10. A sampler shall collect 2 samples at the first collection point for each bulk tank truck load and identify one sample to be used as the temperature control for all samples subsequently collected for that load and placed in the truck's sample compartment. The sample container of the temperature control, shall show the producer number, date, time of collection and temperature of the milk in the farm bulk tank from which the sample was collected.

(c) *Fresh milk sample size.* The size of the fresh milk sample shall be large enough to permit retesting by the dairy plant, its testing agent or the department, but in no case shall the sample size be less than 2 ounces (60 milliliters) without prior written approval of the department. Approval may be granted only where the plant can demonstrate to the satisfaction of the department that the sample taken will permit thorough mixing and at least one retest for every test conducted on the milk sample.

(d) *Composite samples.* A composite sample of a patron's milk shall consist of a representative sample from each delivery of milk by the producer to a dairy plant within a given pay period. A minimum of 10 milliliters of milk for each day's production shall be included in the composite sample from every delivery. In no event shall a completed composite sample consist of less than 150 milliliters for a 15 day milk delivery period. When milk is frozen or otherwise delivered in a condition which prevents adequate mixing, a sample of the milk shall not be taken and a notation shall be made on the collection sheet that a sample was excluded from the composite sample. The composite sample shall be built up as follows:

1. For bulk milk deliveries, by transferring a minimum of 10 milliliters of milk for each day's production from each fresh milk sample to the composite sample container. Such transfer shall be made on the day of receipt or by 12:00 o'clock noon of the following day.

2. For can milk deliveries, by transferring 10 milliliters of milk from each day's production directly from the milk weigh tank to the composite sample container immediately after the milk is poured into the weigh tank.

(3) CARE AND STORAGE OF SAMPLES. (a) All milk samples received directly from the farm shall be kept tightly covered and maintained at a temperature between 32 and 40° Fahrenheit at all times during transportation and while held for testing at the dairy plant or a testing laboratory.

(b) No composite sample shall be kept out of refrigeration at the dairy plant for a longer time than necessary to continue building the composite sample from fresh milk samples of a producer, or the completion of weighing and sampling operations for each truck load delivery of can milk. Transporting composite samples from the dairy plant or laboratory to a dairy farm is prohibited. Each dairy plant or laboratory shall use a preservative approved by the department in building each composite sample. The preservative may consist of a bichloride of mercury or corrosive sublimate tablet which shall weigh not more than one gram and contain not less than 2.5 nor more than 3.5 grains of bichloride of mercury. Potassium dichromate or other chemical preservatives may be used to preserve composite samples upon written approval from the department. Not less than 14 grains nor more than 20 grains of potassium

dichromate may be used as a preservative in building composite samples.

Note: After January 1, 1983, mercuric chloride as a preservative will be prohibited.

(c) Fresh and composite milk samples may be transported from a dairy plant to a certified laboratory or other department approved laboratory for milkfat and other component testing only on prior written approval from the department.

(d) Composite samples may be pipetted in duplicate at a dairy plant and transported to a second laboratory for testing on written approval from the department. Only the 2 pipetted portions may be transported to the testing laboratory, and the residual of the composite shall be left at the dairy plant. When any sample is pre-pipetted for testing, all AOAC procedures for warming, mixing and pipetting the sample, shall be followed. All Babcock test bottles shall be properly sealed and legibly identified with the patron number.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76; am. (1), (2), and (3), cr. (2) (b) 9.a. to d. and 10. and (3) (d), Register, September, 1982, No. 321, eff. 10-1-82.

Ag 107.04 Testing of samples. (1) TEST METHODS. Milkfat tests of fresh milk samples may be made by the Babcock test, ether extraction test, the Milko-Tester, or other AOAC test method approved by the department.

(2) ETHER EXTRACTION AND BABCOCK TEST. (a) All ether extraction and Babcock tests shall be conducted as prescribed in "Official Methods of Analysis of the Association of Official Analytical Chemists", 1980 edition, except as provided under pars. (b) and (c). A copy of this reference is on file in the offices of the department, secretary of state, and revisor of statutes, and may be obtained from the Association of Official Analytical Chemists, Inc., P.O. Box 540, Benjamin Franklin Station, Washington, DC, 20044.

(b) Each sample tested by the Babcock test method shall be agitated for at least 3 minutes by the use of a mechanical agitator after pipetting the sample and adding sulfuric acid in accordance with the AOAC procedure. A reader such as a needlepoint divider or other mechnical divider which accurately determines milkfat level in a test bottle shall be used in reading all Babcock tests. All Babcock test readings shall be made against a light-colored surface with adequate natural or artificial light.

(c) The Babcock test may be read to the nearest 0.05% by weight as provided in "Official Methods of Analysis of the Association of Official Analytical Chemists", if the test bottles are graduated to 0.1%. Results obtained from an automated test device may be reported with the same accuracy which the device is capable of reading or reporting.

(3) OTHER APPROVED TEST DEVICES. (a) *Calibration requirements.* All automated test devices shall be calibrated on initial installation and at least every 12 months thereafter by either the Babcock test or the ether extraction test. The device shall be recalibrated whenever the mean deviation on a daily performance check is greater than plus or minus 0.04% or when major repairs are made to the test device.

(b) *Constant voltage.* A constant voltage regulator shall be connected to all automated test devices in line with single phase 115 or 220 volt power supply.

(c) *Calibration procedure.* 1. Twenty representative samples ranging from 3.0% to 6.0% milkfat shall be tested in triplicate on the automated test device and by the Babcock method or ether extraction method. The average of the 3 results for each sample tested by each method shall be calculated to the nearest 0.01% milkfat. The standard deviation of the difference between the automated device's results and the reference test results, calculated in accordance with the Association of Official Analytical Chemists Methods, 13th edition (1980), shall not exceed plus or minus 0.04%. The mean deviation of the results from the automated test device and the Babcock or ether extraction reference test method shall not be greater than plus or minus 0.04% for acceptable calibration.

2. The calibration record shall be maintained on file in the laboratory.

(d) *Daily performance check.* The device shall be checked on a daily performance basis by the use of at least 5 milk sampl;es ranging from 3.0% to 5.5% milkfat. These must be fresh unhomogenized samples. Triplicate Babcock tests must be made on all of the samples and an average recorded on forms approved by the department. If the device varies more than plus or minus 0.04% from the Babcock results and basic adjustment does not bring it to within this tolerance, recalibration of the device is necessary.

(e) *Reference check.* 1. A reference check sample must be tested during the course of the performance check and each hour during testing. The reference sample may be one of the samples used for the daily performance check or may be a homogenized milk sample.

2. If a homogenized milk sample is utilized, at least 10 tests must be run on the initial reference check sample before the start of producer sample testing. The average of those results and the hourly reference check sample results must agree within plus or minus 0.03%.

3. If the reference sample has not repeated within tolerance, adjustments shall be made to the device to agree with the average of the reference samples and all producer samples tested since the previous complying reference check shall be retested.

(f) *Test accuracy and recordkeeping.* All Babcock test results used in the calibration, daily performance check, or reference check of an automated test device shall be read to the nearest 0.05%. Records of all checks, calibration data and daily performance checks shall be maintained on file in the laboratory and be available for department inspection for a period of at least one year.

(4) FRESH MILK TESTING. Fresh milk samples shall be tested for milkfat or other components no later than the third day following the day the sample was taken. No fresh milk sample may be tested for milk quality as defined in s. Ag 30.01 (7), if the sample is held for longer than 36 hours.

(5) COMPOSITE SAMPLE TESTING. (a) Milkfat tests of composite milk samples may be made by the Babcock test or ether extraction test methods. No mechanical device may be used in testing composite samples without prior written approval of the department. Approval, when granted, shall be limited to a specific location for samples from specified producers.

(b) Composite samples shall be tested within 3 days, Saturdays, Sundays and holidays excluded, after the build-up of the sample has been completed. Time for completion of the tests may not be extended without prior written approval from the department. Replacement of a set of composite samples with fresh milk samples shall not be done without prior approval from the department. Composite samples shall be built and run for the periods specified in s. 98.13, Stats. Loss of an individual composite sample due to a laboratory accident shall be replaced with 3 subsequent fresh milk samples.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76; am. (1), (2), (4) and (5), r. and recr. (3), Register, September, 1982, No. 321, eff. 10-1-82.

Ag 107.05 Sample retention and retesting. The residual of each composite sample shall be removed from the water bath immediately after each sample is pipetted. The residual of each composite sample shall be held intact after initial testing and kept refrigerated at a temperature between 32 and 40° F. on the premises where tested for a period of not less than 5 days. After completion of all testing, fresh milk samples shall be retained in the laboratory for at least 4 hours. The department may retest any composite or fresh milk samples on the premises where they were tested or remove them to a department laboratory for this purpose. The department shall, upon written request of the licensed tester or the employer give notice of the time and location for retesting the plant samples, providing the request is made at the time the samples are collected by the department for retesting. Notice of department retesting of a dairy plant's samples is not required to be given to a marketing association engaged in testing composite or fresh milk samples for its member patrons, unless a written request, signed by the marketing association tester who executed the official test record, is left at the dairy plant. The department may retain retested samples for investigative or evidentiary purposes, or return them in a sealed condition to the dairy plant upon written request.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76.

Ag 107.06 Test records. (1) Each licensed tester, immediately after testing a sample, shall record in duplicate, on a form approved by the department, the name or identification letters or number of the patron whose milk or cream was tested, the date of test and the test results. The record and all copies shall be signed by the tester. One copy shall be retained at the testing laboratory and one copy made available at the dairy plant. All original test records shall be kept for a period of not less than 2 years. No test records may be altered except that errors, if made, shall be corrected by striking through the original entry and inserting the correct entry immediately adjacent to the original, along with the initials of the tester who made the corrective entry.

(2) When using fresh milk tests for payment to patrons, the arithmetic average of 3 or more milkfat or other component test results shall be used for each 15 or 16 day pay period or the arithmetic average of 4 or more milkfat or other component tests results for a one month pay period. The frequency of conducting milkfat or other component tests shall be evenly distributed throughout a pay period. In averaging milkfat or other component test results, decimal fractions may be rounded to the nearest 0.01%.

(3) Calibration records, daily performance checks and routine checks of automated instruments, Milko-tester calibration records, and Babcock test results shall be kept on forms prescribed by the department.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76; am. (1) and (2), r and recr. (3), Register, September, 1982, No. 321, eff. 10-1-82.

Ag 107.07 Milk component sampling and testing. (1) All test methods and equipment used in testing milk to determine its value for payment by testing for components other than milkfat, shall conform to test methods and equipment approved by the Official Association of Analytical Chemists, Standard Methods for Examination of Dairy Products, or other test methods and equipment approved by the department. Test methods approved by AOAC for protein analysis include:

(a) Keldahl method number 16036 for total nitrogen.

(b) Dye binding method number 1637 acid orange 12.

(c) Pro-milk method for determination of milk and protein, annato black 10B, first action by AOAC.

(d) Infra-red milk analysis method number 16079 and number 16080, part 2, protein, first action by AOAC.

(2) Devices used for testing milk for components other than milkfat shall be calibrated as outlined in the 13th edition (1980) of the Association of Official Analytical Chemists manual and shall consist of a comparison of 20 representative milk samples ranging from 2.4 to 4.0% protein. One sample shall be present in triplicate and at least 2 samples shall be in duplicate. The Keldahl results and the instrument results shall have a mean and standard deviation of not more than .02% on components. There shall be no more than .05% difference from any instrument results with the reference method.

(a) The calibration record shall be maintained on file in the laboratory and repeated whenever major parts are replaced, rebuilt or adjusted.

(b) A daily performance check shall be made and reported before the daily testing of producer samples. Five samples from the previous day's testing shall be held over and repeated the start of the second day. The average mean deviation on the two runs shall not exceed plus or minus .02%.

(c) A reference check sample shall be tested during the course of the performance check and each hour during testing. The reference sample may be one of the samples used for the daily performance check.

(3) All reagents used in any of these methods shall be used in accordance with the AOAC method and shall be clearly and fully labeled to insure they are the reagents required by the method. All testing equipment shall be calibrated as prescribed in the AOAC methods, and shall be subject to initial calibration with the department's standards and at least annual calibration on split samples provided by the department.

(4) When milkfat and milk protein or other component tests are to be conducted from the same sample, the milkfat test may be run on one day and other component tests no more than 24 hours following the initial milkfat test. If other components tests will be made within 2 hours after

the initial milkfat test, those samples need not be refrigerated. All samples used for multiple testing shall not be less than 3 ounces.

(5) If abnormal milk standards or other quality tests are used to deny payment for components of producer milk, only the direct microscopic somatic cell count (DMSCC) or electronic somatic cell count (ESCC) shall be used to confirm the accuracy of the denial of payment for components based on abnormal milk.

History: Cr. Register, September, 1982, No. 321, eff. 10-1-82.

Ag 107.08 Accuracy of samples and tests. No sampler, tester, cheesemaker, buttermaker or any other person shall falsely identify milk samples, submit false samples of milk to a dairy plant, make any false record or report concerning a milk sample or the test results, or the quality or quantity of milk, or violate any other provision of these rules.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76; renum. from Ag 107.07 and am. Register, September, 1982, No. 321, eff. 10-1-82.

Ag 107.09 Authority. This chapter is adopted under authority of ss. 93.07(1), 97.24(4) and 98.13(5), Stats.

History: Cr. Register, September, 1982, No. 321, eff. 10-1-82.