Standard Operating Procedure for:

pH
using Oakton pH 5+ Handheld pH Meter

Missouri State University

and

Ozarks Environmental and Water
Resources Institute (OEWRI)

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OEWRI-SOP-003

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1 Identification of the test method

Measurement of pH using a laboratory pH meter.

2 Applicable matrix or matrices

This method is suitable for the determination of pH in potable and surface waters and wastewaters with a pH between 0 and 14.

3 Detection Limit

The practical detection limit is 1 pH unit.

4 Scope of the test method

This standard operating procedure provides Missouri State University (MSU) 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 and 14 pH units.

5 Summary of test method

pH is the measure of a solution’s hydrogen ion activity. The pH of a neutral solution is 7. Natural waters generally have a 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.

6 Definitions

6.1 Analytical batch: The set of samples processed at the same time

6.2 Field duplicates (FD): Two samples taken at the same time and placed under identical circumstances and are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage as well as laboratory procedures. Collect one set of field duplicates for every ten samples collected.

6.3 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.

7 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.

8 Health and safety

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

9 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.

10 Equipment and supplies

10.1 Oakton pH 5+ Handheld pH Meter with probe

11 Reagents and standards

11.1 pH 7.00 buffer: Fisher SB107-500, color code = yellow, or equivalent
11.2 pH 4.00 buffer: Fisher SB101-500, color code = red, or equivalent
11.3 Deionized water (DI): for rinsing the probes

12 Sample collection, preservation, shipment and storage

12.1 See the SOP for water sample collection procedures (SOP: 1040R01 Water Sampling.doc).

12.2 Samples should be refrigerated as soon as possible after collection.

12.3 There is no holding time for pH (Standard Methods, 2005), analyze samples as soon as possible.

13 Quality control

13.1 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.

13.2 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:

Equation 1:

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

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

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

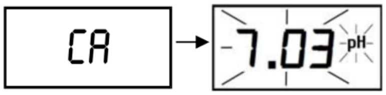
13.3 pH check standard: analyze a certified pH standard (not one of the calibration standards). Record this value in the instrument log book. If this reading is not within 0.02 pH units of the expected value, the analyst must recalibrate the instrument or perform maintenance to ensure that the meter is operating correctly.

14 Calibration and standardization

14.1 The calibration procedures listed here are for the Oakton pH 5+ Handheld pH Meter with. Review the manufacturer's instructions for other meters.

14.2 Turn on the meter and select pH mode by pressing MODE if necessary.

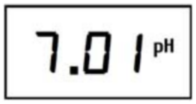
14.3 Press CAL to begin pH calibration mode. "CA" (calibrate) will display briefly. Notice that the pH reading and "pH" annunciator will both blink.



14.4 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).

14.5 When the pH reading has stabilized, the pH annunciator will stop blinking. Press HOLD ENTER to confirm the value. "CO" (confirm) will display briefly. 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.

14.6 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 14.4 & 14.5 with additional pH buffer calibration standards. When you have completed the preset number of calibration points, the meter will automatically save the calibration, cease blinking, and begin pH measurement.



15 Procedure

15.1 Rinse the electrode and probe with DI water and blot dry with a lint-free tissue.
15.2 Place both pH and temperature probes into test sample.
15.3 Allow the reading to stabilize.
15.4 Record the pH reading and temperature.
15.5 Repeat steps 15.1 to 15.4 for all samples.

16 Data acquisition, calculations, and reporting

16.1 Record the pH values for each sample on the bench sheet (see below).

16.2 Record any other calculation results (e.g., duplicate RPD) in the comments section of the bench sheet.

17 Computer hardware and software

17.1 Word: This document and attached bench sheet are prepared using Microsoft Word. The Word document file name for this SOP is: pHR01.doc.

17.2 Excel: Quality control charts are created using Excel.

18 Method performance

18.1 The desired performance criteria for this measurement are:

- a. Detection limit: 2 and 12 pH units (Note: a pH of < 2 or > 12 is highly acidic or basic, respectively, and is not likely to be encountered with environmental samples.)
- b. Precision: ± 20% RPD
- a. Minimum Quantification Interval: 0.01 pH units

19 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. These adjustments should be made to reduce waste. The pH buffer solutions are not hazardous and can be disposed of in the laboratory sinks.

20 Data assessment and acceptable criteria for quality control measures

20.1 The analyst should review all data for correctness (e.g., calculations).

20.2 Precision values are calculated for pairs of duplicate analyses.

20.3 Record the precision values as a percent on the bench sheet.

20.4 The desired precision is ± 20% RPD.

20.5 The desired detection limit is 2 and 12 pH units

20.6 The completed bench sheet is reviewed by the analyst’s supervisor or the OEWRI QA coordinator

21 Corrective actions for out-of-control or unacceptable data

21.1 Quality control charts will be created for charting precision values.

21.2 The results for precision are compared to the acceptable values for this analysis and should be ± 20%.

21.3 If a precision value exceeds 20% RPD then the analyst should write in the 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%).

21.4 The samples can be reanalyzed because the sample volume will not have been depleted after the initial analysis.

21.5 If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.

21.6 Maintenance steps may include cleaning the probe, changing the reference solution or replacing the probe. All maintenance steps must be recorded in the instrument log book.

22 Waste management

The wastes generated in this method are not hazardous. They can be discarded in the following manner: the water can be discarded in the laboratory.

23 References

23.1 Standard Methods for the Examination of Water and Waste Water. Method 4500-H^+. APHA, 21st Edition, 2005.

23.2 pH Measurement: Meters and Electrodes. Chemistry course instructions (CHM175). 2006. Missouri State University

23.3 Water Sample Collection. 2006. OEWRI SOP. Document name: 1040R01 Water Sampling.doc

24 Tables, diagrams, flowcharts and validation data

24.1 There are no tables, diagrams, flowcharts or validation data for this method.

24.2 See page 8 for the pH, conductivity, and turbidity bench analysis sheet. The analyst should make a copy of this form for each batch of samples analyzed.

24.3 See page 9 for the pH procedure bench sheet.

Ozarks Environmental and Water Resources Instititute (OEWRI) at Missouri State University
pH, Conductivity, and Turbidity Bench Sheet

Project: ________________ Date:______________ Analyst: ____________________________

Site ID	**Temperature** (°C)	**Sp. Cond** (µS/cm)	**pH**	**DO** (ppm)	**Turbidity** (NTU)	**Stage** (cm)

pH - Bench Procedures

2 Step Calibration

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.
7. Press HOLD ENTER to confirm the value.
8. Repeat with pH 7 buffer for 2 point calibration.

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.