Measuring pH in Acidified Foods

Find out all you need to know about measuring pH in acidified foods.

HANNA® instruments

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Introduction

To succeed in creating high quality foods, analysis should be at the heart of your strategy. Quantitative analysis needs to be a driving force behind decision-making for food formulations, quality control, and food safety.

Analytical testing can easily be integrated into the food production process. From hobbyists to food scientists, many involved in food quality and safety now use a variety of analytical techniques to create stable, safe, and most of all, great tasting foods.

Analyzing data is of no use unless the data represents the actual condition of the food. The quality of analytical data depends on proper sampling, appropriate method selection, and effective measurement techniques.

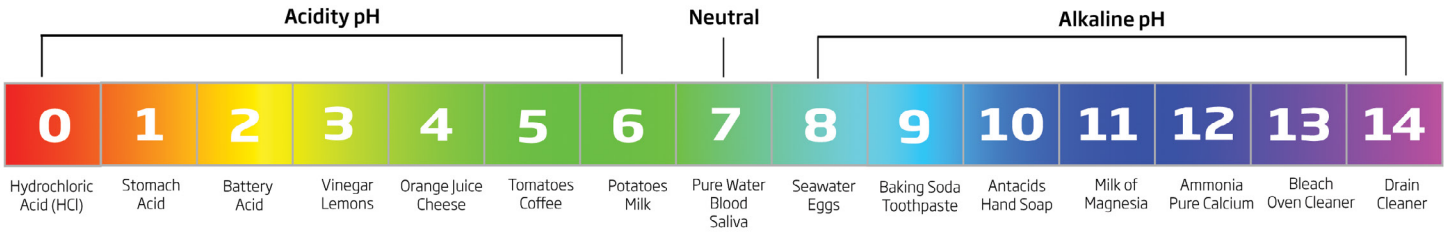
This eBook will discuss how to utilize pH testing to ensure food quality and safety. It will cover how pH affects food quality, as well as the necessary tools and suggested measurement procedures, which will be highlighted for liquid, semisolid, and solid food samples.

Why pH Matters

What is pH?

In technical terms, pH is the hydrogen ion activity in a solution. The equation is commonly written as: pH = -log [H+]

This equation will determine pH according to a scale from pH 0 to 14, with solutions less than pH 7 being acidic and solutions greater than 7 being basic. A pH of 7 is neutral and is neither an acid nor a base.



How does pH affect food quality?

Flavor	Organic acids, such as citric acid, can provide a tart or sour flavor to foods.
Fermentation	pH affects bacteria used in food production to make cheese, yogurt, vinegar, and soy sauce, to name just a few. Yeast performs best at a pH of 4.5-6.0.
Texture	Texture is particularly susceptible to pH changes. Low pH will result in a cheese without shape or hold while high pH causes cheese to crumble.
Appearance	pH plays a role in haze formation and contributes to pigment. Anthocyanins, the red pigments found in berries, turn blue, green, or yellow in alkaline conditions.
Shelf stability	pH prevents spoilage by inhibiting bacterial growth. Bacteria, such as *E.coli*, require pH higher than 4.6 to thrive; anything lower will inhibit growth.

Regulations

The Code of Federal Regulations (CFR) is an annual edition of general and permanent rules produced by various US government agencies including the Food and Drug Administration (FDA). The methodology of pH measurement for food products and how they are processed can be found in 21CFR114.

The classification of food, based on pH, is found in 21CFR114.3. This section divides foods into multiple groups based on their pH value, stating:

Acid Foods are foods that naturally have a pH of 4.6 or below.

Low-acid Foods are any foods, except alcoholic beverages, with a finished equilibrium pH values greater than pH 4.6 and a water activity greater than 0.85a_w.

Low acid foods can be pH adjusted by the addition of acid or acid foods to lower the pH to 4.6 or below.

Acidification reduces the risk of contamination by bacteria such as *C. botulinum.*

Even though a low-acid food can be acidified, high heat and pressure may still be required to adequately sterilize the product.

Regulations (cont.)

A low acid food that is pH adjusted is known as an Acidified Food.

They are defined as:

- Having a water activity greater than 0.85a_w
- Are stored and distributed under non-refrigerated conditions

21CFR114.8 states that frequent testing and recording of results should be performed. Manufacturing must be in accordance with a scheduled method, and acidified foods shall be thermally processed to destroy bacteria.

21CFR114.9 cites methodology for pH measurements:

- Use commercially prepared buffer.
- pH meter with accuracy to be approximately + 0.1 pH unit.
- Determine pH of samples at room temperature (25 °C).

Preparation of samples:

- Direct insertion into liquid samples.
- Solids and semisolids should be processed as a paste of uniform consistency.
- Oil layers may be discarded to reduce clogging.

What You Need

1. pH meter

Must have –Minimum 2 point calibration, automatic temperature compensation, 0.01 pH resolution, and a mV scale

Good to have – Built in electrode diagnostics, Cal Check, GLP - Good Laboratory Practice, data logging capability, and a USB port for transferring data.

2. pH electrode

Must have – Combination pH electrode (both the glass and reference electrode are found in one body)

Good to have – Application specific electrode made for taking direct measurements of food products with a built-in temperature sensor for automatic compensation of temperature variations.

Hanna Note

CAL Check™ alerts users to potential problems during the calibration of the pH electrode. Indicators include "Electrode Dirty/Broken," "Buffer Contaminated," as well as the overall probe condition.

GLP data includes date, time, buffers /standards used for calibration and slope/offset characteristics. This and the ability to log data are helpful in maintaining good record keeping as cited in 21CFR114.8.

What You Need

Juice

HI10480 | Wine/Juice Reference Electrode for Samples with Suspended Solids

Milk

FC101D | Amplified pH electrode with PVDF body

Cheese

FC240B | Small Diameter pH Electrode with Stainless Steel Body

Semi-solids

FC230B | pH Electrode for Meat and Semi-Frozen Products

Meats

FC231 | pH SMART Electrode for Meat and Semi-Frozen Products
+ FC099 Stainless Steel Blade (1.4" blade)

3. Calibration Solutions

The calibration buffers used should bracket (see note) the expected sample pH. Generally, pH 4.01 and 7.01 buffers will bracket most food samples.

– The calibration is only as good as the buffer used. Any changes in pH of the buffer will result in an inaccurate reading.

– Once opened, buffers should be changed at least every six months.

Hanna Note

When calibrating a pH electrode it is important to choose buffers that "bracket" the expected reading. For example, if the expected reading is pH 4.2 then it would be best to calibrate to pH 4.01 and pH 7.01.

If the expected reading is pH 3.2 then it would be best to use a pH meter that allows for custom buffers. There are many other buffers available other than the pH 4.01, 7.01 and 10.01.

Hanna Instruments offers buffers from pH 1.00 through pH 13.00.

4. Cleaning Solutions

– Contamination on the glass sensing surface and clogged junctions are two of the most common reasons for poor electrode performance.

– In foods, this is particularly common because fat and protein residues can easily coat the electrode and clog the junction of the pH electrode.

– Application specific cleaning solutions are formulated to clean the pH electrode based on the sample composition. For example, the HI7073 is an enzymatic cleaner for cleaning proteins.

Application specific cleaning solutions made by Hanna include general, protein coating, inorganic soak, and an oil and grease rinse.

Hanna Note
Handling the glass tip of the electrode or wiping it clean with a cloth can damage the sensor, resulting in inaccurate measurements.

5. Storage Solutions

– A dry pH electrode can contribute to poor performance. A pH electrode will display one pH value when dry and a different value when hydrated.

– Every glass pH electrode will form a hydrated layer on the glass. It takes around 3-4 hours for the hydration layer to completely form.

– A storage solution is designed to keep the electrode bulb hydrated. Storage solutions are also formulated to prevent organic growth which can coat the electrode bulb.

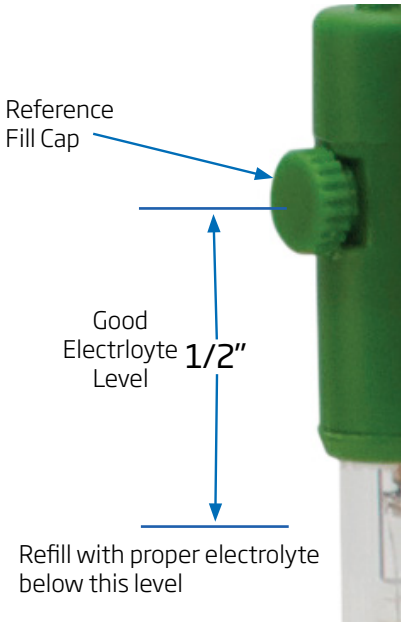
Hanna Note

Never use deionized (DI) water for a storage solution. The DI water will have an osmotic and diffusion effect on the reference cell.

The concentrated salt solution found in the reference cell will change. If altered too much, the solution will have to be replenished. For a gel-filled electrode, the probe will have to be replaced.

6. Fill Solutions

Fill Solution: The filling solution of a pH electrode diffuses slowly from the inside of the pH electrode, losing anywhere from 15 – 45 µL/hour (0.33 to 1 drops/hour). The fill solution must be topped off to maintain a proper level.



Hanna Note

Refillable pH electrodes are available in a single junction and double junction design. Single junction pH electrodes use a 3.5 M KCl solution with AgCl while a double junction electrode uses 3.5M KCl without AgCl.

What You Need

Additional items for a good testing set up:

HI181-1

Magnetic stirrer: A stirrer should have variable speed control to allow for proper sample mixing. Mixing will ensure a faster electrode response, greater stability, and more accurate measurements.

HI76405

Electrode holder: An electrode holder will help hold the electrode in the correct position for measurement. A holder will also help to avoid damage to the glass pH bulb by holding it in a secured position.

HI70036P

Labware: 100 mL beakers for samples to be tested, 500 mL waste beaker, and a laboratory wash bottle with deionized or distilled water.

An Ideal pH Meter Makes Testing Easy

Flexibility: Today's meters offer a wider range of flexibility. A portable meter offers the ability of in-field use with the performance of a benchtop.

Ease of use: Measurement, configuration, calibration, and logging should be easy to do with data management features including direct / USB data transfer.

Simple maintenance: Advanced pH meters have diagnostics capability to inform of problems during the calibration process. Diagnostic messages should include the overall probe condition and whether the probe should be cleaned, or if the buffer is contaminated.

HI98190

What You Need

HI99161

HI98190

HI2002

HI5222

General Information

HI99161	HI98190	HI2002	HI5222
Type	**Type**	**Type**	**Type**
Portable & Waterproof	Portable & Waterproof	Portable/Benchtop	Benchtop
Perfect for: Field Use	Perfect for: Field Use	Perfect for: Field Use/Lab	Perfect for: Lab

Data

HI99161	HI98190	HI2002	HI5222
Connectivity	**Connectivity**	**Connectivity**	**Connectivity**
N/A	USB Port	2 USB port for PC and storage	USB Port
Logging	**Logging**	**Logging**	**Logging**
No	200 samples	1,000 samples	100,000 data point storage

Special Features

HI99161	HI98190	HI2002	HI5222
ATC	**ATC**	**ATC**	**ATC**
Yes	Yes	Yes	Yes
Cal Check	**Cal Check**	**Cal Check**	**Cal Check**
No	Yes	Yes	Yes
GLP	**GLP**	**GLP**	**GLP**
No	Yes	Yes	Yes
Calibration Point(s)	**Calibration Point(s)**	**Calibration Point(s)**	**Calibration Point(s)**
One or two calibration points	Up to five calibration points	Up to five calibration points	Up to five calibration points

ATC - (Automatic Temperature Compensation)

Cal Check - Alerts users to potential problems during the calibration of the pH electrode (unique to Hanna meters)

GLP - (Good Lab Practices) Ensures consistency and reliability of your tests

Calibration Point(s) - The more calibrations you have the more accurate your readings will be

2.5-inch (diagonal)

2.75-inch (diagonal)

5.5-inch (diagonal)

3.5-inch (diagonal)

1. Calibration

The following procedure is based on the best practice of using two beakers for each calibration buffer.

a. Prepare the electrode.

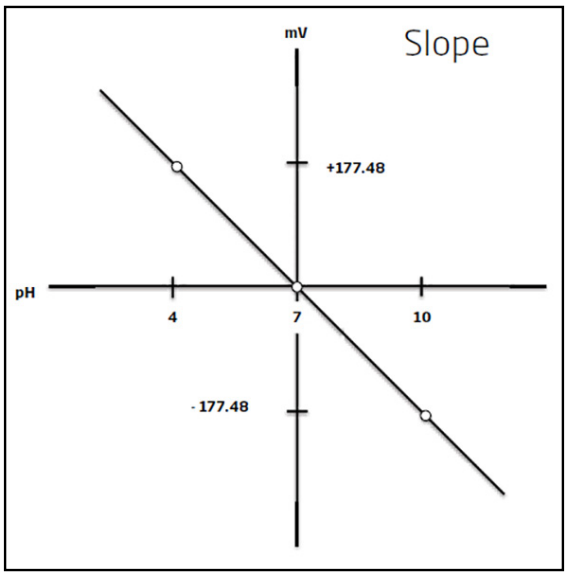
- Remove the protective/storage cap from the electrode.
- If the pH bulb is dehydrated, place in storage solution for at least 3-4 hours.
- If the pH electrode is a refillable design, then remove the fill hole screw cap.

b. Prepare the calibration buffers.

- Fill two beakers with enough pH buffer solution 7.01 to cover the pH electrode junction (approximately 75 mL in a 100 mL beaker).
  One of the beakers will be used to rinse the pH electrode and the other for the actual calibration.
- Repeat for any other pH buffer (i.e. pH 4.01) that is used.

c. Perform a two point calibration

- Rinse the pH electrode with Deionized water.
- Rinse the pH electrode in the pH 7.01 rinse beaker.
- Place the electrode in the pH 7.01 calibration beaker and stir.
- Wait for the reading to stabilize and confirm the calibration.
- Repeat this procedure for the second point with pH 4.01 buffer.



Hanna Note

Calibration parameters to monitor include slope and offset. When a pH probe is placed in a solution, a voltage is generated. The offset of a probe is the mV reading in pH 7 buffer. The slope of a probe is based on the mV difference between two buffer solutions. A probe should always have a slope minimum of 85%, ideally greater than 90% slope and any probe with an offset outside +/- 30 mV should be cleaned and conditioned prior to use. These parameters provide information on the probe's overall condition.

To learn more about how to calculate your slope and offset click here to download **Calculating Your Slope and Offset Cheat Sheet** or **watch the video** on " How to check the slope and offset of a pH meter"

2. Measure

a. Procedure for liquid samples

- Do not start taking measurements unless the pH electrode has been properly hydrated and calibrated.
- Fill two beakers with enough sample to cover the pH electrode junction (approximately 75 mL in a 100 mL beaker). Use one of the beakers to rinse the pH electrode and the second for the actual measurement.
- Rinse the probe with deionized water
- Place the electrode in the rinse sample beaker.
- Place the sample beaker to be measured on a magnetic stirrer and drop a clean magnetic stir bar into it.
- If available, place the pH electrode into the electrode holder and lower it until the junction is fully immersed.
- Wait for the reading to stabilize before recording the measurement.

b. Procedure for semi-solid and solid samples

- Direct Measurement with application specific pH electrodes
- Do not start taking measurements unless the pH electrode has been properly hydrated and calibrated.
- If a solid, use a knife or auger to make a hole for the pH electrode.
- Insert the tip of the probe into the hole. Ensure that the electrode junction is covered by placing the electrode at least 2cm (0.75") into the sample.
- Wait until the pH reading is stable before recording.

Hanna Note

The measurement of solid and semi-solid samples is difficult. Either a specialized electrode that is designed to take a direct measurement is needed, or the sample has to be pretreated (i.e. made into a slurry) so that a standard pH electrode can be used.

Meats

FC231 | pH SMART Electrode for Meat and Semi-Frozen Products

+ FC099 Stainless Steel Blade (1.4" blade)

Hanna Note

Some Food Care pH electrodes offer the option to use a stainless steel blade. The blade is used to pierce the sample and protect the glass pH bulb.

b. Procedure for semi-solid and solid samples (cont.)

Slurry Method for pH Measurement

- The slurry method involves taking a sample mixed with deionized/distilled water to make a solution that can be tested.
- 21CFR114.9 (Council for Federal Regulation) recommends 10-20 mL of distilled water should be added to each 100 grams of product. The mixture is homogenized and the resultant slurry tested.
- The testing procedure would then be the same as a liquid sample.

Hanna Note

The addition of distilled/deionized water should have little effect on the pH of the sample since the water has no acidity/alkalinity (buffering capacity). It takes very little effort to change the pH of high purity water.

Click Here to Read Our Case Study: "Comparison of pH Measurements of Food Products Using Various pH Electrode Designs and Sample Preparations"

3. Cleaning a pH electrode

• Once you are finished measuring your samples, slide the pH electrode from the electrode holder and rinse it with water until all food residues are removed.

• Periodically the pH electrode should be cleaned with a pH electrode cleaning solution. The cleaning procedure is:

– Fill a 100ml beaker with approximately 75 mL cleaning solution.
– Place the pH electrode into the cleaning solution, making sure the junction is covered.
– Soak for 10 to 15 minutes.
– Rinse the electrode with DI water.
– Soak in storage solution for at least two hours before next use.

Hanna Note
Knowing the offset (mV value in pH 7.01 buffer) is a great way to know if the probe needs to be cleaned. A new pH electrode will have an offset of +/- 15 mV. A large change is an indication that the probe is coated and requires maintenance. A meter with GLP option will display the offset.

4. Storing a pH electrode

- Fill the storage cap of the pH electrode to the half point with storage solution and replace the storage cap on the electrode.

- Make sure there is enough storage solution in the cap to cover the tip of the pH electrode.

- For refillable pH electrodes, check to see if the probe needs to be refilled. The fill solution should be less than a half-inch below the fill.

THANKS FOR READING!

Our experts are here to help you.

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