

pH (Slurry)

PRINCIPLE

pH is a measure of active acidity or alkalinity of solutions as contrasted with the titratable acidity or alkalinity. The pH value of a sample solution or dispersion is determined by measuring the potential difference between two immersed electrodes.

SCOPE

This procedure is generally applicable to modified and unmodified starches which are substantially insoluble in water at room temperature.

SPECIAL APPARATUS

1. pH Meter: A modern instrument with both pH and millivolt (mV) readouts, equipped with glass and reference electrodes, capable of measuring pH values in the range of 1 to 10 (accurate to 0.01 pH unit), is recommended. Satisfactory instruments and electrodes are available from several suppliers.
2. Stirring Apparatus: A magnetic stirrer is usually preferred. Connect the chassis of the stirrer electrically to the ground circuit of the pH meter. An additional ground between the starch slurry and the pH meter ground circuit may be necessary to minimize erratic readings caused by rapid stirring.

REAGENTS

Standard Buffers: Two buffer solutions, having known pH values of about 4 and 7, are necessary (Note 1).

PROCEDURE

Standardization (Note 2):

Follow the manufacturer's instructions in preparing the meter for pH measurements. Connect to a power source with a ground, and connect shorting strap to meter. Switch meter to mV mode; a reading of 0 mV should be observed.

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(If the meter has no mV mode, it should read 7.00 + 0.05 pH.) Zero the instrument (mV = 0 or pH = 7.0) by adjusting the meter movement.

Plug prepared (hydrated) pH electrodes into the instrument. Rinse with purified water, and remove excess by blotting with clean, dry tissue (do not wipe!). Place electrode probe in "fresh" standard buffer having a known pH value of about 7.0, and stir at a rate sufficient to produce a small vortex at the solution surface. Switch meter to mV mode; observe and record the mV reading after a steady response is obtained.

Switch meter to the pH mode and adjust the meter to read the stated pH value of the standard buffer by moving the "ZERO" or "CALIBRATE" knob, after a steady response is obtained.

Remove electrode probe from the pH 7 buffer, rinse with purified water, and remove excess by blotting with a clean, dry tissue. Place the electrode probe in the second standard buffer having a known pH value of about 4.0, and stir as before. Switch meter to mV mode; observe and record the mV response after the reading has stabilized.

Calculate meter sensitivity as follows:

$$\text{mV per pH} = \frac{\text{pH 4.0 Buffer mV} - \text{pH 7.0 Buffer mV}}{\text{pH 4.0} - \text{pH 7.0}}$$

Meter sensitivity should equal 59 + 3 mV per pH unit. If not, refer to the manufacturer's instruction manual.

When sensitivity is satisfactory, switch meter to pH mode and adjust the instrument to read the stated pH value of the second standard buffer by moving the "SLOPE" or "TEMPERATURE" knob. If the meter does not have the mV measuring mode, disregard the mV measurements and the sensitivity calculation, and adjust the instrument to read the stated pH value of the second standard buffer by moving the "SLOPE" or "TEMPERATURE" knob.

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Remove electrodes from the second standard buffer, rinse with purified water, and remove the excess by blotting with a clean and dry tissue prior to sample analysis. Store electrodes in fresh pH 4 buffer when not in use. Do not store in water!

Analysis:

Starch samples containing hard granular particles should be ground. In most cases, however, grinding is not necessary.

Weigh 20 g (+ 0.1 g) of sample, transfer to a glass beaker and add 100 mL of purified water at room temperature (about 25 °C). Stir about 5 minutes (Note 3) at a rate sufficient to produce a small vortex at the slurry surface and to disperse the sample completely. Observe and record the pH value to the nearest 0.1 pH unit, after a stable reading is achieved. If erratic and/or unstable readings are observed, refer to the manufacturer's instruction manual.

NOTES AND PRECAUTIONS

1. Both liquid and dry stock buffers are available commercially, and can be used with confidence when handled according to the manufacturer's instructions.
2. The meter and electrodes should be standardized daily in both the pH and mV modes to assure accurate measurements.
3. Starches containing additives may require longer than 5 minutes to reach a stable pH reading. Stir until a steady pH reading is observed (as much as 30 minutes may be required with some samples).