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Revision Page

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4/23/02	0	Initial Approval	
5/31/02	1	Changed DO requirement to < 1.0mg/l	5.5.1.8

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1.0 Scope and Application

The purpose of this standard operating procedure (SOP) is provide a framework for calibrating sondes used to measure water quality parameters for ground water and surface water. Water quality parameters include temperature, pH, dissolved oxygen, conductivity/specific conductance, turbidity, and chlorophyll.

This SOP is written specifically for the YSI model 6-Series Sondes (which include the 600R, 600XL, 600XLM, 6820, 6920 and 6600 models), and the YSI 650 MDS (Multi parameter Display System) display/logger. The general calibration processes discussed herein are applicable to other manufactures sondes and displays/loggers. Consult the manufacture's instruction manuals for specific procedures.

2.0 Summary of Methods

This document describes a process for calibrating and performing water quality field measurements using YSI 6-Series Sondes.

3.0 Health and Safety Warnings

- 3.1 All proper personal protection clothing and equipment is to be worn.
- 3.2 The standard solutions for calibrating conductivity contain Iodine and Potassium Chloride. When using the standards, avoid inhalation, skin contact, eye contact or ingestion. If skin contact occurs remove contaminated clothing immediately. Wash the affected areas thoroughly with large amounts of water. If inhalation, eye contact or ingestion occurs, consult the Material Data Safety Sheets (MSDS) for prompt action, and in all cases seek medical attention immediately.
- 3.3 The standard solutions for calibrating turbidity contain Styrene divinylbenzene copolymer spheres. While the material is not volatile and has no known physical effects on skin, eyes, or on ingestion, general health and safety precautions should be adopted to minimize unnecessary contact. If skin contact occurs remove contaminated clothing immediately. Wash the affected areas thoroughly with large amounts of water. If inhalation, eye contact or ingestion occurs, consult the Material Data Safety Sheets (MSDS) for prompt action, and in all cases seek medical attention immediately.
- 3.4 All standard solutions for calibration pH contain the following compounds:
- | | |
|------------------|---------------------------------------------------------------------------------------------------------------------|
| pH 4 Solutions: | Potassium Hydrogen Phthalate, Formaldehyde, Water |
| pH 7 Solutions: | Sodium Phosphate (dibasic), Potassium Phosphate (Monobasic), Water |
| pH 10 Solutions: | Potassium Borate (Tetra), Potassium Carbonate, Potassium Hydroxide, Sodium (di) Ethylenediamine Tetraacetate, Water |

Avoid inhalation, skin contact, eye contact or ingestion. If skin contact occurs remove

contaminated clothing immediately. Wash the affected areas thoroughly with large amounts of water. If inhalation, eye contact or ingestion occurs, consult the Material Data Safety Sheets (MSDS) for prompt action, and in all cases seek medical attention immediately

3.5 Follow the Boat Safety SOP when conducting sampling from a boat.

4.0 Equipment and Supplies

4.1 Thermometer (with NIST trace)

4.2 pH Standards of 4, 7, and 10

4.3 Conductivity standards (concentration dependent upon expected field conditions)

4.4 Turbidity standards (concentration dependent upon expected field conditions)

4.5 Deionized water

4.6 YSI Sonde with attached pH, Conductivity, Dissolved Oxygen, and Turbidity probes

4.7 YSI 650 MDS Multiparameter Display System (display logger)

4.8 Sonde communications cable

4.9 Notebook

4.10 Pen

5.0 Calibration

All instrument probes must be calibrated before they are used to measure environmental samples. Before performing any calibration procedure the sonde and display/logger must stabilize (warm-up) at least 15 minutes. During the warm-up period check the display/logger to determine the battery level in the display/logger to see if recharging is necessary.

Prior to calibration, all instrument probes on the sonde must be cleaned according to the manufacture's instructions. Failure to perform this step can lead to erratic measurements. The probes must also be cleaned by rinsing with deionized water before and after immersing the probe a calibration solution. For each of the calibration solutions provide enough volume so that the probe and the temperature sensor are sufficiently covered (see the manufacture's instructions for required volumes of calibration solutions).

5.1 Temperature

For instrument probes that rely on the temperature sensor (pH, dissolved oxygen/specific

conductance, and oxidation-reduction potential), the sonde temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). This accuracy check should be performed at least once a year, and the date and results of the check kept with the instrument. Below is the verification procedure.

- 5.1.1 Allow a container filled with water and the sonde to come to room temperature.
- 5.1.2 Place a thermometer that is traceable to the NIST into the water and wait for both temperature readings to stabilize.
- 5.1.3 Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer within the accuracy of the sensor ($\pm 0.15^{\circ}\text{C}$). If the measurements do not agree, the instrument may not be working correctly and the manufacturer should be contacted.

5.2 pH

The pH of a sample is determined electrometrically using a glass electrode. Choose the appropriate standards that will bracket the expected values at the sampling locations. For this procedure three standards will be used (pH 4, pH7, & pH10).

- 5.2.1 Allow the buffered samples to equilibrate to the ambient temperature.
- 5.2.2 Clean all of the probes on the sonde with deionized water. Shake off excess water.
- 5.2.3 Place the probes on the sonde into the pH 7 buffer.
- 5.2.4 On the display/logger use the up/down arrow keys to highlight the "Calibrate" option and press the enter key.
- 5.2.5 Highlight the "pH" option and press enter.
- 5.2.6 Highlight the "3-point" option and press enter.
- 5.2.7 Input the value of the buffer, which is 7.00 and press enter.
- 5.2.8 Wait for the value of pH to stabilize and then press enter. Wait for "Calibrated" message. If an "Out of Range" message appears, do not accept, check the probe and refer to operators manual.
- 5.2.9 Rinse probe with Deionized water and shake off excess water.
- 5.2.10 Place the pH probe into a pH buffer of 4.00.
- 5.2.11 Press enter key to continue calibration

- 5.2.12 When prompted, enter the pH of the second buffer, "4.00". Wait for "Calibrated" message, and press any key to continue.
- 5.2.13 Rinse probe with Deionized water and shake off excess water
- 5.2.14 Place the pH probe into a pH buffer of 10.00.
- 5.2.15 Press any key to continue calibration
- 5.2.16 When prompted, enter the pH of the third buffer, "10.00". Wait for "Calibrated" message, and press any key to continue.
- 5.2.17 Rinse probe with Deionized water and shake off excess water.
- 5.2.18 Insert probe into pH 7 buffer and make sure it is reading correctly (± 0.05). If buffer reading is not correct, repeat the calibration procedure.

5.3 Specific Conductance

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected at 25°C.

- 5.3.1 Place the cleaned probes into the specific conductivity standard solution, making sure that the specific conductivity probe is fully submerged.
- 5.3.2 For field calibration go to 5.3.3, for a more accurate laboratory calibration continue with the procedure below. For calibration in the laboratory place the display/logger in "Sonde Run" mode, and check the temperature of the standard solution. For calibration of specific conductivity the standard must be at 25°C ($\pm 0.5^\circ\text{C}$). If the temperature of the solution is not within this range, adjust the solution temperature by placing the container (with lid firmly tightened), into a bath of warmer or colder water (depending on standard's temperature). Check on the progress of temperature change by placing the instrument probes into the solution. Once the temperature falls within $\pm 0.5^\circ\text{C}$ of 25°C continue the calibration procedure.
- 5.3.3 Return to the display/logger main menu and select "Calibrate" and press enter.
- 5.3.4 Select "Conductivity" and press enter.
- 5.3.5 Select "spCond" and press enter.
- 5.3.6 Enter the standard concentration in mS/cm^3 and press enter. The standard concentration should be close to the concentrations you expect to measure.
- 5.3.7 After the specific conductivity reading has stabilized press enter to calibrate. Wait for the

“Calibrated” message to appear.

5.3.8 Rinse probe with deionized water and off excess water.

5.3.9 Insert probe back into the standard concentration and make sure it is reading within 10%.

5.4 Turbidity

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference solution. Critical to the instrument's operation is that the lens covering the detection unit is kept clean both during calibration and field use. The turbidity probes used on the YSI 6-Series sondes include an automated optics wiper. This wiper can be activated using the display/logger. A 2-point calibration procedure is recommended.

The manufacturer recommends that the YSI 6-Series Turbidity probe be calibrated using the calibration cup provided with the sonde. This method is preferred however, one major drawback to this is that the standard solutions must be discarded after calibration due to possible contamination. An alternative is to place the standard solutions in secondary containers with openings large enough to allow the turbidity probe to be placed into the standard. These containers should have similar physical properties as the calibration cup (ie. clear to opaque, plastic). The sides of the container should not have any material such as tape or writing on them.

5.4.1 Allow the standard samples to equilibrate to the ambient temperature.

5.4.2 Clean all of the probes on the sonde with deionized water. Shake off excess water.

5.4.3 Place the probes on the sonde into the 0.0 NTU standard (which can be deionized water)

5.4.4 From the “Calibrate” Menu, on the display/logger, select the “Turbidity” option and press enter.

5.4.5 Select the “2-point” option and press enter.

5.4.6 Enter “0.0” as the first calibration standard and press enter.

5.4.7 Select the “clean optics” option to activate the automated wipers. Once the cleaning process is completed, wait for the turbidity measurement to equilibrate, and then press the enter key.

5.4.8 Place the probe in the 10 NTU standard. Do not clean the probe before placing into the second standard.

5.4.9 Press enter to continue calibration.

- 5.4.10 Enter "10.0" as the second calibration standard and press enter.
- 5.4.11 Again, select the "clean optics" option to activate the automated wipers. Once the cleaning process is completed, wait for the turbidity measurement to equilibrate, and then press the enter key.
- 5.4.12 Clean all of the probes on the sonde with deionized water. Shake off excess water.
- 5.4.13 Insert probes back into the 10.0 NTU standard and make sure it is reading between 9.5 and 10.5 NTU. If the buffer reading is not correct, repeat the calibration procedure.

5.5 Dissolved Oxygen

Dissolved oxygen (DO) content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according to manufacturer suggestions. (After changing the membrane you should wait 12 hours before use to allow the membrane to equilibrate) YSI 6-Series DO probe be must calibrated using the calibration cup provided with the sonde.

Calibration of the DO probe requires inputting the current barometric pressure. The YSI 650 display/logger has a barometer within the unit and automatically provides this during the calibration procedure. Other display/loggers do not supply the barometric pressure, and this must be obtained from other sources. Do not use barometric pressure obtained from meteorology reports as these are usually corrected to sea level.

Two calibration procedures are listed below for dissolved oxygen, one for sampling applications and one for long-term monitoring applications.

5.5.1 Calibration Procedure for Sampling (non deployment) Applications

The dissolved oxygen probe should be calibrated in the field prior to use. An initial inspection and calibration should be performed the day before to assure the membrane is in good shape the instrument is working properly. Follow the procedure below to calibrate.

5.5.1.1 Clean all of the probes on the sonde with tap(or clean ambient water) water. Shake off excess water.

5.5.1.2 Place approximately 1/8 inch of water in the bottom of the calibration cup. Place the probe end of the sonde into the cup. Engage only 1 or 2 threads of the calibration cup to insure the DO probe is vented to the atmosphere. Make sure that the DO and temperature probes are NOT immersed in water and that the Sonde cup is not in direct sunlight. Wait approximately 10 minutes for the air in the

calibration cup to become water saturated and for the temperature to equilibrate.

5.5.1.3 For sampling applications the dissolved oxygen probe is continuously pulsing, therefore the "Autosleep RS232" function should be deactivated. From the "Main" menu on the display/logger, select the "System Setup" option and press enter. Then select the "Advanced" option and press enter. Select the "Autosleep RS232" option and press enter to obtain the "off" setting. Then press the "ESC" button until returning to the main menu.

5.5.1.4 From the calibration menu select the "Dissolved Oxy" option, then the DO% option (Note: For the YSI 6-Series Sondes, calibration of dissolved oxygen by the DO% procedure also results in the calibration of the DO mg/l mode and vice versa.)

5.5.1.5 Enter the current barometric pressure in mm of Hg. The correct pressure will often be provided but double check with the reading provided in the lower right hand corner of the display.

5.5.1.6 Press enter and then wait for the DO% reading to equilibrate. Press enter to accept the calibration. Press enter again to return to the calibration menu.

5.5.1.7 Immediately enter the "Sonde Run" mode and record the temperature, dissolved oxygen in mg/l and %, and the barometric pressure used for calibrating.

5.5.1.8 For some applications it may be necessary to verify the probe with a zero DO solution. If so continue with the following.

- 1) Place the probe in a zero DO solution.
- 2) Verify the probe reads < 1.0mg/l.
- 3) Rinse probe and store the probe in tap water.

5.5.1.9 Fill the calibration cup half way with tap water and screw on to the sonde. The sonde is now ready for use.

5.5.2 Calibration Procedure for Continuous Monitoring (deployment) Applications

When the instrument will be used for longer term monitoring applications, the "Autosleep RS232" function must be activated before calibration. After making sure this function is on, follow steps 5.5.1.1-5.5.1.9 (skipping 5.6.1.3) in "Calibration Procedure for Sampling Applications".

6.0 Troubleshooting

6.1 Occasionally problems are encountered during a calibration and the instrument must be uncalibrated to return the instrument to factory settings. Uncalibration can be performed following these steps

- 6.1.1 Access the desired parameter to uncalibrate in the calibrate menu.
- 6.1.2 When prompted to input a number for a standard, hold the enter key down and press the escape key. Highlight the “yes” key and press enter.
(Please note: This procedure is the equivalent of entering the command “uncal” from the YSI 610 logger at the numeric calibration prompt.)

- 6.2 For additional troubleshooting refer to the operations manual or call YSI technical support at 1-800-897-4151 and ask for technical support.

7.0 Post Sampling Check and Data Evaluation

During use of the sondes in the field, the instrument probes experience “drift” and may operate outside of their expected range. To determine the amount of drift the probes must be checked against their calibration standards.

7.1 Instrument Dissolved Oxygen Performance Check

The dissolved oxygen should be checked in the field at least at every 5 stations or every couple of hours and after the last sampling point.

- 7.1.1 Clean all of the probes on the sonde with tap(or clean ambient water) water. Shake off excess water.
- 7.1.2 Place approximately 1/8 inch of water in the bottom of the calibration cup. Place the probe end of the sonde into the cup. Engage only 1 or 2 threads of the calibration cup to insure the DO probe is vented to the atmosphere. Make sure that the DO and temperature probes are NOT immersed in water and that the Sonde cup is not in direct sunlight. Wait approximately 10 minutes for the air in the calibration cup to become water saturated and for the temperature to equilibrate.
- 7.1.3 Record the temperature, dissolved oxygen in mg/l and %, and the barometric pressure. The dissolve oxygen value should be within 0.5 mg/l or of the saturation value (which is based on barometric pressure and temperature). If the data does not meet this requirement it should be deleted or reported as estimated data.

7.2 Instrument pH, Conductivity, Turbidity Performance Check

- 7.2.1 Allow the standards equilibrate to the ambient temperature.
- 7.2.2 Clean all of the probes on the sonde with deionized water. Shake off excess water.
- 7.2.3 Place sonde instrument probes in reference standard solution of the particular parameter (eg. pH, specific conductivity, etc.).

- 7.2.4 From display/logger main menu, select the “Run” option and press enter. Allow measurements to equilibrate, then write down result of measurement in log book.
- 7.2.5 Before and after placing the probes in the standard clean all of the probes on the sonde with deionized water and shake off excess water. Repeat for each reference standard solution.
- 7.2.6 These results should be compared with the below quality control goals. Data not meeting this criteria should be deleted or reported as estimated.

Table 7.1 Quality Control Goals for Sondes

PARAMETER	Post Calibration check accuracy goals
pH	+0.3 with pH 7 buffer and other bracketing buffer (pH4 or pH10)
Conductivity	± 10% of standard
Dissolved Oxygen	± 0.5 mg/l of sat. value
Turbidity	+/- 2 NTUs

7.3 Adjacent Measurement Check (for deployed sondes)

- 7.3.1 When sondes are deployed adjacent measurement checks shall be performed with a second instrument. The number of adjacent measurement check will depend on the quality of the monitored water and the project objectives. At a minimum these shall be performed during sonde retrieval. Adjacent measurements shall be measured at the depth of the sonde and at the surface. (Surface reading are taken to assess stratification that may exist).
- 7.3.2. At the same depth, the difference between the adjacent measurement and the recorded values by the sonde should not be greater than the below quality control goals. Data not meeting this criteria should be deleted or reported as estimated.

Table 7.2 Quality Control Goals Between the Adjacent Measurements and Deployed Sonde

PARAMETER	Adjacent Measurements accuracy goals
Temperature	0.5 °C
pH	+0.5
Conductivity	± 0.15 %
Dissolved Oxygen	+ 0.7 mg/l
Turbidity	+/- 2 NTUs

8.0 Measurements

Sondes can be used for either discrete sample measurements or be deployed for a period of time to record measurements. Each of these types of measurements require different configurations of the sonde memory and display logger. Each procedure for configuration and operation of the sondes is discussed below. The procedures described below involving using the sonde memory to log data. Display/loggers can be used to store data, however this requires the display/logger to remain with the sonde during monitoring.

8.1 Discrete sample measurements

8.1.1 From the main menu select the “Sonde Run” option and press enter.

8.1.2 Select the “Run/Discrete Sample” option and press enter.

8.1.3 Place the Sonde into the water to be analysed, and watch the variations in temperature, D.O., pH and conductivity.

8.1.4 When the variations are less than:
0.1°C temperature
0.02su pH
0.02mg/l D.O.
5 uS/cm conductivity
0.5 NTU

Log the measurements in the project’s log book.

8.1.5 If the measurement is to be logged in the sonde memory, the select the “Log one sample” option from the Sonde Menu, and press enter.

8.1.6 If a series of measurements from one site is to be logged in the sonde memory, select the “Start Logging” option from the Sonde Menu and press enter. After a pre-determined amount of time select the “Stop Logging” option to stop logging measurements.

8.2 Deploying Sonde for Unattended Logging

While similar to discrete sampling in operation of the sonde, unattended logging requires setting up the memory of the sonde to record data.

8.2.1 From the Sonde menu select the “Run/Unattended sample” option and press enter.

8.2.2 Follow the prompts on the screen to prepare the sonde for unattended sampling including:
- sample interval time
- logging start date

- logging start time
- logging duration (days)
- file name to store data (no more than 8 characters)
- Site name (associated with file name but not critical)
- battery life (check to make sure it will cover length of time sampling)
- memory space
- View parameters to log

8.2.3 Once these items have been reviewed and are correct, toggle down to “Start Logging”.

8.2.4 The sonde will now begin logging parameters at the next sample interval. If not already attached, place the probe protector on the sonde. Turn the display/logger off and disconnect the communications cable sonde. Place the communications port plug on the sonde. Place the sonde in the desired sample location and securely anchor sonde using the bail provided on top of the sonde.

8.2.5 The sonde is now in place and will continue to record until reaching the specified end time of logging.

9.0 Data Management and Records Management

All results of calibration must be documented and kept in a project’s log book. At a minimum the following should be kept as part of the documentation: the instrument’s manufacture, model number, instrument identification number, standards used to calibrate the instruments, calibration date and the instrument readings.

10.0 References

Wagner J.W., and others, 2000, Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Site Selection, Field Operation, Calibration, Record Computation, and Reporting U.S. Geological Survey Water-Resources Investigation Report 00-4252

YSI, 2001, 650 MDS Operations Manual

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