Calibration and Use of Mettler Toledo SevenMulti® for Measuring pH, Conductivity, Salinity and TDS

(Based on Mettler Toledo SevenMulti® Operating Instructions)

1. SCOPE AND APPLICATION

1.1. This method is based on the Mettler Toledo SevenMulti® operating instructions. It is used to calibrate the SevenMulti® and to measure conductivity, salinity, and total dissolved solids (TDS) in aqueous solutions. It is also used to ensure the reliability of pH measurements made with the pH expansion unit and attached electrode.

2. SUMMARY OF METHOD

2.1. The SevenMulti® is set up and calibrated. After the initial set-up, daily calibrations are performed and recorded in the calibration logbook. A one point calibration is performed for conductivity, salinity, and TDS. A multi-point (minimum 2, maximum 9) calibration is performed for pH. To perform the calibrations, the meter is turned on and the desired mode (COND, SAL, TDS, pH) is selected. The probe is placed in the aqueous standard solution, briefly swirled then held still until the display stabilizes (locks). The probe is removed from the solution, rinsed, and placed in the storage solution. This is repeated with a second standard for the pH calibration, and then checked with a third. End-of-day measurements are made and recorded to ensure the probe readings have not drifted.

2.2. The instrument setup calibration is performed when the value of the setup calibration standards change. The operator must check the standard container when the standard solution is replaced and compare the "new" value with the "old" value. If those values differ, an instrument setup calibration is needed. This calibration might also be done when a new probe is installed, to test a suspect probe, when a daily-check reading is out of tolerance (see *Daily Physical-Chemistry Calibration Log*), suspicious readings, etc.

2.3. Calibration checks are performed each day prior to the first use of the meter and after last use of the meter for parameters that are being measured. If more than two hours pass after calibration in which the meter has not been used, standards are rechecked.

2.4. Use of this SOP constitutes acknowledgement that the user has read the Mettler Toledo Manual.

2.5. Interferences: temperature, probe not rinsed thoroughly, not waiting for meter to stabilize, contaminated standards.

2.6. Definitions:

2.6.1. ATC: automatic temperature compensation

2.6.2. NIST: National Institute of Standards and Technology

2.6.3. Stabilized: 1) Conductivity, salinity, TDS—when the endpoint icon stops blinking. 2) pH—when the reading has not changed over a 5-6 second time

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

span or the second digit to the right of the decimal moves back and forth between two digits.

3. EQUIPMENT AND SUPPLIES

3.1. Mettler Toledo SevenMulti® with conductivity and pH expansion units and ATC conductivity and pH probes

3.2. 30 mL Plastic cups

3.3. Waste cup

3.4. Rinse cups

3.5. Electronic thermometer

4. REAGENTS AND STANDARDS

4.1. Deionized (DI) Water and rinse bottle

4.2. Fisher® calibration buffers: pH 5.00, pH 7.00 and pH 10.00

4.2.1. Additional buffers such as pH 4, pH 9 or pH 13 can be used as needed.

4.3. Conductivity standards; very low-NIST 10 µmhos-cm, low-NIST 100 µmhos-cm, medium-NIST 1,000 µmhos-cm, and high-NIST 10,000 µmhos-cm

4.3.1. Actual values will vary.

4.3.2. A 100 mmhos-cm standard may be useful for extremely high (>50 mmhos) conductivity samples.

4.3.3. NIST traceable standards are purchased from Fisher Scientific.

4.4. Salinity Standard, 35 ppth (This can be used as a 50,000 µmhos-cm conductivity standard)

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1. See SOP FS 1000 for sample collection and preservation.

6. INTIAL SET-UP CALIBRATION

6.1. Turn the meter on by pressing the [On] button.

6.2. For initial conductivity calibration:

6.2.1. Press [Right]. Select **Menu** and then **2. Calibration Setting**. Use up/down arrow keys to highlight **1. Set Calibration Standard**. Press [Enter].

6.2.2. Use the up/down arrow keys to highlight **6. Enter Your Special Standard**. Press [Select]. Using the numerical keypad, enter your chosen standard (usually the 1,000 µmhos-cm standard). Press [Save] and then press [Exit] twice.

6.3. For initial pH calibration:

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

6.3.1. Select two standard buffers that bracket the expected pH of the solutions to be measured. Typically, Fisher® pH standards 5.0 and 10.0 are used for calibration and pH 7.0 standard is used for verification.

6.3.2. Label two cups with the chosen pH standard values. Pour 20-25 mL of standard into the appropriately labeled cup.

6.3.3. Use the electronic thermometer to measure the temperature of the buffers. Note the temperature. It will be entered into the meter later.

6.3.4. Press [Left] then select **Menu**. Use the up/down arrow keys to highlight **1. Calibration Setting**. Press [Enter]. Then, highlight **1. Set Calibration Buffer** and press [Enter].

6.3.5. Use the up/down arrow keys to highlight **2. Set a Customized Buffer Group** and press [Enter]. Press [Edit]. Use the numerical keypad to enter the buffer temperature measured in Step 6.3.3. Press [Next]. Use the numerical keypad to enter your first pH standard. Continue to press [Next] and enter the next standard until the customized buffer group has been entered.

6.3.5.1. The buffers should be entered in order from low to high.

6.3.5.2. A maximum of nine standards can be entered.

6.3.6. After the last entry, press [End], and then press [Exit] four times to return to the main screen.

7. DAILY CALIBRATION

7.1. Conductivity

7.1.1. Pour ~25 mL each of low, medium, and high conductivity standards into labeled 30 mL plastic cups.

7.1.2. Turn the meter on. Press [Right]. Remove the probe from the storage bottle, rinse with DI water and shake off the excess water.

7.1.3. Place probe into the conductivity calibration standard with the same value as the standard used for Instrument Setup Calibration, usually 1,000 µmhos-cm. Swirl the probe then hold it still. Do not let the probe touch the cup.

7.1.4. Press [CAL] to start calibrating. When the endpoint icon stops blinking, press [End] and then [Save].

7.1.5. Remove probe, rinse with DI water, and record the result in the *Daily Physical-Chemistry Calibration Log*.

7.1.6. Immerse the probe into the next conductivity standard to be measured. (Measurements are made from low to high.) Swirl the probe then hold it still. Press [Read]. When the endpoint icon stops blinking, record the value in the *Daily Physical-Chemistry Calibration Log*.

7.1.7. Repeat Step 7.1.6 with the third conductivity standard. Rinse the probe with DI water and return the probe to the storage bottle.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

7.1.7.1. Probe is stored in a BOD bottle ½-¾ full of DI water.

7.2. pH

7.2.1. Select two standard buffers that bracket the expected pH of the solutions to be measured. Typically, Fisher® brand pH 5 and pH 10 standards are used.

7.2.2. Select a third standard buffer with pH between the two buffers selected above.

7.2.3. Pour ~25 mL of each standard into appropriately labeled 30 mL cups.

7.2.4. Turn the meter on. If a single screen appears with the word "left" on it, go to the next step. If not, press [Dual], then press [Left]. The pH screen will appear. Continue on to the next step.

7.2.5. Place the electrode in the lowest value pH standard being used. The electrode tip should be immersed without touching the bottom of the cup. Swirl the electrode then hold it still.

7.2.6. Press the [CAL] button. When the displayed value is stable, record the result in the *Daily Physical-Chemistry Calibration Logbook*. Remove the electrode from the cup and rinse with DI water.

7.2.7. Repeat Steps 7.2.5-7.2.6 substituting the highest value pH being used. Press [End] and then [Save].

7.2.8. Place the electrode in the meter check pH standard (the third standard), swirl and then hold still. Press [Read]. When the displayed value is stable, record the result in the *Daily Physical-Chemical Calibration Logbook*.

7.2.8.1. This value must be within ±0.1 pH units of the expected value on the container label. If not, then the daily calibration should be repeated.

7.2.9. Remove the electrode from the cup and rinse with DI water. Return the electrode to the storage bottle.

7.2.9.1. Electrode is stored in a BOD bottle ½-¾ full of pH 7 buffer solution.

8. DAILY MEASUREMENTS

8.1. Ensure that the instrument is in the correct mode.

8.1.1. Conductivity: [Right] or [Dual] then [Right]

8.1.2. pH: [Left] or [Dual] then [Left]

8.1.3. Salinity: in conductivity mode, press [Mode] then [Sal]

8.1.3.1. Salinity can also be determined by conversion using the conversion table kept in the culture lab or by going to http://www.fivecreeks.org/monitor/sal.html Conductivity and temperature values are needed for conversion.

8.1.4. TDS: in conductivity mode, press [Mode] then [TDS]

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

8.2. Remove the appropriate probe (conductivity or pH) from its storage bottle. Rinse the probe and shake off the excess.

8.2.1. Conductivity probe is also used to measure salinity and TDS.

8.3. Immerse the probe in the solution, swirl, hold still then press [Read]. Avoid touching the sides and the bottom of the cup. When the reading stabilizes, record the measurement in the appropriate logbook. Remove the probe from the sample and rinse with DI water.

8.4. Repeat Step 8.3, until all samples have been read.

8.4.1. Read one parameter at a time, one sample at a time, starting with the lowest expected value and ending with the highest expected value per sample. Rinse probe between measurements with deionized water. When the one parameter has been read on the last aliquot of the last sample, select the next parameter and repeat.

8.4.1.1. It is suggested that conductivity be measured before pH due to possible leaking of electrode fill solution from the pH electrode into the sample.

8.5. When the last sample is measured and recorded, remove the probe from the sample, rinse with DI water and return to the appropriate storage bottle.

8.6. At the end of the day, or after the last measurement has been taken, perform an end-of-day calibration check.

8.6.1. Conductivity: measure the conductivity of the 1,000 µmhos-cm standard, and record the reading in the *Daily Physical-Chemical Calibration Logbook.*

8.6.2. pH: measure the pH of one standard (typically 7.00), and record the standard name and reading in the *Daily Physical-Chemical Calibration Logbook.* If the day-end measurement is more than ±0.2 SU from the buffer, contact the tox lab coordinator.

8.7. Turn the meter off by pressing the **on/off** button.

9. DATA ARCHIVAL

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

9.2 Record daily calibrations in the *Daily Physical-Chemical Calibration Logbook.* Record daily sample and control measurements in the appropriate bioassay logbooks.

9.3 To correct raw data entries, place a single line through the incorrect or illegible entry. Write the corrected entry near the error, date and initial the correction. Do not overwrite entries.

9.3.1 The corrected entry is written as near its appropriate space as possible or else in the comment box on the appropriate bench sheet.

9.3.2 If the correction requires an explanation, write a comment and reference the error. Initial and date the explanation.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

9.3.3 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.).

10. QUALITY CONTROL

10.1 During the daily conductivity calibration check, if the value obtained from any of the conductivity standards is not within ±5% of the listed value, the calibration check should be repeated. If a value still is not within ±5% of the listed value, then an initial calibration procedure should be performed. The additional daily calibration must be recorded in the *Daily Physical-Chemistry Calibration Logbook*. If the problem is not resolved, notify the tox lab supervisor.

10.2 During the daily pH calibration check, if the value obtained from any of the pH standards is not within ±0.2 pH units of the listed value, the calibration check should be repeated. If a value still is not within ±0.2 pH units of the listed value, then an initial calibration procedure should be performed. The additional daily calibration must be recorded in the *Daily Physical-Chemistry Calibration Logbook*. If the problem is not resolved, notify the tox lab supervisor.

10.3 Samples, standards and controls should be warmed to near 25 ˚C before being poured into their cups for measurement.

10.4 Sample parameters are read from lowest value to highest value to minimize the potential for contamination. (Probe is rinsed with deionized water between measurements).

10.5 If a measured sample pH is more than 1 SU less than the lowest daily calibration standard or more than 1 SU greater than the highest daily calibration standard, an additional day-end reading should be performed on a pH calibration standard having a pH value nearest the sample value. Ex.: sample reads pH 4.8, meter calibrated with 7.0 SU standard, the pH 4 SU standard should be measured at the end of the day. Secondary measurements are recorded in the comments section of the *Daily Physical-Chemistry Calibration Logbook.*

10.6 If a problem occurs during the sample processing or testing, the following steps are performed:

10.6.1 Lab supervisor is notified.

10.6.2 Client is notified (client makes decision for additional testing).

10.6.3 Section administrator is notified.

10.6.4 A non-conformance report (NCR) is submitted describing the problem and the corrective action.

10.6.4.1 To create an NCR: open LIMS and go to Laboratory/Administrative/Non-Conformance Report

10.7 Refer to http://www.dep.state.fl.us/water/sas/sop/sops.htm and the FDEP Laboratory Quality Manual for corrective action and handling out-of-control data.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.

11. REFERENCES

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

11.2 Refer to FDEP Laboratory Quality Manual for sample disposal.

11.3 Refer to the FDEP Laboratory Health and Safety Plan.

11.4 Read MSDS information for reagent handling and disposal.

11.5 Mettler Toledo SevenMulti® Instruction Manual.

11.6 FDEP laboratory Quality Manual http://www.dep.state.fl.us/labs/library/lab_sops.htm

11.7 Laboratory Health and Safety Plan (https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents).

11.8 MSDS information sheets.

Appendix of Changes

08/19/2013 Combined SOPs TA-06.24-1.9 and TA-06.25-1.9 and reformatted and saved the resulting SOP as TA-06.25-2.0.

03/15/2016 Updated links. Corrected manual references.

03/28/2017 Updated links.

The OFFICIAL COPY is the on-line version. All other copies are considered unofficial and uncontrolled.