Calibration and Use of Accumet Model AR10 pH Meter

1. SCOPE AND APPLICATION

1.1. This procedure is based on the *Fisher Scientific Accumet Model AR10 pH Meter* User Manual. It is used to calibrate the meter and measure pH of aqueous solutions.

2. SUMMARY OF METHOD

2.1. The meter is turned on and allowed to warm up (~ 5 minutes). Two pH buffers are selected which will bracket the expected pH of the aqueous solutions that are to be measured. The temperature of the lower value pH buffer is measured. The temperature knob is set to the measured temperature. The meter slope is set to 100%. The meter probe is placed into an aliquot of the lower value pH buffer. After the pH meter value stabilizes, the standardization knob is used to set the meter display to the value of the lower pH buffer. The temperature of the higher value pH buffer is measured. The temperature knob is set to the measured temperature. The meter probe is placed into an aliquot of the higher value pH buffer. After the pH meter value stabilizes, the slope knob is used to set the meter display to the value of the higher value pH buffer. A secondary standard (pH 7 buffer) is measured. After the last measurement of the day, the secondary standard is measured again. All calibration results are recorded in the pH logbook in the Laboratory Information Management System (LIMS).

2.2. If using an ATC probe, temperature is automatically accounted for; thus, the temperature of each individual standard and sample does not need to be measured. When an ATC probe is not used, the temperature of all standards and samples must be measured and the temperature knob adjusted to match the measured temperature. When an ATC probe is attached to the meter, the temperature knob on the meter is disabled.

2.3. Interferences

2.3.1. Wiping rather than blotting the electrode may leave an electrical charge on the electrode bulb. Failure to rinse and blot the electrode may result in carry-over of solutions. Failure to maintain the electrode in accordance with the manufacturer's instructions may result in inaccurate readings.

Note: Use of this SOP indicates that the user has read the Fisher Scientific AR10 User Manual and is familiar with the meter. The manual must be read prior to use of this SOP.

3. EQUIPMENT AND SUPPLIES

3.1. Accumet model AR10 pH meter

3.2. pH electrode, Fisher model 13-620-298 (different pH probes may be used as needed)

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3.3. 30 mL disposable plastic cups

3.4. Thermometer, calibrated per SOP TA-06.13 (Can be electronic or liquid filled. Mercury-filled thermometers are NOT used in this lab.)

3.5. Rinse water cup

3.6. Kimwipes®

3.7. ATC probe (if available)

3.8. Computer with access to the Laboratory Information Management System (LIMS)

4. REAGENTS AND STANDARDS

4.1. Deionized (DI) Water with 500 mL wash bottle

4.2. Standard buffer solutions, pH 4 or 5, 7, 9 or 10, and 13 (Fisher Scientific certified pH calibration solutions.)

4.3. Second-source buffer, pH 7 (Orion® pH 7.0 buffer solution, or another brand that is not the Fisher brand.)

5. CALIBRATION

5.1. Turn meter [**Mode**] knob from **Standby** (**off** on screen) to **pH**. Set the [**% Slope**] knob to 100%.

5.2. Select two standards that bracket the expected pH of the solution to be measured.

5.2.1. To greatly decrease the need for recalibration, **pH 5** and **pH 10** are the primary calibration standards used. (Most samples received in this lab are measured at pH >7.0 and <9.0 ± 1 pH unit.)

5.2.2. If sample pH information is available prior to pH calibration, use the appropriate buffer solutions to bracket the pH.

5.3. Label two 30mL cups (one cup per standard) with the pH of the selected calibration standards. Label a third cup for the Orion® pH 7.

5.4. Pour 20-25 mL of standard into the appropriately labeled cup.

5.5. Check that the fill hole on the side of the probe is exposed. If not, turn the plastic sleeve on the pH probe to expose the fill hole.

5.5.1. The fill hole must be uncovered when the electrode is being used.

5.6. Take the temperature of the first pH buffer solution and set the [**Temp ºC**] knob on the meter to the measured temperature.

5.7. Immerse the probe tip into the first pH buffer standard. Swirl the probe for 10 seconds. Stop swirling and allow the meter readout to stabilize. When the tenths place of the pH readout stops changing, the meter is stable.

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5.7.1. The first pH standard is usually pH 5. Other buffers can be used as needed.

5.8. Determine the exact pH of the buffer solution from the table of buffer pH versus temperature that is usually found on the buffer bottle label. Use the [**Standardize**] knob to adjust the meter to display the appropriate numerical value of the buffer solution.

5.9. Record the meter reading.

5.10. Remove the probe from the solution and rinse with DI water. Blot the electrode with a Kimwipe® if necessary. Do not rub.

5.11. Take the temperature of the second pH buffer solution and set the [**Temp ºC**] knob on the meter to this temperature.

5.12. Immerse the probe tip into the second pH standard. Swirl the probe for 10 seconds. Stop swirling and allow the meter readout to stabilize. When the tenths place of the pH readout stops changing, the meter is stable.

5.12.1. The second pH standard is usually pH 10. Other buffers can be used as needed.

5.13. Use the [**% slope**] knob to adjust the meter display to the pH value of the standard as determined by temperature/pH table on buffer bottle label.

5.14. Record the meter reading.

5.15. Remove the probe from the solution and rinse with DI water. Blot the electrode with a Kimwipe® if necessary. Do not rub.

5.16. Take the temperature of the Orion® pH 7 (or another secondary-source) buffer. Adjust the [**Temp ºC**] knob to this temperature.

5.17. Determine the exact pH of the buffer solution from the table of buffer pH versus temperature that is usually found on the buffer bottle label.

5.18. Immerse the probe tip in the Orion® pH 7 buffer. Swirl the probe for 10 seconds. Allow the meter reading to stabilize.

5.19. Record the meter reading. If the pH is > ±0.1 SU (Standard Units) from the exact pH of the buffer determined in Step 5.18.:

5.19.1. Repeat the calibration procedure, or

5.19.2. Use new buffers and recalibrate, or

5.19.3. Change the probe and recalibrate

5.19.4. In case of emergency, make a note of the uncertainty, and measure the pH of the solutions.

5.20. Remove the probe from the solution and rinse with DI water. Blot the electrode with a Kimwipe® if necessary. Do not rub.

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6. SAMPLE MEASUREMENT

6.1. Measure and record the temperature of the sample solution (usually in a 30-mL disposable plastic cup).

6.1.1. Temperature of sample is typically recorded on bioassay bench sheets/ LIMS logbooks (TOX_pH_Meter per SOP TA-06.42).

6.2. Immerse the pH probe tip into the solution.

6.2.1. If the measured pH value is < the lower or > the higher pH buffers used for calibration, then recalibrate the meter substituting appropriate low and/or high buffers.

6.2.1.1. In lieu of recalibration, a pH buffer which would cause the measured value of the sample to fall between the buffer and a calibration point can be measured. The buffer information (lot # / expiration date) would need to be recorded on the appropriate bench sheet.

6.3. Swirl the probe for 10 seconds, then stop swirling and allow the meter to stabilize. Record this reading.

6.3.1. Depending on how much the sample pH differs from the last reading, and depending on the ionic strength of the sample, stabilization can take several minutes.

6.4. Remove the probe from the solution, and rinse with DI water. Blot the electrode with a Kimwipe® if necessary. Do not rub.

6.5. Repeat Steps **6.1.-6.3** for each sample to be tested.

6.6. When finished with the last sample, rinse the probe with DI water. Place the probe in the probe-storage container. Make sure that the lower 2 cm of the probe is immersed in the storage solution (i.e. pH 7 buffer).

6.6.1. Renew the storage solution every 30 days.

6.7. After the last pH measurement of the day, measure and record the pH and temperature of the verification standard. Record this value in the pH logbook in LIMS and make a comment in the comment box that this is the “end of day (EOD) check”. The value must be within 0.1 SU for the meter to be in calibration range.

7. DATA ARCHIVAL

7.1. Use a black, waterproof pen for all bench sheets and logbooks.

7.2. To correct raw data entries, place a single line through the incorrect or illegible entry. Write the corrected entry near the error, label with corresponding sequential alphabetical annotation, initial and date the correction. Do not overwrite entries.

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7.2.1. The corrected entry is written as near as possible to the error or in the comment box on the appropriate bench sheet.

7.2.2. If the correction requires an explanation, write a comment and reference the error with a letter. Initial and date the explanation. Do not attempt to put all information (correction and comments) into a small space.

7.2.3. Use sequential alphabetical annotation to identify errors and corresponding comments (use A for first error and use A for corresponding explanatory comment; use B for second error, etc.).

7.3. Record calibration check and measurements in pH logbook in LIMS.

8. QUALITY CONTROL

8.1. A two-point calibration check is performed on the meter. The points should bracket the expected pH values of the received samples. A second-source buffer is used to check the calibration.

8.1.1. If a measured sample pH is outside of the bracketed range, the meter should be recalibrated to include the measured pH.

8.2. The second-source pH buffer should read < ±0.1 SU of the exact pH value at the measured temperature. If the reading is outside of this range, repeat the calibration procedure. If the analyst believes the problem is with the buffers, new buffers can be used. If the probe is the issue, the probe can be changed before recalibrating.

8.3. If something goes wrong with the meter and all maintenance and troubleshooting has been done, the lab supervisor is notified. The meter should not be used for test measurements if calibration can not be done correctly.

8.4. A pH 7 buffer is used to perform an end-of-day calibration check to show that the meter did not fall out of calibration and that there was no drift. It is done after the last measurement of the day and before the meter is shut down. The measurement should be < ±0.1 SU of the exact pH value at the measured temperature.

8.5. For each lot number of pH buffer solution, a certificate of analysis must be obtained. They are saved in the “pH certificate of analysis” folder, located in a binder next to the telephone in B-246 (Tox Lab). They are also saved in X:\Labs\Lab5\Biology\BenchCommon\ToxCommon\Certificates of Analysis. The certificate of analysis label can be removed from the side of the container or obtained from the www.Fishersci.com website.

8.6. Probe should be stabilized (at thermal equilibrium) before a measurement is recorded. Swirling helps speed probe acclimation. It can require up to 2 minutes for the probe to stabilize; however, buffers are kept at room temperature (~25 ºC), and most samples are warmed to ~25 ºC, so thermal equilibrium usually occurs within seconds.

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8.7. Certified or calibrated thermometers are used to measure temperatures. The Thermo Scientific Orion Star A213 Benchtop RDO/DO meter or a Control Corporation digital thermometer can also be used.

8.8. The electrode is blotted with a Kimwipe® between measurements. It is never rubbed. An electrical charge could form and cause interference with a measurement.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Proper laboratory protection, including lab hood or ventilation system, lab coat, gloves and safety glasses, is required when working with chemicals and unprocessed samples.

9.2. Refer to FDEP Laboratory Quality Manual for sample disposal.

9.3. Refer to the FDEP Laboratory Health and Safety Plan and Hazardous Waste Management.

9.4. Read MSDS information for reagent handling and disposal.

10. REFERENCES

10.1. *Fisher Scientific Accumet model AR10 pH Meter* User's Manual.

10.2. Fisher Scientific Website, www.Fishersci.com.

10.3. DEP Laboratory Quality Manual https://floridadep.gov/dear/florida-dep-laboratory/content/dep-laboratory-quality-assurance-manual-and-sops.

10.4. DEP Laboratory Safety Manual https://www.floridadep.net/resources/health-safety-manual DEP SOP TA-06.42, LIMS Entry for Laboratory Parameters

10.5. DEP SOP TA-06.13, Thermometer Accuracy Check and Use

10.6. MSDS information sheets.

APPENDIX OF SIGNIFICANT CHANGES

09/08/2008 Inserted Scope and Application, Summary of Method, Interferences, References and Appendix of Changes. Modified comments for Materials Step 4 and Step 6 and Calibration Step 2, Step 7 and Step 13.

06/14/2013 Reformatted SOP.

02/09/2015 Formatted for 508 compliance.

06/13/2016 Updated links.

03/16/2017 Updated links.

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05/07/2018 Changed font to Calibri; added references to temperature adjustments; included saving the certificates of analysis to ToxCommon; added end-of-day check to the QC section.

04/26/2019 Updated links.

4/24/2020 Updated links, took out information on recording measurements in the *Daily Physical and Chemical Calibration Log* and replaced it with pH logbook in LIMS, replaced "pH units" with SU (standard units).

3/12/2021 Updated links and updated text to reflect universal language.

3/30/2022 Updated references to the DO meter and other SOPs.

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