NC DEQ/DWR LABORATORY CERTIFICATION BRANCH

LABORATORY NAME:		CERT #:	
PRIMARY ANALYST:		DATE:	
NAME OF PERSON COMPLETING CHECKLIST (PRINT):			
SIGNATURE OF PERSON COMPLETING CHECKLIST:			

Parameter: **Ammonia Nitrogen**
Method: **EPA Method 350.1, Rev. 2.0, 1993**

EQUIPMENT:

	Automated Instrument:
	pH Meter or Short-Range pH Paper
	All Borosilicate Glass Distillation Equipment

DISTILATION REAGENTS:

	Borate Buffer
	Boric Acid Solution, 20 g/L

ANALYSIS REAGENTS:

	Ammonia free water
	NaOH, 1N
	Dechlorinating Reagent (if needed): Sodium thiosulfate Sodium Sulfite
	Sulfuric Acid, 5*N*
	Sodium hypochlorite solution
	Sodium phenolate
	Sodium nitroprusside
	EDTA buffer, 5%

PLEASE COMPLETE CHECKLIST IN INDELIBLE INK

Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice and in the column labeled SOP to indicate whether it is addressed in the SOP.

	GENERAL	**LAB**	**SOP**	**EXPLANATION**
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 2H .0805 (a) (7)] **Date:**			Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made. Verify proper method reference. During review notate deviations from the approved method and SOP.
2	Are all review/revision dates and procedural edits tracked and documented? [15A NCAC 2H .0805 (a) (7)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents.
3	Is there North Carolina data available for review?			If not, review PT data.
	PRESERVATION and STORAGE	**LAB**	**SOP**	**EXPLANATION**
4	Are samples checked for Residual Chlorine at the time of collection and prior to pH preservation adjustment? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2] If no, skip to question 7			Testing for residual chlorine must be performed at a neutral pH (i.e., before pH preservation adjustment). DPD screening and test strips are not reliable at extreme pHs.
5	What action is taken if chlorine is present? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2] **Answer:**			Remove the residual chorine in the sample by adding dechlorinating agent (Section 7.5) equivalent to the chlorine residual.

6	Is the residual chlorine check and any necessary mitigation documented? [NC WW/GW LC Policy]			Dechlorinating agents used at the time of sampling must be documented to have been effective (either by the sample collector or the receiving laboratory) by verifying a chlorine residual <0.5 mg/L at a neutral pH. If measuring chlorine concentration in an acidified sample, pour off a small portion of the sample and neutralize the pH prior to testing. Use sufficiently strong base to not dilute the sample. Discard that portion after testing.
7	Are samples preserved at time of collection with H_2SO_4 to pH of <2 S.U.? [40 CFR 136.3 Table II]			Preservation not required if analyzed within 15 minutes.
8	Are samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136.3 Table II and footnote 18]			
9	Is pH checked and documented to be <2 S.U. upon receipt in the laboratory? [40 CFR 136.3 Table II]			
10	What action is taken if pH is >2 S.U.? [15A NCAC 2H .0805 (a) (7) (M)] **Answer:**			Sample preservation shall be verified and documented. If a laboratory receives a sample subject to G.S. 143-215.1 and 143-215.63 that does not meet sample collection, holding time, or preservation requirements, the laboratory shall document the incident, notify the sample collector or client, and secure another sample that meets the regulatory requirements, if possible. If another viable sample cannot be secured, the original sample may be analyzed but the results reported shall be qualified with the nature of the sample collection, holding time, or preservation infractions and the laboratory shall notify the State Laboratory of the infractions. The notification shall include a statement indicating corrective action taken to prevent future infractions.
11	Are samples refrigerated above freezing to 6°C during storage? [40 CFR 136.3 Table II and footnote 18]			
12	Are samples analyzed within 28 days of collection? [40 CFR 136.3 Table II]			
	PROCEDURE – Sample Preparation	**LAB**	**SOP**	**EXPLANATION**
13	If residual chlorine is not checked prior to acidification in the field, is a portion of the preserved sample neutralized in the laboratory and checked for residual chlorine prior to distillation or analysis if distillation is not required? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2]			Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent (Section 7.5) equivalent to the chlorine residual. Note: Guidance from EPA Region IV confirms that the residual chlorine removal is not required to be performed at collection. It may be removed prior to distillation, or analysis if distillation is not required. Removal is not required at all if the permittee does not use chlorine for disinfection.
14	Is the residual chlorine check documented? [15A NCAC 02H .0805 (a) (7) (M)]			Sample preservation shall be verified and documented. If a laboratory receives a sample subject to G.S. 143-215.1 and 143-215.63 that does not meet sample collection, holding time, or preservation requirements, the laboratory shall document the incident, notify the sample collector or client, and secure another sample that meets the regulatory requirements, if

				possible. If another viable sample cannot be secured, the original sample may be analyzed but the results reported shall be qualified with the nature of the sample collection, holding time, or preservation infractions and the laboratory shall notify the State Laboratory of the infractions. The notification shall include a statement indicating corrective action taken to prevent future infractions.
15	If the sample is determined to contain chlorine at a level above 0.5 mg/L, is it removed prior to distillation and/or analysis by adding dechlorinating agent equivalent to the chlorine residual? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2]			Remove the residual chorine in the sample by adding dechlorinating agent equivalent to the chlorine residual.
	PROCEDURE – Distillation	**LAB**	**SOP**	**EXPLANATION**
16	If manual distillation is not performed on all samples is a distillation comparison study on file? [NC WW/GW LC Policy] **NOTE: This question may not be applicable to Commercial laboratories.** **Attach a copy of the study to this checklist.**			Manual distillation may not be required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. A comparison study may be performed in-house or contracted to another certified laboratory. Permittees that do not perform the analyses in-house, and contract the analyses or the distillation study to a NC WW/GW LC certified commercial laboratory must obtain a copy of the initial comparison data and all subsequent comparison data, keep it on file at their facility and make these records available to the Department upon request. The following frequencies are required: Initially, compare a minimum of 9 samples, spiked in duplicate, both with and without the distillation step (a total of 36 samples), to evaluate the need for distillation.
17	How is the distillation equipment cleaned? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.1] **Answer:**			Preparation of equipment: Add 500 mL of reagent water to an 800 mL Kjeldahl flask. The addition of boiling chips that have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia.
18	Is the sample pH adjusted to 9.5 S.U. with 1*N* NaOH? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2]			To 400 mL of sample add 1 N NaOH (Section 7.4), until the pH is 9.5 S.U.
19	How is the sample pH checked while adjusting to 9.5 S.U.? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2] **Answer:**			Check the pH during addition with a pH meter or by use of a short-range pH paper.
20	What sample volume is distilled? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.3] **Answer:**			Method bases reagent additions off 400 mL sample distilled
21	Is 25ml of borate buffer solution added to the sample? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.3]			Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 mL Kjeldahl flask and add 25 mL of the borate buffer (Section 7.3).
22	Is the distillate collected in 50 ml of 2% boric acid solution? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.3]			Distillation: Distill 300 mL at the rate of 6-10 mL/min. into 50 mL of 2% boric acid (Section 7.2) contained in a 500 mL Erlenmeyer flask.

23	Is the condenser outlet tip submerged below the surface of the receiving acid solution? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.3]			The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.
24	Is the sample volume to be distilled documented? [15A NCAC 2H .0805 (a) (7) (F) (xviii)]			All laboratories shall use printable laboratory benchsheets. Certified Data shall be traceable to the associated sample analyses and shall consist of: any other data needed to reconstruct the final calculated result.
25	Is the final volume documented? [15A NCAC 2H .0805 (a) (7) (F) (xviii)]			All laboratories shall use printable laboratory benchsheets. Certified Data shall be traceable to the associated sample analyses and shall consist of: any other data needed to reconstruct the final calculated result.
	PROCEDURE – Analysis	**LAB**	**SOP**	**EXPLANATION**
26	Is the pH of the wash water and standards adjusted to approximate that of the samples? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.4]			Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples.
27	Are ammonia standards analyzed in order of decreasing concentration of nitrogen? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.6]			Arrange ammonia standards in sampler in order of decreasing concentration of nitrogen. EPA Region IV recommended not changing the calibration standard order directed by the method. If the method goes through the detail of specifying a particular order, there is likely an underlying reason for having it in the method.
	QUALITY ASSURANCE	**LAB**	**SOP**	**EXPLANATION**
28	Has an MDL been established according to 40 CFR 136 Appendix B? [Method 350.1, Rev. 2.0 (1993), Section 9.2.4]			Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates.
29	Is ongoing MDL data being collected quarterly? [Procedure for the Determination of the Method Detection Limit, Rev. 2, (3) (a)]			During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked samples on each instrument, in separate batches, using the same spiking concentration used in Section 2.
30	Are MDL values verified at least every 13 months according to the ongoing MDL determination requirements and updated if necessary? [Procedure for the Determination of the Method Detection Limit, Rev. 2, (4) (a)]			At least once every thirteen months, re-calculate MDLs and MDLb from the collected spiked samples and method blank results using the equations in Section 2.
31	Is a standard curve produced consisting of a blank and 3 non-zero standards daily? [15A NCAC 2H .0805 (a) (7) (I)] [EPA Method 350.1, Rev. 2.0 (1993), Section 10.1]. **List values of standards used for Standard Curve.**			North Carolina Rule requires that the instrument be calibrated each day of use with a minimum of a blank and three non-zero standards. 350.1: Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solutions (Sections 7.12 and 7.13) to 100 mL with reagent water.
32	Does each standard curve have a correlation coefficient of ≥0.995? [NC WW/GW LC Policy]			When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of determination, r^2, of 0.99) is required.
33	Is each initial calibration verified before sample analysis with a second source standard? [15A NCAC 2H .0805 (a) (7) (H) (ii)] [EPA Method 350.1, Rev. 2.0 (1993), Section 10.7]			Rule: Laboratories shall analyze one known second source standard to verify the accuracy of standard preparation if an initial

	What is the concentration of the second source standard used for verification? **Answer:**			calibration is performed and in accordance with the referenced method requirements thereafter. 350.1: After the calibration has been established, it must be verified by the analysis of a suitable QCS. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards.
34	What is the acceptance criterion of the second source standard (QCS)? [EPA Method 350.1, Rev. 2.0 (1993), Section 10.7] **Answer:**			Must not exceed ±10% of the established QCS value.
35	What corrective action is taken if the second source standard varies by greater than 10%? [EPA Method 350.1, Rev. 2.0 (1993), Section 10.7] **Answer:**			If measurements exceed ±10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check.
36	Is a laboratory reagent blank (LRB) analyzed with each batch of samples? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.1]			The laboratory must analyze at least one LRB with each batch of samples. Definition of LRB (Section3.6): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
37	What is the acceptance criterion of the LRB and calibration blanks? [15A NCAC 2H .0805 (a) (7) (H) (i)] [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.1] **Answer:**			Rule: The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method. 9.3.1: Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.
38	What corrective action is taken if the blanks do not meet the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B)] **Answer:**			If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
39	Is a mid-range Instrument Performance Check (IPC) standard and calibration blank analyzed immediately following calibration, after every 10th sample, and at the end of sample analysis? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4]			For all determinations the laboratory must analyze the IPC (instrument performance check) (a mid-range check standard) and a calibration blank immediately following daily calibration, after every 10th sample (or more frequently, if required) and at the end of the sample run.
40	What is the acceptance criterion for the mid-range IPC standard? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4] **Answer:**			Analysis of the IPC solution immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%.

41	What corrective action is taken if the mid-range IPC standard recovery is not within specified limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4] **Answer:**			If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed.
42	Is a laboratory fortified blank (LFB) analyzed with each batch of samples? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2]			Definition of LFB (Section 3.4): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
43	What is the acceptance criterion for the LFB? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2] **Answer:**			Recovery within 90%- 110%
44	What corrective action is taken if the LFB does not meet specified limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2] **Answer:**			If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
45	Does the laboratory analyze duplicate samples at a rate of 5%? [15A NCAC 2H .0805 (a) (7) (C)]			Except where otherwise specified in an analytical method, laboratories shall analyze five percent of all samples in duplicate to document precision. Laboratories analyzing fewer than 20 samples per month shall analyze one duplicate during each month that samples are analyzed. **NOTE: A Laboratory Fortified Matrix Duplicate (LFMD) can satisfy our Rule requirement for a sample duplicate but should be analyzed at the same frequency as the LFM.**
46	What is the acceptance criterion for duplicates? [15A NCAC 2H .0805 (a) (7) (A)] **Answer:**			Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses.
47	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits or method precision limits? [15A NCAC 2H .0805 (a) (7) (B)] **Answer:**			If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
48	At what frequency is a Matrix Spike (MS) analyzed? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.1] **Answer:**			Also called Laboratory Fortified Matrix (LFM). The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples.
49	How is the MS prepared? [NC WW/GW LC Matrix Spike Technical Assistance.] **Answer:**			See Matrix Spike Technical Assistance document.

50	What is the acceptance criterion for MS recovery? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.2] **Answer:**			designated LFM recovery range 90-110%
51	What corrective action does the laboratory take if the MS results are outside of established control limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.3] **Answer:**			If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.
52	Is a lower reporting limit standard analyzed or back-calculated with each analysis? [15A NCAC 2H .0805 (a) (7) (H)]			Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day samples are analyzed.
53	What is the acceptance criterion for the lower reporting limit standard? [15A NCAC 2H .0805 (a) (7) (A)] **Answer:**			Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses.
54	What corrective action does the laboratory take if the lower reporting limit standard does not meet the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B)] **Answer:**			Recalibrate/re-verify the curve
55	Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)]			If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such. If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data.

Additional Comments:

Stock Standard – Dissolve 3.819 g anhydrous NH_4CL (dried at 100^0 C) in water and dilute to 1000 mL.
1.00mL = 1.00 mg N = 1.22 mg NH_3.

NOTE: Data is reported as NH_3 –N, that is Ammonia as Nitrogen, so 1.00 mL of stock standard equals 1 mg of Ammonia nitrogen

Additional Comments:

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Inspector: _____Date: _____