

(4) Testing and examinations of containers shall occur often enough to ensure that the container suitably protects the food from leakage or contamination.

(b) *Coding.* Each container or product shall be marked with an identifying code permanently visible to the naked eye. If the container does not permit the code to be embossed or inked, the label may be legibly perforated or otherwise marked, as long as the label is securely affixed to the product container. The required identification shall specify in code the establishment where the product was packed, the product contained therein, and the year, day, and period during which it was packed. The packing period code shall be changed often enough to enable ready identification of lots during their sale and distribution. Codes may be changed periodically on one of the following bases: intervals of 4 to 5 hours; personnel shift changes; or batches, as long as the containers constituting the batch do not represent those processed during more than one personnel shift.

§ 114.83 Establishing scheduled processes.

The scheduled process shall be established by a qualified person who has expert knowledge acquired through appropriate training and experience in the acidification and processing of acidified foods.

§ 114.89 Deviations from scheduled processes.

Whenever any process operation deviates from the scheduled process for any acidified food and/or the equilibrium pH of the finished product is higher than 4.6, the commercial processor of the acidified food shall either: (a) Fully reprocess that portion of the food by a process established by a competent processing authority as adequate to ensure a safe product; (b) thermally process it as a low-acid food under part 113 of this chapter; or (c) set aside that portion of the food involved for further evaluation as to any potential public health significance. The evaluation shall be made by a competent processing authority and shall be in accordance with procedures recognized by

competent processing authorities as being adequate to detect any potential hazard to public health. Unless the evaluation demonstrates that the food has undergone a process that has rendered it safe, the food set aside shall either be fully reprocessed to render it safe, or be destroyed. A record shall be made of the procedures used in the evaluation and the results. Either upon completion of full reprocessing and the attainment of a safe food, or after the determination that no significant potential for public health hazard exists, that portion of the food involved may be shipped in normal distribution. Otherwise, the portion of the food involved shall be destroyed.

§ 114.90 Methodology.

Methods that may be used to determine pH or acidity for acidified foods include, but are not limited to, the following:

(a) *Potentiometric method for the determination of pH—(1) Principles.* The term “pH” is used to designate the intensity or degree of acidity. The value of pH, the logarithm of the reciprocal of the hydrogen ion concentration in solution, is determined by measuring the difference in potential between two electrodes immersed in a sample solution. A suitable system consists of a potentiometer, a glass electrode, and a reference electrode. A precise pH determination can be made by making an electromotive force (emf) measurement of a standard buffer solution whose pH is known, and then comparing that measurement to an emf measurement of a sample of the solution to be tested.

(2) *Instruments.* The primary instrument for use in pH determination is the pH meter or potentiometer. For most work, an instrument with a direct-reading pH scale is necessary. Battery and line-operated instruments are available commercially. If the line voltage is unstable, line-operated instruments should be fitted with voltage regulators to eliminate drifting of meter-scale readings. Batteries should be checked frequently to ensure proper operation of battery operated instruments. An instrument using an expanded unit scale or a digital readout system is preferred since it allows more precise measurements.

(3) *Electrodes.* The typical pH meter is equipped with a glass membrane electrode and a reference electrode or a single probe combination electrode. Various types of electrodes designed for specific uses are available. The most commonly used reference electrode is the calomel electrode, which incorporates a salt bridge filled with saturated potassium chloride solution.

(i) *Care and use of electrodes.* Calomel electrodes should be kept filled with saturated potassium chloride solution or other solution specified by the manufacturer because they may become damaged if they are allowed to dry out. For best results, electrodes should be soaked in buffer solution, distilled or deionized water, or other liquid specified by the manufacturer for several hours before using and kept ready by storing with tips immersed in distilled water or in buffer solution used for standardization. Electrodes should be rinsed with water before immersing in the standard buffers and rinsed with water or the solution to be measured next between sample determinations. A lag in meter response may indicate aging effects or fouling of the electrodes, and cleaning and rejuvenation of the electrodes may be necessary and may be accomplished by placing the electrodes in 0.1 molar sodium hydroxide solution for 1 minute and then transferring them to 0.1 molar hydrochloric acid solution for 1 minute. The cycle should be repeated two times, ending with the electrodes in the acid solution. The electrodes should then be thoroughly rinsed with water and blotted with soft tissue before proceeding with the standardization.

(ii) *Temperature.* To obtain accurate results, a uniform temperature should be maintained for the electrodes, the standard buffer solutions, and the samples. Tests should be made at a temperature between 20° and 30 °C, the optimum being 25 °C. Any temperature determinations made without meter compensation may affect pH values. An automatic temperature compensator may be used.

(iii) *Accuracy.* The accuracy of most pH meters is stated to be approximately 0.1 pH unit, and reproducibility is usually ± 0.05 pH unit or less. Some meters permit the expansion of any pH

unit range to cover the entire scale and have an accuracy of approximately ± 0.01 pH unit and a reproducibility of ± 0.005 pH units.

(4) *General procedure for determining pH.* When operating an instrument, the operator should use the manufacturer's instructions and should observe the following techniques for pH determinations:

(i) Switch the instrument on and allow the electronic components to warm up and stabilize before proceeding.

(ii) Standardize the instrument and electrodes with commercially prepared standard 4.0 pH buffer or with freshly prepared 0.05 molar potassium acid phthalate buffer solution prepared as outlined in "Official Methods of Analysis of the Association of Official Analytical Chemists" (AOAC), 13th Ed. (1980), section 50.007(c), under "Buffer Solutions for Calibration of pH Equipment—Official Final Action," which is incorporated by reference. Copies may be obtained from the AOAC INTERNATIONAL, 481 North Frederick Ave., suite 500, Gaithersburg, MD 20877, or may be examined at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html. Note the temperature of the buffer solution and set the temperature compensator control at the observed temperature (room temperature is near 25 °C).

(iii) Rinse the electrodes with water and blot, but do not wipe, with soft tissue.

(iv) Immerse the tips in the buffer solution and take the pH reading, allowing about 1 minute for the meter to stabilize. Adjust the standardization control so that the meter reading corresponds to the pH of the known buffer (for example, 4.0) for the temperature observed. Rinse the electrodes with water and blot with soft tissue. Repeat procedure with fresh portions of buffer solution until the instrument remains in balance on two successive trials. To check the operation of the pH meter, check the pH reading using another standard buffer such as one having a

pH of 7.0, or check it with freshly prepared 0.025 molar phosphate solution prepared as outlined in the AOAC, 13th Ed. (1980), section 50.007(e), which is incorporated by reference. The availability of this incorporation by reference is given in paragraph (a)(4)(ii) of this section. Expanded scale pH meters may be checked with pH 3.0 or pH 5.0 standard buffers. Buffers and instruments can be further checked by comparison with values obtained with a second properly standardized instrument.

(v) Indicating electrodes may be checked for proper operation by first using an acid buffer and then a base buffer. First standardize the electrodes using a pH 4.0 buffer at or near 25 °C. Standardization control should be adjusted so that the meter reads exactly 4.0. Electrodes should be rinsed with water, then blotted and immersed in a pH 9.18 borax buffer prepared as outlined in the AOAC, 13th Ed. (1980), section 50.007(f), which is incorporated by reference. The availability of this incorporation by reference is given in paragraph (a)(4)(ii) of this section. The pH reading should be within ± 0.3 units of the 9.18 value.

(vi) The pH meter can be tested for proper operation by shorting the glass and reference electrode inputs, thereby reducing the voltage to zero. In some meters this shorting is done by switching the instrument to standby, and in other instruments by use of a shorting strap. With the instrument shorted out, standardization control should be turned from one extreme to another. This operation should produce a deflection greater than ± 1.5 pH unit from center scale.

(5) *Determining pH on samples.* (i) Adjust the temperature of the sample to room temperature (25 °C), and set the temperature compensator control to the observed temperature. With some expanded scale instruments, the sample temperature must be the same as the temperature of the buffer solution used for the standardization.

(ii) Rinse and blot the electrodes. Immerse the electrodes in the sample and take the pH reading, allowing 1 minute for the meter to stabilize. Rinse and blot the electrodes and repeat on a fresh portion of sample. Oil and grease

from the samples may coat the electrodes; therefore, it is advisable to clean and standardize the instrument frequently. When oily samples cause fouling problems, it may become necessary to rinse the electrodes with ethyl ether.

(iii) Determine two pH values on the well-mixed sample. These readings should agree with one another to indicate that the sample is homogeneous. Report values to the nearest 0.05 pH unit.

(6) *Preparation of samples.* Some food products may consist of a mixture of liquid and solid components that differ in acidity. Other food products may be semisolid in character. The following are examples of preparation procedures for pH testing for each of these categories:

(i) *Liquid and solid component mixtures.* Drain the contents of the container for 2 minutes on a U.S. standard No. 8 sieve (preferably stainless steel) inclined at a 17- to 20-degree angle. Record weight of the liquid and solid portions and retain each portion separately.

(a) If the liquid contains sufficient oil to cause electrode fouling, separate the layers with a separatory funnel and retain the aqueous layer. The oil layer may be discarded. Adjust the temperature of the aqueous layer to 25 °C and determine its pH.

(b) Remove the drained solids from the sieve, blend to a uniform paste, adjust the temperature of the paste to 25 °C and determine its pH.

(c) Mix aliquots of solid and liquid fractions in the same ratio as found in the original container and blend to a uniform consistency. Adjust the temperature of the blend to 25 °C and determine the equilibrated pH. Alternatively, blend the entire contents of the container to a uniform paste, adjust the temperature of the paste to 25 °C, and determine the equilibrated pH.

(ii) *Marinated oil products.* Separate the oil from the solid product. Blend the solid in a blender to a paste consistency; it may become necessary to add a small amount of distilled water to some samples to facilitate the blending. A small amount of added water will not alter the pH of most

food products, but caution must be exercised concerning poorly buffered foods. No more than 20 milliliters of distilled water should be added to each 100 grams of product. Determine the pH by immersing electrodes in the prepared paste after adjusting the temperature to 25 °C.

(iii) *Semisolid products.* Food products of a semisolid consistency, such as puddings, potato salad, etc., may be blended to a paste consistency, and the pH may be determined on the prepared paste. If more fluidity is required, 10 to 20 milliliters of distilled water may be added to 100 grams of product. Adjust the temperature of the prepared paste to 25 °C and determine its pH.

(iv) *Special product mixtures.* For special product mixtures such as anti-pasto, pour off the oil, blend the remaining product to a paste, and determine the pH of the blended paste. If more fluidity is required, add 10 to 20 milliliters of distilled water to each 100 grams of product and blend. Adjust the temperature of the prepared paste to 25 °C and determine its pH.

(7) *Process pH determination.* Obtain sample portions of material for pH determination.

(i) For process liquids, adjust the temperature of the liquid to 25 °C and determine the pH by immersing the electrodes in the liquid.

(ii) Drain solid materials on a sieve and blend to a workable paste. Adjust the temperature of the prepared paste to 25 °C and determine its pH.

(iii) If enough solid materials are available to make a paste, blend representative aliquots of liquid and solid materials to a workable paste. Adjust the temperature of the prepared paste to 25 °C and determine the equilibrated pH. Alternatively, blend the entire contents of the container to a uniform paste, adjust the temperature of the paste to 25 °C, and determine the equilibrated pH.

(b) *Colorimetric methods for the determination of pH.* This method may be used in lieu of the potentiometric method if the pH is 4.0 or lower.

(1) *Principle.* The colorimetric method for pH involves the use of indicator dyes in solutions that gradually change color over limited pH ranges. An indicator that has the greatest color

change at approximately the pH of the sample being tested is selected. The pH is determined by the color of the indicator when exposed to the sample under test.

(2) *Indicator solutions.* Most indicator solutions are prepared as a 0.04 percent solution of the indicator dye in alcohol. In testing, a few drops of indicator solution are added to 10-milliliter portions of the sample solution. Colors should be compared using a bright background. Approximate determinations can be made on white porcelain spot plates, the test colors being compared thereon with a set of color standards. More accurate colorimetric tests can be made using a comparator block fitted with sets of tubes of standard indicator solutions of known pH.

(3) *Indicator paper.* A paper tape treated with indicator dye is dipped into the sample solution. Depending upon the pH of the solution, the tape will change color and an approximate pH can be determined by comparison with a standard color chart.

(c) *Titrateable acidity.* Acceptable methods for determining titrateable acidity are described in the AOAC, 13th Ed. (1980), section 22.060, under “Titrateable Acidity—Official Final Action,” for “Indicator Method,” and section 22.061 for “Glass Electrode Method—Official Final Action,” which is incorporated by reference. The availability of this incorporation by reference is given in paragraph (a)(4)(ii) of this section. The procedure for preparing and standardizing the sodium hydroxide solution is described in the AOAC, 13th Ed. (1980), sections 50.032–50.035, under “Sodium Hydroxide—Official Final Action” by the “Standard Potassium Hydroxide Phthalate Method,” which is also incorporated by reference and available as set forth in paragraph (a)(4)(ii) of this section.

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Subpart F—Records and Reports

§ 114.100 Records.

(a) Records shall be maintained of examinations of raw materials, packaging materials, and finished products,