

pH Analysis

1) Applicable Matrices

- a) pH analysis is applicable for surface water, ground water, and wastewater analysis.

2) Scope and Application

- a) pH analysis is performed using an ion selective electrode which measures electrical potential between the electrode and the ion of interest.
- b) This analysis can be performed in combination with conductivity analysis by using the same poured sample for each analysis. Perform conductivity first, then pH.

3) Interferences

- a) It is possible for other ions dissolved in a sample to interfere with accurate H⁺ ion detection, however, this is very rare and the interfering ions must be in high concentration.

4) Equipment and Supplies

- a) Thermo Scientific Orion Star A211 pH meter with pH electrode
- b) Sample Cups
- c) Ultrapure water squirt bottle
- d) KimWipes
- e) Warm water bath if samples are cold

5) Reagents and Standards

- a) Certified pH 4, 7, 10 buffers for calibration

6) Sample Collection, Preservation, Shipment, and Storage

- a) Collect samples in clean plastic bottles. Store at 0-4°C. Analyze ASAP.

7) Quality Control

- a) A duplicate sample shall be analyzed once for every 20 samples. Control limits shall be calculated using the data obtained from these duplicates. Any duplicate falling outside of these control limits shall be reanalyzed.

8) Calibration

a) pH Meter Calibration Procedure

- (1) Turn on pH meter
- (2) Remove Plug on electrode
- (3) Remove the electrode from the storage solution, and rinse it with ultrapure water. Blot dry with a Kim[®] Wipe.
- (4) Press the button beneath the word "Cal" on the screen.
- (5) Fill a small cup with pH 7 buffer.
- (6) Place electrode into cup so it is fully submerged in buffer.
- (7) Press the button beneath the word "Start"
- (8) Allow the meter to stabilize. Once stabilized, make sure the calibrated reading is correct. If not, press the button beneath "Edit" and change the value to 7.00.
- (9) Press the button beneath "Accept."
- (10) Rinse the electrode with ultrapure water and blot dry with a Kim[®] Wipe.
- (11) Press the button beneath "Next."
- (12) Fill a cup with pH 4.01 buffer.
- (13) Follow step 8 but change the pH value to 4.01 if needed.
- (14) Fill a cup with pH 10.01 buffer.
- (15) Follow step 8 but change the pH value to 10.01 if needed.
- (16) pH 10.01 buffer is the last buffer. Press the button beneath "End Cal." The slope of the calibration will be displayed. Be sure it is between 92 and 102%. If it is outside this range, recalibrate. If after recalibration, the slope is still out of range, be sure the electrode is filled with electrode filling solution. If it is low, fill the electrode to just below the opening where the plug was.

9) Procedure

- a) Make a list of samples requiring pH analysis. Locate and collect samples. Unpreserved samples are used for pH analysis.
- b) Allow samples to warm to room temperature, by either allowing to warm on the bench, or placing in a warm water bath. If using a water bath, be sure samples are capped tightly, and are not floating.

- c) Print a pH/Conductivity benchsheet located on the Arikaree Shared drive.

(Procedure Continued)

- d) Record the sample ID #'s to be analyzed on the benchsheet.
- e) Locate a clean cup (triple rinsed w/ ultrapure water) and rinse the cup with a few mL's of sample. Be sure the sample coats all inside surfaces of the cup.
- f) Pour enough sample into the cup to cover the bottom inch of the pH electrode.
- g) Rinse electrode with ultrapure water. Gently blot the electrode dry with a KimWipe.
- h) Place cup with sample under the pH electrode. Place electrode as far as possible into sample without the electrode contacting the bottom of the cup.
- i) Press the "measure" button on the pH meter. The word "stabilizing" and "AR" will flash above the pH value. Allow "stabilizing" to change to a solid "ready" and "AR" to stop flashing. Record the pH value.
- j) Repeat from step c for all remaining samples recorded on benchsheet.
- k) After all samples have been analyzed, rinse electrode with ultrapure water and dry with a KimWipe. Replace plug in electrode, and place electrode in KCl storage solution.
- l) Turn off pH meter, and wipe up any spills on the surrounding bench. Make the work station cleaner than when you started.
- m) Record sample results on data sheets in office. Be sure to record the date analyzed at the top of the pH column, and write your initials under the pH data. Write legibly!

10) Corrective Action for Out-of-Control Data

- a) Any duplicate outside of the control limits will be reanalyzed. If this does not correct the issue, meter will be recalibrated, and all samples will be reanalyzed.

11) Contingencies for Handling Out-of-Control Data

- a) Samples with a failed duplicate will be qualified back to the last sample with a controlled duplicate.

12) Waste Management

- a) All waste produced from pH analysis is considered non-hazardous and can be disposed of down the sink.

13) References

- a) Standard Methods for the Examination of Water and Wastewater, 22nd ed. Clesceri, L.S.; Greenberg, A.