STANDARD OPERATING PROCEDURE (SOP)
Title: Analysis of *In Situ* Turbidity in Ambient Surface Water at Pine Island Bayou for the Environmental Monitoring Response System Using Yellow Springs Instrument EXO Multi-probes
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1.0 PURPOSE

This document describes sampling, analytical, and quality control (QC) procedures for *in situ* continuous monitoring of turbidity for the Lower Neches Valley Authority (LNVA) Pine Island Bayou Environmental Monitoring and Response System (EMRS) using Yellow Springs Instruments (YSI) EXO multi-probes.

2.0 SCOPE AND APPLICABILITY

2.1 This procedure is intended for use in the Texas Commission on Environmental Quality (TCEQ) Continuous Water Quality Monitoring Network (CWQMN).

2.2 The LNVA operates Continuous Ambient Monitoring Station 749 at Pine Island Bayou and uses turbidity data on a near real-time basis for water management decisions. Turbidity measurements from the station provide LNVA general water quality information. No EMRS notification levels have been set for this station.

2.3 This SOP is specific to turbidity. Procedures within this SOP are generally limited to calibration and QC. Turbidity sensor QC check criteria are used to ensure measurement equipment is operating within limits. See TCEQ's Main YSI EXO SOP for other multi-probe procedures and turbidity calibration and calibration verification worksheets.

2.4 Leading Environmental Analysis and Display System (LEADS) data records for turbidity are not validated. Multi-probe turbidity sensor fouling measurements are not required, however the LNVA may collect fouling measurements for informational purposes.

2.5 LEADS reports multi-probe EXO turbidity measurements units in Nephelometric Turbidity Units (NTU). The American Society for Testing and Materials (D7315 method) designates measurement units for the EXO turbidity sensor to be reported as Formazin Nephelometric Units (FNU).

2.6 The working range of the EXO turbidity sensor is listed in Table 1.

Table 1

Parameter	Working Range
Turbidity	0 to 100 NTU/FNU

3.0 METHOD SUMMARY

3.1 Multi-probes are deployed via deployment structures. Discrete *in situ* Turbidity measurements are collected every 15-minutes by a data logger and data are transmitted every 15-minutes to TCEQ's LEADS in Austin Texas.

3.2 Turbidity is the measurement of the content of suspended solids in water. The EXO optical turbidity sensor measures turbidity using a near-infrared light source and detects scattering of particles at 90 degrees of the incident light. The wavelength of the light emitted is 860 ± 15 nm. EXO turbidity sensors use an internal thermistor for temperature compensation.

3.3 EXO multi-probe sensors have three different averaging and filtering capabilities used for monitoring. For continuous water quality monitoring applications, the Factory **Default** for data averaging is used. The Factory Default allows for a rolling 40 seconds of averaging and filtering.

3.4 *In-situ* optical turbidity sensors are highly susceptible to various forms of fouling. To date, YSI has three types of EXO multi-probes, EXO1, EXO2, and EXO3. Only EXO2 and EXO3 multi-probes can be equipped with a central wiper to brush the turbidity sensor.

4.0 LIMITATIONS

4.1 Multi-probe deployment structures can be susceptible to damage due to high water events. Data loss can occur because of damage to deployment structures. Replacement can be expensive and require significant resources.

4.2 Over deployment periods, the interface between measurement sensors and the environment can become fouled by a variety of organisms, sedimentation, and hard scale fouling. Sensor fouling can compromise data quality. In some water bodies (or due to seasonal or event driven changes in water quality), sensor fouling can occur rapidly, decreasing deployment periods. EXO multi-probes can be equipped with various anti-fouling measures to increase deployment periods.

5.0 SAFETY

This procedure includes processes that can be hazardous. Therefore, before attempting this process, operators should review the *TCEQ Chemical Hygiene Plan* for proper equipment and procedures necessary for the safe completion of this procedure. Operators must also read and be familiar with the Material Safety Data Sheets for the various chemicals that can be used in this procedure. Lab coats, safety glasses with side shields

and/or splash goggles and chemical resistant gloves should be worn when handling chemicals.

6.0 EQUIPMENT AND REAGENTS

6.1 Equipment

- EXO2 or EXO3 Multi-probes
- EXO Handheld (v2) or Electronic Device with KorEXO PC software
- EXO Calibration cable
- EXO2 and EXO3 Central Wiper Assembly
- EXO2 and EXO3 Replaceable central wiper brushes
- EXO Calibration cup
- EXO plastic or brass anti-fouling sensor guards
- Calibration Worksheets
- Instrument logbook
- C-spray anti-fouling nanopolymer (optional)
- Ring stand and clamp
- PC with internet access
- Multi-probe deployment structure
- Class A 1,000 ml volumetric flask
- Class A 25 ml pipette

6.2 Cleaning equipment/supplies for measuring sensor fouling

- All-purpose cleaner in spray bottle
- Water and white vinegar solution (hard fouling)
- Five-gallon bucket
- Kimwipes
- Large soft-bristle brush
- Small soft-bristle brush
- Cotton swabs
- Paper towels

6.3 Standards and Reagents (All reagents/chemicals must be analytical reagent grade).

- Hach Formazin Turbidity Stock Standard 4000 NTU (see section 11.1 for disposal).
- De-ionized (DI) water
- Reagent Grade Water (E-pure)

7.0 PROCEDURE

Before the multi-probe is deployed, the sensors are cleaned and calibrated. At the end of the deployment period, multiprobe sensor analytical drift is measured.

7.1 Service intervals, Turbidity Sensor calibration, and QC.

7.1.1 The station is serviced, and sensor is calibrated at a minimum of once every month. More frequent service visits may be needed depending on sensor fouling rates for a given water body, time of year, and employed anti-fouling measures. Station operators will need to determine appropriate deployment periods for their water bodies.

7.1.2 During routine station visits, the station operator brings a newly calibrated multi-probe to the station and exchanges the currently deployed multi-probe with the newly calibrated multi-probe. The multi-probe is taken back to a temperature-controlled environment where Calibration Verification Samples are (CVS) analyzed to determine the amount of analytical drift that occurred over the deployment period.

7.2 Turbidity Sensor Calibration

Sensor calibrations are performed immediately prior (within a day) to each deployment period. A two-point calibration at 0 and 100 NTU is used and generally covers most water quality conditions. A Hach Formazin 4000 NTU stock standard is diluted to 100 NTU. Calibration information is recorded on the EXO Multi-probe Calibration Worksheet (see Appendix A).

General Calibration Procedures

- Multi-probe sensor calibration commands for this SOP are described using the EXO Handheld v2. The EXO Handheld v2 or PC KorEXO PC software may be used to calibrate multi-probe sensors.
- Sensor must be cleaned before calibration or analyzing CVSs.
- The multi-probe must equilibrate at room temperature for at least two hours (as close to 25 °C as possible) prior to calibrating or analyzing CVSs.
- All multi-probe components that come in contact with standards must be clean. Solid particulates will contaminate standards and cause calibration errors.
- The EXO's sensor guard is used to attach the calibration cup to the multi-probe. The calibration cup fits over the sensor guard and the guard comes in direct contact with the Formazin standards. Ideally a station will have three sensor guards. The extra sensor guard is kept in the laboratory and is only used for calibrations. If an extra sensor guard is not available, sensor guards must be thoroughly cleaned before they are used in multi-probe calibrations.
- New or cleaned EXO Central Wiper brushes must be installed prior to each

calibration/deployment.

- Anti-fouling C-spray nanopolymer can be applied to the turbidity sensor bodies prior to calibration. Applying C-spray is optional. **Do not apply C-spray to optical sensor interfaces.**
- Care must be taken to minimize any air bubbles with all turbidity calibration standards.
- If air bubbles are noticed on the probe face or the initial readings do not look accurate, manually activate the turbidity wiper 1-2 times to clear any trapped air.
- A ring stand, and clamp can be used to secure the multi-probe body.
- The turbidity sensor must be rinsed twice with DI water before and between the introduction of turbidity standards.
- Initial turbidity calibration value must be 0 NTU.
- Operators should have several clean absorbent paper towels available. Shake the excess rinse water off the sensor. This will reduce carry-over contamination of calibration solutions.
- A small amount of calibration solution should be used to pre-rinse the sensors. Expired calibration solutions may be used for this.

Formazin Standard Preparation

- Stock Turbidity standards must be within expiration date.
- Prepare 100 NTU standard by partially filling a 1000 mL volumetric flask with reagent grade water. Pipette 25 mL of the 4000 NTU Hach Formazin standard to the volumetric flask and fill with reagent grade water to volume. Gently swirl flask while filling.
- Hach reports the following about the stability of Formazin standards.
  - Standards greater than 400 NTU are stable for more than a year.
  - Standards between 20 – 400 NTU are stable for approximately one month.
  - Standards should be stored in cool dark place or refrigerator. Keep standards sealed in containers.
- Calibration standards must be allowed time to equilibrate to room temperature before calibration or initial readings

Turbidity Calibration Acceptance Criteria

The EXO has color codes for accepting or rejecting calibrations (Green, Yellow, and Red). After calibration, color codes can be obtained by pushing the **Calibrate button** and selecting Smart QC. Only calibrations with Green or Yellow QC are accepted. If a red QC score is indicated, the calibration is rejected, and corrective action is performed before continuing.

Indicates successful calibration.
Indicates a poor or unsuccessful Calibration. Numeric criteria have

been exceeded. A calibration error message will also occur.

⚠ Indicates a successful calibration that is nearing the edge of acceptable limits.

7.2.1 C-spray Anti-Fouling Sensor Application (optional)

Prior to sensor calibration C-spray nanopolymer can be applied to the sensor body. **DO NOT apply C-spray to optical sensor interfaces.** Ensure Sensor body and sensor interface is clean and dry. Apply C-spray to sensor and allow to air dry. Apply a second coat of C-spray and allow to air dry. Proceed with calibration.

7.2.2 Two-Point Turbidity Calibration

Calibration of the turbidity sensor consists of a two-point calibration with DI water and a 100 NTU turbidity standard.

7.2.2.1 Rinse a clean calibration cup, sensor guard and sensors twice with DI water.

7.2.2.2 Pour enough DI water (0 NTU) standard into calibration cup to immerse sensors. Immerse the probe end of the multi-probe into the water.

7.2.2.3 In the calibrate menu, select turbidity, then select Turbidity FNU.

7.2.2.4 From the **Dashboard** screen. Push the **Calibration Button** on the v2 Handheld, then select **Turbidity** from the list of parameters. In the next screen select **Calibrate**. In the Calibrate turbidity screen screen select **Calibration Value** [0] and enter the value of the turbidity standard in and select **enter**.

7.2.2.5 Select the wipe sensor menu option to activate the wiper to remove bubbles. Allow the sensor to stabilize for three minutes and click Accept calibration from the menu option.

7.2.2.6 Rinse calibration cup, sensor guard and sensors twice with 100 NTU Formazin standard.

7.2.2.7 Pour enough Formazin (100 NTU) standard into calibration cup to immerse sensors. Immerse the probe end of the multi-probe into the standard. In the Calibrate turbidity screen select **Calibration Value** [100] and enter the value of the turbidity standard in and select **enter**

7.2.2.8 Observe reading under Current and Pending Data Point. Select the

wipe sensor menu option to activate the wiper to remove bubbles. Allow the sensor to stabilize for three minutes and click apply to accept calibration.

7. 2.2.9 Select Finish Calibration. If the calibration was successful a "Calibration successful" will be displayed in the message area. If a calibration is unsuccessful a calibration error measles will be displayed. Rinse sensors with tap water.

7.2.2.10 A *View Cal Records* screen will appear. Record the following information in the EXO Multi-probe Calibration Worksheet (Appendix B). This worksheet is found in the main EXO SOP.

- Cal Value
- Pre-Cal Value
- Temperature

Push the **Esc** button to return to the **Dashboard** screen and record the turbidity post calibration value in the Calibration Worksheet's (Appendix B).

7.2.2.11 Obtain the QC color score for the calibration by pushing the **Calibration Button**. Then select **Smart QC** and record the QC color score into the calibration worksheet's (Appendix B).

8.0 Turbidity Probe Cleaning and Storage

Note: Section 8.1.1 details a nominal cleaning procedure. Station-specific sensor cleaning procedures may need to be developed when sensors are fouled by chemical coatings.

8.1 The deployment multi-probe is gently removed from the deployment tube. After removal, the multi-probe is cleaned. Field cleaning includes the through cleaning of the sensor interface, sensor body, and multi-probe body using cleaning supplies listed in Section 6.3

8.1.1 Spray multi-probe and turbidity sensor body with an all-purpose cleaning solution.

8.1.2 Clean sensor body with a soft cloth or brush. Clean the optical surface with moist lens cleaning paper.

8.1.3 Thoroughly rinse the multi-probe, sensor body, and sensor interface. Inspect multi-probe (especially sensory interface) and repeat steps 8.1.1, 8.1.2, and 8.1.3 if necessary.

8.2 No special precautions are necessary for either the short or long-term storage of turbidity probes. For long-term storage, the user may wish to remove the probe from the multi-probe, install a port plug, and store the probe in dry air to minimize any cosmetic degradation and to maximize the life of the wiper.

8.3 QC Calculations

Measurement Accuracy

8.3.1 Measurement accuracy is expressed in terms of the Absolute Error (AE) or Relative Percent Error (RPE) and is defined as:

$$AE = MeasuredValue - ActualValue$$

$$RPE = \frac{Measured\ value - Actual\ Value}{Actual\ Value} * 100$$

Sensor Calibration Drift Calculations

8.3.2 Multi-probe turbidity sensor calibration drift (C_d) is evaluated using RPE:

$$C_d = \frac{(S_r - S_v)}{S_v}(100)$$

Where:
S_r = Sensor response; and
S_v = Turbidity NTU theoretical value.

8.3.3 Turbidity C_d is evaluated using AE:

$$C_d = (S_r - S_v)$$

Where:
S_r = Sensor response; and
S_v = Turbidity NTU theoretical value.

9.0 QUALITY CONTROL

9.1 QC Samples

QC samples are used to ensure that acceptable data quality is maintained throughout the measurement process.

9.2 Turbidity Sensor Drift Measurement Procedures

Sensor CVSs are analyzed to assess analytical drift from the previous calibration. Analyze CVSs as close to 25.0 °C as possible. Allow the multi-probe to equilibrate to room temperature for a minimum of two hours before analyzing CVSs. The Formazin 100 NTU CVS should be the same standard used to generate the initial calibration.

Turbidity Sensor CVS

9.2.1 DI (0 NTU) water and the 100 NTU Formazin standards are introduced using the calibration cup. **Note:** Rinse calibration cup and sensor twice with DI water before measuring DI (0 NTU) water standard. Rinse calibration cup and sensors twice with 100 NTU Formazin standard before measuring standard. Allow at least 40 seconds in time to elapse before recording measurements.

Results are entered into the Multi-probe Calibration Verification Worksheet (Appendix B). This worksheet is found in the main EXO SOP. Results may also be entered into the Post Deployment Excel Worksheet (PDW).

Table 9-1: QC Checks

QC Check	Purpose	Frequency	Acceptance Criteria	Response Action
Two-Point Formazin Calibration	To establish slope used for quantitation	Prior to deployments	Green or Yellow QC score	1) Analyze standard again and re-calibrate 2) Check for contaminated standard 3) Clean sensor 4) Replace sensor
DI water (0 NTU) standard (CVS)	To Assess Sensor Drift	At the conclusion of every deployment period. A minimum of once monthly	± 3 NTU/FNU AE	1) Perform corrective action as necessary 2) Replace sensor
Formazin Calibration Verification Samples (CVS)	To Assess Sensor Drift	At the conclusion of every deployment period. A minimum of once monthly	± 5% RPE	
Turbidity Sensor Cleaning	Improve Sensor Responses	As needed and at the conclusion of deployment periods	None	Clean sensors more frequently

9.0 DEFINITIONS

See Appendix A of the *Continuous Water Quality Monitoring Network Quality Assurance Project Plan.*

10.0 REFERENCES

TCEQ Continuous Water Quality Monitoring Network Quality Assurance Project Plan
EXO User Manual, Rev. H
EXO Handheld Operation Manual E117 Mini-Manual Revision A
TCEQ YSI CWQMN SOP: Analysis of In Situ Dissolved Oxygen, Electrical Conductivity, pH, Water Temperature, and Sample Depth in Ambient Surface Water Using Yellow Springs Instrument 6-Series Multi-probes, Rev. 4
LNVA YSI CWQMN SOP: Analysis of Turbidity in Ambient Surface Water at Pine Island Bayou for the Environmental Monitoring and Response System Using Yellow Springs Instrument 6-Series Multi-probes, Rev. 0
Turbidity Standards: Technical Information Series – Booklet No.12
TCEQ Operating Policies and Procedures, Chapter 6.13

11.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Supervisors, sampling personnel, and laboratory analysts should identify and implement innovative and cost-saving waste reduction procedures as part of the method development, review, and revision of standard operating procedures. Wastes that do result from these

procedures are managed and disposed of in accordance with appropriate state and federal regulations.

Refer to Chapter 6.13 of the *TCEQ Operating Policies and Procedures* for guidelines on general recycling, waste reduction, and water and energy conservation. Review these procedures for specific employee responsibilities and mechanisms for office-related waste prevention and management. Consult the *Monitoring Operations Hazardous Waste Disposal Plan* for laboratory-specific waste minimization recommendations and requirements for proper handling of hazardous waste that result from laboratory procedures.

11.1 Formazin Disposal Considerations

Dilute the material with excess water making a weaker than 5% solution. Open cold water tap completely, slowly pour the material to the drain. Flush system with plenty of water. For container disposal, rinse three times with appropriate solvent and dispose in normal trash.

Note – These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.