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User Guide

Determination of pH in hummus
by pH electrode measurement

Orion Pro Star PH211
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Introduction

pH testing of prepared food products is an essential part of the food manufacturing process. The pH level of the food during the processing and at packaging determines its quality and consistency. pH levels of the final packaged product can affect the shelf-life, the flavor profile, and the safety of consuming the food. The presence or addition of acids can impart a pleasant tart taste and can function as a preservative by stopping or slowing the growth of food-poisoning and spoilage bacteria. In the United States, the Food and Drug Administration (FDA) has set a maximum pH of 4.6 for packaging certain acid, acidified, and fermented foods. This pH can control the risk of botulism. Acid may not be the sole control measure, but is combined with other control measures such as heat, salt, sugar, preservatives, or other "hurdles" to control growth of unwanted microorganisms.[1,2]

A pH electrode and meter are used for accurate pH testing, especially in the critical range from pH 4.0 to 4.6. [2,3] Temperature measurement is needed to verify the sample temperature and to automatically adjust the pH calibration based on temperature changes.

Key words

pH testing of food, acidified foods, FDA acidified foods, potentiometric method for the determination of pH, acid foods, 21 CFR Part 114, food testing, food safety, food preservation, AOAC 981.12, ISO 11289, NMKL 179.

Equipment

- Thermo Scientific™ Orion™ pH bench meter
- Thermo Scientific™ Orion™ ROSS Ultra™ 8157BNUMD refillable pH/ATC Triode™ or Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ 8172BNWP pH Electrode.
- Thermo Scientific™ Orion™ automatic temperature compensation (ATC) probe
- 50 and 100 mL beakers
- Large plastic beaker
- Laboratory timer
- Lab wipes
- Laboratory wash bottle filled with reagent grade water

Required solutions

- Thermo Scientific™ Orion™ pH buffers: pH 4.01, pH 7.00, and optional pH 5.00 buffer.
- Thermo Scientific™ Orion™ ROSS™ pH electrode filling solution
- Thermo Scientific™ Orion™ ROSS™ pH electrode storage solution
- Reagent grade water (RGW)

Electrode setup

See the electrode user manual for the first time preparation of the electrode. See Table 3 “Best practices for pH electrode care” for a summary of daily, monthly, and as-needed care for the electrode.

Meter setup

1. Connect the pH electrode to the BNC connector on the meter.
2. Connect the ATC or the temperature connection of the triode cable to the ATC connector on the meter.
3. Power up the meter, open the Main Menu / Settings / Instrument Settings, and set the Read Type to Continuous. Save.
4. In Main Menu / Settings, select pH Settings. Set pH Resolution to 0.01 pH, pH Buffer Set to USA, pH Buffer points to 2, and Slope Type to Segmented. Select Slope Guidance. Set Excellent to 95 to 101%. Set Fair Low Value Range to 92 to 95 and Fair High Value Range to 101 to 102. Set Bad Lowest Value to 92 and Highest Value to 102.
5. Select Create Method and type in the desired method name. Save and Load the method.

Temperature is an important factor when testing pH. The temperature affects the calibration of the pH electrode and can change the pH of the food product itself. Use a Triode (having an integrated temperature sensor) or a separate ATC probe to measure temperature and automatically adjust the pH slope to help keep the pH electrode in calibration.

Test the pH of your food product at a consistent temperature to ensure readings are comparable and will fall within desired pH limits when the product is good.

User prepared solutions

- Short term storage solution: Pour 25 mL of the pH 7.00 buffer into a 50 mL beaker. Label as “short term storage”. Pour fresh daily. Using this solution for storage during the day can speed up the testing, reduce drift, and improve precision and accuracy.

Calibration

1. Prior to calibration, condition the pH electrode in the short-term storage solution for about 5 minutes.
2. Prepare the pH buffer standards for testing as follows: for each standard, pour 50 mL of buffer into a clean dry 100 mL beaker.

   a. It is recommended to calibrate at pH 4 and pH 7. The pH 5.0 buffer standard can be used for an independent calibration verification (calibration check standard) after calibration and/or throughout the day.
3. Check that your saved pH of hummus method name is displayed on the Measurement screen. If not, load the method. From the Main Menu, select Methods, and tap the Play icon to load the method of choice.
4. Select Calibrate to begin the calibration. Perform a two point calibration using the pH 4.01 and pH 7.00 buffer standards.
   a. Rinse the electrode (and ATC, if separate) with generous amounts of RGW before and after each pH buffer standard reading. Gently shake to remove excess water drops. Blot lightly and gently, if desired. Do not wipe the glass bulb or touch with fingers or hands.
   b. Lower the electrode (and ATC) into the pH 4.01 pH calibration buffer so that about 1 inch of the electrode (and ATC) is immersed.
   c. Swirl the beaker of pH standard buffer around the electrode(s) for 10 seconds.
   d. Select Start (or Continue) to proceed and start a 1 minute timer. Allow at least 1 minute for the pH standard buffer to reach a stable reading.
      i. To ensure good results, it is important to wait at least 1 minute for a truly stable calibration point. Do not rush the calibration. A pH electrode in good working order will typically take from 1 – 2 minutes to stabilize fully.
   e. After at least 1 minute and when the pH buffer standard reading becomes stable, the meter will automatically display the correct pH buffer value for the recognized standard at the measured temperature.
   f. Select Next to accept the value and proceed to the next calibration point (or Done after both pH buffer standards are measured).
   g. Repeat steps a. through f. for the pH 7.00 pH buffer standard, tapping Continue.in step d to proceed.
5. After calibrating with the two standards, select Done to finish the calibration. The calibration is automatically logged into the meter.
   a. The calibration slope will be displayed. The slope of a well-functioning pH electrode properly calibrated with fresh, unexpired buffers will generally be between 95 and 101 %.
   b. A slope between 92 and 102 % indicates the need for electrode maintenance and the possibility that the buffers are no longer good.
   c. A slope that is not between 92 and 102% is not acceptable. Do not proceed until an acceptable slope is achieved. See the Performance Check section below for corrective actions.
6. If the slope is good, return to Measure mode. Rinse as before, then proceed to the calibration verification or to sample testing. Otherwise, place the pH electrode into short term storage solution until it is needed.
7. If the slope is not acceptable, see the Electrode Performance Check section below for corrective actions.
8. Recommended calibration verification: Read the pH 5.00 buffer standard to verify the calibration. Set a timer and take the reading after 1 minute. If desired, take another reading at 2 minutes to ensure a stable reading has been achieved.

a. After a successful calibration of a well-performing electrode, the calibration check standard will read a value close to the expected value at the measured temperature. Consult your quality department for acceptance limits.
b. Alternately, a simple calibration check can be done by reading one of the calibration buffers (pH 4.01 or pH 7.00) after the calibration is completed to check that it reads as expected.
c. See Table 4 pH vs. temperature of standard calibration buffers to determine the expected value of the buffer at the measured temperature.

Measuring a calibration verification buffer standard has benefits. The best choice is a pH value that differs from the pH buffers used for calibration. After a calibration, a good reading for the verification standard gives confidence that the pH calibration buffers are not compromised and that the calibration proceeded successfully, as expected.

Throughout the day, a good calibration verification reading indicates that the pH equipment is still in calibration and may not require recalibration. Check with our quality department for calibration requirements.

9. If the calibration verification is not acceptable, take corrective actions as follows:
    a. Review the meter calibration log to verify the calibration proceeded as expected. If not, check the buffer standards, as noted in the Electrode Performance Check, below.
    b. Check the electrode for drift, as noted in the Performance Check, below.
    c. Consult the troubleshooting section of the pH electrode user manual.
10. It is best to ensure a good calibration verification reading is achieved before proceeding with sample testing.
11. Proceed to sample preparation and analysis after a good calibration and good calibration verification.

Electrode performance check

If the calibration slope is not between 92 and 102%, conduct a performance check:

1. Check the pH buffer standards: Discard any used standards. Check the expiration date on the buffers and discard any that have expired. Check the date the bottle was opened. Opened bottles are subject to cross-contamination, evaporation, and oxidation which can impact the integrity of the buffer. Open a new bottle or pouch of buffer if the bottle has been open for more than 6 months or has not been stored properly.

2. Check the electrode: Rinse well, and place the electrode in the pH 4.01 buffer. Swirl for 10 seconds. Take a mV reading at 1, 2, and 3 minutes. The 2 minute mV reading should be between 117 and 236 mV and ideally within 1 or 2 mV of the 1 minute reading. If the electrode takes more than 3 minutes to stabilize to 1 or 2 mV/minute or is not within the mV range, perform maintenance as described in Table 3 Best practices for pH electrode care. For more information, consult the troubleshooting section of the electrode user manual. If maintenance does not restore the performance, replace the electrode.

The pH electrode determines the performance of the testing. Follow the electrode care and maintenance recommendations in Table 3 to help ensure the fastest, most reliable pH readings and to extend the usable lifetime of the electrode.

Sample Preparation

1. Depending on your protocol, allow the samples to come to room temperature (RT).
2. However, if your sample needs to be tested at a warm or cool temperature, take measures to stabilize the temperature during the pH testing (since temperature changes during the testing can lead to drift and changes to the pH reading).
3. If the pH will be tested directly in the product package, proceed to the Sample Analysis section that follows.
4. If testing a portion of the product, use a clean spoon or spatula to transfer some of the sample into a small clean, dry beaker. Fill the beaker to about 2 inches deep and make sure to eliminate any visible air pockets that might form in the sample.
5. The sample is ready for analysis.

Sample Analysis

1. Check that your saved pH of hummus method name is displayed on the Measurement screen. If not, load the method. From the Main Menu, select Methods, and tap the Play icon to load your method.
2. Rinse the pH electrode and ATC probe (if using) with generous amounts of RGW and gently shake to remove excess water drops. Blot lightly and gently if desired. Do not touch the glass bulb with fingers or hands.
3. Place probe(s) in the sample, pushing straight down into the sample. Ensure the bottom 1 inch of the pH electrode (and ATC) is fully submerged and that no air pockets have formed around the pH electrode.

   a. If air pockets form around the pH glass bulb and junction, unstable readings may result. Slightly twist or wiggle the beaker to encourage good contact between the sample and the pH electrode. If necessary, remove the electrode and replace in a different location in the sample.

4. Set the timer for 1 minute and start timing.
5. After 1 minute, record the pH and temperature readings or save the readings by selecting Log/Export. If the pH reading is not as expected, allow another minute (2 minutes total) for a more stable pH reading, then record the pH and temperature results.
   a. If the pH reading at 2 minutes is not within 0.05 pH unit of the 1 minute reading, the electrode is responding slowly. Allow another minute and collect the 3 minute pH and temperature readings. Before overnight storage of the electrode, do maintenance on the electrode to restore quick response.
   b. If the temperature reading at 2 minutes is not within 1.0 degree of the 1 minute reading, the sample temperature is changing. Take action to stabilize the temperature before collecting the final pH and temperature readings.
6. After the measurement, use a lab wipe to gently remove excess hummus from electrode. Then, swirl the pH electrode (and ATC) in a large plastic beaker of tap water to remove all the sample from the glass pH bulb and the reference junction. Then rinse the pH electrode (and ATC) with generous amounts of RGW. Gently shake off excess water drops. Blot lightly and gently, if desired.
7. Repeat steps 2 through 6 until all the samples have been measured.
8. For storage between samples or throughout the day, place the electrode(s) into the prepared short-term storage solution.

After Sample Analysis

1. When testing is done for the day, rinse the electrode thoroughly with RGW. If necessary, clean the electrode before storage. Swirl the pH electrode in 1% warm (not hot) lab detergent solution until clean, then rinse thoroughly with RGW until all traces of detergent are removed.
2. Replace the fill hole cover (if using a refillable electrode) and place the electrode into the pH electrode storage solution.
3. Discard used pH buffer standards at the end of the day. Do not pour standards back into the bottle or save for reuse. Reusing standards can lead to alteration and/or contamination of the standard, which can cause incorrect readings.

Quality control (QC)

Recommended QC procedures may include: calibration verification (described above); analysis of a prepared or purchased QC sample; and/or sample duplicates. Consult your Standard Operating Procedures (SOPs) or your quality department for requirements.

pH Testing performance demonstration

To demonstrate the accuracy, reproducibility, and stability of the pH electrode in hummus, five readings (replicates) were taken of a packaged hummus product using 8157BNUMD and 8172BNWP electrodes. A calibration verification (calibration check) standard at pH 5.00 was measured after each calibration and another calibration verification standard at pH 4.01 was measured after the hummus readings.

See Tables 1 and 2 for the demonstration of performance. The calibration standard readings before and after hummus testing were accurate and the replicate sample readings of the hummus were repeatable, showing good agreement. Both the 8157BNUMD and the 8172BNWP electrodes showed good performance for pH testing of hummus.

Table 1. Performance of Orion 8157BNUMD in hummus

Calibration				
Calibration slope	99.2% ✓			
Calibration Verification				
	pH measured*	**Temperature, °C**	**Expected buffer pH**	**pH difference**
Before testing	5.02	20.5	5.00	0.02 ✓
After testing	3.99	20.5	4.01	0.02 ✓
Hummus testing				
	pH measured*	**Temperature, °C**		
Reading 1	4.46	19.3		
Reading 2	4.47	19.2		
Reading 3	4.46	19.3		
Reading 4	4.46	19.3		
Reading 5	4.46	19.7		
Average	4.46			
Standard deviation	0.01 ✓			

*all readings at 60 seconds

Table 2. Performance of Orion 8172BNWP in hummus				
Calibration				
Calibration slope	99.1%			
Calibration Verification				
	pH measured*	**Temperature, °C**	**Expected buffer pH**	**Difference from expected**
Before testing	4.99	20.6	5.00	0.01 ✓
After testing	3.97	20.6	4.01	0.04 ✓
Hummus testing				
	pH measured*	**Temperature, °C**		
Reading 1	4.46	17.9		
Reading 2	4.43	18.6		
Reading 3	4.43	18.6		
Reading 4	4.39	18.7		
Reading 5	4.41	18.7		
Average	4.42			
Standard deviation	0.03 ✓			

*all readings at 90 seconds

Hints and tips for pH testing in hummus and semi-solid foods

- Refer to the electrode user manual for details on cleaning, storage, and maintenance recommendations to keep the electrode performing well. Main points for electrode care are summarized below in Table 3.
- Conditioning the electrode in a portion of a pH buffer standard for a few minutes before calibration will speed up the calibration and improve stability and accuracy. Short-term storage in this solution during the day will also speed up sample results and improve precision and accuracy.
- Semi-solid food samples may have limited flow and therefore may not have ideal contact with the pH glass bulb and the electrode reference junction. Give attention to pushing the electrode straight down into the food and avoid creating air pockets that will interfere with good results. If the pH reading is not as expected, remove the pH electrode and try inserting the electrode in another location in the sample.

- Use a timer and allow at least 1 minute wait time before accepting a pH measurement. This applies to the calibration and to testing semi-solid foods. If the reading has not stabilized or is not as expected, wait another minute.
- Don't rush the calibration or the sample readings. It is worth the extra minute, and your results will be better for it.
- Semi-solid foods can be challenging to remove from the pH electrode after testing. Using a lab wipe, gently wipe away the excess sample from the electrode. Then swirl the electrode in a large plastic beaker of tap water to remove every trace of the food product from around the glass pH bulb and the reference junction. Finish by rinsing thoroughly with generous amounts of RGW.
- pH standards are meant to be poured fresh each day of testing. Discard used standards at the end of the day. Do not pour standards back into the bottle or save for reuse. Reusing standards is not advised. It can lead to contamination or alteration of the standard and can compromise your results.

Electrode Care

Refer to the electrode user manual for details on cleaning, storage, and maintenance recommendations to keep the electrode performing well. Main points for care are summarized below.

Table 3. Best practices for pH electrode care

Daily Care	Monthly Care	As Needed
•Open the fill hole cover and top up the appropriate electrode filling solution to the bottom of the fill hole. Leave the fill hole cover off during testing. •Prepare short term storage solution by pouring 25 mL of the pH 7.00 buffer into a 50 mL beaker. •Before and after each pH reading, rinse with generous amounts of RGW. Also direct the spray between the pH bulb protective prongs to wash the bulb, junction, and thermistor. •After testing, store the clean electrode in the pH electrode storage solution. Replace the fill hole cover prior to storage.	•Drain the fill solution and rinse the chamber with RGW*. Replace the electrode filling solution. •Discard the used overnight storage solution. Use fresh solution. •If electrode becomes drifty or slow, soak 15 minutes in warm (not hot) 1% laboratory detergent. Rinse thoroughly, drain the fill solution, and refill (if refillable), then store in overnight pH electrode storage solution, as usual.	•For long term storage (more than 1 or 2 weeks), follow the instructions in the pH electrode user manual. •If crystals form inside a refillable electrode or the fill solution becomes discolored, drain the fill solution. Then rinse the chamber until crystals or discoloration are gone. Replace the filling solution. •See the electrode user manual for more maintenance details.

*See the electrode user manual for details on all electrode care procedures.

Table 4. pH vs. temperature of standard pH calibration buffers					
pH Buffers	**15°C**	**20°C**	**25°C**	**30°C**	**35°C**
pH 4.01 buffer value	4.00	4.00	4.01	4.02	4.02
pH 5.00 buffer value	5.00	5.00	5.00	5.00	5.01
pH 7.00 buffer value	7.03	7.01	7.00	6.98	6.97

Ordering information

Product	Description	Cat. No.
Bench meter kit options	Orion Pro Star PH211 pH/ORP bench meter durable kit (comes with 8157BNUMD pH/ATC Triode)	PSTAR2116
	Orion Pro Star PH211 pH/ORP bench meter difficult sample kit (comes with 8172BNWP pH electrode and an ATC)	PSTAR2114
Electrode option	Orion ROSS spear tip electrode for solid samples (meat, fruit, semi-soft cheese, dough) + ATC	8163BNWP 927007
Solutions	Orion pH 4.01 buffer standard	910104
	Orion pH 7.00 buffer standard	910107
	Orion pH 5.00 buffer standard	910105
	Orion ROSS pH Electrode Storage Solution	810001
	Orion ROSS pH electrode filling solution	810007
Reagent Grade Water	Barnstead Smart2Pure 12 UV Water Purification System	50129890*

*Please contact your local Thermo Scientific™ representative for support to order a suitable water purification system for your application.

References

1. Carson City Health and Human Services, Carson City, NV USA. Acidification and Fermentation HACCP Guidance.
2. 21 CFR § 114.90 (2017). Food and Drugs, Part 114 – Acidified foods, Subpart E - Production and process controls.
3. Pennsylvania Department of Agriculture, Bureau of Food Safety and Laboratory Services, Harrisburg, PA USA. Canning of Acid, Acidified, & Fermented Foods Guidance for Commercial Food Establishments Licensed by PDA. Rev 12/2022.
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For technical support, contact us today at wlp.techsupport@thermofisher.com

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