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Methods of Determining the Total Solids in Fluid Milk

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OKLAHOMA A&M COLLEGE DIVISION OF AGRICULTURE
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There is continued interest in developing methods for determining the total solids content of milk which are accurate but simpler and faster than the methods now in use. This interest is evidenced by a number of publications on the subject which have appeared in recent years (1, 2, 5, 6, 10, 12).

This publication presents an evaluation of two methods for determining the total solids content of milk which seemed promising: (a) A drying procedure using a Cenco moisture balance and (b) a direct titration method using the Karl Fischer reagent. The Mojonnier procedure was used as the control.

Preliminary reports using portions of these data were published by Loewenstein (7, 8).

Procedure

Two trials were conducted during the course of this experiment. For Trial 1, 1448 daily composite samples were collected from 63 Holstein cows over a 20 month period of time. The percent of total solids in these samples was determined with Mojonnier and Cenco equipment.

Single determinations were run on most of the samples, however, duplicate or triplicate determinations were run on 187 samples with the Cenco moisture balance and 111 samples with the Mojonnier. The duplicate and triplicate samples were taken at random.

One quart of homogenized milk was sampled for Trial 2. Forty replicate total solids determinations were run from it by each of three methods; the Mojonnier, the Cenco and the Fischer titration. Two days were required to finish all 40 determinations on the Mojonnier and Cenco equipment and three days were required to finish 40 replicates by the Fischer Method.

The procedure used to determine the total solids content of milk with the Cenco Moisture Balance was as follows (7):

1. Place an 11 cm. filter paper on the sample pan and a cotton filter disc on top of the filter paper.

2. Put the sample pan on its support in the balance, close the lamp housing, adjust the voltage output from the variable transformer to 100 volts, and move the toggle switch to the "on" position.

3. Leave the infra-red lamp on until the pointer comes to rest indicating that the sample bed is at constant weight. Rotate the knurled wheel so that the 100% line on the graduated scale is lined up with the index line. Move the zero adjusting knob up or down so that the pointer is in line with the index line, then loosen the zero adjusting knob and let it rest on the bottom of its slot. The instrument is then prepared to receive the sample.

4. Move the toggle switch to the "off" position, open the lamp housing, and while the sample bed is cooling, rotate the knurled wheel until the 0% line on the graduated scale is lined up with the index line. Allow about one minute for the sample bed to cool, then add approximately 5 g. of properly prepared sample to the absorbent cotton mat, distributing it evenly over the entire surface. The exact amount of sample is that amount required to line-up the pointer with the index line.

5. When the sample is weighed, close the lamp housing, move the toggle switch to the "on" position, and rotate the knurled wheel until the 80% line on the graduated scale is lined up with the index line. This keeps the sample pan level and allows for even drying.

6. In 4 to 4½ minutes, the movement of the pointer will indicate that 80 % of the initial sample weight has been evaporated as moisture. At that time reduce the voltage from the transformer to 90 and allow drying to continue until complete i.e., until the pointer remains motionless for ½ to 1 minute. During the final stages of drying continuously adjust the position of the pointer to line up with the index line by rotating the knurled wheel.

7. When the sample is dried read the percent of moisture from the graduated scale opposite the index line.

For the direct titration method, two pellets of NaOH were added to 5 g. of milk and the mixture was diluted to 100 ml. with absolute methanol. A 5 ml. aliquot of this solution was titrated (11) with the Karl Fischer reagent using a Fisher titrimeter.

Except where otherwise noted, statistical analyses of these data were conducted according to procedures outlined by Snedecor (9).

Results and Discussion

In Trial 1, mean values of 12.39 and 12.25% total solids were obtained for the Mojonnier and Cenco procedures, respectively. The results obtained by these two methods were statistically different. However, the 95% confidence limits on this difference were 0.11 to 0.17%, which was less than the smallest graduation on the scale of the Cenco moisture balance (0.2%).

Variances were calculated for the duplicate determinations and for all samples (Table I). The total variance of all the samples was due mainly to differences in the composition of the samples (3). Differences due to methods, different operators, sampling techniques and methods of sample storage also were included in this value which is quite large. The variance obtained from the duplicate and triplicate determinations included only the errors due to sampling, technique, and the balance itself. The Mojonnier variance was larger than that of the Cenco method when all 1488 samples were considered. However, the situation was reversed for the duplicate samples.

In Trial 2, values obtained by each of the three methods were not statistically different, at the 95% level, when all 40 replicates were considered. There was a significant difference, at the 95% level, between methods for the February 12 data but no statistically significant differences for the February 15 data (Table II).

For each method, there was a statistical difference between the values obtained on February 12 and those obtained on February 15, 1955. This difference was significant at the 95% level for the Mojonnier values and at the 99% level for the Cenco and Fischer values. Confidence limits of the differences between the means obtained for each method are recorded in Table III.

Table I.—Variance of Mojonnier and Cenco methods—Trial 1.

	Mojonnier	Cenco
	(%)	(%)
Duplicate Determinations (187 Cenco, 111 Mojonnier)	0.033	0.172
All Samples (1448)	1.320	1.250

Table II.—Mean Total Solids Values — Trial 2.

	Mojonnier	Cenco	Fischer
	(%)	(%)	(%)
February 12	12.76	13.17	12.13
February 15	12.86	12.87	13.17
All Samples	12.80	13.05	12.83

The variances for each method pooled over the two days were: 0.014, 0.007 and 0.382 for the Mojonnier, Cenco and Fischer methods respectively.

The average time required to run one determination was determined for each of the three methods. The Cenco method required an average of 11 minutes per sample, the Mojonnier method took 20 minutes per sample, and the Fischer titration required 24 minutes per sample.

The variances obtained for the Cenco and Mojonnier methods in Trial 2 were used to calculate the differences from the true mean which would be expected on any sample when various numbers of replicate determinations were run. These values are shown in Table IV along with the labor and reagent cost for various numbers or replicate determinations.

An effort was made to explain the differences between days in Trial 2. Several trials were run on the Cenco machine using a quart of homogenized milk as the sample. The two Cenco machines used in this work were tested to determine if they gave different results, and the effects of changing transformers and light bulbs were evaluated. None of these variations caused any significant differences in the results obtained providing the equipment was used according to the directions given on procedures page 4.

However, changing the voltage output of the variable transformer from that recommended in the directions did cause significant differences in the results obtained. It was observed that large differences (0.2-0.3%) also could be caused by errors in reading the graduated scale of the Moisture Balance. This may be caused by the operator not observing the scale from the same angle each time a reading was taken. Errors also resulted when the milk was unevenly distributed on the sample bed, when the sample was burned, or when it was weighed incorrectly.

It was impossible to repeat trial 2 since the sample used during those four days could not be duplicated. Therefore, any explanation of the differences between days in this trial, of necessity is somewhat theoretical. However, most of the things which could cause differences in the Cenco results are directly or indirectly controlled by the operator. In view of this, it would seem that the Cenco machine requires as skilled an operator as does the Mojonnier.

Table III.—95% Confidence Limits on the Differences Between Means—Trial 2.

	Mojonnier—Cenco	Mojonnier—Fischer
	(%)	(%)
February 12	0.27 to 0.55	—0.48 to —0.78
February 15	0.38 to —0.36	0.73 to —0.11

This work also indicated that it was necessary periodically to standardize the Cenco instrument against a procedure giving results of known accuracy. This was done by adjusting the voltage output of the variable transformer until the Cenco results agreed with those of the standard procedure. The voltage setting used throughout this work was determined by preliminary trials using the Mojonnier as the standard.

The results of this study seem to substantiate Heinemann's report (5), which indicated that the Fischer titration was not satisfactory for products containing more than 20% moisture.

Summary

Two methods of determining the total solids content of fluid milk were evaluated: A drying procedure using a Cenco Moisture Balance and a direct titration method using the Karl Fischer reagent. The Mojonnier procedure was used as a control.

The data indicated that the Fischer titration required more time and had a larger variance than did the Mojonnier procedure. In addition, the results of the Fischer titration did not agree as closely with those of the Mojonnier as did the results of the Cenco procedure.

The Cenco procedure, on the other hand, required less time and was less costly than the Mojonnier method. Its results, in most cases, were reasonably close to those of the Mojonnier and the variance of this procedure was usually less than that of the Mojonnier.

Table IV.—Labor and reagent cost* and differences from the true mean obtained with selected numbers of replicate total solids determinations on the Mojonnier and Cenco Moisture Balance — Trial 2.**

Replicate Determinations	Mojonnier		Cenco	
(N + 1)	Difference	Cost	Difference	Cost
	(%)	($)	(%)	($)
2	1.80	0.77	1.23	0.42
4	0.28	1.53	0.19	0.83
6	0.18	2.30	0.12	1.25
8	0.14	3.07	0.09	1.67
10	0.11	3.83	0.08	2.08

* Labor calculated at $1.00 per hour. Average time per sample was 20 minutes and 11 minutes for the Mojonnier and Cenco respectively.

** Formula (4): $\frac{d^2}{s^2} = \frac{t^2_{(n)} \quad F_{(n,39)}}{n+1}$

d = Difference of actual mean from the true mean.

$t_{(n)}$ = Student's 5% t value with n degrees of freedom.

$F_{(n,39)}$ = Snedecor's 10% F value with n and 39 degrees of freedom.

n + 1 = Number of samples necessary.

s^2 = Variance estimate based on 39 degrees of freedom. Pooled daily variances of 0.0066 and 0.0141 used for the Cenco and Mojonnier respectively.

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