at all times. Bottles not sterilized at the beginning.		
Sample 1	4.18	.12
Sample 2	4.15	.15

The samples that were not handled according to standardized procedure were heated to 100° F., then cooled to 68° F. before testing. The fat column in each duplicate test was measured by two men, and the average of the two measurements is given.

(C). **Effect of the method of handling and storing composite samples on the percentage of fat.** The results obtained from using different methods in the handling and storing of composite samples are shown in Tables 9 and 10. For this study two groups of composites, one 7-day and one 15-day, of eight samples each, were carried as follows:

A ¾-ounce dipper of the same sample of well-mixed milk was added to each of the composite samples daily until tested.

Two samples from each set of composites were handled according to standardized procedure; that is, they were stored in a dark place at 40° F. at all times and were only taken out each day to add the daily portion; they were rotated gently to reincorporate the cream, and the bottles were tipped to reincorporate any moisture that had lodged on the inside of the bottles. They were then immediately returned to the cooler and kept at 40° F.

Two samples from each set of composites were stored at 40° F. but were left out at room temperature each day for 4 hours before being returned to the cooler. Also, they were rotated only slightly, not sufficiently to reincorporate the cream and moisture.

Two samples from each set of composites were kept at room temperature for 4 hours each day after adding the daily portion and were shaken vigorously in an up-and-down motion to reincorporate the cream and moisture. The remainder of the time they were stored at 40° F.

Two samples from each set of composites were stored at room temperature throughout the period. The bottles were not sterilized to begin with and were not rotated thoroughly each day to mix the cream and milk serum.

It is evident from these results that even when composite samples are made up over a period of 7 days it is necessary to use a low temperature (40° F.) for storing the samples and also to mix the contents of the bottles

Table 10. The Effect of Different Methods of Handling and Storing Composite Milk Samples on the Final Fat Test.

Mercuric chloride preservative used
15-day composite samples
Babcock method

Method used in handling the samples	Average test	Lower than fresh daily test
	Per cent fat	
Tested fresh	4.10	......
Handled according to standardized procedure. Stored at 40° F.		
Sample 1	4.10	0.00
Sample 2	4.10	.00
Not sufficiently stirred daily to reincorporate cream and moisture. Remained at room temperature 4 hours daily before returning to 40° F. storage		
Sample 1	4.05	.05
Sample 2	4.03	.07
Shaken vigorously up and down. Remained out at room temperature 4 hours daily before returning to 40° F. storage		
Sample 1	4.00	.10
Sample 2	4.00	.10
Only slightly stirred daily. Kept at room temperature at all times. Bottles not sterilized at the beginning		
Sample 1	4.00	.10
Sample 2	4.00	.10

Before testing all samples were heated to 100° F. in a water bath maintained at 110° F. and pipetted for the Babcock test at that temperature after mixing the cream and serum by pouring from one container to another a total of six times. The samples kept at 40° F. and handled according to standardized procedure, the only ones that could have been sampled without heating due to the ease of reincorporating the cream, were also heated to 100° F. in order to be assured of comparative results. The fat column in each duplicate test was measured by two men, and the average of the two measurements is given.

daily. Samples held at room temperature and not handled according to the standard procedure as outlined had cream and dried material on the sides of the bottles and stoppers. This would not come off when pouring from one bottle to another at a temperature of 100° F., and it was necessary to scrape it off by means of a rubber policeman. It was difficult to get this material reincorporated with the milk, even after the milk had been heated to 100° F. All the samples except those handled according to the standardized procedure showed signs of slight "oiling off" on heating. The method of mixing the samples daily by shaking vigorously proved to be satisfactory in this experiment; this is not an approved method, however, and is not recommended. It may result in a considerable amount of churning.

With the 15-day composite samples, the experiment showed the desirability of handling these in accordance with the formerly mentioned standardized procedure. By handling the samples in accordance with this procedure, it is usually unnecessary to heat the samples before testing in order to effect a complete remixing of the cream and the underlying milk. This can usually be accomplished satisfactorily by rotating and tilting the bottle before the milk is heated to a temperature of from 60° to 70° F. before measuring the milk.

The appearance of the 15-day composite milk samples at the end of the 15-day period before they were used for testing is shown in Figure 6.

The samples handled according to the standardized procedure were in a satisfactory condition.
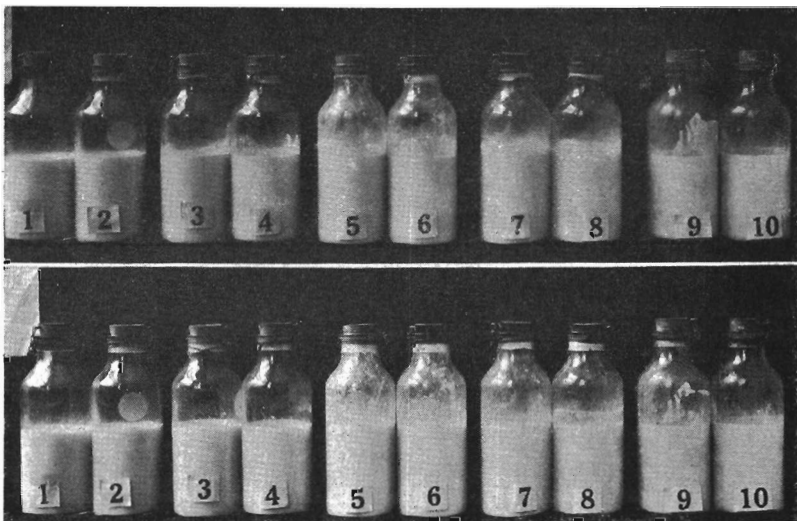
Figure 6.   Comparison of fresh and 15-day composite samples.   *Top:* Before stirring.
Bottom: After rotating bottles 15 seconds.

1-2   Fresh milk.
3-4   Composites handled in accordance with standardized procedure. Kept at 40° F.
5-6   Composites held at room temperature 4 hours each day, balance of time at 40° F. Bottles not rotated each day to reincorporate cream and milk.
7-8   Composites handled as 5-6, but shaken vigorously daily when adding the daily portion.
9-10 Composites stored at room temperature during the 15-day period. Only slightly stirred daily. A tough cream layer had formed on the surface, and moldiness was observed on surface and on the underside of the stopper.

(D). The effect of the preservative used on the fat percentage. In this experiment it was the purpose to determine the effect of mercuric chloride and of formalin as preservatives for milk samples that were stored under different conditions of temperature and light.

One-gallon samples of milk were obtained from each of four different dairy farms. Each sample of the well-mixed milk was tested in duplicate for fat by the Babcock method. Each of the four samples was then divided and placed into 24 rubber-stoppered sample bottles. Each batch of 24 bottles was treated and held as follows: Twelve samples from each batch were preserved with two No. 1 mercuric chloride tablets, 4 were stored at 40° F. in darkness, 4 were stored at 70° to 80° F. exposed to daylight, and 4 were stored at 70° F. in darkness. The other 12 samples from each batch were preserved with 0.5 cc. formalin. These were divided into groups of 4 and stored as with the mercuric chloride. The results obtained in this experiment are shown in Table 11. The results for the 15-day and 30-day periods only are shown.

Table 11. EFFECT OF TIME, METHOD OF HOLDING, AND TYPE OF PRESERVATIVE ON THE FINAL FAT TEST OF COMPOSITE MILK SAMPLES*

Lot number	Original fat test Babcock	Preserved with mercuric chloride held in darkness at 40° F.	Preserved with formalin held in darkness at 40° F.	Preserved with mercuric chloride exposed to daylight at 70° to 80° F.	Preserved with formalin exposed to daylight at 70° to 80° F.	Preserved with mercuric chloride at 70° F. in darkness	Preserved with formalin at 70° F. in darkness
	Per cent fat	Per cent fat	Per cent fat	Per cent fat	Per cent fat	Per cent fat	Per cent fat
15 days							
1	5.27	5.27	5.22	5.22	5.15	5.22	5.22
2	4.30	4.27	4.30	4.22	4.10	4.25	4.25
3	5.30	5.30	5.27	5.25	5.20	5.25	5.25
4	4.10	4.07	4.07	4.00	4.02	4.05	4.05
Average	4.742	4.727	4.715	4.672	4.617	4.692	4.692
Decrease in average test as compared with original	.000	.015	.027	.070	.125	.050	.050
30 days							
1	5.27	5.27	5.22	5.17	5.17	5.20	5.17
2	4.30	4.27	4.22	4.17	4.20	4.20	4.22
3	5.30	5.27	5.27	5.20	5.25	5.22	5.22
4	4.10	4.05	4.05	4.02	4.05	4.05	4.07
Average	4.742	4.715	4.690	4.640	4.667	4.667	4.670
Decrease in average test as compared with original	.000	.027	.052	.102	.075	.075	.072

* When tested all samples were heated to 110° F. in a controlled-temperature water bath. All samples were measured at this temperature and tested according to standardized procedure, the average of duplicates being taken as the final fat test.

It was found that mercuric chloride was as effective as formalin for preserving samples kept at a low holding temperature (40° F.). The time of holding the composites at the low temperature did not seem to change the results materially. There were no consistent results to show that one preserva-

tive was superior to the other in preventing a decline in the test at the higher temperatures, either when stored in darkness or in daylight. The results did not show that it was advantageous to store composites in darkness instead of in daylight.

(E). **The use of saponin and cholesterol in composite milk samples.** To study whether or not the addition of saponin,* which is an emulsifying agent, to the milk would aid in preventing a decrease in the test of composite samples, a comparison was made that involved the testing of 180 daily samples and 36 15-day composite samples in which mercuric chloride and saponin were used with one set of samples and mercuric chloride alone with another set of samples. One No. 1 mercuric chloride tablet and 0.5 gram saponin were added to each bottle in the one set of samples and one No. 1 mercuric chloride tablet was added to each bottle in the other set of samples. When handled and tested by the Babcock method in accordance with the standardized procedure, it was found that the average test of the composite samples that contained saponin was 0.020 lower than the average daily test, and the test of the composite samples that contained no saponin was 0.021 lower. When tested by the Mojonnier method the average test of the composite samples that contained saponin was 0.010 higher than the average daily test and the test of the composite samples that contained no saponin was 0.038 lower. These results show that no material benefit was derived from the use of saponin for composite samples.

In another comparison when 0.5 gram cholesterol was added to each sample at the beginning of the 15-day holding period, it was found that the samples that contained cholesterol were badly curdled at the end of the 15 days and could not be tested. The samples had been held at a temperature of 40° F.

(F). **Comparison of the effect on the fat percentage of storing at 40° F. Preserved milk in rubber-stoppered 8-ounce sample bottles and in tightly stoppered 8-per-cent milk test bottles for periods of 7 and 14 days.** Milk from the same lot was divided into 24 8-ounce sample bottles, using 6 ounces of milk for each bottle. Two No. 1 mercuric chloride tablets were added to each bottle. The milk was tempered to 70° F. After thorough mixing, by means of a 17.6 cc. pipette, milk from each bottle was transferred to two sets of 8-per-cent milk-test bottles. The sample bottles were closed by means of rubber stoppers and the test bottles by means of cork stoppers, tightly inserted. One-half of the sample bottles and one-half of the test bottles were

Table 12. COMPARISON OF THE EFFECT ON THE FAT PERCENTAGE OF STORING AT 40° F. PRESERVED MILK IN RUBBER-STOPPERED 8-OUNCE SAMPLE BOTTLES AND IN CORK-STOPPERED 8-PER-CENT MILK TEST BOTTLES FOR PERIODS OF 7 TO 14 DAYS.

Number of samples	Days stored	Average test				
		Fresh milk	Kept in sample bottles	Change from test of fresh milk	Kept in test bottles	Change from test of fresh milk
		Per cent	Per cent	Per cent	Per cent	Per cent
12..............	7	4.142	4.146	0.004 increase	4.162	0.020 increase
12..............	14	4.183	4.183	.000	4.158	.025 decrease

* A glucoside, which occurs in soap bark.

kept in a refrigerator maintained at a temperature of 40° F. for 7 days. The others were stored under the same conditions for 14 days. The samples were tested by the Babcock method at the end of each period. The results obtained are shown in Table 12.

The difference obtained between the average of the fresh samples and the average of the preserved, stored samples was of no significance. The average fat percentages of the milk stored in sample bottles for 7 and 14 days and then tested were little different from those of the milk stored in rubber-stoppered 8-per-cent test bottles for the same periods. These results indicate that where relatively large differences in daily and composite samples are obtained such differences are due to difficulties and carelessness in sampling and storing samples rather than due to a loss of fat in properly stored samples.

2. VARIATIONS IN TESTING PROCEDURE

(A). **Effect of varying the temperature of the milk at the time of pipetting on the fat test.** The effect of measuring the charge of milk by the standard 17.6 cc. pipette for the test bottle at a temperature other than at 68° F. is shown in Table 13.

Table 13. EFFECT OF TEMPERATURE WHEN PIPETTING MILK ON FAT PERCENTAGE.

Number of tests, each comparison	Average per cent of fat when milk pipetted at a temperature of					
	68° F.	80° F.	68° F.	100° F.	55° F.	120° F.
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
12..............	4.500	4.500	4.346	4.300	4.541	4.458
Difference		.00		.05		.08

The data obtained show that the temperature at the time of pipetting the sample for the Babcock test is important. There was no measurable difference in the final fat test with a small difference of 12° (68° to 80° F.). There was a decrease in the test of 0.05 when the milk was measured at 100° F. instead of at 68° F. With a difference of 65° (55° to 120° F.), there was a difference in the average test of 0.08. The lowest test was obtained when the higher temperatures were used. This can be explained by referring to the coefficient of expansion of milk in accordance with its temperature. In accordance with the work done by the U. S. Bureau of Standards* when the volume of 4-per-cent milk at 68° F. is 1.0, it is 0.9975 at 50° F., 1.0020 at 80° F., 1.0040 at 90° F., 1.0065 at 100° F., 1.0085 at 110° F., and 1.0115 at 120° F.

This is evidence to show the importance of using a temperature at the time of measuring the charge of milk that is strictly in accordance with that specified by law.

(B). **Effect of a variation in the capacity of the graduated portion of milk test bottles on the fat percentage.** The present Oregon law† specifies

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* Bearce, H. W. Studies in the Expansion of Milk and Cream. Journal of Agricultural Research. Vol. III, No. 3, 1914.
† Section 41-703 and 41-711, Oregon Code, 1930.

that milk test bottles shall be tested for accuracy by Oregon State College and only those bottles can legally be used that do not vary more than 0.1 in the percentage (0.02 cc.) at any point in the graduated portion of the bottle neck. Some plant operators believe that more accurate bottles should be used.

Of a total of 524 bottles examined, using the certified mercury-calibrating burette as previously described, it was found that 46 per cent of the bottles had a capacity of the graduated portion of from 1.582 to 1.599 cc. inclusive and 54 per cent contained from 1.600 to 1.615 cc. inclusive. The theoretically correct capacity of the graduated portion of the 8-per-cent milk-test bottle is 1.600 cc.

In order to find out how significant a variation in the content of the graduated portion might be in influencing the results obtained, two sets of bottles, 12 to a set, were selected for this test. In one set were placed the bottles that were found to contain in the graduated portion from 1.585 to 1.595 cc. inclusive. In the other set were placed the bottles that contained from 1.605 to 1.615 cc. inclusive. By means of a 17.6 cc. pipette, well-mixed milk from the same sample of fresh milk was pipetted into each bottle in the two sets. The testing was done in accordance with the standardized procedure and the fat columns were measured, using the reading lamp and a magnifying glass, by two different men reading independently of each other. The experiment was repeated, using a different sample of milk containing a higher percentage of fat. The results obtained are shown in Table 14.

Table 14. EFFECT ON FAT TEST WHEN USING TEST BOTTLES OF VARYING CAPACITIES OF THE GRADUATED PORTION.

(Average test)

Comparison	Number of tests	Graduated portion 1.585 to 1.595 cc.	Graduated portion 1.605 to 1.615 cc.	Difference in average test
		Per cent	Per cent	Per cent
1	12	3.977	3.910	0.067
2	12	4.796	4.719	.077

There was an average difference of 0.067 in the percentage in the first comparison, and 0.077 in the second comparison. The tests were the highest with the bottles of the smaller neck capacity. This checks fairly well with the theoretical computation.

In order to determine if similar results could be obtained at another laboratory, the same test bottles were taken to the Central Testing Laboratory at Portland where the regular assistants made a similar comparison. The results obtained are shown in Table 15.

Table 15. EFFECT ON FAT TEST WHEN USING TEST BOTTLES OF VARYING CAPACITIES OF THE GRADUATED PORTION. TESTS MADE IN COMMERCIAL PRACTICE.

(Average test)

Comparison	Number of tests	Graduated portion 1.585 to 1.595 cc.	Graduated portion 1.605 to 1.615 cc.	Difference in average test
		Per cent	Per cent	Per cent
1	24	2.969	2.931	0.038
2	24	3.000	2.955	.045

The measurements were made to the nearest 0.1 per cent. It will be noted that the results obtained at the two laboratories were substantially similar.

(C). **The use of sulphuric acid of unknown or incorrect strength in testing milk.** Some operators of the Babcock test claim that it is possible to obtain accurate results when sulphuric acid of variable strength and temperature is used, provided the amount of acid added to the milk is varied to allow for the differences. This puts testing on a guess basis, and consistent and uniform results cannot be obtained. It was found in this study that if uniform, accurate results were to be obtained the strength and temperature of the acid should be properly standardized.

In Figure 7 are shown tests obtained with acid that had not been standardized for strength and temperature, and tests obtained when the acid had been standardized to the right strength and the correct temperature. The use of too strong acid caused charring of the fat. Inaccurate results were obtained.
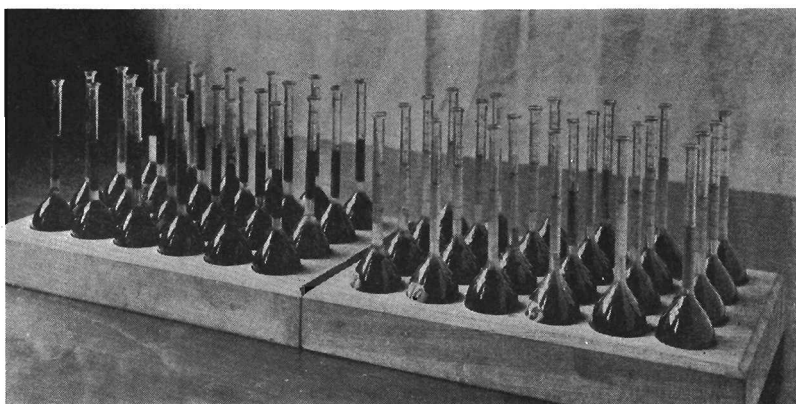
Figure 7.   Results from using sulphuric acid of correct and incorrect strength.   *Left:* Acid too strong.   *Right:* Acid correct strength, specific gravity 1.825.

A study was made of the sulphuric acid used by several dairy plants at Corvallis and in the dairy products laboratory at the College to determine the variation, if any, in the specific gravity and acid concentration (per cent H_2SO_4) of the different lots. Six different lots of acid were examined. Differences were slight. The per cent acid by weight varied from 93.24 to 93.70. The theoretical specific gravity calculated from assaying ranged from 1.834 to 1.838, while the specific gravity as determined by a hydrometer ranged from 1.835 to 1.838.

(D). **Effect of varying the speed of the centrifuge on the fat percentage.** The Babcock centrifuge is usually one of three different types; namely, motor driven, steam driven, or hand operated. The correct number of revolutions per minute that a centrifuge should make is dependent on the diameter of the wheel measured from the bottom of opposite cups when they are in an extended position. Variations from the correct speed may occur, however, either due to carelessness of the operator or to unavoidable causes, such as a sudden low steam pressure in the case of the steam turbine machine,

voltage drop in the case of the electric machine, and belt slippage in the case of belt-driven machines.

It was found by Babcock* that to obtain a maximum separation of fat it was necessary with a centrifuge wheel of a diameter of 18 inches to revolve this at seven or eight hundred revolutions per minute. The centrifugal force to which the contents of the test bottle were subjected was calculated to be 30.65 pounds per square inch. On this basis the speeds at which centrifuge wheels of various diameters should revolve were calculated.

Results obtained from the use of three different speeds in 12 determinations are shown in Table 16. The correct speed of the 20-inch wheel of the centrifuge used in this set of comparisons was, in accordance with the calculations, 759 revolutions per minute.† For convenience, 760 revolutions were used.

Table 16. EFFECT OF VARYING THE SPEED OF A CENTRIFUGE 200 R.P.M. ABOVE AND BELOW THE SPECIFIED SPEED ON THE FAT PERCENTAGE OF THE SAME SAMPLE OF MILK.

Comparison number	Specified R.P.M. standard (760)	200 R.P.M. decrease from standard (560)	200 R.P.M. increase from standard (960)
	Per cent fat	Per cent fat	Per cent fat
1	3.85	3.70	3.85
2	3.80	3.70	3.80
3	3.80	3.65	3.80
4	3.75	3.70	3.80
5	3.80	3.75	3.85
6	3.80	3.65	3.80
7	3.80	3.70	3.80
8	3.80	3.75	3.85
9	3.85	3.70	3.80
10	3.80	3.70	3.80
11	3.80	3.65	3.80
12	3.85	3.70	3.85
Average	3.808	3.696	3.817
Difference	......	—0.112	+0.009

It would appear from these data that the speed of the centrifuge is of extreme importance. A speed below that which is required for the diameter of centrifuge wheel had a very definite effect in lowering the fat percentage approximately 0.1 per cent. A speed above that required resulted in little difference from the normal.

(E). **Effect of varying the temperature of the air in the centrifuge during centrifuging on the final results.** A number of Babcock centrifuges in milk plants are steam driven. Occasionally these machines become very hot during centrifuging. If, contrary to state law, the fat columns are measured immediately after the bottles are removed from the centrifuge without first tempering the bottles and contents in a water bath maintained at the correct temperature, incorrect results may be expected. The effect of this on the fat percentage was therefore investigated.

In Table 17 are shown the results that were obtained when the readings of 24 tests from the same sample of milk were made with the fat at a temperature of 138° F. and at 180° F. An increase of 0.046 in the average

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* Farrington, E. H., and Woll, F. W. Testing Milk and Its Products, published by Mendota Book Company, Madison, Wisconsin. 26th ed., 1924.
† See Methods of the Association of Official Agricultural Chemists.

test was obtained when the fat columns were measured at the higher temperature.

Table 17. Effect of the Temperature of the Fat in Milk Test Bottles on the Test Obtained.

	Results of tests read before and after tempering in 138° F. water bath		
Number of tests	Read from tester at 180° F. (too high)	Read from bath at 138° F. (correct)	Increase in test due to reading at too high temperature
	Average per cent	Average per cent	
24	4.554	4.508	0.046

(F). Effect of the temperature of the fat at the time of measuring the fat column on the fat percentage. This series of comparisons involved the following: 24 different samples of milk were tested, and the test bottles after centrifuging were placed in a water bath, with the water at a temperature of 150° F., for a period of 5 minutes. The fat columns were then measured by means of a pair of needle-pointed calipers, using the illuminated reading lamp equipped with a magnifying lens as previously described. The test bottles were returned to the water bath immediately after the fat columns were measured. After the 24 bottles had been returned to the bath, the temperature of the water was reduced to 140° F. The fat columns were then again measured. This was repeated with water-bath temperatures of 130°, 120°, and 110° F. The results obtained are shown in Table 18.

Table 18. Effect of the Water Bath Temperature on the Final Fat Test.

	Average per cent fat when read from a bath maintained at				
Number of tests	150° F.	140° F.	130° F.	120° F.	110° F
	Per cent fat	Per cent fat	Per cent fat	Per cent fat	Per cent fat
24	3.950	3.904	3.894	3.893	3.891
Variation from test at 140° F.	+0.046	0.000	—0.010	—0.011	—0.013

Since a temperature of from 130° to 140° F. is standard, a higher reading would be obtained with a temperature higher than this and a lower reading would be obtained at a temperature lower than this.

(G). Effect of the length of time of holding completed tests in the water-tempering bath before measuring on the fat percentage. The Oregon law specifies that the fat column shall be measured at from 130° to 140° F. and at no other temperature. Tempering the completed tests in a water bath for 5 minutes may be considered the minimum time. An observation was made to determine the influence of holding the test bottles in the water bath for 5 minutes and for 1 hour before reading. The results obtained are shown in Table 19.

Table 19. EFFECT OF TIME OF TEMPERING THE FINISHED TESTS IN A WATER BATH ON THE FINAL FAT TEST

Group	Number of tests in each group	Fat column measured after tempering at 138° F. for	
		Five minutes	One hour
		Average per cent fat	Average per cent fat
1......	12	3.87	3.86
2......................	12	3.99	3.99
3......................	12	4.28	4.28
4......................	12	4.22	4.23

It will be observed that the average test was practically identical whether the samples had been held for 5 minutes or for 1 hour before the fat column was measured.

(H). **Effect of adding water at a different temperature to test bottles after second centrifuging on the final fat percentage.** When hot water at a temperature considerably higher than that used for tempering the finished tests in the water bath was used for adding to the test bottles before the last period of centrifuging, it was found that the fat column would be lowered from 1 to 1½ inches in the neck of the bottles during the subsequent tempering in the water bath. A series of 128 comparisons was made at Corvallis and at Portland in order to determine the effect of this on the readings. The results are shown in Table 20.

Table 20. EFFECT OF THE TEMPERATURE OF THE WATER ADDED TO TEST BOTTLES AFTER THE SECOND CENTRIFUGING PERIOD ON THE FINAL TEST. BOTTLES KEPT IN WATER BATH AT 138° F. FOR 5 MINUTES BEFORE MEASURING THE FAT.

Group	Samples tested	Average fat test. Last water added 145°-150° F.	Average fat test. Last water added 175°-180° F.	Increase due to fat migration
		Per cent fat	Per cent fat	
1 (Corvallis)......................	56	4.864	4.883	0.019
2 (Portland)........................	72	4.460	4.470	0.010
Downward migration of fat in necks (inches).....	....	(¾ to ½)	(1 to 1½)	....

The results obtained when the high-temperature water was used were on an average slightly higher than when the lower-temperature water was used. It was observed that when the high-temperature water was used, the meniscus at the bottom of the fat column was more concave than when the lower-temperature water was used. This would in part account for the higher reading that was obtained with the former.

(I). **Effect of the addition of glymol to the surface of the fat column on the fat percentage.** Because of the difficulty of locating the exact upper point of the meniscus on the surface of the fat column in the milk-test

bottle when reading, a comparison was made to determine to what extent the elimination of the meniscus would have on the reading. This was accomplished by the addition of glymol (a colored mineral oil) to the surface of the fat column.

In 36 comparisons it was found that the addition of glymol caused a reduction in the average test of 0.192.

It should be pointed out here that the addition of glymol is not permitted for milk tested under the provisions of the Oregon Babcock milk-testing law.

(J). **Agreement of duplicate tests made by the Babcock method.** The question often arises how closely duplicate tests made on the same sample of milk, tested in accordance with the Babcock method, agree.

The results obtained on 1,044 duplicate tests made in the study that involved a comparison of daily and composite testing are shown in Table 21. The standardized procedure was used in testing. The measurements for fat were made by the same person. It is seen that a variation of 0.1 in the percentage of the duplicates occurred with 18 of the 1,044 samples. This was the maximum variation obtained.

Table 21.   CHECKING DUPLICATE TESTS BY THE BABCOCK METHOD.   SPECIAL READING LIGHT USED.

	Number exact checks	Number that varied in the percentage		
		0.05	0.10	More than 0.10
1,044 duplicate samples tested..........	646	380	18	0
Per cent of total.......	61.9	38.4	1.7	0

The bottles used were accurate to within 0.005 cc. of the total correct capacity of 1.600 cc. of the graduated portion of the neck. The measurements were all read to the nearest 0.05 per cent.

An examination of the data obtained when the fat column in the duplicates was measured by a second person was also made. The readings obtained by the two persons in the majority of instances agreed exactly or within 0.05 in the percentage.   Variations of 0.1 between the readings of the individual tests by the two persons occurred 27 times with the 834 samples in which the fat columns in the completed tests were measured by two persons. The second reader obtained a higher reading 17 times and a lower reading 10 times.   No greater difference than 0.10 in the percentage in the 4 measurements from each sample of milk (each sample tested in duplicate and the fat measurements made by two persons) occurred.

DIRECTIONS FOR SAMPLING, PRESERVING, AND TESTING MILK FOR FAT BY THE BABCOCK METHOD

On the basis of the findings from the research reported in this bulletin, there is presented below a set of directions for the sampling, preserving, and testing of milk by the Babcock method. Accurately following these directions will result in the seller and buyer receiving as near equity as it is believed possible to obtain under practical commercial operation.   There is nothing in

these directions that conflicts with the provisions of the present Oregon law that governs the sampling and testing of milk, except as indicated.

The methods and procedures used by milk plants in determining the fat content of the thousands of cans of milk received daily from dairy farmers must be such that: (1) an accurate test is obtained, (2) the test is practical, and (3) the cost of each determination is not excessive.

Making composite samples over a period of 15 or 16 days and testing these by the Babcock method at the end of that period was the system used by Portland milk plants until the accuracy of this practice was questioned. With the establishment of the Portland Central Testing Laboratory the method of making composite samples for each 7- to 8-day period and storing the samples under refrigeration was adopted.

The method of making composite samples has some disadvantages. If not properly handled the milk may sour and even curdle, churning may take place if the milk is shaken too much, the milk may freeze, evaporation may occur, a tough cream layer may form on the surface of the milk, and mold may grow on the surface of the milk and on the upper inside surface of the bottle. Composite samples that are in a poor physical condition cannot be correctly tested.

It was found in this investigation that when composite samples were properly prepared and handled it was possible to maintain the milk in a satisfactory condition for testing. The method used was outlined under the heading of "standardized procedure in preparing and handling composite samples."

It is believed that the 7-to-8-day composite method of preparing composites, and storing and testing them in accordance with the directions given below, is a practical method to use.

SAMPLING, PRESERVING, AND STORING SAMPLES

1. **Sample bottle.** The 8-ounce sample bottle with tightly fitting rubber stopper should be used for the composite sample. The stopper should be chained to the neck of the bottle. The stopper should be replaced when it becomes hard or cracked. A permanent metal number plate for easy identification should be attached to the neck or stopper of the bottle. A sample bottle with a cylindrical body approximately 140 millimeters high, with bottom approximately 60 millimeters outside diameter, and opening in neck approximately 35 millimeters inside diameter, with gradual sloping neck from top of bottle to body for easy cleaning, is recommended.

2. **Stirring the milk.** Unless a satisfactory mechanical stirrer is used, the milk in each can must be thoroughly stirred by means of a hand stirrer before it is poured into the weighing vat. The milk must be poured out into the weighing vat within 5 minutes after it has been stirred. A stirring rod with a long handle and a perforated disk-shaped bottom should be used. The disk, if tinned steel or stainless steel, should either have a sanitary rolled edge or be made of a material that will not scratch the tin coating on the inside of the milk cans during stirring.

3. **Mechanical stirrer in milk-weighing vats.** If it can be demonstrated that a mechanical stirrer or other mixing device will satisfactorily stir the milk so that a uniform sample can be obtained, stirring the milk in the cans may be omitted.

4. **Sampling dipper.** A ¾-ounce capacity, cylindrical, flat-bottomed, non-corrosive metal dipper should be used to measure the milk for the composite sample.

5. **Sample bottle crates.** Suitable crates should be provided for the storing of the sample bottles containing milk.

6. **Obtaining the sample.** A ¾-ounce portion of each producer's milk from each delivery shall be transferred to the composite sample bottle. When the shipment consists of more milk than the weighing vat will hold, the milk shipment should be divided into two or more equal parts. By means of a dipper, an equal amount of the well-mixed milk is taken from each of these parts from the milk-weighing vat and placed in a clean, dry container. These portions are then thoroughly mixed and from this a ¾-ounce amount is then transferred to the composite sample bottle.

After the addition of each daily portion to the sample bottle the stopper should be placed tightly on the bottle and the bottle given a rotary motion until all of the cream has been thoroughly incorporated with the milk. The bottle may also be gently tipped in order to incorporate any moisture and cream that may have adhered to the inside top of the bottle or to the inside of the stopper. Rough handling must be avoided, otherwise churning will take place.

7. **Preservative used.** One mercuric chloride tablet of the No. 1 size, or equivalent, is sufficient for preserving a composite sample of first-quality milk made up over a period of 8 days. When the milk is of a poorer quality, two tablets may be necessary.

8. **Storing the composite samples.** The samples should be stored in a refrigerator at a temperature between 35° and 45° F. They must never be frozen. They should be removed from the refrigerator only long enough for the addition of the daily portions. In no case should the samples be allowed to remain out on the milk-receiving platform or elsewhere for more than 2 hours. They should be protected from radiating heat from steam pipes or heaters and from direct sunlight. The samples should be stored in a specially constructed cabinet provided with a lock. The keys to this cabinet should be in the possession of the licensed sampler and tester.

9. **Frequency of testing.** All samples should be tested at the end of each consecutive 7- or 8-day sampling period.

10. **Numbering bottles.** The bottles should be legibly numbered. Permanent metal numbers attached to the neck or stopper of the bottles are recommended. Identification numbers can also be placed on the bottles by pasting gummed labels on the clean, dry bottles and writing the numbers on the labels with India ink. After the ink has dried, a coating of clear varnish or shellac is applied over the numbers and label. Such a label will remain in good condition for long periods and will withstand washing in soapy water.

11. **Sampling abnormal milk.** No attempt should be made to sample churned, frozen, curdled, or otherwise abnormal milk.

GLASSWARE

All the glassware used in testing must conform to the specifications given in the Oregon State law.* It must be clean and in good condition before it is used. (The milk-measuring pipette should be graduated to hold 17.6 cc.)†

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* Even though the milk-test bottles conform to the standard required by law, slight variations between the results may be obtained on account of small variations in the content of the graduated portion of the neck.
† See Methods of the Association of Official Agricultural Chemists.

CENTRIFUGE

1. **Speed**. The centrifuge should rotate, when filled to capacity, at the correct speed. The centrifuge should not be permitted to vibrate. The following are the speeds that must be used.* They vary slightly from those given in the present state law.

SPEED OF CENTRIFUGE WHEEL.

Diameter of wheel in inches	Revolutions per minute
10	1,074
12	980
14	909
16	848
18	800
20	759
22	724
24	693

By diameter of wheel is meant the distance between the inside bottoms of opposite cups measured through the center of rotation of the centrifuge wheel, while the cups are horizontally extended.

2. **Speedometer**. An accurate speedometer should preferably be permanently installed on the centrifuge.

3. **Temperature of tester**. The interior of the tester during centrifuging should be maintained at a temperature of from 130° to 140° F. It is desirable that the tester be equipped with an accurate Fahrenheit thermometer, permanently attached, so that the temperature within the centrifuge can be observed at all times.

MISCELLANEOUS EQUIPMENT AND REAGENTS

1. **Acid dispenser**. The burette or measuring device employed to deliver suphuric acid should be constructed to allow a 17.5 cc. portion of the acid to be delivered to the test bottle accurately and without spilling.

2. **Temperature of the acid**. Some means should be provided whereby the sulphuric acid can be maintained at a temperature of from 60° to 70° F. at the time it is used for testing.

3. **Acid hydrometer**. An accurate acid hydrometer should be available so that the specific gravity of the acid used can be determined.

4. **Sulphuric acid**. The specific gravity of the sulphuric acid used should be 1.825 or 1.830 at 60° F. Sulphuric acid of a specific gravity below 1.825 should not be used. If the specific gravity of the acid is greater than 1.830 it

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* See Methods of the Association of Official Agricultural Chemists for correct speeds to use.

may be standardized by mixing it with distilled water. Usually only a small amount, about 1 or 2 cc. for each 100 cc. acid, is sufficient to lower the specific gravity to the density desired. Do not add the water to the acid, as this is dangerous. The acid should always be added to the water slowly. The temperature of the standardized acid should then be adjusted to 60° F. for determination of the specific gravity.

5. **Water bath for tempering composite samples.** A water bath for tempering composite samples before they are used in testing should be provided. When a considerable number of samples are to be tested, it is convenient that the tank be equipped with a thermometer and also with a regulating device for maintaining the temperature of the water at 60° to 70° F.

6. **Water for adding to the test bottles.** Distilled water or pure soft water must be used for adding to the test bottles.

7. **Hot-water tank.** A water tank should be provided for distilled or soft water used for adding to the test bottles. This tank should be constructed of nonrust material and equipped with an indicating thermometer. For convenience it may also be equipped with an automatic heating device for heating the water to and maintaining it at the proper temperature. Oil, rust, and other foreign material must be kept out of this water.

8. **Water bath for test bottles.** A water bath for tempering the test bottles and contents before measuring the fat column should be provided. This should be equipped with an indicating thermometer, preferably permanently attached. When a large number of determinations are made, it is desirable to equip this tank with an automatic heating device for maintaining a temperature of 130° to 140° F.

9. **Dividers or calipers.** The dividers or calipers used for measuring the fat column should be of the needle, or sharp-pointed type having a hinge joint and thumb-screw tightener.

10. **Reading lamp.** A lamp equipped with a magnifying glass is very convenient for making accurate fat measurements.

11. **Time alarm clock.** A time alarm clock or interval timer should be provided so that it is possible accurately to determine the length of the three centrifuging periods and also the length of time of keeping the test bottles in the water bath prior to measuring the fat column.

PREPARING THE MILK SAMPLES FOR TESTING

1. **Preparing composite samples.** With properly handled composite samples, ordinarily there should be no cream adhering to the side and stopper of the bottles after these have been gently rotated for a few seconds. The samples should be kept in a water bath with the water maintained at a temperature of 60° to 70° F. for at least ½ hour before the charge is transferred to the test bottle. If it is found, however, that some cream adheres to the side of the bottle and stopper after gently rotating them for a few seconds, the samples shall be handled as follows. Place the bottles in a water bath with the water at a temperature of not over 110° F. The samples should be kept in this bath until the temperature of the milk reaches 100° F. With a bath equipped with automatic tem-

perature control, this will probably require at least 30 minutes. The bottles should then be rotated until all of the cream from the sides and stoppers of the bottles has been removed. The samples are then placed in the water-tempering bath at from 60° to 70° F. and cooled to this temperature before they are used in testing.

2. **Preparing single samples.** When it is desired to test single samples of fresh milk, it is only necessary to place the samples in the water bath maintained at 60° to 70° F. for half an hour before the sample is used in testing.

DIRECTIONS FOR TESTING MILK BY THE BABCOCK METHOD

1. **Obtaining the charge for the test.** Remove the sample from the water-tempering bath and mix thoroughly by pouring from one container to another until six separate pourings have been accomplished. Care should be exercised to prevent foaming. The extra container used in mixing—a lipped glass beaker is satisfactory—should be inverted over a suitable screen to drain between measuring the different samples.

Immediately after mixing, by means of a standard milk-measuring pipette, transfer the charge of the milk to the test bottle. This is done in the following manner: With the pipette tip below the surface of the milk the pipette is filled until the milk passes about 1 inch above the mark on the neck of the pipette. Then with the pipette held above the bottle and with the mark on the pipette horizontal with the eyes, the milk is allowed to run out until the bottom of the meniscus of the milk in the pipette is even with the graduated mark on the pipette. This charge of milk without loss is allowed to drain into the test bottle by inserting all of the lower stem of the pipette inside the neck of the bottle. The last drop remaining in the tip of the pipette must be gently blown into the test bottle.

2. **Adding sulphuric acid to the milk.** Sulphuric acid of correct strength should be added to the milk in the test bottle and mixed as follows: When a single test is made, the full 17.5 cc. portion of acid is slowly added, without spilling, to the test bottle. The bottle is held in a tilted position during the addition of the acid and carefully rotated so that all milk is removed from the inside neck of the bottle by the acid. Care should be exercised to prevent undue shaking of the bottle during the addition of the acid. The acid and milk are then thoroughly mixed by rotating the bottle in a circular motion until all of the curd has been completely dissolved and the acid-milk mixture acquires a dark chocolate-brown color. When more than one test is to be made, the acid must be carefully added to each bottle before mixing. It is necessary that the acid and milk be of the correct temperature, from 60° to 70° F. The addition of the acid must be done with dispatch and skill, avoiding jostling or jarring the milk and acid before the mixing operation. The whole lot is then mixed at one time. This may be accomplished by placing the bottles in a wooden rack and shaking the rack with bottles in a rotary motion. When an automatic mixer is used the bottles can be placed in the mixer after the addition of the acid to each bottle, provided the mixer is kept operating continuously.

3. **Centrifuging.** The bottles should be centrifuged as soon as possible after the reaction between the milk and the acid has been completed. The centrifuge should be properly balanced and the temperature of the interior

should be adjusted to 130° to 140° F. before the bottles are placed in it. It should be maintained at this temperature at all times during the centrifuging. The length of time of centrifuging should be accurately determined by the interval timer. The centrifuging periods should be 5 minutes for the first, 2 minutes for the second, and 1 minute for the third.

4. **Addition of water to the test bottles.** Distilled or other soft water free from oil, rust, or other foreign material at a temperature of 140° to 150° F. should be added to the bottles after the first centrifuging period until the fat rises to the bottom of the neck. After the second period of centrifuging, water at the same temperature should be added until the contents of the bottle reach the 7- or 8-per cent mark on the graduated scale.

5. **Adjusting the temperature of the fat before measuring.** After centrifuging, the bottles should be placed in a water bath with the temperature of the water maintained at 130° to 140° F. The top of the water should extend above the top level of fat in the test bottles. All the bottles should remain in this water for not less than 5 minutes until the fat column is in equilibrium and the lower fat surface has assumed final form before the measurements are made.

6. **Measuring the fat.** The fat column should be measured in its entirety by means of the calipers from its lower surface to the highest point of the upper meniscus. The measuring should be done immediately after removing each bottle from the bath. Measuring the fat when test bottles are removed from the centrifuge will cause variable results and is contrary to the state law.

7. **Recording results.** The results should be immediately recorded on approved record blanks.

8. **Retesting.** Retests should be made when charred material or undissolved curd is present in the fat column, or when the fat column is milky or brownish in color. The ideal fat column has a translucent amber or golden-yellow color.

9. **Testing churned, frozen, or sour milk.** Milk that is churned, frozen, or sour should not be used for testing.

10. **Washing bottles used** for composite samples. All the sample bottles used should be washed free of all milk and fatty material, using a soap solution or other cleaning solution and a brush. They should be thoroughly rinsed in clean running water and should finally be sterilized by exposing them for at least 1 minute in a solution containing not less than 100 parts per million of available chlorine. The bottles must be dry before they are used for the milk samples. While not in use the clean sample bottles should be stored in a clean, dry place in an inverted position without the stoppers inserted.

11. **Cleaning test bottles.** The Babcock milk-test bottles should be thoroughly cleaned after the test has been completed. The following method of cleaning is recommended: The bottles should be emptied while the contents are still warm. The bottles should be shaken while emptying to remove the sediment from the bottom of the bottles in order to facilitate efficient cleaning. If a special bottle-washing apparatus is not available, the following method can be used satisfactorily. A clamp with holes, preferably made of wood, constructed to fit over the necks of the bottles is placed over the whole set of

bottles held in a rack. Using a locking device to hold the clamp over the bottles, the whole rack of bottles is submerged upright in a solution of alkali or in a soap solution containing from 4 to 6 ounces of soap powder in 2 gallons of water. The temperature of the solution should be 140° F. or above. As it is not necessary to place the hands in the water, it is possible to use this high temperature. A container for holding the cleaning solution should be constructed so that the necks of the bottles will be completely submerged. Filling the bottles about one-half full with solution, the rack with bottles is then removed, shaken, and emptied into the drain by inverting the whole rack at one time. This washing process is repeated for at least three times. Before emptying the bottles the last time, a test-bottle brush is used to remove any material that may still adhere to the inside of the necks. The bottles are then rinsed in soft, clear water, with the water at a temperature of 140° F. No soap or alkali is used in this water. All traces of cleaning solution must be removed from the bottles. The bottles are placed in an upright position for a few minutes until they have cooled. They are then placed in an inverted position on a rack for complete drainage. The cleaned bottles should be entirely free from fat or foreign matter and should have a clean, shiny appearance.

CAUSES OF VARIATION IN MILK TESTS

1. A curdy or milky fat column may be caused by one or more of the following:
  a. Insufficient acid.
  b. Weak acid.
  c. Too low temperature of acid.
  d. Incomplete mixing of milk and acid.
  e. Placing bottles in centrifuge before reaction is completed.

2. A dark or charred fat column may be caused by one or more of the following:
  a. Too much acid.
  b. Too strong acid.
  c. Too high temperature of acid or milk.
  d. Uneven mixing of acid and milk.
  e. Too long interval between mixing and centrifuging when the samples are not being continuously shaken.

3. Too low results may be caused by one or more of the following:
  a. Improper sampling of milk when received.
  b. Improper mixing of milk samples.
  c. Water in sample bottle when adding the sample.
  d. Sample handled so that it is churned or "oiled off."
  e. Moldy or sour sample.
  f. Inaccurate glassware.
  g. Improper measuring of milk into test bottles.
  h. Too low temperature of acid and milk. Some of the fat is held by the undissolved curd.
  i. Insufficient time of centrifuging.
  j. Insufficient speed of centrifuge.
  k. Measuring the fat column at a temperature below 130° F.

4. Too high results may be caused by one or more of the following:

a. Improper sampling of the milk when received.
b. Improper measuring of the milk for the test bottle.
c. Measuring the milk charge at temperatures below 60° F.
d. Inaccurate glassware.
e. Sample handled so that it is churned.
f. Loose stoppers on composite sample bottles, causing evaporation of water from the milk.
g. Too high centrifuging temperature.
h. Too hot water added to bottle in running tests.
i. Measuring the fat column at temperatures above 140° F.
j. Charred particles in fat column.

5. Moldy surface on milk samples may be caused by one or more of the following:

a. Using unsterilized bottles for storage of samples.
b. Insufficient preservative added.
c. Too high storage temperature.
d. Improper mixing of the daily portion and failure to incorporate the moisture and milk remaining on the side of bottle and stopper daily. This leaves part of the milk unpreserved.

6. Check testing. It is wise to make occasional check tests on the same sample of milk in order to judge one's ability to obtain consistent results. Duplicate analyses varying by more than 0.1 in the per cent fat should be rejected and a retest should be made.

IMPORTANT POINTS IN TESTING MILK

The State law that governs testing milk, and methods of the Association of Official Agricultural Chemists give certain requirements that must be observed when testing milk. They are:

1. Standard glassware.
2. Sampling milk. Preparing, preserving, and storing composite samples.
3. Speed of centrifuge wheel and speed indicator.
4. Temperature of tester during centrifuging.
5. Hot water bath.
6. Measuring calipers.
7. Specific gravity of sulphuric acid and size of acid measure.
8. Temperature and condition of milk when measuring sample for testing.
9. Method of adding acid and mixing it with the milk.
10. Method of whirling the test bottles.
11. Temperature of water to add to test bottles.
12. Tempering finished tests in water bath.
13. Measuring the fat column.

Elaborate equipment is not necessary in order to observe the above. When a large number of determinations are to be made it will be found desirable to use certain specially constructed apparatus equipped with automatic control devices.

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