

Scope and Application

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TITLE: pH, EPA 150.1

INSTRUMENTATION: HACH EC10 pH Meter

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1. Scope and Application

- 1.1. This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).
- 1.2. It is applicable to field as well as laboratory analysis.

2. Summary of Method

- 2.1. Instrument setup procedure is followed.
- 2.2. The meter is calibrated using certified standards.
- 2.3. The calibration is verified with an independent certified standard (initial calibration verification).
- 2.4. The pH of a sample is determined electrometrically using a combination electrode.
- 2.5. Continuing calibration verification is carried out using the independent certified standard.

3. Sample Handling and Preservation

- 3.1. Samples should be analyzed as soon as possible preferably in the field at the time of sampling.
- 3.2. The sample containers should be filled completely and kept sealed prior to analysis because high-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere.
- 3.3. Stable pH, conductivity and temperature conditions must be demonstrated prior to groundwater sample collection (see SOP #SP002- Groundwater Sampling).

4. Interferences

- 4.1. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.
- 4.2. Coatings of oily material or particulate matter can impair electrode response. Gentle wiping or detergent washing, followed by distilled water rinsing can usually remove these coatings. An additional treatment with hydrochloric acid (1 + 9) may be necessary to remove remaining film.
- 4.3. Variation in temperature causes pH values to differ. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples.
- 4.4. If compensation or calibration at the same temperature is not possible, it should be noted by reporting both the pH and the temperature at the time of analysis.

5. Apparatus

- 5.1. HACH EC10 Portable pH/mV/Temperature Meter Model 50050
- 5.2. HACH One Combination pH Electrode Model 48600-22
- 5.3. Magnetic stirrer and Teflon-coated stirring bar (for lab analysis)

6. Reagents/Supplies

- 6.1. VWR pH 7.00 Buffer Solution Yellow (Cat. No. 34170-130)
- 6.2. VWR pH 7.00 Buffer Solution Yellow (Cat. No. 34170-130)-different lot
- 6.3. VWR pH 4.00 Buffer Solution Red (Cat. No. 34170-127)
- 6.4. VWR pH 10.00 Buffer Solution Blue (Cat. No. 34170-133)
- 6.5. Ricca Deionized Water (Cat. No. 9150-5)
- 6.6. Kimberly-Clark Kimwipes EX-L (Cat. No. 34120)

7. Procedure

- 7.1. *Instrument Setup* (Refer to *HACH* EC10 Manual)
 - 7.1.1. Power-up and Self-Diagnostics Checkout
 - 7.1.1.1. Disconnect the electrode and temperature probe from the meter.
 - 7.1.1.2. Attach BNC shorting cap to the BNC connection on the meter.
 - 7.1.1.3. Press the **I/O** key to turn the instrument on.
 - 7.1.1.4. Install new 9V battery when low battery indicator appears on the display.
 - 7.1.1.5. Press **I/O** key to turn the meter off.
 - 7.1.1.6. Press and hold the **YES** key while pressing the **I/O** key.
 - 7.1.1.6.1. The instrument automatically performs electronic and hardware diagnostic tests, and a system countdown will display 1-8.
 - 7.1.1.6.2. The meter will stop on test 7.
 - 7.1.1.7. When "0" is displayed, press each key (including the **I/O** key) within 10 seconds to complete this test.
 - 7.1.1.7.1. The numeric digits will change.
 - 7.1.1.7.2. If all keys are not pressed within 10 seconds, or if a key is not responding properly, *E-7* will appear.
 - 7.1.1.7.3. If problems are found during self-test, the meter will display the error code until **YES** is pressed.
 - 7.1.1.7.4. If there is an error, begin Power-up and Self-Diagnostics Checkout again (7.1.1).
 - 7.1.1.7.5. Refer to Table 2 *Error Codes* on page 19 of the instrument manual.
 - 7.1.1.8. After the keypad test (7), the meter will display test 8 and then the meter will turn off.
 - 7.1.1.9. Make sure that the BNC shorting cap is securely attached to the BNC connection of the meter and then press the **I/O** key. The meter will be in the MEASURE mode as indicated on the display.
 - 7.1.1.10. Press the **MODE** key until the pH mode indicator is displayed.
 - 7.1.1.11. Press the **CAL** key.
 - 7.1.1.12. When the display flashes 7.00, press the **YES** key.
 - 7.1.1.13. Press the **MODE** key. The current slope (SLP) will be displayed.
 - 7.1.1.14. Press the **YES** key.
 - 7.1.1.15. The meter advances to MEASURE and reads a steady 7.00.
 - 7.1.1.16. Turn meter off.
 - 7.1.1.17. Remove the BNC shorting cap from the input connector.
 - 7.1.2. Install the HACH One combination pH electrode.
 - 7.1.2.1. Attach the BNC connector of the electrode to the BNC connection of the pH meter.
 - 7.1.2.2. Attach the ATC/DIN Connector of the electrode to the ATC/DIN connection of the pH Meter.
 - 7.1.3. Thoroughly rinse electrode with deionized water.
 - 7.1.4. Blot with Kimwipe.
 - 7.1.5. Instrument is ready for calibration.

7.2.Instrument Calibration

- 7.2.1. Allow standard pH buffer solutions to equilibrate to room temperature (20-30° C).
- 7.2.2. Turn the pH meter on.
- 7.2.3. Using the MODE key, set the meter to "pH" mode.
- 7.2.4. Depress the dispenser button on the electrode until a "click" is heard, then release.
 - 7.2.4.1.Repeat until electrolyte gel emerges from the reference outlet (See Model 48600 HACH One Combination pH Electrode Instruction Manual).
 - 7.2.4.1.1. If no gel emerges, the hole may need to be unplugged with a needle.
 - 7.2.4.1.2. If this does not help, the gel unit may need to be replaced if low.
 - 7.2.4.2. Rinse the electrode with deionized water and blot dry with Kimwipe.
- 7.2.5. Place the pH electrode into 7.00 buffer solution (Yellow).
 - 7.2.5.1. Make sure that the electrode tip is at least 1 inch below the surface of the solution.
 - 7.2.5.1.1. If access to magnetic stirrer is available, place clean magnetic stir rod into buffer solution.
 - 7.2.5.1.2. Wait for reading to stabilize while stirring on magnetic stirrer.
 - 7.2.5.1.3. If not magnetic stirrer is used, gently agitate probe in buffer manually.
- 7.2.6. Press the **CAL** key.
- 7.2.7. **P1** will be displayed under pH reading.
- 7.2.8. Wait until "ready" is displayed and then press the **YES** key.
- 7.2.9. Remove electrode, rinse with deionized water and blot dry with Kimwipe.
- 7.2.10. Click electrolyte pump button two (2) times.
- 7.2.11. Place electrode into either 4.00 or 10.00 buffer solution depending on the expected pH range of the sample to be measured.
 - 7.2.11.1. If expected pH range of the sample is greater than 7.00, use the 10.00 buffer (Blue).
 - 7.2.11.2. If the expected pH range of the sample is less than 7.00, use the 4.00 buffer (Red).
- 7.2.12. Make sure that the electrode tip is at least 1" below the surface of the solution.
 - 7.2.12.1. If access to magnetic stirrer is available, place clean magnetic stir rod into buffer solution.
 - 7.2.12.2. Wait for reading to stabilize while stirring on magnetic stirrer.
 - 7.2.12.3. If no magnetic stirrer is used, gently agitate probe in buffer manually.
- 7.2.13. Wait until "ready" is displayed and then press the **YES** key.
- 7.2.14. Slope is displayed. Record slope in HACH ph Meter Log, Logbook #30 A.
- 7.2.15. Remove electrode, rinse with deionized water and blot dry with Kimwipe.
- 7.2.16. Click electrolyte pump button two (2) times.
- 7.2.17. Take pH of a second standard pH 7.0 buffer as a calibration check (*initial calibration verification-ICV*).
 - 7.2.17.1. If pH is within 0.05 pH units of the expected value (acceptable *precision*), calibration is verified. Proceed to 7.2.6.6.
 - 7.2.17.2. If pH is outside this range, reread pH of calibration check standard.
 - 7.2.17.3. If pH is within range, proceed to 7.2.6.6.
 - 7.2.17.4. If pH is still outside range, reread fresh buffer standard.
 - 7.2.17.5. If pH is outside range, recalibrate (7.2).



- 7.2.18. Retake pH of calibration standards and record reading for each in HACH pH Meter Log, Logbook #30 A.
- 7.2.19. Dispense gel electrolyte if the reading becomes unstable, erratic, or if stabilization takes too long (greater than two minutes).
- 7.3. Take sample measurements.
 - 7.3.1. For laboratory analyses, allow samples and buffer solutions to come to room temperature $(20-30^{\circ}\text{C})$.
 - 7.3.2. Thoroughly rinse pH electrode with deionized water and blot dry with Kimwipe.
 - 7.3.3. Insert rinsed and dried electrode into the sample.
 - 7.3.4. Record the pH value and the temperature of samples in appropriate bound logbook (Surface Water Monitoring Log, Logbook #21 A, or Groundwater Monitoring Log, Logbook #13 A).
 - 7.3.5. Analyze a calibration check standard (*continuing calibration verification- CCV*) with every ten samples to re-check the validity of the calibration curve.
 - 7.3.5.1. If the CCV is within 0.05 of the expected value, continue with sample analysis.
 - 7.3.5.2.If the CCV differs by more than 0.05, follow the steps below.
 - 7.3.5.2.1. If the CCV does not come within 0.05 of the expected value, then the calibration check standard should first be reread.
 - 7.3.5.2.2. If the repeat analysis result still differs by greater than 0.05, a fresh calibration check standard should be read.
 - 7.3.5.2.3. If this calibration check standard does not meet the criteria, a new calibration must be performed and all samples analyzed since the last valid calibration must be rerun.

8. Calculation

8.1. The pH meter reads directly in pH units. Report pH to the nearest 0.01 unit and the temperature to the nearest 0.1°C.

9. Bibliography

- 9.1. HACH EC10 Portable pH/mV/Temperature Meter Model 50050 Manual.
- 9.2. Model 48600 HACH One Combination pH Electrode Instruction Manual.
- 9.3. EPA Method 150.1.
- 9.4. Standard Methods for the Examination of Water and Wastewater, 20th Edition, Method No. 4500-H+, p 4-87, (1998).