Ozarks Environmental and Water Resources Institute (OEWRI)
Missouri State University (MSU)

Standard Operating Procedure for:

pH using Oakton pH 5+ Handheld pH Meter

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Missouri State University

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Identification of the method
Measurement of pH using a laboratory pH meter.

Applicable matrix or matrices
This method is suitable for the determination of pH in potable, surface waters, and wastewaters with a pH between 0.00 and 14.00, with an accuracy of 0.01.

Scope of the test method
This standard operating procedure provides OEWRI laboratory personnel with guidance on the procedure for determining pH in water samples. This method is limited to the determination of pH in water samples collected from natural bodies of water with values between 0.00 and 14.00 pH units.

Summary of method
pH is the measure of a solution's hydrogen ion activity. The pH of a neutral solution is 7. Natural waters generally have pH value between 4 and 9 (Standard Methods, 2005). In this method, a calibrated pH meter is used to measure the pH value for water samples in the laboratory. The meter is calibrated with pH 4.0 and pH 7.0 buffers. To analyze a sample, insert the probe into a beaker containing an aliquot of the sample. Allow the reading to stabilize. Record the pH value on the bench sheet.

Definitions
1. Analytical batch: The set of samples processed at the same time
2. Laboratory duplicates (LD): Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures. Analyze one set of laboratory duplicates with every ten samples analyzed.

Interferences
The glass electrode is relatively free from interference from color, turbidity, colloidal matter, oxidants, reductants, or high salinity. pH measurements can be affected by temperature in two ways: a mechanical effect causing changes in the properties of the electrodes and chemical effects caused by equilibrium changes.

Health and safety

This analysis involves handling freshwater samples that may contain live microorganisms and therefore pose some threat of infection. Lab personnel who are exposed to such water samples are encouraged to protect themselves from waterborne illnesses by wearing clean disposable gloves and washing their hands frequently.

Personnel qualifications

Laboratory and field personnel shall have a working knowledge of this analytical procedure and will have received training from an MSU employee knowledgeable of the proper sample analysis procedures. Prior to the first batch of sample analyses, the analyst will complete a demonstration of capability exercise as described below in the Quality control section.

Equipment and supplies

Oakton pH 5+ Handheld pH Meter with probe.

Reagents and Standards

1. pH 7.00 buffer: Fisher SB107-500, color code = yellow, or equivalent
2. pH 4.00 buffer: Fisher SB101-500, color code = red, or equivalent
3. pH 10.00 buffer: Fisher SB115-4, color code = blue, or equivalent
4. Deionized water (DI): for rinsing the probes

Sample collection, preservation, shipment, and storage

1. See the SOP for water sample collection procedures (OEWRI-SOP: 1040R01).
2. Samples should be refrigerated as soon as possible after collection.
3. There is no holding time for pH (Standard Methods, 2005), but samples should be analyzed as soon as possible.

Quality Control

1. Demonstration of capability; a new analyst should:
    a. Read this standard operating procedure,
    b. Demonstrate to a previously trained analyst that the procedures outlined here are being followed, and
    c. Successfully complete a report using data collected with these procedures.
2. Duplicate reproducibility:
    a. Measure two replicates on the same sample.

b. The relative percent difference (RPD) between the two pH values should be ≤ 20% of their average value.
c. Use equation 1 to calculate RPD:

$$\text{Equation 1:} \quad \text{RPD (\%)} = \frac{(A - B)}{(A + B)/2} \times 100\%$$

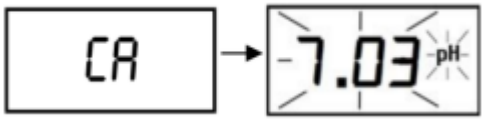
Where: A = first measurement, and
B = duplicate measurement

d. Analyze one set of duplicates for every 10 samples analyzed.

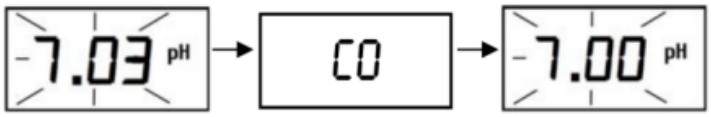
3. pH standard check test with pH 10 calibration solution.
   a. Record this value on the calibration sheet.
   b. If this reading is not within 0.2 pH units of the expected value, the analyst must recalibrate the instrument or perform maintenance to ensure that the meter is operating correctly.

Calibration and standardization

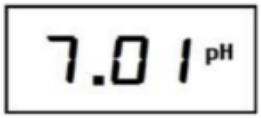
1. The calibration procedures listed here are for the Oakton pH 5+ Handheld pH Meter. Review the manufacturer's instructions for other meters.
2. Press CAL to begin pH calibration mode. "CA" (calibrate) will display briefly. Notice that the pH reading, and "pH" annunciator will both blink.



3. Pour pH buffer calibration standard solution into a clean, dry container and dip your pH electrode and temperature probe in the solution. Swirl gently or stir and wait for reading to stabilize (approx. 30 seconds depending on your electrode condition).
4. When the pH reading has stabilized, the pH annunciator will stop blinking (this often doesn't happen, just allow the reading to stabilize). Press HOLD ENTER to confirm the value. "CO" (confirm) will display briefly.
   a. The pH value is automatically adjusted to the buffer value shown from your selected buffer group. The example below shows a successful pH 7.00 calibration at 25ºC.



5. For a one-point calibration with pH 7.00 or pH 6.86 only, press CAL to return to measurements mode. However, for highest accuracy – perform a multiple-point calibration, beginning with pH 4.00 and repeat steps 2-4 with additional pH buffer calibration standards.
    a. When you have completed the preset number of calibration points, the meter will automatically save the calibration, cease blinking, and begin pH measurement.



6. Check pH calibration with the 10 pH calibration standard to ensure its measures between pH 9.8 and 10.2.

Procedure

1. Rinse electrode and probe with DI water and blot dry with a lint-free tissue.
2. Place both pH and temperature probes into test sample.
3. Allow the reading to stabilize.
4. Record the pH reading and temperature.
5. Repeat steps 1-4 for all samples.

Data Acquisition

1. Record pH values for each sample on the bench sheet (see below).
2. Record any other calculation results (e.g., duplicate RPD) in the comments section of the bench sheet.

Pollution Prevention

All wastes from these procedures shall be collected and disposed of according to existing waste policies within the MSU Geography, Geology, and Planning Department. Volumes of reagents made should mirror the number of samples being analyzed, adjusting to reduce waste. The pH buffer solutions are not hazardous and can be disposed of in the laboratory sinks.

Computer Hardware and Software

Word: This document and attached bench sheet are prepared using Microsoft Word.

Data assessment and acceptable criteria for quality control measures

1. Lab analyst should review all data for correctness (e.g., calculations).
2. Precision values are calculated for pairs of duplicate analyses.
3. Record precision values as a percent on the bench sheet.
4. The desired precision is ± 20% RPD.
5. The desired detection limit is 2 and 12 pH units
6. Completed bench sheet is reviewed by the lab manager.

Corrective actions for out-of-control or unacceptable data

1. Quality control charts will be created for charting precision values.
2. Results for precision are compared to the acceptable values for this analysis and should be ± 20%.
3. If a precision value exceeds 20% RPD then lab analyst should write in comments section of the bench sheet: “These data are associated with an out-of-control duplicate analysis. The UCL = 20%.” Note: “UCL” is the Upper Control Limit (i.e., 20%).
4. The samples can be reanalyzed because the sample volume will not have been depleted after the initial analysis.
5. If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.
6. Maintenance steps may include cleaning the probe, changing the reference solution or replacing the probe. All maintenance steps must be recorded in the instrument logbook.

Waste Management

The wastes generated in this method are not hazardous. They can be discarded in the in the laboratory sink.

References

1. Standard Methods for the Examination of Water and Wastewater. Method 4500-H+. APHA, 21st Edition, 2005.
2. pH Measurement: Meters and Electrodes. Chemistry course instructions (CHM175). 2006. Missouri State University
3. OEWRI. Standard Operating Procedure for: Water Sample Collection. Ozarks Environmental and Water Resources Institute, Missouri State University. 2007. 1040R03.

Tables, Diagrams, and Flowcharts

1. See page 9 for the pH and conductivity calibration log sheet.
2. See page 10 for the pH and conductivity bench analysis sheet. The analyst should make a copy of this form for each batch of samples analyzed.

Ozarks Environmental and Water Resources Institute (OEWRI)
at Missouri State University
pH/SC Calibration Log

Date of Calibration: _____ Analyst: _____

Oakton pH Meter

pH 4 Pre_____ Post_____ Lot # _____

pH 7 Pre_____ Post_____ Lot # _____

pH 10 (pH standard check) Is it between 9.8 and 10.2? ☐ Yes ☐ No

Cole-Parmer Conductivity Meter

Conductivity (µS/cm) Pre_____ Post_____ Lot # _____

DI Check is it <2.0 µS/cm? ☐ Yes ☐ No

Comments _____

_____

Date of Calibration: _____ Analyst: _____

Oakton pH Meter

pH 4 Pre_____ Post_____ Lot # _____

pH 7 Pre_____ Post_____ Lot # _____

pH 10 (pH standard check) Is it between 9.8 and 10.2? ☐ Yes ☐ No

Cole-Parmer Conductivity Meter

Conductivity (µS/cm) Pre_____ Post_____ Lot # _____

DI Check is it <2.0 µS/cm? ☐ Yes ☐ No

Comments _____

Ozark Environmental and Water Resource Institute (OEWRI) Lab Bench Sheet		
Project: _____	Date: _____	Analyst: _____
Sample ID	***Sp. Cond (µS/cm)***	***pH (std.)***
DI Blank		
DI Blank		
Lab Dup _____		
DI Blank		

**Lab duplicates need to be ≤20% relative percent difference (RPD)*

*** DI blank should have SC <2 uS/cm and pH of around 7.0*

Oakton Handheld pH - Bench Procedures

Two Step Calibration Procedure

1. Turn on meter.
2. Select pH mode by pressing MODE.
3. Press CAL to begin pH calibration (pH reading and “pH” annunciator will both blink).
4. Pour pH 4 buffer into container.
5. Place pH electrode and temperature probe in the solution, swirling gently and wait for reading to stabilize (approx. 30 seconds).
6. When stabilized the pH annunciator will stop blinking (sometimes it won’t stop blinking so just allow it to stabilize before recording).
7. Press HOLD ENTER to confirm the value.
8. Remove probe and electrode and rinse with DI and wipe with lint-free tissue.
9. Repeat with pH 7 buffer for 2-point calibration.
10. Check pH calibration with the 10 pH calibration standard to ensure it is within 9.8 and 10.2

Analysis Procedure

1. Rinse the electrode and probe with DI water and blot dry with a lint-free tissue.
2. Place both pH and temperature probes into test sample and allow the reading to stabilize.
3. Record the pH reading and temperature.
4. Repeat steps 1 to 3 for all samples taking a lab duplicate every 10 samples.