Eleven causes of inaccurate pH measurement

by Fritz Buss, Laboratory Products Manager, Nelson-Jameson Inc.

Since becoming Laboratory Products Manager at Nelson-Jameson eight years ago, I've become particularly aware of two aspects of pH testing. First, acid development is the most critical parameter measured during the manufacturing process of cheese and other cultured dairy products. Second, pH values for given lots of product measured in different laboratories are among the most disputed of test results. For example, a recent informal survey conducted by a major cheese buyer found that split-sample test results obtained by several suppliers spanned a standard deviation exceeding 0.3 pH units.

Why do such large differences exist, and what can be done to make pH test results more accurate? It seems so simple — sticking a glass combination electrode into a piece of cheese and recording the value displayed by a digital meter — yet numerous factors can alter the test results. The following hypothetical account of a pH measurement of Cheddar cheese contains at least 11 possible sources of error. Can you find them? Read through the technician's procedures in the following example, then compare your ideas for improving the accuracy of this analysis with my list of recommendations on the next page.

Before beginning the first test of the day, a technician examined the pH meter and electrode. Our technician observed that the reference electrode chamber was half-full with fill solution, and also determined that the crystals that had formed at the bottom of the cavity were not excessive. The technician then calibrated the meter using two buffers — a pH 7 buffer in which the electrode was stored, and a pH 4 buffer kept in a closed jar at room temperature. The calibration adjustment of the meter was used to set 7, and 4 was entered using the slope control. Next the meter was placed in the absolute millivolt mode while the electrode remained in pH 4 buffer. The display indicated 145 millivolts. (errors 1-6) Following meter calibration, our technician removed a sample of Cheddar cheese from the refrigerator, and cut a half-inch slice from one side of the block to expose a fresh surface. The electrode was gently pressed into the freshly cut sample. At first, the numbers on the meter's display changed rapidly. After about 10 seconds, the numbers continued to change, but very slowly. The new-style meter's automatic end point recognition feature locked-in the display, and the pH value was recorded. (errors 7-9)

The technician noted that the electrode tip had acquired a coating of residue from the sample. This was washed away using a squirt bottle containing deionized water. The probe was then wiped dry with a piece of tissue and returned to the pH 7 buffer for storage. (errors10- 11)

The technician in this example probably used more care than most, yet there are at least 11 instances where technique could be improved. Turn to the next page to see what they are.

Eleven recommendations for more accurate pH testing

1. Keep the reference electrode chamber filled to the fill port with electrolyte solution. This creates a slight hydrostatic pressure that helps the solution flow through the junction.

2. Any crystals in the reference component of a combination electrode can cause problems by clogging the junction. Remove crystals by flushing with warm deionized water.

3. Keep calibration buffers in separate, closed containers at room temperature, and replace them after a few days of use. Keep reserve supplies refrigerated in tightly closed bottles marked with the dates they were first opened. Once opened, buffers should be used within two months and must be discarded if any cloudiness or specks appear in the liquid.

4. After setting 4 and 7 calibration points to bracket the anticipated pH range of the cheese samples, use an intermediate buffer such as pH 5 to check the accuracy of the calibration. A displayed value that deviates more than ± .03 pH units from 5 indicates an inaccurate meter setting for the pH 4 or pH 7 buffer.

5. Before using the absolute millivolt mode of a pH meter to check electrode slope, set the meter to zero millivolts using a shorting strap or plug.

6. A responsive electrode normally has a slope of ± 50-60 millivolts per pH unit. Given a value for pH 7 buffer of zero millivolts (neutral — neither acid nor alkaline), the electrode in the example should have produced a reading of at least 150 millivolts while in the pH 4 buffer.

7. Sample and buffer temperatures should be the same. If the temperature difference between the buffers and sample is excessive (more than 5°C), recalibrate the meter using the temperature of the sample as a basis.

8. Blend cheese samples, especially those from recent production, before measuring pH. The probe will then contact the sample in a location that represents its average composition.

9. Even slow drifting of displayed pH values after 10 seconds of contact with the sample indicates a problem with the electrode. In the example, the junction of the electrode is probably clogged. While new meters with automatic endpoint recognition are convenient, they may conceal drifting by artificially creating a stable reading before the "best" value has been reached.

10. Use a fat solvent followed by an acidic solution to dissolve the film of fat, protein, and minerals that accumulates on the electrode tip when cheese and other milk products are tested. While a squirt bottle is good for removing cheese particles, it is ineffective for removing this film.

11. Avoid rubbing the glass or plastic electrode body while drying it, as this may create a static charge. Simply blotting the end of the probe with a tissue or cloth will remove droplets without affecting the electrode's sensitivity.

Fritz Buss demonstrates procedures for pH measuring and electrode maintenace in a new training videotape produced by Nelson Jameson Inc. See Dairy Resource Center *(page 10) or call CDR Videotapes at (608) 262-2217 for information.*

Article Taken from:

