



## COMMONWEALTH OF PENNSYLVANIA MILK MARKETING BOARD

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### STUDY MATERIAL FOR TESTERS



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**I**ntroduction - The Pennsylvania Milk Marketing Law and Official General Price Orders, as issued by the Milk Marketing Board, fix minimum prices for milk purchased by Milk Dealers or Handlers from producers primarily on the basis of a price per hundredweight and component contents.

The importance of an accurate weight, sample and component test of producers' milk, can easily be determined since the weight and test have a direct relationship to the value of the milk as priced by the Board's Order.

The responsibilities of the weighing and testing of milk generally lie with the qualified person who determines these weights and takes the sample of the milk when received from producers at the processing plants, or from a bulk farm tank on the producers' premises, and the person who tests the milk to determine the component content of the sample.

Any persons who weigh and sample milk, and those that test milk samples and report results from which payment is made, must exercise every care and skill that will result in accuracy by proper methods. For this purpose, all such persons are required to undergo an examination for proficiency in weighing and sampling milk and a separate examination for testing milk. If successful in completing such examinations, they are eligible to apply for a Weigher and Sampler and/or Tester's license, renewable annually, which will qualify them to act as a Weigher and Sampler or Tester. These study materials, along with the Weigher/Sampler study materials, will prepare a candidate to take the tester examination. While the emphasis of the tester examination is on testing procedures, it is essential that certified testers have an understanding how the milk is picked up at the farm and how an accurate sample is taken. For a candidate for the tester examination to be successful, they must have a command of the information in both these and the Weigher/Sampler study materials.

**DEFINITIONS**

**Authorized Laboratory** – Laboratory sanctioned by the Federal Market Administrator's Office to produce reference and control samples for calibration and accuracy checks of electronic testing equipment. Authorized laboratories include laboratories operated by the United States Department of Agriculture, Federal Market Administrators and commercial laboratories approved by the Federal Market Administrator's Office.

**Accuracy Check** – A test made at the beginning of each testing session and once per hour thereafter to determine the continued accuracy of the electronic testing apparatus. To perform the Accuracy Check, the Tester tests the first four of the reference samples. If the results of the

four Accuracy Check tests are 0.05 or less than the results obtained by the Reference Method, the Accuracy Check is successful.

**Approved Preservative** – Chemical approved by the Market Administrator used to improve the longevity of milk samples. One of the currently approved preservatives is Potassium Dichromate.

**Calibration** – The adjustment of an electronic instrument so that the results for a given payment component match the results of an Association of Official Analytical Chemists (AOAC) or Intersociety Council on Standard Methods for the Examination of Dairy Products (ICSMEDP) approved Reference Method.

**Certified Tester** – A Milk Marketing Board certified technician as referenced in §144.2 (relating to certification requirements) operating electronic instruments and/or a person certified to perform specific Reference Methods for determining the components in raw milk.

**Control Milk/Control Samples** – Samples produced by a commercial laboratory or by the United States Department of Agriculture (USDA) Market Administrator's Office or its successor agency, used for determining the calibration of an electronic instrument. Typically the control samples are identical to the first four reference samples and are used to perform the hourly accuracy check of the electronic equipment.

**Electronic Method** – A method for determining the components in raw milk using an electronic apparatus.

**Electronic Testing Equipment** – Approved equipment used to test for milk fat content or other components of milk. Maintenance records, calibration and accuracy test records must be maintained for at least one year.

**Meniscus** - The curved surface of liquids in tubes, whether concave or convex, caused by the surface tension of the liquid.

**Milk Component/Component** – Unique compound within milk whose relative mass within the milk may be used to determine the payment to producers. Component parts of milk include, but are not limited to, butterfat, protein, lactose, solids non-fat, other solids, and total solids.

**Reference Method** – Standard method using analytical chemistry or other approved techniques by which all other electronic methods of testing milk are compared for determining the accuracy of the machine testing for the components in milk.

**Reference Samples** – Milk samples produced by a Reference Method in laboratory recognized by the United States Department of Agriculture and or the Pennsylvania Milk Marketing Board and used to calibrate electronic milk testing equipment. Typically there are 12 reference samples used to calibrate electronic equipment.

**Repeatability Check** – A test run at the beginning of each testing session in order to demonstrate the ability of a given machine or piece of equipment to meet the requirements for repeatability. The repeatability check requires ten consecutive tests on a single well-mixed sample. If the range of the results do not exceed 0.07 or less, the repeatability check is acceptable.

**Zero Check** – Test performed on Electronic Milk Testing Equipment prior to the repeatability to ensure the equipment will yield a “zero” on a non-milk sample. Procedures for the zero check vary by manufacturer.

## **TESTERS**

It shall be unlawful for any Milk Dealer to buy or receive milk from producers, or to sell or deliver milk to stores or consumers on the basis of, or with reference to, the amount of butterfat or milk components contained in such milk, unless the butterfat or component test is conducted by a Tester licensed by the Board.

The Tester shall keep an accurate record of all tests made, and copies of such record shall be kept for a period of two years by the Tester and by the Milk Dealer.

To become a licensed Tester, a person is required to pass an examination in testing conducted by the PA Milk Marketing Board. The cost of the examination is twenty-five (\$25.00) plus an annual testing license costing twenty dollars (\$20.00). The tester license is issued from January 1st through December 31st and must be renewed annually. Renewal applications will be mailed by the PA Milk Marketing Board to current license holders. These applications shall be mailed back to the PA Milk Marketing Board office along with the twenty dollar (\$20.00) annual license fee by December 1st for the following year.

## **REFUSAL, SUSPENSION AND REVOCATION OF LICENSES**

The Board may decline to grant, or may suspend or revoke a Weigher and Sampler license or a Tester license, where the applicant therefore, or holder thereof, has violated any provision of the PA Milk Marketing Law, pertaining to the weighing, measuring, testing or sampling of milk, or has engaged in any practice with respect thereto, which causes stores or consumers to overpay for such milk, or which otherwise defrauds or tends to defraud milk producers, stores or milk consumers.

This procedure before the Board and of the Board, with respect to the refusal, suspension or revocation provided for herein, and the procedure governing appeals from such action of the Board, shall be as that prescribed in the PA Milk Marketing Law.

## **TESTING: NOTIFICATION OF PRODUCERS**

- (a) A Milk Dealer buying or receiving milk from a producer on the basis of, or with reference to, the amount or percentage of components contained in such milk shall, within five days after the end of each sampling period, test composite samples and within two days thereafter, notify the producer of the result of such test, stating the period of time during which samples were taken.
- (b) Notice to the producer shall be in writing delivered to the producer.
- (c) For the purposes of implementing the Milk Marketing Law, it may be interpreted that “Fresh Samples” may be used for the determination of component content

for payment to producers, providing that not less than two random representative fresh milk samples, evenly spaced, shall be taken and tested in each one-half month period, or a total of not less than four component tests in each calendar month. Producers shall be notified of the results of such tests, stating the sampling period, within two days after the end of each period.

- (d) After samples have been tested, their residues shall be held intact under refrigeration at a temperature of between 33 ° F and 40° F for a period of not less than ten days.

### **ACCEPTABLE TEST METHODS**

For the purposes of implementing the act regarding tests as to the amount of components contained in milk or cream, the term “Babcock Test”, as used in the act shall include: all component testing methods, including the automated Light Scattering Method, and Infra-Red Method; have prior approval of the Board; and shall be performed in accordance with the standard established by the Association of Official Analytical Chemists (AOAC) in that organization’s current edition of Official Methods of Analysis. When using electronic testing methods, for testing milk fat, results are recorded to the nearest 0.01%. Care must be taken when using Electronic Testing Equipment to insure that there is no air in the sample. Air in the sample may cause the results to be inaccurate. The milk fat in milk may be determined by butyrometric, gravimetric, light scattering, or infra-red methods. The infra-red testing is the most commonly used method. Although older, the Babcock method is still used widely and provides a dependable result. Mojonnier and Roese-Gottleib are also used to determine milk fat content.

### **REFRIGERATION OF SAMPLES**

Samples shall be maintained under refrigeration at a temperature of between 33° F and 40° F. Samples shall not be un-refrigerated for more than 30 minutes.

### **TRANSPORTATION OF SAMPLES**

- (a) Samples are collected at the farm where the milk is picked up in plastic vials, glass bottles or in plastic bags and stored in a cooler with ice. The samples must be maintained at a temperature of between 33° F and 40° F (.5° and 4.4° C) at all times.
- (b) The daily fresh samples shall, upon return of the tank truck from the producer’s farm to the dealer’s plant must continue to be maintained at the dealer’s plant under refrigeration at a temperature between 33° F and 40° F for testing.

### **OFF-PREMISES TESTING**

Samples may be tested off the premises of the Milk Dealer by a recognized laboratory if the following conditions are complied with:

- (1) Samples shall be transported under refrigeration at a temperature of between 33° F and 40° F.
- (2) Records of all tests of producer milk shall be maintained by the tester for a period of not less than two years.

### REJECTION OF PRODUCER'S MILK

- (a) No handler under the act or person holding any license, permit or certificate issued by the Board shall return milk to a producer or reject any milk of a producer for any cause except:
  - (1) Inferior quality of the milk.
  - (2) Non-compliance with the regulation of other governmental health or sanitation agencies.
- (b) The return or rejection of the milk shall be supported by a signed statement to the producer, setting forth the reasons for which the milk was returned or rejected. A copy of the statement shall be retained by the rejecting handler (purchaser) as a part of his permanent records.
- (c) In the case of bulk tank pick-up, the signed statement shall be delivered to the producer in person at the time of rejection or, in the event neither the producer nor his representative is available at the time, it shall be posted in a conspicuous place in the milk house.

Milk is defined in the current Grade A Pasteurized Milk Ordinance Recommendations of the U. S. Public Health Service as the "Lacteal secretion, practically free from colostrums, obtained by the complete milking of one or more healthy cows, which contains not less than 8.25 percent milk solids, not fat, and not less than 3.25 percent milk fat."

In Pennsylvania, whole milk offered for sale must contain at least 3.25 percent fat and 11.5 percent total milk solids. Minimal standards for other states vary from 3.00 to 3.80 percent milk fat and 11.00 to 12.00 percent milk solids.

### COMPOSITION OF MILK

Components	Average Percentage
Water .....	87.73%
Proteins .....	3.20%
Milkfat .....	3.67%
Lactose .....	4.75%
Ash (minerals) .....	0.65%
Solids, Nonfat .....	9.1%
(Protein, Lactose, Ash)	
Total Solids.....	12.8%
(Protein, Fat, Lactose, Ash)	

## CONSTITUENTS OF MILK

**Water**—The water of milk is identical with any other water in nature. The water of milk serves as the medium in which all other constituents are dissolved, suspended and emulsified.

**Fat**—Milk fat is also called butterfat. When warmed, it becomes liquid oil. The yellow color of milk fat is due to the carotene in it.

Chemically, milk fat is very complex. About 99 percent of it is a mixture of hundreds of different triglycerides. The remaining 1 percent consists of phospholipids and traces of diglycerides, monoglycerides, free fatty acids, steroids and fat-soluble vitamins (A, D, E, and K). All these substances are collectively called lipids (fatty or fat-like material). All lipids or fats are soluble in the so-called fat solvents, such as gasoline, ether, chloroform and dry cleaning fluid. They do not mix or rinse with water, a property to be aware of when cleaning testing glassware.

Milk is synthesized in the cow's udder and secreted in the form of globules. These are tiny spheres covered by a very thin lipoprotein membrane. Milk fat globules are so small (0.5 to 10 microns in diameter) that a single drop of milk contains more than 100,000,000 globules. These fat globules are suspended in the water of the milk (temporary emulsion) but rise very slowly toward the surface because fat has a lower specific gravity than the surrounding serum. Milk, upon standing, will therefore eventually show a definite cream layer. Homogenized milk will not do this, because the fat globules have been mechanically broken down to the size of about one micron, so that they no longer respond to the law of gravity.

When milk is exposed to centrifugal force, as in a separator, the fat of the milk will concentrate to cream near the center of the centrifuge. The serum (skim milk) having a higher specific gravity, is propelled to the outside. Of course, skim milk will always contain trace amounts of fat, the amount depending on the speed and efficiency of the separator. Usually the very small fat globules stay in the skim milk portion.

## THE BABCOCK METHOD

The principle behind the Babcock method is to mix milk and concentrated sulfuric acid to release free fat. Generally the Babcock method is as follows:

1. Place raw milk sample in water bath at 32.2° – 37.8° C or 90° – 100° F to disperse the cream layer which forms on top.
2. After the sample reaches the ambient temperature of the water bath, mix sample thoroughly inverting 25 times or shaking whirl bags with 1 foot motions.
3. Pipette 17.5 milliliters (or 18 grams) from the sample container to the Babcock test bottle for milk samples. The milk pipette must have a capacity of 17.6 milliliters but delivers only 17.5 milliliters. When testing cream, use a nine-gram sample. When weighing milk or cream into test bottles, the scales should be on a level, firm place away from drafts. The standard milk test bottle in Pennsylvania is graduated to read a total of

*Heated Water Bath*



*Pipetteing Milk from Sample Vial*



up to 8% fat. A cream test bottle will allow fat readings up to 50%. The smallest division of graduation of a milk test bottle represents .1 of one percent. A cream test bottle is graduated in .5 of one percent.

4. Add 17.5 milliliters (or 18 grams) sulfuric acid into the Babcock bottle. When measuring, never mouth pipette to measure acid. Commercial sulfuric acid with a specific gravity of 1.82 to 1.83 is the kind and strength used in testing milk. The sulfuric acid is used in the Babcock test to digest protein and separate fat. Acid for testing milk should be kept in a glass stoppered bottle. If the bottle is left unstoppered, acid will absorb moisture from the air and become too weak. When diluting acid, to minimize the risk of having acid splash out of the container, always add acid to water; never add water to acid.
5. Gently mix acid and milk sample causing an exothermic (heat is given off) reaction. The sample should turn a golden straw color. A dark muddy or black color indicates the sample is burned and should be discarded.
6. Thoroughly mix the acid and milk sample in a mechanical mixing device or shaker for one minute.

*Centrifuge to Bring Fat to Top of Bottle*

7. Place samples in centrifuge at the prescribed speed for 5 minutes. Fat along with the somatic cells in the milk will naturally, over time, rise to the top of the bottle. The action of centrifuge brings fat to the top of test bottle much more quickly and thoroughly.
8. When centrifuge stops, add sufficient laboratory grade water (soft) at 60° C (140° F) to make the fluid in the Babcock bottle come up to within a ½ centimeter (.2 inches) of the base of the neck. When testing cream, the cream test bottles are centrifuged and water added to bottles in centrifuge the same as milk
9. Centrifuge for an additional two minutes. Both milk and cream will now have been centrifuged three times
10. When centrifuge stops, add sufficient laboratory grade water (soft) at 60° C (140° F)

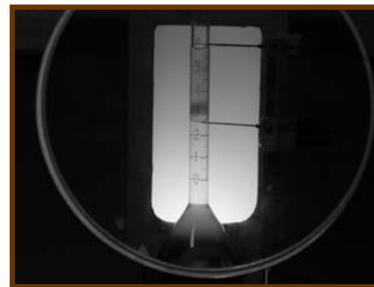


*Babcock Bottle with Visible Fat Column*

to make the liquid fat column in the Babcock bottle come to within the graduated scale in the neck of the bottle. The top of the fat column will be below the uppermost graduation and the bottom of the fat column will be above the lower most graduation in the neck. This is the second time water is added to the Babcock bottle.

11. Centrifuge for an additional one minute.
12. After centrifuge stops, place samples in a tempering water bath at 56.5° – 58.5° C or 133.6°-137.4° F for 5 minutes.
13. Remove sample from bath. Using dividers (like a caliper), measure the distance between the top and bottom menisci of the fat column. In milk, the top and bottom menisci are curved upward. Measure from the lowest point or bottom of the bottom

meniscus to the top or highest point on the curve on the upper meniscus. Express your reading in terms of a percentage to nearest .05 percent. When testing cream, Glymol is added to the fat column of cream test bottles to obtain an accurate reading. The addition of the Glymol will cause the upper meniscus to flatten. When testing herd samples, the maximum allowable standard deviation between the Babcock method and the electronic test is 0.04%.



*Dividers Measuring Fat Column*

## **ETHER EXTRACT METHOD**

Although the ether extract method is one of the more cumbersome procedures for ascertaining milk components, it is among the most dependable and accurate chemical analysis tools. At present it is uncommon to find the ether extract method used for producer payment purposes.

## **ELECTRONIC METHODS**

The most common practice for testing milk for component and fat content is to use specially designed electronic equipment. All electronic equipment must be recognized by the USDA, dairy division, for the analysis of milk and milk components. Laboratories using electronic equipment must make sure their equipment is producing accurate and dependable results.

Electronic testing Equipment will require calibration:

1. Upon initial installation
2. When the Accuracy Check fails
3. When a part of the Electronic Testing Equipment affecting proper operation has been repaired, rebuilt, replaced or adjusted.
4. As required by the manufacturer for proper maintenance or operation of the equipment.

Unless otherwise specified by the manufacturer, use the following procedure for calibrating electronic milk testing equipment.

1. First run a zero check using a non-milk sample to ensure that the machine gives a zero reading.
2. Perform the repeatability test to demonstrate the equipment's ability to test ten consecutive tests on a single well-mixed sample and obtain results within a range 0.07 or less. The actual butterfat level in the



*Electronic Milk Testing Equipment – Milk O Scan*

sample is irrelevant for the repeatability test as long as the results of the ten tests are within the 0.07 range.

3. Test 12 reference samples and compare your test results with the reference test results.
  - a. The average of the 12 differences (called the mean average) is +/- .04 or less; and
  - b. The standard deviation of the ten differences is .04 or less.

**Example:**

<i>Sample ID</i>	<i>Reference Test</i>	<i>Your Test</i>	<i>Difference</i>
RED	3.600	3.632	-0.032
DK BLUE	3.740	3.725	0.015
PURPLE	3.260	3.275	-0.015
CLEAR	3.770	3.789	-0.019
GOLD	3.100	3.091	0.009
DK ORANGE	3.700	3.685	0.015
TURQUOISE	4.350	4.384	-0.034
GREEN	4.070	4.066	0.004
BROWN	5.100	5.132	-0.032
PINK	5.740	5.776	-0.036
WHITE	4.810	4.765	0.045
HOT PINK	4.600	4.619	-0.019
			<i>Average of Differences</i>
			-0.008
			<i>Standard Deviation of Differences</i>
			0.025

Standard deviations are usually obtained from spreadsheet formulas rather than manual calculations. If one wishes to perform the calculation manually, consult a statistical procedures text.

4. Perform an accuracy check to determine the accuracy of the electronic testing apparatus. To perform the Accuracy Check the Tester tests the first four of the reference samples. If the results of the four Accuracy Check tests are 0.05 or less than the results obtained by the Reference Method, the Accuracy Check is successful and the equipment is ready to test milk. **Note:** The Accuracy Check must be run each hour that machine is in operation and testing farm milk samples. If samples used for the Accuracy Check called the control samples were not provided with the reference samples, the Tester may subdivide the first four reference samples into several smaller control samples for use throughout the testing session. Using an approved preservative, reference and control samples may be kept under refrigeration for up to ten days.

5. If a machine has failed any of the above tests, the cause of the failure must be corrected or the machine adjusted and all tests rerun to ensure that the equipment is properly calibrated.
6. Once the equipment is properly calibrated, the Tester need only run the accuracy check and repeatability check at the beginning of each testing session. When using electronic equipment, make sure air is not present in the samples as this will affect accuracy.

Records of all zero checks, repeatability checks, tests on reference samples and accuracy tests must be kept for at least one year.