FREEZING POINT OF BUTTERMILK AS A MEASURE OF ADDED WATER IN CREAM

The official method (Association of Official Agricultural Chemists 1950) for the determination of added water in cream is similar to the method used for milk except that the percentage of added water is calculated by the formula:

$$\text{Added water} = \frac{\text{Percentage serum in cream} \times (T - T_1)}{T}$$

where T = f.p. of undiluted cream ($-0{\cdot}550°C$)

T_1 = observed f.p. of given sample

Percentage serum = 100% − (% fat + % protein)

Owing to the high viscosity of some creams, this determination can be difficult and unreliable results may be obtained. Doan (1927) showed that the freezing point of cream and skim-milk was the same as the original milk from which the two constituents were derived. He noted when making the readings a relative sluggishness of the mercury thread in comparison with the movements with milk. Shipe (1959) also observed differences in the apparent freezing rates for cream and milk. His results suggest that the rate is related to viscosity. Shipe also pointed out that in practice the analyst should be sure to wait long enough after seeding the samples to insure that the maximum temperature has been reached.

Kendall and Leedham (1959) suggested that a possible solution to the problem was to churn a sample of cream and to determine the freezing point depression on the resultant buttermilk.

No attempt has been made to correlate the freezing point of buttermilk produced from cream separated from milk known to contain added water. In order to test the accuracy of this method of determining added water in cream, trials were carried out using milks and creams containing known amounts of added water.

Methods

Bulk milks known to be free from added water were used in 40-gal quantities during the first stage of the trials. The first 20 gal were separated and various amounts of water were then mixed with the remainder, which was subsequently separated. Representative samples of the original milks as well as the adulterated milks, creams and skim-milks were taken.

In the second stage of the trials, cream from unwatered wholemilk was adulterated with known amounts of water and samples were taken. Precautions were taken during sampling and testing to avoid factors likely to affect the results.

The freezing point depression was determined in duplicate for all samples and where necessary the samples were analysed for fat, solids-not-fat, and protein contents. The buttermilk for freezing point tests was obtained by churning 250 ml of cream at 7–10°C with a mixer until there was distinct separation of the buttermilk.

Results and Discussion

The average freezing points of samples of wholemilk, skim-milk and buttermilk are shown in Table 1. These samples were derived from bulk wholemilk which had been diluted with various concentrations of water prior to separation. The results indicate that the freezing point of skim-milk and buttermilk from the cream is similar to that of the wholemilk from which it is derived.

TABLE 1

COMPARISON OF THE FREEZING POINTS OF ADULTERATED WHOLEMILK WITH THOSE OF THE SKIM-MILK AND BUTTERMILK

Approximate Percentage Added Water	No. of Samples	Average Freezing Point Depression (−°C)		
		Wholemilk	Skim milk	Buttermilk
0	24	·548	·548	·548
1	3	·542	·542	·541
2	4	·536	·536	·536
3	7	·531	·531	·531
4	2	·521	·523	·523
5	5	·516	·517	·517
10	7	·487	·487	·487
15	5	·456	·455	·456
20	2	·427	·427	·425

Table 2 shows the freezing point depressions of buttermilk obtained from creams of known fat content which were adulterated with known percentages of water by weight. The percentage of extraneous water as calculated by the official formula is also shown with the freezing point.

The results show the calculated amounts of added water to be close to the actual figure, the average algebraic variation being 0·5 per cent. It is mentioned that the results were calculated with $T = -0{\cdot}550°C$ and not the actual freezing point of the original milk. This would be necessary from a practical aspect when analysing unknown samples of cream.

As the solids-not-fat content of the serum of the cream can be taken with sufficient accuracy as the solids-not-fat content of the buttermilk, and as the freezing points of the milk, skim-milk, and cream are dependent on the concentration of soluble substance in the serums, the results in Table 1 are to be expected. Although the milk prior to separation contained known concentrations of water, no attempt was made to determine the amounts of added water in these creams because the amount of serum in the skim-milk and cream varies considerably as the richness of the cream increases.

Doan's work was limited to creams of low fat content. These do not present as great a difficulty as higher fat creams.

TABLE 2

FREEZING POINT DEPRESSION (−C°) OF BUTTERMILK AND THE CALCULATED AMOUNT OF ADDED WATER IN ADULTERATED CREAM

Fat Percentage of Original Cream	——	Actual Percentage Added Water by Weight											
		1	2	3	4	5	6	7	8	9	10	15	20
58	f.p. dep.	·526	·517	·494	·488	·467	·460	·439	·435	·422	·417	·369	·347
	calc. water	1·6	2·4	4·2	4·7	6·4	7·0	8·8	9·3	10·5	11·1	16·1	19·1
53	f.p. dep.	·539	·524	·511	·507	·485	·476	·462	·452	·448	·437	·390	·360
	calc. water	0·8	2·0	3·2	3·6	5·5	6·4	7·7	8·7	9·1	10·2	15·3	19·1
45	f.p. dep.	·535	·529	·517	·503	·494	·483	·474	·465	·459	·455	·407	·358
	calc. water	1·0	1·9	3·1	4·6	5·5	6·7	7·7	8·6	10·4	10·9	15·5	21·6
41	f.p. dep.	·533	·529	·521	·507	·498	·487	·483	·476	·466	·460	·419	·380
	calc. water	1·6	2·0	2·9	4·4	5·4	6·7	7·2	8·0	9·3	9·9	15·0	20·2
38	f.p. dep.	·536	·531	·522	·518	·502	·492	·486	·478	·471	·456	·425	·385
	calc. water	1·3	1·9	2·9	3·3	5·1	6·3	7·1	8·0	8·8	10·5	14·9	20·1

This method of determining the freezing point of the buttermilk from the cream is a useful one as it is practical and determines with certainty watered cream. The magnitude of temperature rise with the addition of water gives a good approximation of the proportion of added water in cream.

REFERENCES

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS (1950).—"Methods of Analysis of the Association of Official Agricultural Chemists". 7th Ed. (Association of Official Agricultural Chemists: Washington).

DOAN, F. J. (1927).—Some observations on the freezing point of cream and its use in detecting added water. *J. Dairy Sci.* 10:353-60.

KENDALL, V. W., and LEEDHAM, L. F. (1959).—A simple control method for the detection of water in fresh cream. Proc. 15th Int. Dairy Congr. 3:1531-3.

SHIPE, W. F. (1959).—The freezing point of milk. A review. *J. Dairy Sci.* 42:1745-62.

V. C. TUCKER,
Queensland Department of Primary Industries.

(Received for publication April 15, 1964)