STANDARD OPERATING PROCEDURE (SOP) – Water Quality Planning Division

Title: Analysis of Turbidity in Ambient Surface Water Using an Greenspan Aqualab Continuous Auto-Analyzer

Team Leader: ______________________________ Date: ______________

Quality Control Review: ______________________ Date: ______________

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1.0 PURPOSE

This describes the sampling and analytical procedure for continuous automated analysis of turbidity in ambient surface water using an Aqualab continuous auto-analyzer.

2.0 SCOPE AND APPLICABILITY

2.1 This procedure is intended for use in the Continuous Water Quality Monitoring Network.

2.2 Due to the extended length of time continuous analyzers are deployed so that the data can be used to establish baseline conditions, identify trends, characterize pollution events, and seasonal variations in water quality.

2.3 The nominal method detection limit (MDL) for this procedure is approximately 6 nephelometric turbidity units (NTU). The working range of the detector is between approximately 6 NTU and 500 NTU.

3.0 METHOD SUMMARY

3.1 Ambient surface water is pumped from the water body through a sample line to a sample reservoir using a submersible pump. A subsample is then collected from the sample reservoir and analyzed by the Aqualab.

3.2 The Aqualab utilizes the nephelometric method. The method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. A photoelectric detector is used to indicate intensity of light scattered at 90° to the path of incident light.

3.3 Each time the Aqualab performs an ambient turbidity analysis the sample concentration is blank corrected.

4.0 LIMITATIONS

4.3 Various Aqualab components are susceptible to frequent fouling when ambient surface waters contain high levels of sediment, nutrients, turbidity or algae. Frequent cleaning and flushing of the various Aqualab components is necessary. Data invalidation can occur as a result of fouling. Please see the Aqualab Preventive Maintenance Inspection SOP MAIN-022 for cleaning and maintenance procedures.

4.4 Turbidity can be determined for any water sample that is free of debris and rapidly settling course sediment. The presence of air bubbles gives false results.

4.5 Water color due to dissolved substances that absorb light, causes measured turbidities to be low.

4.6 The turbidity standards should be at the same temperature as the Aqualab before performing any calibrations.

5.0 SAFETY

This procedure includes processes that can be hazardous. Therefore, before attempting this process, review the *TCEQ Chemical Hygiene Plan* for proper equipment and procedures necessary for the safe completion of this procedure. Operators must read and be familiar with the Material Safety Data Sheets for boric acid. They do not require special handling. However, safety glasses with side shields and/or splash goggles and chemical resistant gloves should be worn when handling these chemicals. These chemicals have the potential to be skin and eye irritants.

6.0 EQUIPMENT AND REAGENTS

6.1 Equipment

- Aqualab Analyzer
- Calibration forms
- Personal computer with Analyzer32 software

- Vinyl or Polyvinyl chloride (PVC) sample line
- Thermometer capable of 0.1 degrees Celsius (°C) resolution
- Sample reservoir (pot)
- Submersible pump capable of delivering ambient surface water to the sample reservoir. 4 gallons/minute

6.2 Standards and Reagents

- De-ionized (DI) Type 1 water
- Polymer turbidity standards traceable to National Institute of Standards and Technology (NIST) with turbidities of approximately 125, 250, 375, and 500 NTU, 100 milliliter (mL) of each. Formazin standards are not recommended because Formazin is a suspected carcinogen

Wash Solution (Container 50)

- Wash solution (½ M Boric acid and DI Type 1 water)
  ½ M Boric acid- 400ml
  Made up to 20 litres with DI water
  Label bottle with date of manufacture and expiration date
  Reagent life 12 months

7.0 PROCEDURE

7.1 Setting up a Schedule

The Analyzer32 software allows the user to set up schedules and calibrate sensors. Schedules control the operation of the Aqualab chemical laboratory and external sensors by describing the tests to be performed and controlling when the tests are performed. To establish a schedule the operator creates a new schedule or edits an existing schedule, selects the test to be performed and their order, chooses the activation mechanism for the schedule, sets the timer if required, and enables the schedule.

7.1.1 Creating and Editing a Schedule

- From the **Main Menu** press **Setup**. From the **Setup Menu** select **Scheduling.** The **Schedule Setup** window will appear.
- To create a new schedule, press the **New** button. If a schedule name already exist and you want to modify it, select that schedule, then press the **Edit** button. It is possible to create more than one schedule.
- To access the schedule editor, press **New** or **Edit** in the Schedule Setup window
- Enter a name for your schedule in the box located at top left of the window (up to 20 characters).
- The **Test in Schedule** list describes the tests to be performed in the schedule as well as the fill and clean operations. The maximum number of tests permitted in one schedule is 20.
- The time it takes for a schedule to execute is displayed in the top right corner of the window. This time is calculated by the software, based on which tests are selected. This information is useful to help plan the timing of multiple schedules.

7.1.2 Editing the Tests for a Schedule

- From the **Main Menu** press **Setup**. From the **Setup Menu** select

Scheduling. The **Schedule Setup** window will appear. Select the **Edit Test** button

- The tests available are shown in a list on the left hand side of the window. To add a test to the current list of tests shown on the right, highlight the test to add and press the **Add Test** button. The same test can be added more than once to a schedule.
- When the test is added to the list it is also given a channel number. The channel number is the code used for the test in the data log.
- A schedule can be rearranged to execute in any order by using the following buttons.

Push Test Up	Move the position of a highlighted test up.
Push Test Down	Move the position of a highlighted test down the list.
Delete Test	Remove a highlighted test.

- If the Automatic box is ticked fills and cleans will be automatically added. The fill/cleans can be manually deselected by unticking the Automatic box. These procedures bring the sample to the internal inlets of the Aqualab and then clean out the lines afterwards. The fill operations occur at the beginning of a schedule and the clean operations at the end of a schedule.

7.1.3 Timing a Schedule

To set the times when a schedule will execute there are four options:

Periodic: A periodic schedule will execute at fixed time intervals.

- Periodic timing is set by selecting **Periodic** from the **Scheduled Times** box in the schedule edit window.
- The schedule will run first at the **start time**. It will run again depending on the on what time is used for the **trigger period**. If the trigger period is larger than the schedule duration then the schedule will not run at the next trigger time. If the trigger period is less than the schedule duration it will run at the next trigger time when the scheduler is not already running. This schedule will run continuously.

Timetable: A timetable schedule will execute at specified times on a daily basis.

- In the Schedule Editor, choose the timetable option under the heading, **Scheduled Times.** Next, press the **Edit Timetable** button. This brings up the timetable editor.
- Add times to the timetable by entering them in the **Enter Time** box and then pressing the **Add** button. The new time will be added to the list in chronological order. To remove a time, highlight it in the list and press the **delete** button. The **Clear All** button removes all of the times listed.

Event: The schedule will start in response to a variation detected in another parameter.

- The list of parameters to use as a trigger can be selected by pressing down the arrow in the **Trigger** box.
- The magnitude of change that is required to cause a trigger is entered into the box labeled **Data Variation Value**.

- When the sample data for the triggering parameter changes by more than the **Data Variation Value** up or down, it will trigger an “Event” and start the second “Event triggered schedule.” The data value at this time becomes the new reference point for the **Data Variation Value**.
- For this option to work, a schedule that tests the trigger parameter must be enabled and started.

External Trigger: A signal from an external line will start the schedule.

7.1.4 Setting the Current Date and Time

The Current Date/Time window shows the setting on both the Aqualab and PC. If the PC settings are incorrect they should be reset. Use the Windows control panel to reset the time and date. The Analyser32 software will update the Aqualab time and date information with that of the PC when the scheduler is started.

7.1.5 Enabling a Schedule and Scheduler Priority

Any schedule, which is due to commence while another is in progress, will not run. If two or more schedules are due to execute at the same time, the schedule earliest in the list will run. The other schedule (s) will not run. To enable a schedule, tick the **Schedule Enable** box. A schedule will not run without this box being ticked.

7.1.6 Starting and Stopping a Schedule Using Analyser32

- The **Start Aqualab** window appears after pressing the **Start** button in the main menu to turn the scheduler on.
- To stop the Aqualab press the **Stop** button in the main menu.

7.2 Chemical Store

The Aqualab is supplied with two complete bottle sets. This is done so that chemicals do not have to be poured into bottles at the site. With two sets, one can be in use at the Aqualab and the other at the laboratory being refilled. The reagents and standards are connected to the Aqualab by polyethylene tubing and Luer fittings. A numbering system is used to identify chemicals and tubes. When replacing reagents and standards always attach the labeled tube to the bottle with like color and numbering. Use the interface software to reset levels for the replaced reagents and standards in the chemical store. The interface software can be used to edit the concentration of the standards and set the chemical level alarms. **Note**: When reagents and standards are not in use they should be stored in a cool dark environment. The shelf life for the wash solution is 12 months. Note: When changing out standards and reagents care should be used not to contaminate reagents and standards and do not allow contents to come in contact with skin or clothing.

7.2.1 Changing wash solution bottles.

- Stop the scheduler if it is running, and wait for any remaining steps to finish.
- Put on safety equipment - a protective coat, gloves and eye protection.
- Open the cabinet front door and connect a PC to the Aqualab. Use the Analyser32 software to check which reagent requires replacement.
- Locate the bottle that requires replacement. Disconnect the tubing Luer fitting.
- Remove the bottle and cap from the Aqualab.

- Check that the color code on the new bottle matches the color code on the inlet tube.
- Swap the caps on each bottle. **Note:** Do not top off bottles, always replace with fresh bottles.
- Place the new bottle back into position. Connect the tubing to the bottle and tighten the Luer fitting finger tight. **Note:** Do not over tighten this fitting.
- Visually check the level of the waste bottle to see if it requires replacement.

7.2.2 Resetting reagent and standard volume levels and editing reagent concentrations.
The Aqualab keeps record of the levels of solutions available in the chemical store. This is done to notify the operator when they are close to empty and also to prevent the Aqualab from running when they are completely empty.

- Turn off the scheduler (see Section 7.16).
- Choose **Setup** from the **Main Menu**.
- Choose **Chemical Store** from the **Setup Menu**.
- If all the bottles are refilled, reset all volumes to full by pressing the **Set All Solutions as Full** button. A message will appear asking for confirmation.
- To change the level of an individual solution, highlight the row for the solution and press the **Edit** button. The **Edit Reagent window** will appear. If the bottle has been completely refilled tick the **Full Solution Bottle Installed** box. To set the level to another level click on the **Current Level** box and edit data.
- In the **Edit Reagent Window** the alarm level in the **Trigger Volume** box should be set to 500 ml with the alarm enabled.
- Also in the **Edit Reagent Window** is the **Active Constituent Concentration**. Visually check that the bottle volumes match the recorded bottle volumes displayed on the PC and edit the volume level in the chemical store if necessary.
- Restart the scheduler.

7.3 Sample Delivery
Ambient surface water is pumped from the water body through a vinyl or PVC sample line by a submersible pump to the sample reservoir. A sample is delivered to the analysis area of the analyzer from the sample reservoir.

7.3.1 A submersible pump should be deployed in a representative section of the water body with adequate flow and sufficient depth to cover the pump with at least one foot of water. Areas where excessive vegetation, turbulence, or silt that may foul the pump or bias sample collection should be avoided.

7.3.2 Secure pump to prevent movement of pump in water body. Options may include: fastening the pump to a trolley mounted on L-track or a pulley system.

7.3.3 Once a month the exhaust line sample flow should be measured. For further details, see SOP MAIN-022, Section 7.6.2.

7.4 Sensor Calibration

A manual turbidity sensor calibration is performed once a year at a minimum. Before commencing any calibration procedures the operator must stop the scheduler (see Section 7.1.6) and wait for any schedules currently in progress to finish. To-access the **Calibration** screen in the system setup menu a **/x** switch must be added to the end of the file path in the **Target** field in the program shortcut. **Note:** rinse (and dry) the "cal port tube" with DI water before and after emersion into calibration standards.

- Connect a PC to the analyzer with a serial cable. A fold down tray is available inside the front door for supporting notebook computers. Start the Analyser32 user interface.
- Upon successful connection to the Aqualab, press the **Setup** button.
- Press **System Setup** and then **Calibrate**.
- Select turbidity from the list of sensors to calibrate.
- Follow the instructions provided on the screen to carry out the calibration. At the end of the calibration steps-ensure that the **Poly Order** is set to 1 less than the number of data points and click **Calculate** on the screen presented.
- Determine the slope of the calibration curve using equation shown in 8.2.6. The curve is considered linear if the r value is >0.99. If the curve is not linear, reanalyze the calibration curve.
- When the fit meets specifications select "Poly Order 3" and click **Send to Aqualab**.
- Enter the calibration information into the turbidity calibration spreadsheet. This spreadsheet will calculate the "r" value.
- When the turbidity calibration is complete thoroughly rinse the cal port tubing with clean water. Cap and rinse the standard solution bottles and clean up any spills.
- For the Aqualab to recognize and use this new calibration data in the calculations for turbidity the Aqualab needs to be reset. To do this, exit the user interface and turn off power to the Aqualab, wait five seconds and turn on power to the Aqualab.

7.5 Sensor Verification Instrument Blanks (IB)

The Aqualab automatically performs a single-point turbidity calibration using DI water (wash solution) every 24-hours (at midnight) for subsequent sample quantitation. The Aqualab provides IB results that do not have the single point calibration applied to it and is used to verify the condition of the sensor.

7.5.1 Every business day the operator will monitor (via modem) IBs and determine if the sensor meets acceptance criteria. If an IB does not meet acceptance criteria in table 9-1, identify and correct the problem before continuing. For further details, see section 9.0.

8.0 CALCULATIONS

8.1 Sample Concentration

The concentration of the sample (*T*) in nephelometric turbidity units (NTU) is calculated using the following equation:

$$T = \sum_{i=1}^{i=4} K_i \left(x_S^i - x_B^i \right)$$

Where:

K_i is the ith 4th order polynomial coefficient determined by the calibration curve,

x_S^i is the counts of the sample raised to the ith power, and

x_B^i is the counts of the base or blank standard raised to the ith power.

8.2 QC Calculations

8.2.1 The mean ($\bar{x}$) is the average of a given set of related data:

$$\overline{X} = \frac{\sum_{y=1}^{n} X_y}{n}$$

Where:

X = individual measurements; and

n = total number of measurements.

8.2.2 The standard deviation (S) is a measure of the average distance of individual observations from the mean. It is usually denoted by "S" and is defined as:

$$S = \sqrt{\frac{\sum_{i=1}^{n} X_i^2 - n\overline{X}^2}{n-1}}$$

Where:

n = number of measurements;

Xi = ith observation in the sample; and

$\overline{X}$ = sample mean.

8.2.3 The relative standard deviation (RSD) is a commonly used measure of variability that is adjusted for the magnitude of the values in the sample:

$$RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \text{ x}$$

The RSD is used most often when the size of the standard deviation changes in proportion to the size of the mean.

8.2.4 The linearity of the calibration curve is determined using the following equation:

$$r = \frac{n\left(\sum XY\right) - \left(\sum X\right)\left(\sum Y\right)}{\sqrt{\left[n\sum X^2 - \left(\sum X\right)^2\right]\left[n\sum Y^2 - \left(\sum Y\right)^2\right]}}$$

Where:

X = response of sensor in area counts; and
Y = the standard concentration in NTU.

9.0 QC

9.1 Initial Demonstration of Analytical Capabilities (IDAC)
Before an analyst may perform these procedures without the oversight of a senior analyst, all aspects of the method must be learned and performed proficiently. Currently, the IDAC consist of on the job training provided by a senior analyst until formalized procedures are developed.

9.2 QC Samples
QC samples are used to ensure that acceptable data quality is maintained throughout the process and to help assess data validation. The QC samples analyzed for this method are instrument blanks.

Any deviation from the procedures documented in the SOP, including any QC samples which do not meet the frequency requirement or acceptance criteria, need to be documented in the operators log. The log entry should contain a description of the exception, the cause (if known), the affected data, and the impact on data. Any affected data should be qualified accordingly.

9.2.1 An IB is analyzed automatically by the Aqualab every 24-hours. An IB is analyzed to ascertain whether and to what extent the system is contaminated with compounds that interfere with the results of analysis and to assess sensor drift. The IB consists of DI water and boric acid that is introduced into the instrument from the wash solution. The turbidity concentration measured in the IB should be between -30 and 50 NTU. If the IB does not meet acceptance criteria, corrective action needs to be performed before continuing sample analysis.

Table 9-1: QC Checks

QC Check	Purpose	Frequency	Acceptance Criteria	Response Action
Calibration Curve (4 concentrations and 1 blank)	To establish slope used for quantitation	Annually, following a major change in the system or after failing IB	1) All four concentration levels are detected 2) r value is >0.99	1) Analyze individual point if needed. 2) Perform corrective action as necessary 3) Re-calibrate
Sample line Flow Rate	To assess operational outlet flow rates	Once a month	Flow, 4 gallons/minute	Perform corrective action as necessary
IB	Used to assess instrument contamination and sensor drift	Once every 24-hours	Must be between (-) 30 and 50 NTU	1) Reanalyze IB 2) If still failing, perform corrective action and/or re-calibrate 3) Qualify data

10.0 DEFINITIONS

See Appendix A of the *Laboratory and Mobile Monitoring Quality Manual* and the *Surface Water Quality Monitoring Quality Assurance Project Plan*

11.0 REFERENCES

- U.S. EPA equivalent method EQSA-0193-092
- *TCEQ Operating Policies and Procedures, Chapter 6.13*
- *Continuous Water Quality Monitoring Network Quality Assurance Project Plan*
- *Laboratory and Mobile Monitoring Quality Manual*
- *Monitoring Operations Hazardous Waste Disposal Plan*
- *TCEQ Chemical Hygiene Plan*

12.0 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, and 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.

The reagents, washes, standards, and waste associated with this procedure do not require special disposal. Before disposing waste into a municipal sewer system check with respective municipal sewer system on what concentration levels are allowed to be put into their system.

13.0 SHORTHAND PROCEDURE

- Set-up Procedures (Section 7.1, 7.2, and, 7.3)
- Create a sampling schedule.
- Change reagents and standards.
- Measure sample exhaust once a month.
- Sampling, Calibration, and Sensor Verification (Section 7.3 and 7.4)
- Calibrate Tubidity sensor at a minimum annually.
- Monitor turbidity IBs daily via modem.
- Return the system to ambient sampling.