

## STANDARD OPERATING PROCEDURE (SOP) - Water Quality Planning Division

### Title: analysis of Ammonia (NH<sub>3</sub>) in Ambient Surface Water Using an Aqualab Greenspan Continuous Auto-Analyzer

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1.0	PURPOSE	

This describes the sampling and analytical procedure for continuous automated analysis of NH<sub>3</sub> 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 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 0.100 parts per million (ppm). The working range of the detector is between 0.100 ppm and 10.0 ppm.

## 3.0 METHOD SUMMARY

- 3.1 Ambient surface water is pumped from the water body through 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 Ammonia-Selective Electrode Method. Dissolved  $\text{NH}_3$  ( $\text{NH}_3$  and  $\text{NH}_4^+$ ) is converted to  $\text{NH}_3$  by raising pH to 10.
- 3.3 The Ammonia-Selective Electrode uses a gas-permeable membrane to separate the sample solution from an electrode internal concentration of chloride ions.  $\text{NH}_3$  diffuses through the membrane and changes the internal solution pH that is measured by a pH electrode. The fixed internal solution of hydroxide ions is measured by a reference electrode.
- 3.4 Before the Aqualab performs a  $\text{NH}_3$  analysis a two-point calibration is automatically generated using onboard standards for subsequent sample quantitation. This ensures that any changes in the sensor due to temperature or drift are effectively compensated for each time a sample is measured.

## 4.0 LIMITATIONS

- 4.1 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 Aqualab Preventive Maintenance Inspection SOP MAIN-022 for cleaning and maintenance procedures.
- 4.2 Amines are a positive interference. High concentrations of dissolved ions affect the measurements.
- 4.3 Standards and reagents should be changed regularly. Expired standards and reagents should not be used.
- 4.4 Standards and reagents should be at the same temperature as the Aqualab before the unit performs any 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 ammonia, nitrate, phosphate, boric acid, and sodium hydroxide. These chemicals 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 ( $^{\circ}\text{C}$ ) resolution
- Sample reservoir (pot)
- Submersible pump capable of delivering ambient surface water to the sample reservoir at 4 gallons/minute or greater.

### 6.2 Standards and Reagents

- Onboard  $\text{NH}_3\text{-N}$  standards (standards 1 and 2) of 1.05 and 3.5 ppm traceable to National Institute of Standards and Technology (NIST)

#### **Standard 1 (container 15)**

Volume 10 liters

Bottle Size 10 liters

Nitrate 3.50 ppm N – 10.00 milliliter (ml) of 3500 ppm N of Nitrate Stock Solution  $\text{NaNO}_3$  Ammonia 1.05 ppm N – 3.00 ml of 3500 ppm N Ammonia Stock Solution  $(\text{NH}_4)_2\text{SO}_4$

Phosphate 155 parts per billion (ppb) P – 10.00 ml of 155 ppm P Phosphate Stock Solution  $\text{KH}_2\text{PO}_4$

$\frac{1}{2}$  M Boric acid- 200 ml

Made up to 10.00 litres 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 liters

Bottle Size 5 liters

Nitrate 7.0 ppm N – 10.00 ml of 3500 ppm N of Nitrate Stock Solution  $\text{NaNO}_3$  Ammonia 3.5 ppm N – 5.00 ml of 3500 ppm N Ammonia Stock Solution  $(\text{NH}_4)_2\text{SO}_4$

Phosphate 310 parts per billion P – 10.00 ml of 155 ppm P Phosphate Stock Solution  $\text{KH}_2\text{PO}_4$

$\frac{1}{2}$  M Boric acid – 100 ml made up to 5.000 liters 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

NaOH solution pH 10 traceable to NIST

### **Ammonia Reagent (container 23)**

Volume 2.0 liters

Bottle Size 2.5 liters

Sodium Hydroxide – 240 grams NaOH

Made up to 2.00 liters with DI water

Label bottle with date of manufacture and expiration date

Reagent life 12 months

De-ionized (DI) Type 1 water

## 7.0 PROCEDURE

### 7.1 Setting up a Schedule

The Analyzer 32 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 all 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

- 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 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 test 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 to eliminate the need to pour standards and reagents 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. 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. The shelf life for the NH<sub>3</sub> standard is two months and 12 months for the wash solution and reagent. **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 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**. 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 the 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.
- 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 Verification, Laboratory Control Sample (LCS), Laboratory Control Samples Duplicate (LCSD), and Equipment Blanks (EB)

For every sample the Aqualab automatically performs a two-point NH<sub>3</sub> calibration using standards 1 and 2 to determine slope used for sample quantitation. LCSs are analyzed automatically and EB samples are analyzed periodically by the site operator.

7.4.1 Every business day the site operator will monitor (via TCEQ website [http://www.tceq.state.tx.us/compliance/monitoring/water/quality/data/wqm/swqm\\_realtime\\_alt.html#data](http://www.tceq.state.tx.us/compliance/monitoring/water/quality/data/wqm/swqm_realtime_alt.html#data)) and screen water quality parameters for anomalies. If problems are identified, a site visit may be needed to correct any problems.

7.4.2 LCSs are analyzed automatically once every 24 hours at 00:22. The site operator monitors LCS results via modem every business day. 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.

7.4.3 EB samples are analyzed by the site operator once every week. If an EB does not meet acceptance criteria in Table 9-1, identify and correct the problem before continuing. For further details, see Section 9.0.

7.4.4 Precision is calculated every day using LCS results from consecutive days. For further details, see Section 9.0

## 8.0 CALCULATIONS

### 8.1 Electrode Response

Hydroxide concentration is proportional to  $\text{NH}_3$  concentration. Electrode response to  $\text{NH}_3$  is calculated by the following:

$$E = E_0 - S \log (\text{NH}_3)$$

Where:

$E$  = measured electrode potential;

$E_0$  = reference potential;

$\text{NH}_3$  =  $\text{NH}_3$  concentration in solution, and;

$S$  = electrode slope (-59.2 mV/decade)

### 8.2 Sample Concentration

The concentration of a sample ( $C_s$ ) in ppmv is calculated as follows:

$$C_s = \frac{x - b}{10a - cva}$$

$$a = \log \frac{cr1 - cr0}{\left( \frac{cv1 + cva}{cv0 + cva} \right)}$$

$$b = cr0 - a * \log(cv0 + cva)$$

Where:

$cv0$  = standard 0  $\text{NH}_3$  concentration;

$cv1$  = standard 1  $\text{NH}_3$  concentration;

$cva$  = addition standard  $\text{NH}_3$  concentration;

$cr0$  = standard 0 area counts, and;

$cr1$  = standard 1 area counts.

### 8.3 QC Calculations

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

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

Where:

$X$  = individual measurements; and

$n$  = total number of measurements.

- 8.3.2 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:

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

Where:  
n =  
number of measurements;

$X_i$  = ith observation in the sample; and  $\bar{X}$  = sample mean.

- 8.3.3 The Relative Standard Deviation (RSD) is a commonly used measure of variability that is adjusted for the magnitude of the values in the sample:

$$\text{RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

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

- 8.3.4 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:

$$\text{RPD} = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100$$

Where:

$X_1$  = duplicate sample measurements; and  
 $X_2$  = duplicate sample measurements.

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

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

## 9.0 QC

### 9.1 MDL

The U.S. Environmental Protection Agency (EPA) procedure for the determination of MDLs in *Appendix B, 40 Code of Federal Regulations (CFR) 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 referenced procedure. **Note:**  $\text{NH}_3$  LCS results can be used for MDL determination. The LCS is automatically analyzed once a day and the LCS concentration is approximately five times (0.500 ppm) the

expected detection limit. When using LCS results for MDL determination seven subsequent LCS results must be used. An additional automated LCS result may be used for Section 9.1.4.

- 9.1.1 Determine the MDL 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. An NH<sub>3</sub> LCS is automatically analyzed once a day. LCS Results from seven subsequent days can be used
- 9.1.3 The MDL is equal to 3.14 times the standard deviation of the seven replicates.
- 9.1.4 Perform a MDL verification study 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 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.

## 9.3 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 include blanks and LCSs.

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 possible), the affected data, and the impact on data. Any affected data should be qualified accordingly. **Note:** A failing LCS can be followed by a single replicate analysis to determine if there is a systematic problem. If the reanalysis meets all acceptance criteria, then the system may be deemed as providing acceptable data. Conducting multiple analyses to obtain a single passing QC sample when no corrective action as a result of an assignable cause or instrument maintenance is performed is not acceptable. In other words, if either the original QC sample or its rerun passes, then the failing QC analysis is considered to be an anomaly, and its results are not used for data assessment.

- 9.3.1 An EB should be analyzed once every week. An EB is analyzed to ascertain whether and to what extent the system is contaminated with compounds that interfere with the results of the analysis. The EB consists of DI water that is introduced directly into the instrument from the sample inlet line (from the sample reservoir). The NH<sub>3</sub> concentration measured in

the EB should be 0.160 ppm. If the EB does not meet the acceptance criteria, corrective action needs to be performed before continuing sample analysis.

9.3.2 An NH<sub>3</sub>-N LCS is analyzed automatically once every 24 hours at 00:22. This is to provide an accuracy assessment of the onboard NH<sub>3</sub> standards. The LCS is prepared from a second source standard and is introduced into the instrument at 0.500 ppm. The recovery of the LCS should be 70.0 % - 130.0 %. If the LCS does not meet the acceptance criteria, corrective action needs to be performed before continuing sample analysis.

9.3.3 An LCSD is an LCS that is used to assess precision and is calculated once every day using consecutive daily LCSs. The RPD between the daily LCSs should be 20.0%.

**Table 9-1: QC Checks**

QC Check	Purpose	Frequency	Acceptance Criteria	Response Action
Sample line Flow Rate	To assess operational outlet flow rates	Once a month	≥4 gallons/minute	Perform corrective action as necessary
MDL	To assess the limits of detection of the analytical system	Annually or following a major change to the system	< 0.135 ppm	Replace sensor
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
EB	Used to assess instrument contamination	Once every week	Must be ≤ 0.160 ppm	1) Reanalyze EB 2) Perform corrective action as necessary 3) Qualify data accordingly
LCS	Used to assess accuracy of the onboard standards and calibration curve	Once every 24 hours	Recovery should be in the range of 70.0 % - 130.0 %	1) Reanalyze LCS 2) If still failing perform corrective action 3) Qualify data accordingly
LCSD	Used to assess precision	Calculated once day using consecutive daily LCSs	RPD ≤20.0 %	1) Perform corrective action as necessary 2) Qualify 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. 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 and Sensor Verification (Section 7.3 and 7.4).
- Monitor NH<sub>3</sub> LCS results daily via modem.
- Analyze an EB once every week.
- Return the system to ambient sampling.