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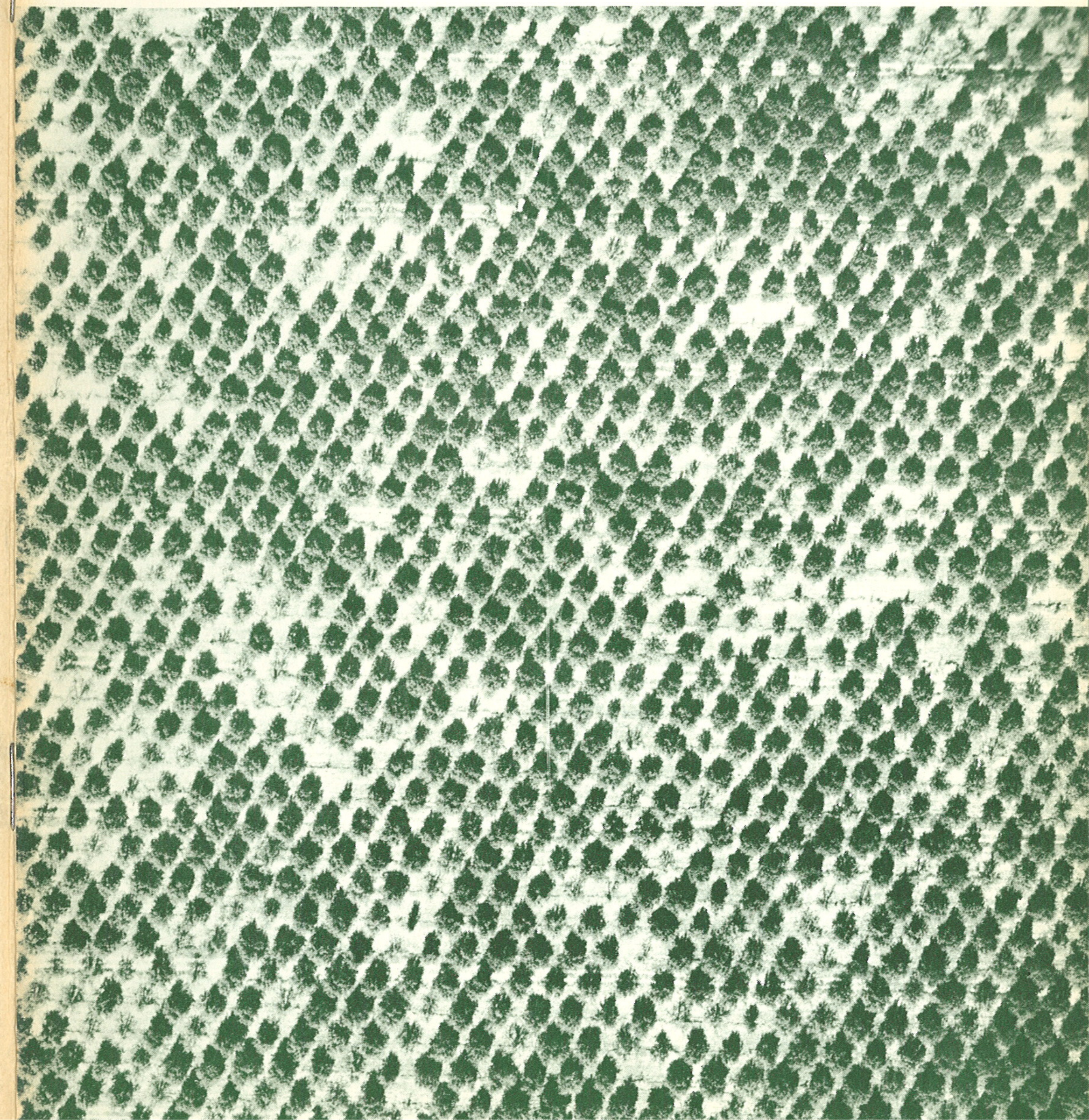

fat component of milk.

The Many Ways of Measuring Solids-Not-Fat In Milk

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The 1961 session of the California Legislature passed a bill which, among other things, provides that the solids-not-fat content of market milk will be a factor in its pricing structures. This bill, now law, has focused the attention of the dairy industry on a problem which it has long considered but never before has implemented.

The fat content of milk has long been the chief yardstick by which its value has been measured. Today increasing emphasis is being given to all milk nutrients, so that the breeder, the dairy farmer, the milk processor, the nutritionist, and the consumer are each interested in both the fat and the solids-not-fat in milk. The solids-not-fat are composed of the protein, milk sugar, and minerals in the milk. An accompanying article in this issue of The Bulletin discusses economical aspects relating to solids-not-fat in milk; this paper will deal with methods used to measure the solids-not-fat content.

Whenever the results of his work are to be used to determine the payment for the product, a person who tests milk for its solids-not-fat content must hold a valid nonfat milk solids tester's license issued by the Bureau of Dairy Service. The procedure used must be one approved by the Bureau of Dairy Service. If a lactometric method is used, the lactometer must be of certified accuracy, as described later in this paper.

An accurate fat test is an essential factor in the calculation of the solids-not-fat. Since accurate and rapid routine fat tests are well known, they will not be discussed here.

The basic method for solids testing is the gravimetric procedure, as outlined in the Official Methods of the Association of Official Agricultural Chemists.[10] This requires chemical laboratory equipment and a skilled technician. It is in reference to this method that the accuracy of all other procedures is compared. In the "A.O.A.C." method a known weight of milk, about 2.5 grams, is placed into an accurately weighed flat-bottomed dish, heated on a steam bath for 10 to 15 minutes and then placed in an air-oven held at 98°-100° C. This procedure drives off the water, leaving the milk solids. After the dish has cooled to room temperature, it is weighed and the percentage of milk solids is calculated. The fat percentage, previously determined, is subtracted from the total solids and the remainder represents the milk solids-not-fat.

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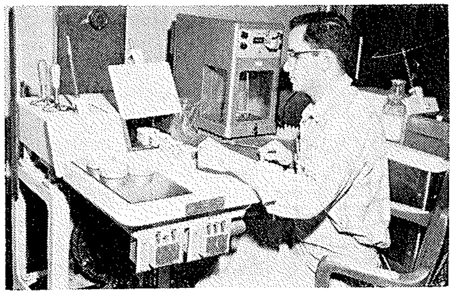


Chemist, M. Monier, making total solids tests on the Mojonnier equipment.

This drying procedure takes too long for routine use. A modified method, devised by T. Mojonnier about 50 years ago is more commonly used by the dairy industry. Here, about two grams of milk are weighed into an aluminum dish and dried to a uniformly light-brown color on a hot plate heated to 180° C. The dried dish is placed for 10 minutes in a vacuum oven held at 100° C. The dish is then cooled to room temperature, weighed and the percentage of total solids calculated.

Other gravimetric procedures have been used to a limited extent. The Dietert-Detroit Moisture Determinator is used in some dairy laboratories in place of the more expensive Mojonnier apparatus. It employs hot-air circulation to dry the sample, and will take four samples at a time, whereas the Mojonnier equipment will dry eight in about the same time. The Brabender Moisture Tester which will handle 10 samples in about 45 minutes, is used in some laboratories, but it does not have the accuracy of the methods just described. It combines a drying oven with a balance which has an accuracy of 0.002 gram, whereas the analytical balance used with other tests will weigh to 0.0001 gram. A different type of moisture-testing apparatus is represented by equipment, such as the Cenco Tester and the Ohaus Moisture Determination Balance, which combine a balance and an infrared drying unit. These instruments handle but one sample at a time, and do not have the accuracy of methods which employ an analytical balance.

Published data show that the A.O.A.C. method and the Mojonnier procedure have essentially the same degree of precision; the other methods are more variable and tend to give results which usually are from 0.1 to 0.2 percent lower than the A.O.A.C. method.

Methods that involve a chemical reaction are useful only for products of comparatively low moisture content and so are not applicable to fluid milk products. Thus, the Karl-Fischer titration for water and the Olivo Moisture Meter which measures the pressure of acetylene gas generated when water reacts with calcium carbide have found little application in the dairy laboratory.

Since gravimetric procedures are time-consuming and require costly equipment, much attention has been given to indirect methods to determine milk solids. One of the best known is that of Jacobson,[6] who in 1936 devised a formula, based on milk from the New England States, which states that the solids-not-fat of a milk is equal to 0.4 times its fat content plus 7.07. For example, if a milk contained 4 percent fat, its solids-not-fat content is (4 x 0.4) + 7.07 or 8.67 percent. Formulas of this type are intended for use with mixed milk as it comes from the herd, and not for milk which may have been standardized in the dairy to a given fat and/or solids-not-fat content. In 1951, a formula relating to California milks was published by Jack and coworkers [5] at the University of California at Davis. This formula is very similar to that of Jacobson and says that the solids-not-fat are equal to 0.444 times fat plus 7.07. Many other formulas have been proposed but have found little application.[8]

Examples of the results obtainable with formulas are given in Tables I and II. The fat tests and gravimetric solids-not-fat figures are taken from a recent study on the composition of California milk.[11]

TABLE I

Estimation of Solids-Not-Fat by Formula

		Jacobson	*Jack*
Fat	3.82 [1]	----	----
SNF	8.58	8.60	8.77
Fat	3.85 [2]	----	----
SNF	8.74	8.61	8.78
Fat	3.90 [3]	----	----
SNF	8.63	8.63	8.80
Fat	4.11 [4]	----	----
SNF	8.71	8.71	8.89
Fat	4.64 [5]	----	----
SNF	8.95	8.92	9.13
Fat	4.96 [6]	----	----
SNF	9.02	9.05	9.27

[1] Average of 41 samples from vicinity of Visalia.
[2] Average of 86 samples from vicinity of Davis.
[3] Average of 36 samples from vicinity of Newman.
[4] Average of 38 samples from vicinity of Willows.
[5] Average of 35 samples from vicinity of Petaluma.
[6] Average of 22 samples from vicinity of Fernbridge.

Statistical examination of much published data indicates that the solids-not-fat in *mixed herd milk* may be estimated to within 0.29 percent of the gravimetric results in two-thirds of the samples tested by the Jacobson formula.

Next to be considered are the formulas for solids-not-fat based upon the fat content and specific gravity of the milk. As in the case of the Jacobson type formula, errors are minimized when mixed milk, rather than that of an individual cow, is tested, although this laboratory did have very good results on milk from individual cows.[4]

The specific gravity of milk may be measured by several methods, such as by the use of the pycnometer, the Westphal balance and the lactometer. The pycnometer gives accurate results, but is slow and awkward for practical use. The Westphal balance gives a result which is a compromise between the accuracy of the pycnometer and the speed of the less accurate lactometer.

The lactometer is a hydrometer with a scale which indicates the specific gravity of milk in terms of lactometer degrees. A lactometer degree is 1,000 times the specific gravity minus 1,000, or more simply, the last two figures of the specific gravity value, read to the third decimal place. Thus, a specific gravity of 1.0325 is equivalent to a lactometer reading of 32.5 The older type of lactometers often read only to a whole degree, but instruments are available with scales divided to 0.1 or 0.2 lactometer degree. The state regulation requires that the instruments to be certified for accuracy must be calibrated to not less than 0.5 lactometer degree and to be accurate to within 0.5 lactometer degree. To facilitate reading the instrument, the writer recommends that the length of scale representing one lactometer degree should be at least 0.5 cm or approximately one-fifth inch. For routine use, the lactometer need only read between 25 and 37 at 60° F. The colored areas on the scale on some lactometers, which are intended to indicate skimming or added water, are of no practical use. The Dairy Laboratory of the Division of Chemistry in the State Department of Agriculture examines lactometers and certifies the accuracy of those that meet these requirements by lightly sandblasting the letters "D.B." on the bulb of the lactometer. A detailed paper on the lactometer and its use was published by the writer several years ago.[7]

The accuracy of the lactometer is determined by comparing its reading when floating in homogenized milk with the known specific gravity of the milk. The specific gravity of the milk is determined by means of a carefully calibrated pycnometer or with a standard lactometer which has been calibrated by comparison with pycnometer readings. Various points on the lactometer scale are checked by diluting the milk with water or by increasing its specific gravity with the addition of evaporated milk. In each case, the exact specific gravity of these test solutions is known.

Most work with the lactometer in this country has made use of the Babcock formula, $SNF = 1/4L + 1/5F$ where L is the lactometer reading at 60°F and F the fat content of the milk tested. By the use of this formula one may, as a rule, expect results well within 0.2 percent of the gravimetric results. As an example, recently four samples of milk were tested here which had been standardized for fat content and fortified with milk solids. The results given in Table II compare the solids-not-fat obtained by Mojonnier and lactometer and the use of the Jacobson and Jack formulas. The erroneous result that may be obtained by these formulas on milk with added solids-not-fat is well indicated in this table.

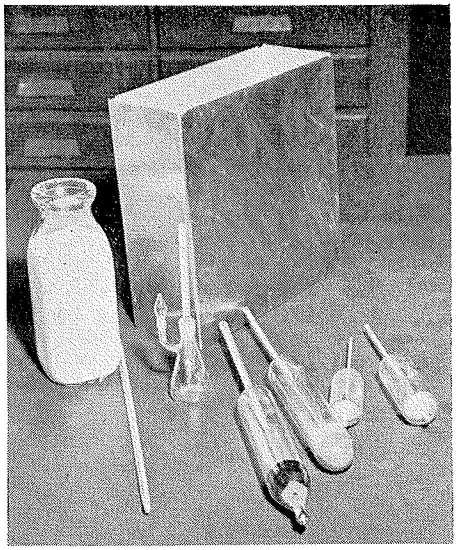
TABLE II

Lactometer Results on Standardized Milk

Fat	*Mojonnier SNF*	*Lactometer 60° F.*	*Jacobson formula*	*Jack formula*
3.70	9.37	9.35	8.55	8.81
3.65	9.97	9.83	8.53	8.69
3.90	9.24	9.22	8.63	8.80
4.05	9.88	9.84	8.69	8.87

Early work done in the laboratory,[4] is in line with the above lactometric results and shows that the average SNF of milk could be determined within 0.02 of the gravimetric results.

The use of the lactometer at 60° F. has been criticized since the specific gravity of a milk sample is not constant. At the time of milking, the fat globules are liquid and, if the milk is not cooled, the globules will remain liquid at 60° for 12 to 15 hours. As the globules cool and gradually solidify, they contract and become smaller in volume. As a result, the volume of milk will also decrease somewhat and so its specific gravity will increase, since the same weight of milk will occupy less volume. This is



A pycnometer (specific gravity bottle) used to check the accuracy of lactometers, such as those shown in foreground.

known as the "Recknagle effect." If this factor is not taken into consideration, a solids-not-fat determination on relatively fresh milk may be 0.1 percent or more too low. However, this factor may not be as important today as it was 10 years ago. Today much milk is held at a low temperature in farm tanks over a period of two, three, or more milkings or has been mixed with so much pooled milk that a sample taken at the processing plant probably has reached its maximum specific gravity. As a result, reliable readings may be expected when the lactometer is used at 60° F. A limited investigation on this point is now being undertaken here.

Various procedures have been proposed to give accurate specific gravity determinations. Some methods involve heating the milk to 95° F. or higher, and then cooling to 60° or 68° before taking the lactometer reading.[3,4,9] The most recently proposed method is to heat the milk to 102° F. and to take the specific gravity at that temperature with a specially calibrated lactometer.[12,13] Since the lactometer is used at 102° F., the usual Babcock formula does not apply and use is made of the formula

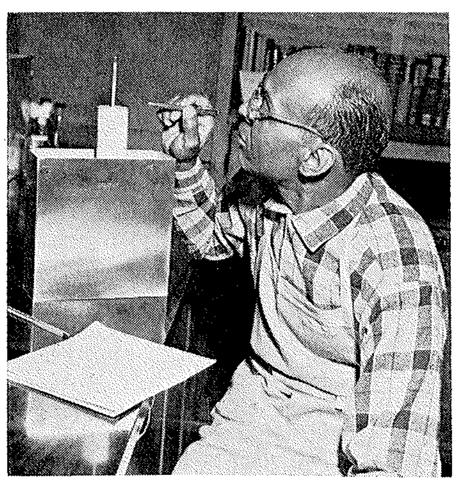
$$SNF = 0.33\,F + \frac{273L}{L + 1{,}000} - 0.40$$

F is the fat content of the milk and L the lactometer reading at 102° F. As in the case of the 60° F. lactometer, tables are also available which make it unnecessary to use the formula in making the solids-not-fat test with the 102° lactometer.

Published results on these procedures are not conclusive. Tests made on milk measured at 102° F show a variation from the gravimetric results of from −0.18 to +0.19 percent. This is essentially the variation that is obtained when milk is measured at 60° F. A factor in favor of the 60° reading is that a variation of about 10° F may be tolerated in the temperature of the milk and corrected for in the test. In the work previously mentioned it was found that the correction of +0.1 lactometer degree for each degree of temperature above 60° F. (up to 65°) and −0.1 lactometer degree for each degree of temperature below 60° F. (down to 55° F.) gave correct results. At temperatures below and above this range the correction is not as satisfactory.[4]

The ability to correct for temperature variation appears to be more limited when the 102° lactometer is used. Published directions indicate that the sample should not vary more than 2° F. from 102° F., in which case the correction factor is 0.2 lactometer degree for each degree of temperature.[12,13]

It would appear that plant-operation conditions would indicate whether the 60° or 102° lactometric procedure should be used.



Laboratory technician, James Moore, making a lactometer reading.

In some places, it may be convenient to warm the milk to about 95° F. for the Babcock fat test and then to 102° for the lactometer test. In other places, daily samples may be warmed to 60° for the lactometer test and then, if necessary, up to about 95° for the fat test. In the case of composite samples, it may be necessary to warm to about 95° F. for the fat test and then cool to 60° for the lactometer reading.

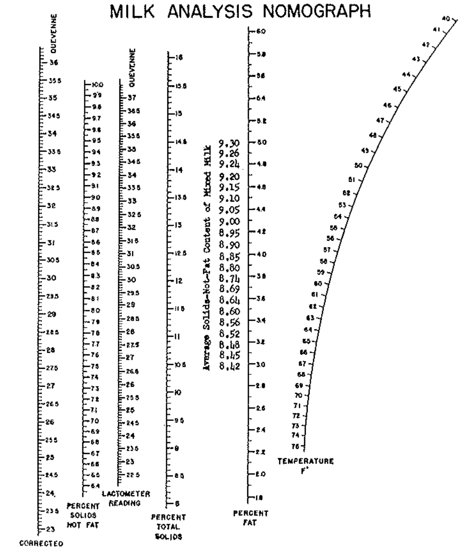
If the milk is heated and then cooled to 60° F. in order to eliminate any element of uncertainty due to the Recknagle effect, the usual Babcock formula for solids-not-fat may not apply. It was found[4] that a correction of +0.2 was needed, so that the modified formula in this case is SNF = 1/4 L + 1/5 F + 0.2. Work is now being undertaken to see how generally this factor applies under present day conditions. If this work indicates that this factor or any other factor is warranted, changes will be made in the formula acceptable for use by licensed non-fat milk-solids testers.

A nomograph, shown in reduced size in Fig. 1, was devised in order to simplify the calculations needed to obtain solids or solids-not-fat.[8]

The plastic bead lactometer is a new development.[2] In this procedure, the milk to be tested is warmed to 104° F. and placed in a small jar. This jar contains 10 small, plastic balls or beads which differ from each other in specific gravity by one lactomer degree. The 10 balls cover the range from 25 to 34 lactometer degrees. When the milk in the jar has cooled to 68° F., the number of beads on the bottom of the jar is counted. The solids-not-fat in the milk sample is determined by a formula which relates the fat content of the milk and the number of beads counted. The formula is SNF = 913 − 0.279 B + 0.307 Fat, where B is the number of beads on the bottom of the jar. Published results[1] indicate the method does not have the accuracy of the lactometric procedure, and of 1,873 samples tested, more than 25 percent of them varied from the gravimetric results by more than 0.2 percent.

Fig. 1

MILK ANALYSIS NOMOGRAPH



The above nomograph is copyrighted.

No temperature correction is made when the plastic bead method is used between 68° and 76° F. This may in part be accounted for by the relative insensitivity of the method, each bead being equivalent to a whole lactometer degree. If one were satisfied with this degree of sensitivity, no correction would be needed with the 60° lactometer, since its readings would vary only one lactometer degree over a range of 10° F.

Another specific gravity procedure has been proposed recently. This is the Banco Gradient Balance, an adaptation of the falling-drop method of measuring density values. This procedure uses a single drop of milk, which is placed into a graduated tube containing a special mixture of standardized light and heavy liquids. This mixture varies uniformly in specific gravity, so that the densest portion is at the bottom of the tube and it gradually becomes lighter towards the top. The drop of milk sinks until it reaches the section which has the same specific gravity as the milk. The specific gravity value is read from the scale on the tube and the solids-not-fat may be calculated by formula or obtained from a table of values. No data is as yet available on the use or accuracy of the procedure. Since it is a specific gravity method, its accuracy would be expected to be in the range of the indirect procedures.

It is possible that in the future some electronic device may be developed which will simplify fat and solids testing. An approach towards this end has been made with the "Darisonometer." This instrument measures fat and solids by applying to a sample of milk high-frequency sound waves, the socalled ultrasonic waves. These sound waves move with different speed through liquids of different composition; the higher the solids content, the faster the waves pass through it. With this instrument, the milk sample is divided into two portions, one part is heated to 122° F., the other cooled to 57° F. The claim is made that at 57° F. the fat content does not influence the speed of the sound waves and the results obtained indicate the solids-not-fat. At 122° F., the result obtained represents the sum of fat and solids-not-fat. The instrument automatically subtracts these readings from each other and the results, read on dials, indicate fat and solids-not-fat. From the limited information available on this procedure, it appears its accuracy is comparable to that of other indirect methods, such as the lactometer, which depend upon physical measurements.

All available information leads one to believe that the lactometric determination of solids-not-fat has the potential accuracy of the gravimetric procedure. Some variation in lactometric results may be attributed to a possible error in the fat test. Although most bottles are accurately calibrated, the California Agricultural Code permits an error not to exceed 0.1 percent in the calibration of any part of the scale on a Babcock milk-fat test bottle. In a 3.5 percent fat milk, an error of 0.1 percent in the fat content is equivalent to 2.85 percent of the fat content of the milk. An error in 0.1 percent in the fat test means a change of only 0.02 percent in the solids-not-fat content of the milk, when the lactometer is used at 60° F.

By similar reasoning, an error of 0.1 lactometer degree, according to the Babcock formula, is equivalent to 0.025 percent of the solids-not-fat. This is equivalent to 0.29 percent of the solids-not-fat in a milk that contains 8.6 solids-not-fat. The Bureau of Dairy Service regulations permit an error up to 0.5 degree in the calibration of a lactometer. This could permit an error of about 1.5 percent in the solids-not-fat determination—nearly one-half that which an error of 0.1 percent in the fat test would cause. Since the dairy industry has long tolerated the possibility of an error of 0.1 percent in its fat tests, it cannot criticize a procedure which may make an error of smaller magnitude in the less costly solids-not-fat.

Thanks are due to Mr. A. E. Reynolds, Acting Chief, and Mr. R. L. Van Buren, Area Supervisor, Bureau of Dairy Service, for suggestions used in the preparation of this paper.

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