Chapter Ag 107

SAMPLING AND TESTING MILK AND CREAM

History: Chapter Ag 107 as it existed on March 31, 1976 was repealed and a new chapter Ag 107 was created effective April 1, 1976.

Ag 107.01 Definitions. As used herein:

(1) "Department" means the state of Wisconsin department of agriculture, trade and consumer protection.

(2) "Calibration" means the complete procedure to arrive at the allowable standard deviation for the test method or instruments, as prescribed in "Official Methods of Analysis of the Association of Official Analytical Chemists," 13th Edition (1980).

(3) "Composite sample" or "composite milk sample" means a milk sample preserved with an approved chemical and built-up as prescribed in this chapter for use in the testing of milk for a given pay period as prescribed in s. 98.13, Stats.

(4) "Fresh milk sample" means an unpreserved sample of producer milk collected aseptically.

(5) "Ounce" means fluid ounce.

(6) "Performance check" means the daily cross-check procedures used for milkfat test determination and approved by the department.

(7) "Preserved fresh milk sample" means a fresh milk sample preserved with potassium dichromate for the purpose of subsequent laboratory analysis in a dairy herd production testing program.

(8) "Sample" means a representative sample of milk or cream used for testing to determine milkfat or protein content, or both, quality and any other properties or characteristics of the milk or cream from which the sample is taken.

(9) "Sampler" means a milk weigher and sampler licensed under s. 98.146, Stats., or a cheesemaker or buttermaker licensed under s. 97.17, Stats.

(10) "Test records" mean the original worksheets for equipment calibration, performance checks and testing on all samples.

History: Cr. Register, March, 1976, No. 243, eff. 4-1-76; am. (1), renum. (2) to (7) to be (8), (5), (9), (4), (7) and (3) respectively, and am. (3), (4), (7) and (8), cr. (2), (6) and (10), Register, September, 1982, No 321, eff. 10-1-82.

Ag 107.02 Licensing milk weighers and samplers. (1) In addition to the initial examination required for the issuance of an original milk weighers and samplers license under s. 98.146, Stats., the applicant may

be required to demonstrate competency to perform the weighing and sampling functions, either on the department's premises or in a field examination. The applicant shall, as a condition to the first biennial license renewal satisfactorily pass a second and more comprehensive examination as prepared by the department.

(2) Every sixth year after a milk weighers and samplers license has been in effect, the department may, as a condition of renewal thereof, require re-examination, unless the applicant has satisfactorily completed a training course approved by the department within the past 6-year period.

(3) Every milk and cream tester or cheesemaker or buttermaker qualified for testing milk with a mechanical device shall have that qualification clearly indicated on the license. Any licensed milk and cream tester found to exceed accepted tolerances for calibration of the mechanical testing device may have his or her license suspended by the department.

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

Ag 107.03 Collection and care of samples. (1) CONTAINER REQUIREMENTS. (a) Sample containers shall be constructed of non-toxic transparent materials, be in a clean, sanitary and dry condition prior to use, and be commercially sterile for all samples intended for bacteriological testing. All glass or rigid plastic containers used for fresh milk sampling shall be equipped with over-the-lip closures, and have an area on the sample container large enough for placing on it the date of collection and the producer number.

(b) 1. Fresh milk sample containers shall have sufficient capacity to hold a quantity large enough to permit 2 tests of the particular test or tests to be applied to the sample, and shall be of sufficient size to permit thorough mixing of the sample prior to its use for any test, or for the residual to be used to build a composite sample.

2. Fresh milk sample containers shall in no event have a capacity of less than 3 ounces (90 milliliters) without written approval from the department. No approval shall be granted unless the plant can demonstrate to the satisfaction of the department that because of test methods and procedures used, a smaller sample container will suffice to meet requirements under subd. 1.

(c) Composite sample containers shall have a minimum capacity of 8 ounces (240 milliliters) and a satisfactory closure permanently affixed to the sample container.

(d) Composite sample and fresh milk sample containers shall bear legible letters or numbers identifying each patron's sample. Fresh milk sample containers shall also bear the date of collection of the sample by the sampler. Identifying letters or numbers and the date of collection shall be placed on the container by the sampler at the time a fresh milk sample is taken. When a patron discontinues shipping milk or cream to any dairy plant, the letters or numbers used by the plant to identify that patron's milk shall not be reused to identify any milk samples of any other plant patron for a period of at least 90 days.

(2) SAMPLE PROCEDURES. (a) *Weigh tank sampling.* If milk is poured into a weigh tank, the tank shall be constructed in such a manner that

the pouring of the milk into the tank results in complete mixing of the milk. A fresh milk sample of each weighing shall be taken immediately after the milk has been poured into the weigh tank. When multiple weighings of a patron's milk shipment is required, the number of cans of milk poured into the weigh tank in any one weighing shall be equalized as nearly as possible, and the entire contents of each can shall be included in each separate weighing. When multiple weighing of a single delivery is required, a separate sample shall be taken of each weighing, using a separate sample container.

(b) *Farm milk tank sampling.* 1. Each sampler shall grade farm milk by appearance and smell prior to accepting it and loading it on the bulk milk truck. The sampler shall reject all off-flavor or off-odor milk and milk which contains any visible evidence of mastitis and extraneous matter. If the quality of milk is in doubt, the sampler, before accepting any milk, shall call the dairy plant and request inspection by a plant quality control officer or other individual designated by the plant, who shall accept or reject the milk.

2. Samplers shall read and record the temperature of the milk prior to accepting and loading it on the bulk milk truck. When the milk temperature is not ascertainable by reading the farm bulk tank thermometer, the sampler shall take and read the temperature by using a pocket type spring dial or other suitable portable thermometer, accurate to plus or minus 2° F. with the smallest gradation not greater than 2° F. Milk shall be rejected unless collected within 2 hours after milking if it exceeds the applicable temperature requirement for the following grades of milk: manufacturing grade: 50° F.; grade A: 45° F.

3. Samplers shall wash their hands before checking the temperature of the milk by use of a portable thermometer or before measuring the quantity of milk in the tank.

4. Samplers shall take and record an accurate measurement of the amount of milk in the farm bulk tank by the reading of a clean gauge rod or other approved measuring device. The gauge rod shall be rinsed with warm potable water and wiped dry with a clean single-service disposable towel immediately prior to its use. The milk shall be allowed to become motionless before the gauge rod is inserted into the bulk tank for a reading. Immediately after the reading, the sampler shall convert the reading to weight or volume using the bulk milk tank manufacturer's conversion chart, and record the reading on a multiple collection record, one copy of which is posted in the milkhouse and one copy submitted to the dairy plant at the time of delivery.

5. The collection record for each patron shall include the patron's identification letters or number, the time and date of collection and sampling, the quantity of milk collected and the temperature of the milk at the time of collection. The collection record for each patron shall be signed by the sampler and delivered with the load of milk at the time of delivery.

6. Milk from which a sample is to be taken shall be agitated by the sampler for at least 5 minutes or more prior to taking a sample. Milk shall be agitated for at least 10 minutes if the farm bulk tank has a capacity greater than 1500 gallons, or when the 3A Standard for a tank requires a longer agitation time. The sampler shall take at least a 2 ounce representative fresh milk sample from each farm bulk tank prior

to collection and delivery of a patron's milk. A sampling procedure which prevents contamination of the sample or sample container shall be used. The dipper shall be cleaned and sanitized in a 100 ppm chlorine or equivalent sanitizing solution and dipped in the milk twice before taking the sample. The milk sample shall be transferred from the dipper to the sample container away from the open port of the farm bulk tank. A sampler shall not commingle fresh milk samples from any other bulk milk tanks on the premises. Patron identification and date of sampling shall be placed on the sample container. The sample shall be placed in a rack in an ice-water bath or ice bath immediately after it is taken to maintain its temperature between 32-and 40° Fahrenheit. No sample of less than 2 ounces may be taken by any sampler without prior written approval of the department. Approval may be granted only when a dairy plant can demonstrate to the satisfaction of the department that accurate test results can be achieved by taking a smaller sample.

7. The sampler shall ensure that the milk truck hose cap is protected from contamination at all times and that the farm bulk tank outlet valve is clean before the hose is connected to the bulk tank. A sampler may sanitize the outlet to the farm bulk tank prior to connecting the truck hose. The truck hose shall enter the milkhouse through the hose port provided in the milkhouse wall. Milk may be pumped only into bulk tank trucks meeting the sanitary and equipment standards of chs. Ag 30, 31, and 80. No milk may be pumped directly or indirectly into bulk tank trucks from containers other than farm bulk tanks.

8. After pumping the milk from the farm bulk tank, the milk hose shall be disconnected from the tank, capped and returned to the bulk milk truck cabinet. The farm bulk tank shall be rinsed with cold or lukewarm water after it has been emptied, but not until after the milk pump has been shut off and the milk truck hose disconnected from the tank and capped.

9. The sampler shall provide the dairy plant purchasing milk from the producer with a representative sample of each patron's milk comprising the load, and an accurate collection record.

a. If a sampler delivers milk to a diverted dairy plant other than the one to which the producers are routinely assigned, the sampler may, upon receipt of written permission from the department, retain the producer samples for 24 hours.

b. All samples retained by a sampler shall be placed in a separate refrigerator until collected by another agent of the receiving dairy plant or the department. The refrigerator shall be maintained at 40° Fahrenheit or less, used for no other purpose than the storage of samples, and shall be accessible to the department at all times.

c. The sampler shall retain dated samples for 24 hours. When a second set of samples is placed in refrigeration, those which exceed the 24 hour time limit must be disposed of and not retained in the same refrigerated storage unit.

d. An agent of the dairy plant purchasing the milk may transport the samples on written approval from the department to the purchasing dairy plant or a central testing laboratory.

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 potassium dichromate or another chemical approved by the department as a preservative for each composite sample. When potassium dichromate is used to preserve composite samples, not less than 100 milligrams (1.5 grains) nor more than 190 milligrams (2.9 grains) may be used in each composite sample, to obtain a concentration of 20 milli-

grams (0.3 grain) per 30 milliliters (one fluid ounce) of milk in the completed sample.

Note: Potassium dichromate for use as a milk sample preservative is commonly available in tablet form containing 40 milligrams of active ingredient per tablet. The use of these tablets at the rate of one tablet per 2 fluid ounces of milk in a completed composite sample is equivalent to the concentration required in par. (b). S. Ag 29.05(1)(c) contains labeling requirements and limitations on disposal of milk samples preserved with potassium dichromate.

After January 1, 1983, the use of mercuric chloride as a preservative for composite milk samples is 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. 248, 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; emerg. am. (3)(b), eff. 5-21-83; am. (3)(b), Register, November, 1983, No. 335, eff. 12-1-83.

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 samples 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 2 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.