

STANDARD OPERATING PROCEDURE (SOP) – Water Quality Planning Division

Title: Analysis of Total Reactive Phosphorus (TRP) in Ambient Surface Water Using a Aqualab Greenspan Continuous Auto-Analyzer

Team Leader: _____ Date: _____

Quality Control Review: _____ Date: _____

Section Manager: _____ Date: _____

Effective Date: 6/16/08

1.0	PURPOSE	1
2.0	SCOPE AND APPLICABILITY	1
3.0	METHOD OR PROCEDURAL SUMMARY	2
4.0	LIMITATIONS	2
5.0	SAFETY	3
6.0	EQUIPMENT, SUPPLIES, REAGANTS, AND STANDARDS	3
7.0	PROCEDURES	6
8.0	CALCULATIONS	10
9.0	QUALITY CONTROL	11
10.0	REFERENCES	13
11.0	DEFINITIONS	13
12.0	POLLUTION PREVENTION AND WASTE MANAGEMENT	13
13.0	SHORTHAND PROCEDURE	14

1.0 PURPOSE

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

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 Data produced by this procedure is used in the Environmental Monitoring Response System (EMRS) pilot program in the Bosque and Leon River basins.
- 2.3 Due to the extended length of time continuous analyzers are deployed and the high frequency of data collection the data can be used to establish baseline conditions, identify trends, characterize pollution events, and seasonal variations in water quality.
- 2.4 Assess improvements after watershed management and implementation plans are in place.
- 2.5 Provide continuous water quality data to the public (via internet) for water bodies of interest.
- 2.6 The nominal method detection limit (MDL) for the low range TRP sensor is approximately 3 parts per billion (ppb). The working range of the detector is between 3 ppb and 600 ppb.
- 2.7 The nominal MDL for high range TRP sensor is approximately 0.015 parts per million (ppm). The working range of the detector is between 0.015 ppm and 4.00 ppm.

3.0 METHOD SUMMARY

- 3.1 Every six hours, ambient surface water is pumped from the water body through sample line to a sample reservoir using a submersible pump. A sub-sample is then collected from the sample reservoir and analyzed by the analyzer.
- 3.2 This method does not filter samples before analysis. Ambient samples analyzed by this method are referred to as TRP. The analyzer is calibrated with dissolved orthophosphate-phosphorus ($\text{PO}_4\text{-P}$) standards.
- 3.4 Measurements are made by the ascorbic acid method. Ammonium molybdate and potassium antimony tartrate react with reactive phosphorus in an acid medium to form an antimony-phosphomolybdate complex, which, on reduction with ascorbic acid yields an intense blue color that is measured photometrically.
- 3.5 Samples are corrected for turbidity and color by measuring the response of un-reacted samples.
- 3.6 A three-point $\text{PO}_4\text{-P}$ calibration is analyzed daily.
- 3.7 Instrument Blanks (IBs) and a $\text{PO}_4\text{-P}$ Laboratory Control Sample (LCS) are analyzed daily.

4.0 LIMITATIONS

- 4.1 Aqualab sample delivery systems can be susceptible to damage due to high water events. Data loss can occur as a result of damage to the sample delivery system.
- 4.2 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 Aqualab components is necessary. Data invalidation can occur as a result of fouling.
- 4.3 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate (PO_4). As much as 50 milligrams/Liter (mg/L) ferric ion, 10 mg/L copper and 10 mg/L silicon dioxide can be tolerated. High silica concentrations cause positive interference. Hexavalent chromium and nitrite negatively bias results by approximately 3%.

- 4.4 Standards and reagents should be changed regularly. Expired standards should not be used.
- 4.5 Standards and reagents should be at the same temperature as the Aqualab before the unit performs any calibrations or sample analysis.

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 standard, reagents, and washes listed in Section 6.2. Lab coat, safety glasses with side shields and splash goggles and chemical resistant gloves should be worn when handling these chemicals. **Note:** some reagents contain concentrated sulfuric acid.

6.0 EQUIPMENT AND REAGENTS

6.1 Equipment

- Aqualab Analyzer with TRP sensor
- Climate controlled trailer
- Personal computer with Analyzer32 software and internet access
- U.S. Robotics modem
- Vinyl or polyvinyl chloride (PVC) sample line
- One horse-power submersible pump capable of delivering ambient surface water to the sample reservoir at 4 gallons/minute or greater
- Sample deliver system and sample reservoir (pot)

6.2 Reagents

- All reagents/chemicals must be AR grade
- De-ionized (DI) water
- PO₄-P standards (standards 1 and 2) traceable to National Institute of Standards and Technology (NIST)
- PO₄-P LCS

Standard 1 (container 15)

Volume 10 L

Bottle Size 10 L

Nitrate 3.50 ppm N – 10.00 milliliter (ml) of 3500 ppm N of Nitrate Stock Solution $NaNO_3$

Ammonia 1.05 ppm N – 3.00 ml of 3500 ppm N Ammonia Stock Solution $(NH_4)_2SO_4$

TRP Low Range 3 - 600 ppb

Phosphate 155 ppb P – 10.00 ml of 155 ppm P Phosphate Stock Solution KH_2PO_4

TRP High Range 0.015 - 4.00 ppm

Phosphate 1.55 ppm P – 10.00 ml of 1550 ppm P Phosphate Stock Solution KH_2PO_4

½ M Boric acid- 200 ml

Made up to 10.00 L with DI water

Label bottle with date of manufacture and expiration date

Standard life 2 months

When not in use store in cool dark environment

Standard 2 (container 16)

Volume 5 L

Bottle Size 5 L

Nitrate 7.0 ppm N – 10.00 ml of 3500 ppm N of Nitrate Stock Solution NaNO_3 Ammonia 3.5 ppm N – 5.00 ml of 3500 ppm N Ammonia Stock Solution $(\text{NH}_4)_2\text{SO}_4$

TRP Low Range 3 - 600 ppb

Phosphate 310 ppb P – 10.00 ml of 155 ppm P Phosphate Stock Solution KH_2PO_4

TRP High Range 0.015 - 4.00 ppm

Phosphate 3.10 ppm P – 10.00 ml of 1550 ppm P Phosphate Stock Solution KH_2PO_4

½ M Boric acid – 100 ml made up to 5.000 L with DI water

Label bottle with date of manufacture and expiration date

Standard life 2 months

When not in use store in cool dark environment

Phosphate 4 Reagent (container 54)

Volume 2 L

Bottle Size 2.5 L

TRP Low Range 3 - 600 ppb

Ascorbic acid- 84 grams (g)

TRP High Range 0.015 - 4.00 ppm

Ascorbic acid- 26g

Made up to 2.00 L with DI water

Label bottle with date of manufacture and expiration date

Reagent life 6 weeks

When not in use store at 4 degrees

Wash Solution (container 50)

Volume 20 L

½ M boric acid – 400 ml

Made up with 20 L with DI water

Label bottle with date of manufacture and expiration date

Reagent life 12 months

When not in use store in cool dark environment

Phosphate 1 Reagent (container 22)

Volume 1 L

Bottle Size 1 L

TRP Low Range 3 - 600 ppb

18 M Sulphuric acid- 97.0 ml (H_2SO_4 - sg = 1.84)

Potassium antimony tartrate- 0.2145g $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6$

Ammonium molybdate- 8.58g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$

TRP High Range 0.015 - 4.00 ppm

18 M Sulphuric acid- 29.4.0 ml (H_2SO_4 - sg = 1.84)

Potassium antimony tartrate- 0.065g $\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6$

Ammonium molybdate- 2.6 g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$

Made up to 1.00 L with DI water

Label bottle with date of manufacture and expiration date

Reagent life 12 months

CAUTION – Always mix acid to water slowly, NEVER water to acid

Method: - Fill 1 L volumetric flask with approx. 750 ml DI

- Slowly and cautiously empty acid into flask

- Allow flask to cool over a period of hours

- Fill to mark and mix thoroughly

Phosphate 2 Reagent (container 20)

Volume 1 L

Bottle Size 1 L

TRP Low Range 3 - 600 ppb

18 M Sulphuric acid - 97.0 ml (H_2SO_4 - sg = 1.84)

Potassium antimony tartrate - 0.2145g $K(SbO)C_4H_4O_6$

TRP High Range 0.015 - 4.00 ppm

18 M Sulphuric acid - 29.4 ml (H_2SO_4 - sg = 1.84)

Potassium antimony tartrate - 0.065g $K(SbO)C_4H_4O_6$

Made up to 1.00 L with DI water

Label bottle with date of manufacture and expiration date

Reagent life 12 months

CAUTION – Always mix acid to water slowly, NEVER water to acid

Method: - Fill 1 L volumetric flask with 750 ml DI

- Slowly and cautiously empty acid into flask

- Allow flask to cool over a period of hours

- Fill to mark and mix thoroughly

Phosphate 3 Reagent (container 53)

Volume 1 L

Bottle Size 1 L

Purpose: This section details the procedure for making 1.0 l of Phosphate reagent 3 for the Aqualab Phosphate sensor. If more or less than 1.0 L is required, multiply the final quantities of materials accordingly.

TRP Low Range 3 - 600 ppb

2.2% m/v Sodium laurel sulphate in DI water

TRP High Range 0.015 - 4.00 ppm

1.1% m/v Sodium laurel sulphate in DI water

DURING HANDLING, THE DRY CHEMICAL READILY PRODUCES A DUST WHICH IS A MODERATE TO SEVERE IRRITANT TO THE EYES AND RESPIRATORY TRACT.

WEAR SUITABLE PROTECTION WHEN WEIGHING, TRANSFERRING ETC.

Additionally, when dissolved, it is a powerful surfactant which readily forms a stable foam if agitated sufficiently. MINIMIZE THE LATTER AS DESCRIBED BELOW.

1. Weigh by difference from its container, 22 g (TRP Low Range 0.003 - 0.600 ppm) or 11 g (TRP High Range 0.015 - 4.00 ppm) of sodium laurel sulphate directly into a 1000 ml class A volumetric flask equipped with a powder funnel.
2. Flush any dust from the inside of the funnel &/or flask neck with DI water using a low-intensity stream of water to minimize foaming.
3. Tilt the flask to one side and slide a 50 millimeter magnetic stirring bar down the neck.
4. Slowly add sufficient water to the tilted flask to increase the level to about 600 mL. Place the flask on a stirrer-hotplate and adjust the stirring rate to the maximum speed possible that doesn't whip air into the liquid. Adjust as necessary to maintain this rate.
5. Initially set the heater to about 3 and then reduce to setting 1-2 as the flask warms up. When solution is complete, turn off the stirrer-hotplate and cool the flask in running water without agitation.
6. Make to the mark with a low intensity stream of water. Stopper and mix thoroughly in the usual manner.
7. Allow foam to dissipate then carefully transfer to an appropriate HDPE container.
8. Label bottle with date of manufacture and expiration date.
9. Reagent life 12 months.

PO₄-P LCS

LCSs are prepared from a Stock Solution of KH_2PO_4 .

7.0 PROCEDURE

7.1 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 sub-sample is delivered to the analysis area of the analyzer from the sample pot reservoir.

- 7.1.1 The submersible pump should be deployed in a representative section of the water body. The pump intake should be at approximately one foot of water depth. Areas of excessive vegetation, turbulence, shifting stream bottoms, or excessive silt should be avoided.
- 7.1.2 When monitoring rivers and streams, the pump should be located in an area that has greater than 50 percent of the flow. Avoid back-water area with little flow unless this type of area is representative of the water body.
- 7.1.3 The submersible pump is deployed in the water body. Options may include: fastening the pump to a trolley mounted on L-track or a suspended pulley system. The pump may need to be adjusted periodically due to fluctuations in water levels of the water body.
- 7.1.4 Once a month the exhaust line sample flow should be measured. For further details, see Section 9.0. of this SOP and SOP MAIN-022, Section 7.6.2.

7.2 Analyzer Operation

The analyzer is controlled through Analyzer32 software interface. The software allows schedules to be created that automatically control the following prescribed

functions:

1. Ambient sample analysis
2. Calibrations and analyzer cleaning regimes
3. Analysis of IBs and LCSs
4. Starting Schedules

7.2.1 Creating sample collection and analysis schedule

Ambient samples are collected and analyzed every six hours. Once a sample schedule initiates, the submersible pump is activated by the Aqualab and the sample line is flushed for several minutes with ambient water. A sub-sample is collected near the end of the flushing regime and sent to the analysis area of the analyzer. After the sample is collected, the line to the sample reservoir is flushed with wash solution.

- To stop the Aqualab select the **Stop** button in the Main Menu.
- From the Main Menu select **Setup**. The Setup Menu window will appear. From the Setup Menu select **Scheduling**.
- The Schedule Setup window will appear and select **New**.
- The Edit Schedule window will appear.
- Enter the name of the schedule (*Phosphate Every 6 Hours*) in the Name box.
- The Tests in Schedule list describes all the tests to be performed in the schedule as well as the fill and clean operations. Select **Edit Test**. 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 select the **Add Test>>**.
- Add the following tests in the following order and select the **fill/clean** box.
 - 01 – Cabinet Temperature
 - 25 – Measure Phosphate
- Select **OK**.
- Under Scheduled Times select **Periodic**. Enter 02:29:55 in the Period Start window and 06:00:00 in the Trigger Period box.
- To enable the schedule, tick the Schedule Enable box.
- Select **OK** and the Schedule Setup menu will appear.

7.2.2 Creating a daily calibration and system clean schedule

Calibrations are automatically performed once every 24 hours. The system is cleaned before calibration. A three-point calibration curve is generated using DI water and standards one and two.

- Follow procedures in Section 7.2.1 with the following changes.
- Enter the Schedule name as *Daily Calibration and Clean*.
- Choose the following tests.
 - 42 – Phosphate Cal
- In the Scheduled Times window select **Time Table** instead of Periodic.
- Select **Edit Time Table**. The Timetable Editor window will appear.

- Enter 01:22:00 to the timetable by entering them in the Enter Time box and then pressing the **Add>>** button. Then select **OK**.

7.2.3 Creating daily LCS schedule

A LCS is analyzed once every 24 hours.

- Follow procedures in Section 7.2.1 with the following changes.
- Enter the Schedule name as *LCS Schedule*.
- Choose the following tests.
47 – Phosphate LCS
- In the Scheduled Times window select **Time Table** instead of Periodic.
- Select **Edit Time Table**. The Timetable Editor window will appear.
- Enter 23:22:00 to the timetable by entering them in the Enter Time box and then pressing the **Add>>** button. Then select **OK**.

7.2.4 Creating daily IB schedule

IBs are analyzed once every 24 hours. IBs are analyzed after the LCS is analyzed.

- Follow procedures in Section 7.2.1 with the following changes.
- Enter the Schedule name as *Daily IB*.
- Choose the following tests.
54 – Phosphate Base
- In the Scheduled Times window select **Time Table** instead of Periodic.
- Select **Edit Time Table**. The Timetable Editor window will appear.
- Enter 00:22:00 to the timetable by entering them in the Enter Time box and then pressing the **Add>>** button. Then select **OK**.
- Once the prescribed schedules are created, start analyzer schedules according to procedures in Section 7.2.5.

7.2.5 Starting Schedules.

- From the Schedule Setup window select **Close**.
- Setup window will appear and select **Exit Setup Window**. The Main Menu window will appear.
- Select **Start**.
- The Start Analyzer window will appear.
- Enter operator's initials in the Operator 1 Box. Add comments as necessary in the Comment box. Select **Start** to start the schedules.

7.3 Chemical Store

The Aqualab is supplied with two complete bottle sets. 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. Attach the labeled tube to the bottle with like color and numbering when replacing reagents and standards. 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.

7.3.1 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.

7.3.2 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.
- 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 you 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. The concentration values are used in calibration procedures and analysis calculations. **Note:** Do not change concentration values unless the theoretical concentration of the standard(s) has changed.

- The theoretical concentrations of the standards should be used and not the concentrations from a verification report.
 - 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.
- 7.4 Site operators should monitor water quality measurements and Quality Control (QC) parameters daily to ensure the station is operational. If problems are identified, a site visit may be needed to correct any problems.
- 7.4.1 Every business day the site operator will monitor (via TCEQ website www.texaswaterdata.org) the station ensuring site communications are operational and screen measurement data for any anomalies.
- 7.4.2 Every business day the site operator will remotely monitor (via modem analyzer data downloads) the operational status of the analyzer which includes IB and LCS results. If problems are identified a site may be needed to correct any problems. After site visits post operator logs describing site activities.
- LCSs are analyzed automatically once every 24 hours. The site operator monitors LCS results. If an LCS does not meet acceptance criteria in Table 9-1, identify and correct the problem before continuing. For further details, see Section 9.0.
 - IBs are analyzed automatically once every 24 hours. The site operator monitors IB results. 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.
 - Monitor water level measurements. Adjust submersible pump as necessary.
- 7.4.3 Precision is calculated every day using LCS results from consecutive days. For further details, see Section 9.0

8.0 CALCULATIONS

8.1 Sample Concentration

The concentration of the sample (C_s) in ppb is calculated using the following equation:

$$C_s = \frac{(x_1 - x)(x_3 - x)y_2}{(x_1 - x_2)(x_3 - x_2)} + \frac{(x_1 - x)(x_2 - x)y_3}{(x_1 - x_3)(x_2 - x_3)}$$

Where:

- x = Sample machine counts - turbidity area counts + zero area counts;
- x_1 = zero area counts;
- x_2 = standard 2 machine counts;
- x_3 = standard 2 machine counts;
- y_2 = standard 1 concentration (155 ppb or 1.55 ppm) and;
- y_3 = standard 2 concentration (310 ppb or 1.55 ppm).

$$C_s \text{ in ppm} = C_s (\text{ppb})/1000$$

8.2 QC Calculations

8.2.1 The accuracy of analytical laboratory data is usually presented in terms of percent recovery, given by:

$$\% \text{ Recovery} = \frac{\text{Measured Value}}{\text{Actual Value}} \times 100$$

8.2.2 Relative percent difference (RPD) is another commonly used measure of variability that is adjusted for the magnitude of the measured values. It is used when the sample contains only two observations and is given by:

$$\text{RPD} = \frac{|X1 - X2|}{(X1 + X2)/2} \times 100$$

Where:

X1 = duplicate sample measurement; and
X2 = duplicate sample measurement.

9.0 QC

9.1 MDL

The U.S. Environmental Protection Agency procedure for the determination of MDLs in *Appendix B, 40 Code of Federal Regulations Part 136* is used as a guideline for determining the MDL. Due to instrument or calibration standards limitations, it may not be possible to follow the procedure step by step. The following are the major steps for determining the MDL for this SOP. For more details, refer to the cited procedure.

9.1.1 Determine PO₄-P MDLs at initial method set-up, following a major change in the system, or annually.

9.1.2 Introduce seven calibration standards at a concentration of 3 to 5 times the expected detection limit.

9.1.3 The MDL is equal to 3.14 times the standard deviation of the seven replicates.

9.1.4 Perform a PO₄-P MDL verification at a concentration of 2 to 5 times the measured or nominal MDL, whichever is greater. A valid instrument response verifies the MDL values. In a case a valid instrument response is not observed, either perform another MDL study or perform additional MDL verifications studies at higher concentrations. The concentration at which a valid instrument response is observed can be used as the MDL.

9.2 QC Samples

QC samples are used to ensure that acceptable data quality is maintained throughout the process. The QC samples analyzed for this method include IBs and PO₄-P LCSs. Any deviation from the procedures documented in the SOP, including any QC samples which do not meet the frequency requirement or acceptance criteria, should be documented in the operators log. The log entry should contain a description of the exception, the cause (if possible), the affected data, and the impact on data. Any affected data should be qualified accordingly.

- 9.2.1 An IB is analyzed automatically once every 24 hours directly after the analysis of a PO₄-P LCS. An IB is analyzed to ascertain whether and to what extent the system is contaminated with compounds that interfere with the results of the analysis. The IB consists of DI water that is introduced directly into the instrument from the sample inlet line 71 (from the sample reservoir). The concentration measured in the IB should be 6 ppb low range sensor and ≤ 0.015 ppm high range sensor. If interferences exist, the cause of the interferences must be determined.
- 9.2.2 A PO₄-P LCS is analyzed automatically once every 24 hours. This is to provide an assessment of the TRP analysis. The LCS is prepared from a second source standard and is introduced into the analysis area of the analyzers. See Table 9-1 for LCS concentrations. The recovery of the LCS should be 75.0% - 125.0%. If the LCS does not meet the acceptance criteria, corrective action needs to be performed.
- 9.2.3 An Laboratory Control Sample Duplicate (LCSD) is an LCS that is used to assess precision and is calculated once every day using consecutive daily PO₄-P LCSs. The RPD between the daily LCSs should be 20.0%.

Table 9-1 TRP Ranges, Units, and LCS Concentrations

Continuous Ambient Monitoring Station	Location	Analyzer TRP Units	PO ₄ -P LCS Concentration	TRP Range
701	Greens Creek (Bosque River Tributary)	ppb	40 ppb	3 – 600 ppb
702	North Bosque River at Clifton, Tx	ppb	40 ppb	3 – 600 ppb
704	Resley Creek (Leon River Tributary)	ppb	40 ppb	3 – 600 ppb
725	North Bosque River at State Hwy 6	ppm	0.050 ppm	0.015 – 4.00 ppm

Table 9-2 QC Checks

QC Check	Purpose	Frequency	Acceptance Criteria	Response Action
MDL	To assess the limits of detection of the analytical system	Annually or following a major change to the system	<u>Low Range Sensor</u> Must be ≤ 6 ppb <u>High Range Sensor</u> Must be ≤ 0.015 ppm	Replace sensor
Sample line Flow Rate	To assess operational outlet flow rates	Once a month	Must be ≥ 4 gallons/minute	Perform corrective action as necessary

MDL Verification	To verify MDL	Following a MDL	Any instrument response	1) Reanalyze MDL 2) Use an MDL value obtained from a higher concentration MDL verification that produces an instrument response
IB	Used to assess instrument contamination	Once every 24 hours	<u>Low Range Sensor</u> Must be ≤ 6 ppb <u>High Range Sensor</u> Must be ≤ 0.015 ppm	1) Reanalyze IB 2) Perform corrective action as necessary 3) Validate data accordingly
PO ₄ -P LCS	Used to assess accuracy of the on board standards and calibration curve	Once every 24 hours	Recovery should be in the range of 75.0 % - 125.0%	1) Reanalyze LCS 2) If still failing perform corrective action 3) Validate data accordingly
PO ₄ -P LCSD	Used to assess precision	Calculated once day using consecutive daily LCSs	RPD 20.0 %	1) Perform corrective action as necessary 2) Validate data accordingly

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. Geological Survey TWRI Book 9
Greenspan Aqualab Total phosphorus Operator's Manual
TCEQ Operating Policies and Procedures, Chapter 6.13
Laboratory and Mobile Monitoring Quality Manual
Continuous Water Quality Monitoring Network Quality Assurance Project Plan
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

- Procedures (Section 7.0)
 - Create sampling, calibration, IB, and LCS schedules
 - Change reagents, standards, and wash solutions as necessary
 - Reset reagent and standard volumes
 - Every business day remotely monitor station communications, water quality parameters, water level measurements, IB, and LCS results
 - Adjust submersible pump as necessary
 - Conduct site visits as necessary
 - Post operator logs after site visits
 - Measure sample exhaust once a month
 - Conduct MDL studies yearly