# Standard Operating Procedures Field Data Collection

# **1.0 Scope and Application**

This standard operating procedure (SOP) is applicable to wastewater pond sampling for surface water samples, sludge samples and water quality readings. The following SOP will be used for the Legislative-Citizen Commission on Minnesota Resources project wastewater sampling conducted by the Minnesota Technical Assistance Program and Minnesota Rural Water Association.

# 2.0 Sample Preservation, Containers, Handling and Storage

All samples collected will be thermally preserved on ice from the time of collection to the delivery to the laboratory, where they will be thermally preserved in the facility refrigeration system. All samples that require acid preservation will have chemicals in the bottles provided by the laboratory.

# **3.0 Equipment/Apparatus**

Sampling: Water samples will be collected using a submersible pump. The polyethylene (HDPE or LDPE) tubing will be changed out between each pond and transfer station. A Sludge Judge ® will be used to collect sludge samples.

Sonde Equipment: EXO Sonde with probes for conductivity, ORP, pH, turbidity, ammonium, nitrate, temperature, dissolved oxygen, and total algae.

Weather station: Ambient Weather WS-2902B Wifi Smart Weather Station

# 4.0 Procedures

### 4.1 Setup weather station

The field personnel will setup the weather station on land in an open area that will have conditions similar to the pond surface (i.e., not blocked by buildings, trees, etc.). Readings will be recorded on the Weather Data Sheet before sampling, midday and after sampling.

### 4.2 Prepare boats before departing shore

- Cut 15 ft of tubing. Leave extra on shore
- Attach end of tubing to top of submersible pump. Secure other end of tubing to boat with clamp.
- Secure Sonde to submersible pump with zip tie. Secure tape measure to submersible pump with zip tie. At this point, the submersible pump, Sonde and tip of the tape measure should all be connected with zip ties in one unit.
- Put empty cooler with empty sample bottles into boat. Put enough coolers with ice in it to accommodate the sample bottles once filled.
- Check boat for:
  - o Submersible pump
  - Control box for submersible pump
  - Appropriate tubing

- o Sonde and all attachments
- 7 sets of liquid samples bottles
- 1 set of sludge sample bottles
- Filters for collecting ortho phosphorous sample
- Coolers with and without ice
- Ziploc bags for packing samples

## 4.3 Position watercraft in center of pond

Using Google Maps set to satellite imagery, navigate to the center of the pond. The field personnel will stop the watercraft's motor when positioned in the center of the pond. No anchor will be used in order to prevent damage to the pond liner and disturbance of bottom sediments. Instead, a rope will be secured on land, upwind of the pond. Once the boat is at the middle of the pond, that rope will be secured to the boat, preventing the boat from being blown downwind. Upon stabilizing the watercraft, the field personnel will complete the information at the top of the field datasheets prior to collection of samples.

## **4.4 Collection of samples**

## 4.4.1 Collection of pond water samples

When collecting water samples, a submersible pump will be used in coordination with a Sonde for water quality measurements. The pond water sampling will follow the instructions below.

- Don nitrile gloves
- Measure the water depth using the measuring device. Record.
- Calculate the necessary sampling depths (1-7). Record.
- Turn on Sonde and set to record at 1 second interval.
- Moving from the top of the water to bottom of the water column, slowly lower the sampling unit to the desired depth and ensure minimal disturbance in the water column.
- Once at the appropriate depth, secure the unit to the side of the boat.
- Connect the pump wire to the battery.
- Turn on pump slowly with control box. Keep flow as slow as possible.
- Goal is a flowrate around 700 mL per minute. Base the flowrate on minimal disturbance if you see an increase in turbidity, lower the flowrate. This flowrate is only for guidance and is subject to change.
- Discharge the first approximate 700 mL to clear any volume within the hose.
- Prepare sample bottles for the corresponding depth.
- Check sample labels are correct.
- Don new nitrile gloves.
- Begin filling sample bottles.
  - Open first sample bottle
  - Hold sample bottle in one hand over upright bucket
  - In other hand, hold end of tubing with water running out.
  - Hold the hose over the sample bottle mouth until water level is in bottleneck of container.
  - Pass full bottle to sampling partner to cap
  - Finalize and check label
  - $\circ$  Wipe down with paper towel
  - Put in gallon size Ziploc bag
  - Place on ice in cooler

- Get next sample bottle and repeat process for remaining containers at this sample point.
- NOTE that ortho phosphorous need to be FIELD FILTERED. Use syringe from St. Cloud and follow this procedure:
  - Fill the syringe provided by St. Cloud with sample water and rinse the syringe 3 times. This means fill the syringe and push out the contents 3 times
  - After rinsing:
    - Fill syringe
    - Put filter on tip of syringe
    - Push sample out of syringe into appropriately labeled bottle
- Ensure all collected sample bottles have proper labels, preservation and are stored on ice in cooler.
- Prepare next set of sample bottles if not already prepared.
- Lower sonde and submersible pump to next sampling interval and repeat process.
- When water sampling is completed, disconnect polyethylene tubing from pump and dispose of tubing.
- Empty any water from the catch buckets back into the pond.
- Rinse all equipment with clean water when possible.

## 4.4.2 Collection of sludge samples

- At the same location the water samples were collected in the pond, a sludge sample will be collected from the bottom of the pond.
- Prepare all sample bottle labels ahead of time.
- Use the Sludge Judge ® to collect the sludge sample.
- Fill all sample bottles.
- Check all sample bottle labels.
- Put samples on ice.

### 4.4.3 Collection of flow channel water samples

- Open manhole cover of transfer station.
- Sample in middle of depth and width of stream if possible
- Follow same sampling procedures as for the pond
- Connect the pump wire to the battery.
- Turn on pump slowly with control box. Keep flow as slow as possible.
- Goal is a flowrate around 300-700 mL per minute. Base the flowrate on minimal disturbance if you see an increase in turbidity, lower the flowrate. This flowrate is guidance and subject to change.
- Discharge the first approximate 700 mL to clear any volume within the hose.
- Prepare sample bottles for the corresponding depth.
- Check sample labels are correct.
- Don new nitrile gloves.
- Begin filling sample bottles.
- Open first sample bottle
- Hold sample bottle in one hand over upright bucket
- In other hand, hold end of tubing with water running out.
- Hold the hose over the sample bottle mouth until water level is in bottleneck of container.
- Pass full bottle to sampling partner to cap, finalize label, wipe down with paper towel, and place on ice in cooler.

- Get next sample bottle and repeat process for remaining containers at this sample point.
- Pour appropriate preservatives into bottle if applicable
- Cap
- Check label
- Store on ice
- When sampling is completed, disconnect tubing from pump and dispose of tubing.
- Rinse all equipment with clean water when possible.

## 4.5 Collection of water quality readings - Sonde

#### 4.5.1 Sonde Calibration

#### First-Time Setup

- 1. Install KorEXO software on a laptop computer.
  - a. Insert the supplied USB flash drive into a USB port on your computer.
  - b. Double-click Start.exe in the EXO DRIVE window to launch the Installer.
  - c. Click INSTALL DRIVERS and click INSTALL ALL to install all EXO hardware drivers. Follow the prompts to complete each driver installation.
  - d. After drivers are installed, click BACK to return to the KorEXO Installer main menu.
  - e. Click INSTALL APPLICATION and check the box the agree to license terms and conditions, and then click INSTALL.
  - f. After successful install, close the installer.
  - g. Open the KorEXO Software program for the first time. You may be asked if you want to allow a program from an unknown publisher to make changes on the computer. If so, select YES.
- 2. Remove calibration cup and sensor guard from the Sonde if they are attached. Place the Sonde on a clean, flat surface in order to keep it from rolling.
- 3. Visually inspect the Sonde ports for contamination. If the port is dirty or wet, clean it with a KimWipe and/or compressed air.
- 4. Install the Conductivity/Temperature sensor in Port 1.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 5. Install the Dissolved Oxygen sensor in Port 2.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.

- e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 6. Install the pH/ORP sensor in Port 3.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 7. Install the Turbidity sensor in Port 4.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 8. Install the Total Algae sensor in Port 5.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 9. Install the Ammonium sensor in Port 6.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1⁄4 turn until snug.

- 10. Install the Nitrate sensor in Port 7.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Remove and hydration caps or buffer bottles on the probe.
  - d. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - e. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.

### Start of Sampling Season Setup

- 1. Loosen the battery cap on the EXO2 Sonde. Slide the wrench tool's smaller opening over the battery cap on top of the Sonde. Using the tool as a lever, firmly turn the tool counterclockwise until the battery cap is loose.
- 2. Remove battery cap. Clean O-ring sealing surface of the cap with a KimWipe. Make sure the battery tube is dry and clean. If not, use additional KimWipes to clean/dry the tube.
- 3. Insert four (4) new D-cell batteries into the battery well.
- 4. Ensure that the O-rings are not nicked or torn and that there are no contaminants or particles on the O-rings or the sealing surfaces inside the battery cover.
  - a. Apply a thin coat of Krytox lubricant to each O-ring and sealing surface.
- 5. Insert the cap into its recess. With your thumb press down on the pressure relief valve while turning the cap clockwise. Once the cap threads are engaged, use the tool to tighten until snug.

### **Generic Setup**

- 1. Remove the calibration cup and sensor guard from the Sonde.
- 2. Remove the pH/ORP sensor from the storage bottle that it was shipped in. Make sure that it was submerged. If not, allow to soak in 2M KCI (potassium chloride) solution, pH 4 buffer, or tap water overnight.
- 3. Install the pH/ORP sensor in Port 3.
  - a. Remove the port plug. Set aside in a safe space.
  - b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
  - c. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
  - d. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 4. Remove the Ammonium sensor from the storage bottle that it was shipped in. There should be some amount of water in the storage bottle, but the probe should not be submerged. If the storage bottle is completely dry, allow the probe to soak in tap water overnight.
- 5. Install the Ammonium sensor in Port 6.
  - a. Remove the port plug. Set aside in a safe space.

- b. Apply a light coat of Krytox grease to the rubber mating surfaces of the connector (not the O-ring) and a small dab of Krytox grease on the threads of the locking nut.
- c. Insert the sensor into the port by properly aligning the connectors' pins and sleeves (male and female contacts); then press them firmly together.
- d. Taking care not to cross-thread the grooves, finger-tighten the locking nut clockwise. When the nut and O-ring are seated against the bulkhead, tighten the nut with probe tool 1/4 turn until snug.
- 6. Activate Bluetooth wireless capabilities of the Sonde by holding the magnet on the sensor removal tool to the magnetic activation area on the Sonde's bulkhead (identified by the illustrated magnet symbol on the label). Simply hold the magnet within one (1) cm of the symbol until the LED light turns blue.
- 7. Launch KorEXO software.
  - a. Click the "Scan for Bluetooth Devices" button in the Instrument Connection Panel.
    - i. This may need to be repeated several time before the software finds the Sonde.
  - b. Once the EXO Sonde appears, simply click the Connect button to establish communications.
  - c. This blue LED light on the Sonde should start flashing, meaning that a connection has been made.
- 8. Click the "Calibrate Sensors" button on the main screen of the KorEXO software.
- 9. Select "Calibrate" to perform a user calibration.

### Calibration

The sonde will be calibrated once per three-week pond testing interval, prior to sampling. These parameters are:

- Conductivity/Temperature
- Depth
- DO
- pH
- ORP
- Total Algae (TAL)
  - Chlorophyll
  - Phycocyanin
  - o Phycoerythrin
- Turbidity
- Ammonium
- Nitrate

Prior to calibration, remove the ammonium and nitrate sensors from the sonde and install plugs instead, as some calibration solutions will damage the ammonium and nitrate sensors. The sonde will display a warning message if the user selects a calibration with an incompatible probe installed. A pill bottle or similar can be used instead of the sonde calibration cup for pH/ORP, ammonium, and nitrate calibration. The pH/ORP sensor must be installed next to the

Conductivity/Temperature sensor so they can both be submerged into the pill bottle for correct calibration. The algae, turbidity, DO, and conductivity sensors cannot be calibrated in a pill bottle.

- 1. Calibrate the Conductivity/Temperature sensor.
  - a. This calibration will be completed in the calibration cup.
  - b. Clean out the flow cell for the conductivity probe with the included.
  - c. Rinse out the flow cell with tap water.
  - d. Dry the flow cell with a KimWipe, and used compressed air to blow out any moisture remaining in the cell.
  - e. Referencing the KorEXO software, make sure the conductivity is reading ~0.1 uS/cm in the open air.
  - f. Fill up calibration cup with a small amount of conductivity standard solution. Screw the calibration cup onto the Sonde and shake to rinse probe in solution. YSI recommends rinsing three times.
  - g. Pour conductivity standard into clean, dry, rinsed calibration cup. Fill cup to second marked line to ensure standard is above vent holes on the conductivity sensor. Immerse probe end of Sonde into solution, gently rotate Sonde to remove any bubbles from the conductivity cell.
  - h. Allow one minute for temperature equilibration.
  - i. Select the Conductivity sensor from the calibration list.
  - j. Select specific conductance, and enter 1000 uS.
  - k. Once data is stable, observe and record the pre-calibration value on the Sonde calibration form.
  - I. If the data does not stabilize in 40 seconds, gently rotate the Sonde or remove and reinstall the calibration cup to ensure there are no air bubbles in the conductivity cell.
  - m. Click apply to accept the calibration, and record the calibrated value on the Sonde calibration form.
  - n. Rinse the Sonde and sensors in tap water and dry.
- 2. Calibrate the Depth and Level sensor.
  - a. For this calibration, ensure the depth sensor is in the air and not immersed in any solution. Keep the Sonde in a steady orientation matching the deployment orientation for the duration of this calibration.
  - b. In the Calibrate menu, select Depth and then select Calibrate.
  - c. .139 m will be used as an offset for this calibration this is the distance from the pressure transducer to the bottom of the probes.
  - d. Observe the pre-calibration value reading and data stability. When data is stable, click apply to accept the calibration point. This process zeros the sensor with regard to current barometric pressure. Record the pre-calibration and post-calibration values on the field calibration form.
- 3. Calibrate the Dissolved Oxygen sensor.
  - a. This calibration will be completed in the calibration cup.
  - b. Dissolved oxygen is an optical sensor this calibration should always be completed in the official calibration cup with the sensor guard on.
  - c. Ensure there are no water droplets on the DO sensor. Place Sonde sensors into a calibration cup containing 1/8 inch of water that is vented by loosening the

calibration cup threads. Wait 15 minutes before proceeding to allow the temperature and oxygen pressure to equilibrate. Keep out of direct sunlight.

- d. In the Calibrate menu, select ODO, then ODO % sat.
- e. Enter the current barometric pressure in mmHg. This value will be read from the Sonde handheld.
- f. Observe the pre-calibration value and data stability. When stable, record the precalibration value, and apply the calibration. Record the post calibration value. Click complete.
- 4. Calibrate the pH sensor.
  - a. This calibration can be completed in a pill bottle or the calibration cup. If using the pill bottle, submerge the temperature probe as well.
  - b. Select the 3-point option to calibrate the pH probe using three calibration standards. In this procedure, the pH sensor is calibrated with a pH buffer of first 4, then 7, then 10. Pour pH 4 buffer into clean, rinsed calibration cup, up to the first line. Carefully immerse the Sonde into solution, ensuring the sensor's glass bulb is in solution by at least 1 cm. Allow 1 minute for temperature equilibration before proceeding.
  - c. In the Calibrate menu, select pH/ORP, then select Calibrate. Enter the pH value from the pH bottle corresponding with the current temperature.
  - d. Observe the pre-calibration values and data stability. When stable, click apply to accept the calibration. Click "add another cal point" in the software.
  - e. Rinse the sensor in DI water. Pour the correct amount of pH 7 buffer solution into clean, rinsed calibration cup up to the first line and carefully immerse the probe end of the Sonde into solution. Allow 1 minute for temperature equilibration before proceeding.
  - f. Observe the pre-calibration values and data stability. When stable, click apply to accept the calibration. Click "add another cal point" in the software.
  - g. Rinse the sensor in DI water. Pour the correct amount of pH 10 buffer solution into clean, rinsed calibration cup up to the first line, and carefully immerse the probe end of the Sonde into solution. Allow 1 minute for temperature equilibration before proceeding.
  - h. Observe the pre-calibration values and data stability. When stable, click apply to accept the calibration. Click complete.
  - i. Rinse the Sonde in tap water.
- 5. Calibrate the ORP sensor.
  - a. This calibration can be completed in a pill bottle or the calibration cup. If using the pill bottle, submerge the temperature probe as well.
  - b. Pour correct amount of Zobell calibration solution into pre-rinsed calibration cup. Carefully immerse the probe end of the Sonde into the solution.
  - c. In the calibrate menu, select pH/ORP, then select ORP to calibrate.
  - d. Observe the pre-calibration value readings and data stability. When stable, click apply to accept the calibration point and record values on the field calibration form. Note: Do not leave sensors in Zobell solution for a long period of time. A chemical reaction occurs with the copper on the Sonde, which will degrade Sonde materials over time.
  - e. Rinse the Sonde in tap water.
- 6. Prepare calibration solutions for Total Algae probe.

- a. This is an optical probe; the calibration must be completed in the calibration cup with the sensor guard attached.
- b. For any TAL sensor calibration, prepare a 125 mg/L solution of Rhodamine WT. Transfer 5.0 mL of the 2.5% Rhodamine WT solution into a 1000 mL volumetric flask. Fill the flask to the volumetric mark with deionized or distilled water and mix well to produce a solution that is approximately 125 mg/L of Rhodamine WT. Transfer to a storage bottle and retain it for future use.
  - i. This solution can be stored in the refrigerator (4°C). Its degradation will depend upon light exposure and repeated warming cycles, but solutions used 1-2 times a year can be stored for up to two years. Users should implement their own procedures to safeguard against degradation.
- c. For calibration of any chlorophyll channel (on either the TAL-PC or the TAL-PE sensor) and the TAL-PC phycocyanin channel, prepare a 0.625 mg/L solution of Rhodamine WT. Transfer 5.0 mL of the 125 mg/L solution prepared in step one into a 1000 mL volumetric flask. Fill the flask to the volumetric mark with deionized or distilled water. Mix well to obtain a solution that is 0.625 mg/L of Rhodamine WT. Use this solution within 24 hours of preparation and discard it after use.
- d. If using a TAL-PE sensor, additionally prepare a 0.025 mg/L solution of Rhodamine WT for calibration of the phycoerythrin channel. Transfer 0.2 mL of the 125 mg/L solution prepared in step one into a 1000 mL volumetric flask. Fill the flask to the volumetric mark with deionized or distilled water. Mix well to obtain a solution.
- 7. Calibrate the Total Algae probe for Chlorophyll.
  - a. This is an optical probe; the calibration must be completed in the calibration cup with the sensor guard attached.
  - b. Place Sonde loaded with total algae sensor in clean calibration cup containing clean water. It is not required that this be DI or distilled water, but it must be free of particles that might fluoresce and interfere with the calibration process. Distilled water is a good choice to meet this requirement.
  - c. The software will show a graph while the sensor is stabilizing and temperature will also be shown. Temperature is not needed for this zero point; the user must enter a "Standard Value" of 0. When data is stable, apply the calibration. Next select "Add Another Cal Point".
  - d. The same basic procedure will be followed, but using the KOR software will require the user to enter the temperature-compensated standard value for the calibration solution. This will be done using the reading from the EXO temperature sensor. Use the following reference table and enter the ug/L value for chlorophyll corresponding with the temperature reading.

	Chlorophyll 0.625 mg/L Rhodamine		Phycocyanin 0.625 mg/L Rhodamine		Phycoerythrin 0.025 mg/L Rhodamine	
Solution Temperature (°C)	ChI RFU	µg/L chlorophyll	PC RFU	µg/L phycocyanin	PE RFU	μg/L phycoerythrin
30	14.0	56.5	11.4	11.4	37.3	104.0
28	14.6	58.7	13.1	13.1	39.1	109.0
26	15.2	61.3	14.1	14.1	41.0	115.0
24	15.8	63.5	15.0	15.0	43.0	120.0
22	16.4	66	16.0	16.0	45.0	126.0
20	17.0	68.4	17.1	17.1	47.0	132.0
18	17.6	70.8	17.5	17.5	49.2	138.0
16	18.3	73.5	19.1	19.1	51.4	144.0
14	18.9	76	20.1	20.1	53.6	150.0
12	19.5	78.6	21.2	21.2	55.9	157.0
10	20.2	81.2	22.2	22.2	58.2	163.0
8	20.8	83.8	22.6	22.6	60.6	170.0

Table: Temperature-compensated standard solution values for TAL sensors.

- e. For example, when calibrating chlorophyll, if the solution temperature is 22 Celsius, the chlorophyll value in ug/L to be entered into the calibration software will be 66 ug/L.
- f. Upon entering the Table derived value, wait for the sensors to stabilize, then click apply, then complete calibration.
- 8. Calibrate the Total Algae probe for Phycocyanin (PC).
  - a. This is an optical probe; the calibration must be completed in the calibration cup with the sensor guard attached.
  - b. Place Sonde loaded with total algae sensor in clean calibration cup containing clean water. It is not required that this be DI or distilled water, it must be free of particles that might fluoresce and interfere with the calibration process. Distilled water is a good choice to meet this requirement.
  - c. The software will show a graph while the sensor is stabilizing and temperature will also be shown. Temperature is not needed for this zero point; the user must enter a "Standard Value" of 0. When data is stable, apply the calibration. Next select "Add Another Cal Point".
  - d. The same basic procedure will be followed, but using the KOR software will require the user to enter the temperature-compensated standard value for the calibration solution. This will be done using the reading from the EXO temperature sensor. Use the reference table shown in item 9 and enter the ug/L value for phycocyanin (PC) corresponding with the temperature reading.
  - e. Upon entering the Table derived value, wait for the sensors to stabilize, then click apply, then complete calibration.
- 9. Calibrate the Total Algae probe for Phycoerythrin (PE).
  - a. This is an optical probe; the calibration must be completed in the calibration cup with the sensor guard attached.
  - b. Place Sonde loaded with total algae sensor and algae sensor in clean calibration cup containing clean water. It is not required that this be DI or distilled water, it

must be free of particles that might fluoresce and interfere with the calibration process. Distilled water is a good choice to meet this requirement.

- c. The software will show a graph while the sensor is stabilizing and temperature will also be shown. Temperature is not needed for this zero point; the user must enter a "Standard Value" of 0. When data is stable, apply the calibration. Next select "Add Another Cal Point".
- d. The same basic procedure will be followed, but using the KOR software will require the user to enter the temperature-compensated standard value for the calibration solution. This will be done using the reading from the EXO temperature sensor. Use the reference table in item 9 and enter the ug/L value for phycoerythrin (PE) corresponding with the temperature reading.
- e. Upon entering the Table derived value, wait for the sensors to stabilize, then click apply, then complete calibration.
- 10. Calibrate the Turbidity sensor.
  - a. This is an optical probe; the calibration must be completed in the calibration cup with the sensor guard attached.
  - b. Turbidity is an optical sensor this calibration should always be completed in the official calibration cup with the sensor guard on.
  - c. Pour 0 FNU standard into clean calibration cup. Ensure Sonde guard is on.
    Ensure bubbles are not trapped on the face of the turbidity sensors. *Note: 1 FNU* = 1 NTU
  - d. Select Turbidity in the EXO software to calibrate.
  - e. Enter 0.0 for the first calibration value. Once stable, select apply calibration for this point.
  - f. Rinse sensors, Sonde guard, and calibration cup with a small amount of the calibration solution to be used for point two calibration solution (124 NTU). Discard this rinse.
  - g. Fill calibration cup to first line with calibration solution. Click "add another cal point" in the software.
  - h. Place the sensor into the second calibration standard, ensuring there are no bubbles. Input the standard's FNU value, 124 NTU, from the bottle into the software program. Once stable, click apply calibration for this point.
  - i. Rinse sensors, Sonde guard, and calibration cup with a small amount of the calibration solution to be used for point three (1010NTU) calibration. Discard this rinse.
  - j. Place the sensor into the second calibration standard, ensuring there are no bubbles. Input the standard's FNU value, 1010 NTU, from the bottle into the software program. Once stable, click apply calibration for this point.
  - k. Rinse the Sonde with water and discard the calibration solution.
- 11. Calibrate the Ammonium sensor.
  - a. This calibration may be completed in a pill bottle or the calibration cup.
  - b. Before its first use, soak the ammonium sensor in 100 mg/L ammonium standard solution overnight.
  - c. Pour sufficient amount of 1 mg/L NH4 calibration standard solution at ambient temperature in a clean, dry, rinsed calibration cup. Carefully immerse the sensor end of the Sonde into solution, making sure the sensor's tip is in solution by at

least 1 cm. Allow at least 1 minute for temperature equilibration before proceeding.

- d. Observe the pre-calibration value and data stability. When stable, click apply to accept calibration point. Record value on Sonde calibration form.
- e. Rinse sensors in DI water between changes of calibration solution. Pour sufficient amount of 10 mg/L NH4 calibration solution at ambient temperature in a clean, dry, rinsed calibration cup. Carefully immerse the sensor end of the Sonde into solution, making sure the sensor's tip is in solution by at least 1 cm. Allow at least 1 minute for temperature equilibration before proceeding.
- f. Click "add another cal point". Observe the pre-calibration value and data stability. When stable, click apply to accept calibration point. Record value on Sonde calibration form.
- g. Rinse Sonde in tap water.
- h. Remove the ammonium sensor from the Sonde, and place in its storage bottle with a small amount of tap water or calibration standard. Make sure that the sensor is not immersed in the storage solution.
- 12. Calibrate the Nitrate sensor.
  - a. This calibration may be completed in a pill bottle or the calibration cup.
  - b. Pour sufficient amount of 1 mg/L NO3 calibration standard solution at ambient temperature in a clean, dry, rinsed calibration cup. Carefully immerse the sensor end of the Sonde into solution, making sure the sensor's tip is in solution by at least 1 cm. Allow at least 1 minute for temperature equilibration before proceeding.
  - c. Observe the pre-calibration value and data stability. When stable, click apply to accept calibration point. Record value on Sonde calibration form.
  - d. Rinse sensors in DI water between changes of calibration solution. Pour sufficient amount of 10 mg/L NO3 calibration solution at ambient temperature in a clean, dry, rinsed calibration cup. Carefully immerse the sensor end of the Sonde into solution, making sure the sensor's tip is in solution by at least 1 cm. Allow at least 1 minute for temperature equilibration before proceeding.
  - e. Click "add another cal point". Observe the pre-calibration value and data stability. When stable, click apply to accept calibration point. Record value on Sonde calibration form.
  - f. Rinse sensors in DI water between changes of calibration solution. Pour sufficient amount of 100 mg/L NO3 calibration solution at ambient temperature in a clean, dry, rinsed calibration cup. Carefully immerse the sensor end of the Sonde into solution, making sure the sensor's tip is in solution by at least 1 cm. Allow at least 1 minute for temperature equilibration before proceeding.
  - g. Click "add another cal point". Observe the pre-calibration value and data stability. When stable, click apply to accept calibration point. Record value on Sonde calibration form.
  - h. Rinse Sonde in tap water.
  - i. Remove the nitrate sensor from the Sonde, and place in its storage bottle with a small amount of tap water or calibration standard. Make sure that the sensor is not immersed in the storage solution.

## 4.5.2 Collection of Sonde measurements

 Refer to Section A.9 in the QAPP for how to use the Sonde handheld to prepare the Sonde for deployment, as well as for protocols on how to retrieve data from the device. For both depth sampling and profiling, the deployment can be begun before the first point of data collection and finished after the last point of data collection.

### 4.5.3 Shutdown and Storage

- 1. Loosen the battery cap on the EXO2 Sonde. Slide the wrench tool's smaller opening over the battery cap on top of the Sonde. Using the tool as a lever, firmly turn the tool counterclockwise until the battery cap is loose.
- 2. Once the cap is sufficiently loose, remove the cap and old batteries from the well. Always dispose of used alkaline batteries according to local requirements and regulations. Clean the O-ring sealing surfaces of the cap with a KimWipe. Inspect down the battery tube to make sure it is clean and dry. If not, use additional KimWipes to clean/dry the tube.
- 3. Ensure that the O-rings are not nicked or torn and that there are no contaminants or particles on the O-rings or the sealing surfaces inside the battery cover.
  - a. Apply a thin coat of Krytox lubricant to each O-ring and sealing surface.
- 4. Insert the cap into its recess. With your thumb press down on the pressure relief valve while turning the cap clockwise. Once the cap threads are engaged, use the tool to tighten until snug.
- 5. Install a dummy plug on the auxiliary port on top of the Sonde.
- 6. Leave all the probes installed which remain installed at the end of the sampling day. Leave the ammonium or nitrate probe that is not installed in its current storage state.
- 7. Fill the calibration cup with approximately 0.5 inches of water, and install the calibration cup on the Sonde to allow the probes to experience water-saturated air conditions.

## 4.5.4 End of Sampling Season Storage

- 1. Loosen the battery cap on the EXO2 Sonde. Slide the wrench tool's smaller opening over the battery cap on top of the Sonde. Using the tool as a lever, firmly turn the tool counterclockwise until the battery cap is loose.
- 2. Once the cap is sufficiently loose, remove the cap and old batteries from the well. Always dispose of used alkaline batteries according to local requirements and regulations. Clean the O-ring sealing surfaces of the cap with a KimWipe. Inspect down the battery tube to make sure it is clean and dry. If not, use additional KimWipes to clean/dry the tube.
- 3. Ensure that the O-rings are not nicked or torn and that there are no contaminants or particles on the O-rings or the sealing surfaces inside the battery cover.
  - a. Apply a thin coat of Krytox lubricant to each O-ring and sealing surface.
- 4. Insert the cap into its recess. With your thumb press down on the pressure relief valve while turning the cap clockwise. Once the cap threads are engaged, use the tool to tighten until snug.
- 5. Install a dummy plug on the auxiliary port on top of the Sonde.
- 6. Leave the turbidity, total algae, conductivity/temperature, and dissolved oxygen probes attached to the Sonde. Install dummy plugs where the pH/ORP and ammonium/nitrate sensors were. Fill the calibration cup so that all the probes will be submerged in water, and install on the Sonde.

- 7. Remove the pH/ORP sensors from the Sonde and insert its sensing end into the bottle that the sensor was shipped in. Install the bottle's O-ring and cap then tighten. This bottle should contain 2 molar solution of pH 4 buffer. If this solution is unavailable, the bottle can be filled with tap water.
- 8. Remove either the ammonium or nitrate sensor from the Sonde (whichever is installed), and insert its sensing end into the bottle that the sensor was shipped in. Install the bottle's O-ring and then tighten. This bottle should contain either a small amount of DI or tap water or calibration standard. Either of these sensors should not be fully immersed. However, if the vessel becomes dry, the sensor must be soaked overnight in the high-calibration standard.
- 9. Pack the Sonde and accessories into the carrying case.

# 5.0 Health and Safety

During sampling, field personnel will wear all necessary personal protective equipment (PPE) and follow hygiene practices as outlined here.

As outlined by the Center for Disease Control, field personnel will wear the following PPE when handling human waste or sewage:

- Goggles
- Protective face mask or splash-proof face shield (suggested)
- Liquid-repellent coveralls or suit
- Rubber boots or dedicated boots
- Life jackets
- Nitrile gloves

All field personnel will have a Hepatitis A vaccination before commencing field work.

Hygiene practices are very important to ensure health and safety of workers. Following the Center for Disease Control directions, the following hygiene practices should be followed:

- Wash hands with soap and water immediately after handling human waste or sewage
- Avoid touching face, mouth, eyes, nose, or open sores and cuts while handling human waste or sewage
- After handling human waste or sewage, wash your hands with soap and water *before* eating or drinking
- After handling human waste or sewage, wash your hands with soap and water *before* and *after* using the toilet
- Before eating, remove soiled work clothes and eat in designated areas away from human waste and sewage-handling activities
- Do not smoke or chew tobacco or gum while handling human waste or sewage
- Keep open sores, cuts, and wounds covered with clean, dry bandages
- Gently flush eyes with safe water if human waste or sewage contacts eyes
- Use waterproof gloves to prevent cuts and contact with human waste or sewage
- Wear rubber boots at the worksite and during transport of human waste or sewage
- Remove rubber boots and work clothes before leaving worksite
- Clean contaminated work clothing daily with 0.05% chlorine solution (1 part household bleach to 100 parts water)

# 6.0 References

https://www.cdc.gov/healthywater/global/sanitation/workers\_handlingwaste.html