A STUDY OF THE ACCURACY OF TESTING MILK FOR BUTTER FAT USING SAMPLES WITH AND WITHOUT CHEMICAL PRESERVATIVES[1]

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(Received for publication September 25, 1961)

The accuracy of various sampling methods in raw whole milk received at a milk processing plant from dairy farms was compared. Milk samples were collected daily during four 30-day periods representing approximately the four seasons of the year. The Babcock test for butterfat was applied to the following: daily samples, 6-day composite samples without added preservative, seven and 15-day preserved composite samples and periodic samples taken three, four and five times per month. The preservatives, Milkeep and mercuric chloride, were compared in the seven and 15-day composite sample. The daily test, recognized for its accuracy, was used in statistical comparison with the test results from the other samples. When the tests from each sampling method were averaged for the four seasons, no significant differences were found. Tests from the 6-day composite samples and the periodic samples taken five times per month were identical to the daily test. Tests from composite samples preserved with Milkeep averaged closer to the daily test than did those preserved with mercuric chloride in both the seven and 15-day composite samples.

Milk plant operators buy milk from dairy farmers at a price which usually varies according to the fat content of the milk. The per cent fat is determined at the milk plant laboratory by a suitable method such as the Babcock test. In order to save on the cost of testing milk at each delivery, a composite or periodic sampling technique is commonly used. By these methods, only one sample is tested over a period of time, as compared to the necessity of testing a sample at each delivery.

A composite sample is made by taking a sample from each delivery and placing it in a sealed container. The daily samples are combined with the portions from the previous deliveries. The composite sample may cover a period from 5 to 15 days. The per cent fat in the composite sample is applied to the total amount of milk delivered during the period. A periodic sample is one that is taken on designated days and usually tested the same day. These methods, therefore, have been popular from economic, labor-saving standpoints.

Preservative chemicals may be used in the composite sample to retard or prevent microbiological deterioration during storage. In recent years, however, more extensive use of refrigeration and new preservatives has indicated that the accuracy and efficiency of sampling methods may be improved.

Early reports on the accuracy of composite samples were by Farrington *(6)* and Cooke and Hills *(3)*. Judkins *(9)* and Farrington *(6)* found that composite samples tested higher than daily samples. Others *(2, 4, 5, 7, 8, 10, 12,13, 14, 15, 16)* have found that the fat test of composite samples were generally lower than the daily test. Many of the earlier reports lacked a statistical treatment of the data and adequate control or inclusion of certain experimental variables. For example, there is a lack of published information comparing the accuracy of testing by using composite samples stored with and without preservatives and periodic tests.

This study was designed to evaluate the relative accuracy and precision of the Babcock test for butterfat in raw whole milk received at a milk processing plant from a dairy farm. The fat test was applied to composite samples with and without added preservatives and results were compared with those obtained by daily analysis. Results from these were also compared to periodic tests taken at intervals of three, four and five times per month.

METHODS

In this study, milk samples were collected daily during four 30-day sampling periods representing approximately four seasons of the year. The periods were February 20 through March 21, April 14 through May 13, June 13 through July 12 and September 20 through October 19. Ten producers of Grade A raw milk were randomly selected from the Salt River Valley (Arizona) milkshed. Using approved commercial procedure, the tank truck driver collected the samples daily from the farm bulk tank of each producer. The samples were then placed under refrigeration in shaved ice and shipped via milk transport to the milk plant in Tucson. They were then taken to the University of Arizona for testing in duplicate by the standard Babcock method *(1)*. Each daily sample was analyzed for butterfat on arrival at the University.

A 9-ml portion of the daily sample was used to prepare each of the composite samples which con-

[1]A grant from the Arizona Dairymen's League aided in the financial support of this project. In Januray, 1960, a merger of the Arizona Milk Producers and the Arizona Dairymen's League brought about a new organization which is now called the United Dairymen of Arizona.

sisted of two 7-day preserved composite samples, two 15-day preserved composite samples and one 6-day composite sample without a preservative. The composite samples were placed in glass 8-oz plastic screw cap containers and were kept at 35-38°F.

The preservatives compared were Milkeep[2] and mercuric chloride. The mercuric chloride used contained 45.9% bichloride of mercury and the Milkeep contained 100% active ingredient by the manufacturers' labels. In the 7-day preserved composite samples, one tablet of mercuric chloride and one tablet of Milkeep was used in the appropriate containers. In the 15-day composite samples, two tablets were used. The labels indicated that the mercuric chloride and Milkeep tablets weighed 356 and 325 milligrams, respectively.

For comparison with the daily and composite tests, periodic tests were used at intervals of three, four and five times per month. The daily tests corresponding to the appropriate days were used. For three periodic tests a month, the tenth, twentieth and thirtieth days were used. In the case of four periodic tests, the days used were the seventh, fourteenth, twenty-first and twenty-eighth. For five per month, the sixth, twelfth, eighteenth, twenty-fourth and thirtieth days were used.

Daily milk weights were taken by the tank truck driver at the farm bulk tank. The manufacturer supplied an individual stainless steel measuring rod and calibration chart for each tank.

RESULTS AND DISCUSSION

The results of this study are summarized in Table 1. To make a comparison of the various methods for all seasons a column of averages is included. Statistical analysis using the Multiple Range Test to group means with unequal number of replications *(11)* indicates no significant difference among the averages of the five sampling and testing methods. It was observed that the variance of the fall sampling period was greater than the variance of the other sampling periods. This is possibly due to the fact, that, in Arizona, many cows freshen in the fall of the year. During the first two weeks of the lactation period, milk has a characteristically higher per cent fat than during the rest of the lactation period.

Small variations were evident within the seasonal sampling periods. In comparing the tests from 7-day composite samples taken during the summer sampling period, the mercuric chloride sample was found to have a significantly lower test than either the Milkeep sample or the daily sample. During the same period the 6-day composite sample stored without preservative tested significantly lower than the daily sample. The periodic sample taken four times per month showed a significantly higher test than the periodic sample taken either three or five times per month in the summer sampling period. The variations that occurred in the summer sampling period only might be explained from the standpoint of higher environmental temperature. Many times this temperature was 90°F or higher. There were indications that during shipment the ice did not last long enough to keep the samples from warming to the point of partial fat churning. When this phenomenon occurred particles of churned fat clung to the sides of the sample bottle. Even by using recommended procedures of sample preparation it was difficult to bring the fat into complete, uniform suspension.

From the results it appears that, under normal conditions, the most economical method would be by periodic sampling and testing three times per month. Since accuracy as well as economy of testing is a factor, both the periodic samples taken five times per month and the six-day fresh composite samples most closely approximated the true per cent fat obtained by daily analysis. While there is no difference in the number of actual tests needed, there is a difference in sampling time required due to the procedure. With the composite method, a sample must be collected and placed into a container,

TABLE 1—AVERAGE PERCENT BUTTERFAT FOR COMPOSITE SAMPLING WITH AND WITHOUT PRESERVATIVES COMPARED TO PERIODIC AND DAILY SAMPLING

Type of sample	Sampling period				
	Fall	Winter	Spring	Summer	Average
	(%)	(%)	(%)	(%)	(%)
7-day preserved composite ($HgCl_2$)	3.72	3.64	3.61	3.79[a]	3.69
7-day preserved composite (Milkeep)	3.75	3.63	3.62	3.84	3.71
Daily	3.74	3.63	3.63	3.85	3.71
15-day preserved composite ($HgCl_2$)	3.70	3.62	3.59	3.82	3.68
15-day preserved composite (Milkeep)	3.74	3.61	3.61	3.85	3.70
Daily	3.75	3.63	3.63	3.85	3.72
6-day composite (Not preserved)	3.74	3.65	3.66	3.81[b]	3.72
Daily	3.75	3.63	3.63	3.85	3.72
Periodic taken:					
3 per month	3.82	3.65	3.63	3.84	3.74
4 per month	3.80	3.64	3.61	3.91[c]	3.74
5 per month	3.78	3.62	3.64	3.85	3.72
Daily	3.75	3.63	3.63	3.85	3.72

[a]Significantly lower than the other two values at the 1% level.
[b]Significantly lower then the daily sample at the 1% level.
[c]Significantly higher than the other three values at the 1% level.

[2]The chemical composition of this preservative is reported to be disodium phosphate-3, 5-dimethyl tetrahydro-1, 3, 5, 2H-thiadiazine-2 thione.

agitated and refrigerated each day for a period of six days and then tested. This involves a certain amount of labor in taking and handling the samples. The five times per month periodic method requires sampling and testing every sixth day only. A comparison of the 6-day composite samples and the periodic samples taken five times per month shows quite readily that, from the cost and convenience standpoint, the periodic sample taken five times per month would be more favorable.

CONCLUSIONS

In this study, fat tests were compared using daily samples, 6-day composite samples without preservative, seven and 15-day preserved composite samples and periodic samples taken three, four and five times per month. The results show that:

1. There was greater variability in the summer sampling period than during other sampling periods. In the summer sampling period the average test of the 7-day composite samples preserved with mercuric chloride was significantly lower than either the 7-day composite sample preserved with Milkeep or the daily sample. Also in the summer sampling period, the average of the 6-day composite samples without preservative tested significantly lower than the daily samples. The periodic samples taken four times per month tested significantly higher than the daily sample in the summer sampling period.

2. There were no significant differences between various values in the fall, winter or spring period.

3. The larger variance found in the fall sampling period is thought to be due to the freshening of a larger number of cows than is the case during the other periods of the year in Arizona.

4. In averages taken from the four sampling periods, none of the differences were large enough to be significant.

5. The tests from daily samples were consistently higher than the tests from both the seven and the 15-day preserved composite samples in all sampling periods.

6. Seven and 15-day composite samples preserved with Milkeep consistently tested higher than seven and 15-day composite samples preserved with mercuric chloride.

7. Average tests from 6-day composite samples without added preservative agreed exactly with tests from daily samples.

8. Periodic samples taken both three and four times per month tested consistently higher than daily samples in all sampling periods.

9. The average test from the periodic samples taken five times per month were identical to the average test of the daily samples.

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