

Standard Operating Procedure 5:

Core Water Quality – Equipment Maintenance and Data Collection (Version 1.0)

Summary

This Standard Operating Procedure (SOP) describes procedures for accurately measuring core water quality parameters (i.e., temperature, specific conductance, pH, dissolved oxygen) with equipment that the ERMN currently uses. This SOP will be updated accordingly should the ERMN use another product for in-situ core water quality parameter measurement.

Revision Log

Original Version #	Date of Revision	Revised By	Changes	Justification	New Version #
NA	2/23/2016	C. Tzilkowski	NA	Protocol Implementation Plan publication	1.0

Overview

This SOP is based primarily on procedures outlined by the U.S. Geologic Survey (Wilde 2008) and the Klamath Network (Dinger et al. 2013) and includes procedures for measuring “core” water quality parameters (water temperature, specific conductance, pH, and dissolved oxygen) at Eastern Rivers and Mountains Network (ERMN) wadeable stream sites. The ERMN currently uses a Eureka Water Probes (Austin, TX) ‘Sub 2’ water quality multiprobe (with Juniper “Archer” display); thus, procedures outlined here refer to those devices and is based on Eureka Water Probes (<http://waterprobes.com/>) guidance. This SOP will be updated accordingly should the ERMN use another product for in-situ core parameter measurement. Eureka Water Probes should be contacted periodically for software and firmware upgrades.

Sub 2 Maintenance and Calibration

Much of this section is taken directly from the Manta2 manual. If you have questions regarding calibration, refer to the manual which has additional information. Throughout the calibration process, be sure to record all appropriate data on a Field Water Quality Meter Accuracy Check and Calibration Worksheet (Appendix A). A binder with ample copies of the worksheet should be available while sampling benthic macroinvertebrates (BMI) or during any other activity when core parameters are measured. At the end of the calendar year, these worksheets should be electronically scanned (.pdf format), named and stored on the ERMN server.

Accuracy of the dissolved oxygen probe should be checked at each sampling location (because atmospheric pressure changes temporally and spatially) and calibrated if necessary whereas temperature, pH, and specific conductance should be checked daily against a NIST-traceable thermometer and standard calibration solutions.

Sub 2 Maintenance

The Sub 2 multiprobe should be thoroughly checked for damage, lubricated and calibrated in the laboratory immediately prior to field seasons. This process includes inspecting the Sub 2 exterior, lubricating seals (O-rings) with silicone grease, replacing the pH reference electrode solution, calibrating all sensors, and verifying the precision of the calibrated sensors.

After checking the Sub 2 housing for obvious damage (e.g., cracks), disassemble the Sub 2 by removing the four stainless steel hex screws – there are two screws near the cable port and two screws closer to the measurement probes. Be sure to pay close attention to how the housing components were originally assembled so that it can be reassembled in the correct configuration.

Gently remove the cable port end first while paying attention to the internally attached wires; do not pull hard, which could lead to accidentally pulling the attached wires from the internal electronics. Wipe any visible dirt from the inside of the housing then liberally apply new silicone grease to the O-rings. Repeat the process for the other end of the housing where the sensors are situated. Reassemble in the correct configuration

Sub 2 Calibration

The first step of Sub 2 calibration is to connect it to a PC or Archer display.

1. If necessary, attach the communication cable to the Manta2 USB adapter (Fig. 1). The Archer PDA does not require the adapter because it has a nine-pin connection.
2. Unscrew the top of the Sub 2 and remove the cable port plug.
3. Attach the communication cable to the Sub 2 – feed the cable through Sub 2 cap, plug the cable into the Sub 2, then screw the cap on. The plug remains separate until the cable is removed for SUB 2 storage.

Plug the Manta2 USB adapter into the computer and open Manta2 Control software.

The Manta2 Control software should find and communicate with the Sub 2 automatically. Instantaneous readings should be displayed once every second (the interval between readings can be changed but one second intervals work well for this application).



Figure 1. Adapter used for connecting Eureka multiprobes to USB port on personal computers.

Sensor Response Factor (SRF):

During calibration, the Sensor Response Factor (SRF) for all sensors is stored in the Calibration Record. Suppose that a typical Conductivity sensor reports 100 μA in a 1413 $\mu\text{S}/\text{cm}$ standard. If the Conductivity sensor reports 100 μA in that same calibration solution, then the SRF is 100% (some parameters, such as pH, have a more complex SRF calculation, but the effect is the same).

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If the response is 80 μ A, the SRF would be 80%. When you press the OK button to accept a calibration, the Sub 2 automatically accepts the calibration if the SRF is between 60% and 140%. If the SRF falls outside that range, you will be cautioned to check your standard value, make sure the sensor is clean, and make sure the reading has stabilized.

Temperature:

The Temperature sensor is an electrical resistor (thermistor) for which resistance changes predictably with temperature. The sensor is protected by a stainless-steel tube. Thermistors are very stable with time, and so do not require calibration. The thermistor should be checked against a NIST-traceable thermometer – record the reported values on the Field Water Quality Meter Accuracy Check and Calibration Worksheet. If the Sub 2 thermistor is not operating properly, it must be returned to Eureka for repair or replacement. The thermistor is not only important for accurate temperature readings, it is also crucial for the accurate calculation of several temperature-dependent core parameters (e.g., pH).

Specific Conductance:

The ERMN calibrates specific conductance using 100 μ S/cm standards, then checks this calibration with 10 and 1,000 μ S/cm standards. The zero point for the sensor is set electronically, so you only have to set the “slope” point: The general procedure follows:

1. Fill the calibration cup with approximately 2” of de-ionized (DI) water. Replace the calibration cup top and tighten.
2. Gently swirl the DI water around the cup to rinse the sensors. Make sure the water comes in contact with all sensors. Empty and repeat.
3. Empty the DI water from the calibration cup.
4. Fill the calibration cup $\frac{1}{4}$ of the way with 100 μ S/cm conductivity standard. Place the cap on the calibration cup and tighten.
5. Gently swirl the standard to ensure that the sensors are rinsed with the standard solution.
6. Empty the calibration cup.
7. Repeat steps 4, 5, and 6 two times (triple rinse in conductivity standard).
8. Fill the calibration cup with 100 μ S/cm conductivity standard so that the conductivity and temperature sensors are submerged.
9. In Manta2 Control software, click on “Manta2”, then “Calibrate...”, then “SpCond μ S/cm...”.
10. Enter “100” and press enter; when the reading has stabilized, press enter to calibrate. The Manta2 will report the resulting Sensor Response Factor (SRF); then press Y to accept the calibration, N to back up one step, or Exit to leave the sensor uncalibrated.

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11. Repeat steps 1-8 with the 10 $\mu\text{S}/\text{cm}$ and 1,000 $\mu\text{S}/\text{cm}$ standard solutions but do not calibrate with these! Simply check that the calibration is accurate with these standards and record the values.

pH Reference Electrode:

A key to reliable pH measurement is a frequently-maintained reference electrode. The reference electrode is required to complete voltage measurement for pH readings. Reference electrode maintenance is simple and should be conducted annually or when pH needs calibrated during deployment.

1. Remove the reference cap by unscrewing it from the reference sleeve (Fig. 2) and discard old reference electrolyte.
2. Fill the sleeve completely with fresh pH reference electrolyte (KCl saturated with silver chloride). Be careful not to introduce air bubbles.
3. With the sensors facing up, screw or press the reference cap back on to the sleeve. Air should be purged through the Teflon junction when replacement is completed.
4. As you screw the sleeve into place, the excess electrolyte is forced out of the sleeve through the reference electrode junction (the white, porous circle at the end of the sleeve). This not only purges bubbles from the electrolyte, but also cleans the junction.



Figure 2. To remove the pH reference electrode cap, insert a coin or appropriate screwdriver into the slot on the cap to loosen it (left) then unscrew and remove it using your fingers (right).

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pH:

The ERMN uses a two-point calibration with 4.00 and 7.00 buffered standard solutions using the following procedure.

1. Fill the calibration cup with approximately 2” of DI water. Replace the calibration cup cap and tighten.
2. Gently swirl the DI water around to rinse the sensors. Make sure that the water comes in contact with all sensors. Empty and repeat.
3. Empty the DI water from the calibration cup.
4. Fill the calibration cup $\frac{1}{4}$ of the way with pH 4.00 buffer. Place the cap on the calibration cup and tighten.
5. Gently swirl the standard around to ensure that the sensors are rinsed with the standard solution.
6. Empty the calibration cup.
7. Repeat steps 4, 5, and 6 twice.
8. In Manta2 Control software, click on “Manta2”, then “Calibrate...”, then “pH units...”.
9. Enter “4.00” as the “Sample 1 pH” and press enter. After the reading has stabilized, **press ENTER to calibrate**. The Manta2 will report the resulting Sensor Response Factor (SRF); then press Y to accept the calibration, N to back up one step, or Exit to leave the sensor uncalibrated.
10. Repeat steps 1-7, and 9 with pH 7.00 buffer and enter it as the “Sample 2 pH”. The calibration tab will remain open because pH requires a two-point calibration.
11. Repeat steps 1-7 with the pH 10.00 buffer but do not calibrate with this! Simply check the calibration with this and record the value.

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Dissolved Oxygen:

Optical sensors have a very stable zero calibration; consequently, Eureka recommends calibrating only at another, non-zero point. The Manta2 manual recommends that the point be 100% saturation in air-saturated water for Hamilton Dissolved Oxygen (HDO) sensors. ***Note that calibration of the HDO sensor should be done at the sampling location due to fluctuations in barometric pressure (e.g., inside/outside pressurized buildings, elevational changes).***

The general procedure follows.

1. Fill the calibration cup with approximately 2” of DI water. Replace the calibration cup cap and tighten.
2. Gently swirl the DI water around to rinse the sensors. Make sure the water comes in contact with all sensors. Empty and repeat.
3. Empty the DI water from the calibration cup.
4. Set the instrument’s Barometric Pressure **with the calibration cup empty**:
 - a. In Manta2 Control software, click on “Manta2”, then “Calibrate...”, then “Set BP...”.
 - b. Click on the “Get BP” tab and the correct reading will be displayed.
 - c. Click “OK” and proceed with DO calibration check.
5. Shake the bottle of DI water (with the lid on) vigorously for one minute. Take the lid off of the bottle and let the water stand for approximately five minutes to let the air bubbles float out.
6. Fill your calibration cup until the aerated water covers the DO sensor by approximately 1 cm.
7. In Manta2 Control software, click on “Manta2”, then “Calibrate...”, then “HDO %Sat...”.
8. Press the “Cal” tab and then the “100” tab.
9. Once the reading stabilizes, press “enter”. The Manta2 will report the resulting Sensor Response Factor (SRF); then press Y to accept the calibration, N to back up one step, or Exit to leave the sensor uncalibrated.

Sub 2 Accuracy Check

After the Sub 2 has been calibrated it is possible that only the HDO sensor will need routine calibration (at each site) during the field season because the other sensors (particularly temperature and specific conductance) remain very accurate. Even though the sensors may not need recalibrated, it is very important to verify and document that the sensors are indeed accurate. The Sub 2 accuracy check can be conducted in approximately 10 minutes and should

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be done at either the beginning or end of each day during the field season. All associated data should be recorded on a Field Water Quality Meter Accuracy Check and Calibration Worksheet.

The accuracy check is essentially an abbreviation of the calibration process where the thermistor (water temperature), specific conductance sensor and pH sensor are checked against known standards to verify their accuracy. If accuracy criteria are not met, sensors are recalibrated. Temperature is checked against a NIST-certified or -traceable thermometers whereas specific conductance and pH sensors are checked against 100 $\mu\text{S}/\text{cm}$ and pH 7.0 standard solutions respectively. See the Sub 2 calibration process above for details on how to check the sensors.

Core Water Quality Parameter Data Collection

1. After you have arrived at a sampling location:
 - a. Connect the Sub 2 to the Archer;
 - b. Turn the Archer on and open the Manta2 Control Software;
 - c. Check the dissolved oxygen sensor calibration using the instructions in the “Sub 2 Sensor Calibration” section above; calibrate if necessary.
 - d. Remove the Sub 2 calibration cup and attach the Sub 2 sensor guard;
 - e. Place the Sub 2 in the stream and allow the sensor measurements to stabilize for 10 minutes. If the Sub 2 can be situated at a cross-section upstream of BMI sampling locations, it can be allowed to stabilize while BMI are collected to maximize efficiency.
2. Locate a stream cross-section that:
 - a. Has moderate to fast velocities;
 - b. Appears well-mixed; and
 - c. Has minimal turbulence.
3. Determine whether the stream cross-section is well-mixed and that a single measurement location will be representative of the cross-section.
 - a. For narrow streams (e.g., < 3 m) it is usually most effective and sufficient to place the Sub 2 probe in the water at mid-depth and slowly cross the stream while monitoring the core parameters (primarily specific conductance).
 - b. If the stream is wider, it may be necessary to measure the stream width and then visually break the stream into seven equidistant points and measure at those locations.
 - i. The best way to determine these measurement locations is to use the width at the cross-section, divided by eight. This is the distance from the near shore that the first reading should be taken. For example, if the width is 2.5 m, the first point measurement should be at 0.31 m from the shore. The next measurement should be at 0.62 m (0.31 m from the first measurement).
 - c. While monitoring the measurements, look for low variation in specific conductance readings to ensure mixing.

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- d. When entering the water, position your body downstream of the probe. Disturbance upstream could bias measurements.
4. If measurements are consistent across the cross-section, place the Sub 2 at mid-depth near the maximum flow (typically near the middle of the stream) and record the core parameter measurements on the data sheet.
 - a. If specific conductance measurements (in particular) are variable, the cross-section is not well-mixed, a new location should be found and evaluated for sufficient mixing (i.e., repeat the measurement process).
5. After core parameters have been recorded, power down the Sub 2, fill the storage cup with ½" of stream water, remove the sensor guard and replace it with the storage cup.

Literature Cited

- Dinger, E. C., D. A. Sarr, S. R. Mohren, K. M. Irvine, and C. E. Stanley. 2013. Integrated aquatic community and water quality monitoring of wadeable streams in the Klamath Network: Narrative and standard operating procedures. Natural Resource Report NPS/KLMN/NRR—2013/669. National Park Service, Fort Collins, Colorado.
- Eureka Water Probes. 2011. Manta 2 water quality multiprobe manual. Austin TX. waterprobes.com (accessed January 20, 2015).
- Wilde, F.D., 2008, Guidelines for field-measured water-quality properties (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6, section 6.0, October, available only online from <http://pubs.water.usgs.gov/twri9A/>.

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Appendix A. Field Water Quality Meter Accuracy Check and Calibration Sheet

Be sure to fill in all cells, using "NA" for "not applicable".

Calibrated by:		Park:
Date:	Time:	Activity:
Meter Make and Model:		
S/N:		

Temperature - Single Point Check with NIST-certified or NIST-traceable Thermometer

NIST Water Temp.:	Meter Water Temp.:	Error:	Calibration criteria: $\pm 0.2^{\circ}\text{C}$
			Replace Thermistor?: YES NO

Specific Conductance Error Check (100 $\mu\text{S}/\text{cm}$) and Calibration (if necessary) - triple rinse sensors

Standard	Pre-cal. Reading	Post-cal. Reading	Error	Cal. Criteria	Std. Maker:	Lot #:	EXP Date:
100 $\mu\text{S}/\text{cm}$				$\pm 5 \mu\text{S}/\text{cm}$			
10 $\mu\text{S}/\text{cm}$				$\pm 5 \mu\text{S}/\text{cm}$			
1,000 $\mu\text{S}/\text{cm}$				$\pm 30 \mu\text{S}/\text{cm}$			

pH Error Check and/or Calibration (if necessary); replace electrolyte routinely (2 mo.)

Error check at pH = 7.0. Calibration criteria: ± 0.2 standard units.							
Standard	Pre-cal. Reading	Post-cal. Reading	Error	Std. Maker:	Lot #:	EXP Date:	
7.0							
10.0							
4.0							

Hamilton Optical (HDO) Dissolved Oxygen (DO) Error Check and/or Calibration

Check/calibrate in 100% air-saturated water – (1) Shake $\frac{1}{2}$ L-jar of water for 1 min, remove lid and let stand 5 min, (2) Fill cal. cup w/water, covering DO sensor > 1 cm, (3) Ensure BP is accurate, (4) Allow temp. to equilibrate, (5) Follow Manta instructions. Calibration criteria: 0.3 mg/L or 3% saturation.				
Water Temp $^{\circ}\text{C}$	Baro. Press. mm Hg	DO Table Reading mg/L	DO Reading (pre-cal)	DO Reading (post-cal)

Turbidity Error Check and/or Calibration

Depth Error Check and/or Calibration

Standard (approx. 100 NTU) must be used within 24 hr of preparation. Calibration criteria: ± 3.0 NTU or 30%				Be sure that all sensors are exposed to air then calibrate w/Manta Control software		
Standard	Pre-cal. Reading	Post-cal. Reading	Error	Pre-cal. Reading	Post-cal. Reading	Error
0						