NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

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

Parameter: **Nitrogen, Nitrite**
Method: **Automated - Cadmium Reduction Bypassed**
NO_2 EPA 353.2, Rev. 2.0, 1993 (Aqueous)

Equipment & Reagents:

	Automated continuous-flow analyzer. **Make/Model:**		Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.
	Color reagent: To approximately 800 mL of reagent water, add, while stirring, 100 mL conc. phosphoric acid (CASRN 7664-38-2), 40 g sulfanilamide (CASRN 63-74-1) and 2 g N-1-naphthylethylenediamine dihydrochloride (CASRN 1465-25-4). Stir until dissolved and dilute to 1 L. Store in brown bottle and keep in the dark when not in use. This solution is stable for several months.		Reagent water: Because of possible contamination, this should be prepared by passage through an ion exchange column comprised of a mixture of both strongly acidic-cation and strongly basic-anion exchange resins. The regeneration of the ion exchange column should be carried out according to the manufacturer's instructions.
	Wash solution: Use reagent water for unpreserved samples. For samples preserved with H_2SO_4, use 2 mL H_2SO_4 (CASRN 7764-93-9), per liter of wash water.		Dilute hydrochloric acid, 6N: Add 50 mL of conc. HCl (CASRN 7647-01-0) to reagent water, cool, and dilute to 100 mL.
	Ammonium chloride-EDTA solution: Dissolve 85 g of reagent grade ammonium chloride (CASRN 12125-02-9) and 0.1 g of disodium ethylenediamine tetraacetate (CASRN 6381-92-6) in 900 mL of reagent water. Adjust the pH to 9.1 S.U. for preserved or 8.5 S.U. for non-preserved samples with conc. ammonium hydroxide (CASRN 1336-21-6) and dilute to 1 L. Add 0.5 mL Brij-35 (CASRN 9002-92-0).		Stock nitrate solution: Dissolve 7.218 g KNO_3 (CASRN 7757-79-1) and dilute to 1 L in a volumetric flask with reagent water. Preserve with 2 mL of chloroform (CASRN 67-66-3) per liter. Solution is stable for six months. 1 mL = 1.0 mg NO_3 -N.
	Stock nitrite solution: Dissolve 6.072 g KNO_2 in 500 mL of reagent water and dilute to 1 L in a volumetric flask. Preserve with 2 mL of chloroform and keep under refrigeration. 1.0 mL = 1.0 mg NO_2 -N.		Standard nitrite solution: Dilute 10.0 mL of stock nitrite solution to 1000 mL. 1.0 mL = 0.01 mg NO_2 -N. Solution is unstable; prepare as required.

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 collected and stored in polyethylene, Teflon®, or glass containers? [40 CFR 136 Table II]			
5	Are samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136 Table II]			
6	Are samples refrigerated above freezing but ≤ 6 º C during			

	storage? [40 CFR 136 Table II]			
7	Are samples analyzed within 48 hours of collection? [40 CFR 136 Table II]			
	PROCEDURE – Calibration	**LAB**	**SOP**	**EXPLANATION**
8	Is the continuous-flow manifold equivalent to that in the figure at the end of this checklist with the cadmium column bypassed? [EPA Method 353.2, Rev. 2.0 (1993), Section 10.2]			Set up manifold as shown in Figure 1. Bypass the cadmium column.
9	Is the instrument calibrated with at least three standards and one blank, each day? [EPA Method 353.2, Rev. 2.0 (1993), Section 10.1 and 9.3.4]			Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard nitrite solution (Section 7.13). 9.3.4 states: For all determinations the laboratory must analyze the IPC (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.
10	What are the concentrations of the standards used to calibrate the instrument? **Answer:**			
11	What is your laboratory's reporting limit? [15A NCAC 2H .0805 (a) (7) (H)] **Answer:**			For analytical procedures requiring analysis of a series of standards, the concentrations of these standards shall bracket the range of the sample concentrations measured. One of the standards shall have a concentration equal to or less than the laboratory's lowest reporting concentration for the parameter involved.
12	Are standards analyzed in order of decreasing concentration? [EPA Method 353.2, Rev. 2.0 (1993), Section 10.3]			Place appropriate standards in the sampler in order of decreasing concentration and perform analysis.
13	Is the correlation coefficient at least 0.995 (or a coefficient of determination, r^2, of 0.99)? [NC WW/GW LCB 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.
14	Is each initial calibration verified before sample analysis with a second-source Quality Control Standard (QCS)? [15A NCAC 2H .0805 (a) (7) (H) (ii)] [EPA Method 353.2, Rev. 2.0 (1993), Section 10.5] What is the concentration of the second-source standard used for verification? **Answer:**			Rule: Laboratories shall analyze one known second source standard to verify the accuracy of standard preparation if an initial calibration is performed and in accordance with the referenced method requirements thereafter. 353.2: 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.
15	What is the acceptance criterion of the second-source standard (QCS)? [15A NCAC 2H .0805 (a) (7) (B)] [EPA Method 353.2, Rev. 2.0 (1993), Section 10.5] **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.
16	What corrective action is taken if the second-source standard recovery is outside of established control limits? [15A NCAC 2H .0805 (a) (7) (B)] [EPA Method 353.2, Rev. 2.0 (1993), Section 10.5] **Answer:**			Rule: 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. 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. 353.2: 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.
	PROCEDURE – Interference Mitigation	**LAB**	**SOP**	**EXPLANATION**
17	Are samples high in particulate matter filtered prior to analysis? [EPA Method 353.2, Rev. 2.0 (1993), Section 4.1]			Buildup of suspended matter in the reduction column will restrict sample flow. Since nitrate and nitrite are found in a soluble state, samples may be pre-filtered.
18	Are samples with residual chlorine concentrations greater than 0.5 mg/L at a neutral pH treated with sodium thiosulfate prior to analysis? [EPA Method 353.2, Rev. 2.0 (1993), Section 4.2]			Residual chlorine can produce a negative interference by limiting reduction efficiency. Before analysis, samples should be checked and if required, dechlorinated with sodium thiosulfate.
	PROCEDURE – Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
19	If not already, is sample pH adjusted to between 5 and 9 S.U. with either conc. HCl or conc. NH_4OH? [EPA Method 353.2, Rev. 2.0 (1993), Section 11.1]			If sample pH is below 5 S.U. or above 9 S.U., adjust to between 5 and 9 S.U. with either conc. HCl or conc. NH_4OH. The acid/base must be sufficiently concentrated to not dilute the sample.
20	Are the initial and adjusted pH values documented? [15A NCAC 2H .0805 (a) (7) (E)]			All analytical records, including original observations and information necessary to facilitate historical reconstruction of the calculated results, shall be maintained for five years. All analytical data and records pertinent to each certified analysis shall be available for inspection upon request.
21	Is analysis performed according to manufacturer's instructions, and without the Cu-Cd reduction step? [EPA Method 353.2, Rev. 2.0 (1993), Sections 11.3 and 2.1]			Allow system to equilibrate as required. Obtain a stable baseline with all reagents, feeding reagent water through the sample line. Separate, rather than combined nitrate-nitrite values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.
	QUALITY ASSURANCE	**LAB**	**SOP**	**EXPLANATION**
22	Are MDLs determined prior to performing analyses by this method? [EPA Method 353.2, Rev. 2.0 (1993), Section 9.2.1 and 9.2.4] [EPA MDL Procedure, Rev. 2 (December 2016)]			The initial demonstration of performance is used to characterize instrument performance (determination of LCR and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method. Method Detection Limit (MDL) -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. 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.
23	Is ongoing quarterly MDL data being analyzed and documented? [EPA MDL Procedure, Rev. 2 (December 2016), Section (3) (a)]			MDL Procedure: 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. If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method (see Section 2(c) of this procedure), then this is an indication

				that the spiking level is not high enough and should be adjusted upward. Note that it is not necessary to analyze additional method blanks together with the spiked samples, the method blank population should include all of the routine method blanks analyzed with each batch during the course of sample analysis.
24	Is the MDL evaluated at least every 13 months and updated, if required? [EPA MDL Procedure, Rev. 2 (December 2016), Section (4)]			
25	Is a laboratory reagent blank (LRB) analyzed with each batch of samples? [EPA Method 353.2, Rev. 2.0 (1993), Section 9.3.1]			The laboratory must analyze at least one LRB with each batch of samples. Definition of LRB (Section 3.6): 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.
26	What is the acceptance criterion of the LRB and calibration blanks? [15A NCAC 2H .0805 (a) (7) (H) (i)] [EPA Method 353.2, Rev. 2.0 (1993), Section 9.3.1] **Answer:**			Rules: 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. 353.2: Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. May choose which criterion to follow. Must be established in the SOP.
27	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.
28	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 353.2, Rev. 2.0 (1993), Section 9.3.4]			For all determinations the laboratory must analyze the IPC (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.
29	What is the acceptance criterion for the mid-range IPC standard? [EPA Method 353.2, 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%.
30	What corrective action is taken if the mid-range IPC standard recovery is not within specified limits? [EPA Method 353.2, 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.
31	Is a Laboratory Fortified Blank (LFB) analyzed with each batch of samples? [EPA Method 353.2, 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.

32	What is the acceptance criterion for the LFB? [EPA Method 353.2, Rev. 2.0 (1993), Sections 9.3.2 and 9.3.3] **Answer:**			Recovery within 90%-110%, or optional control limits may be developed when sufficient internal data (usually a minimum of 20-30 analyses) become available. The optional control limits must be equal or better than the required control limits of 90-110%
33	What corrective action is taken if the LFB does not meet specified limits? [EPA Method 353.2, 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.
34	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.**
35	If no sample duplicate is analyzed, is a Laboratory Fortified Matrix Duplicate (LFMD) analyzed at a frequency of 5% of samples? [15A NCAC 2H .0805 (a) (7) (C)]			See Note above.
36	What is the acceptance criterion for duplicates or LFM/LFMD? [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.
37	What corrective action does the laboratory take if the duplicate samples/LFMD 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.
38	At what frequency is a Matrix Spike (MS) analyzed? [EPA Method 353.2, 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.
39	How is the MS prepared? [NC WW/GW LCB Matrix Spiking Policy and Technical Assistance] **Answer:**			Volume of spiking solution must not exceed 5% of the total volume.
40	If the volume of spike solution added to the sample is greater than 1% of the total volume, is the recovery calculation adjusted? [NC WW/GW LCB Policy]			It is preferable that the spike solution constitutes < 1% of the total MS volume so that the MS can be considered a whole volume sample with no adjustment (i.e., volume correction) by calculation necessary. If the spike solution volume constitutes >1% of the total sample volume, the sample concentration must be adjusted by calculation.
41	What is the acceptance criterion for MS recovery? [EPA Method 353.2, Rev. 2.0 (1993), Section 9.4.2] **Answer:**			Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%.
42	What corrective action does the laboratory take if the MS results are outside of established control limits? [EPA Method 353.2, Rev. 2.0 (1993), Section 9.4.3]			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

	Answer:			(Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.
43	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.
44	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.
45	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:**			
46	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:

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Inspector: _____Date:_____

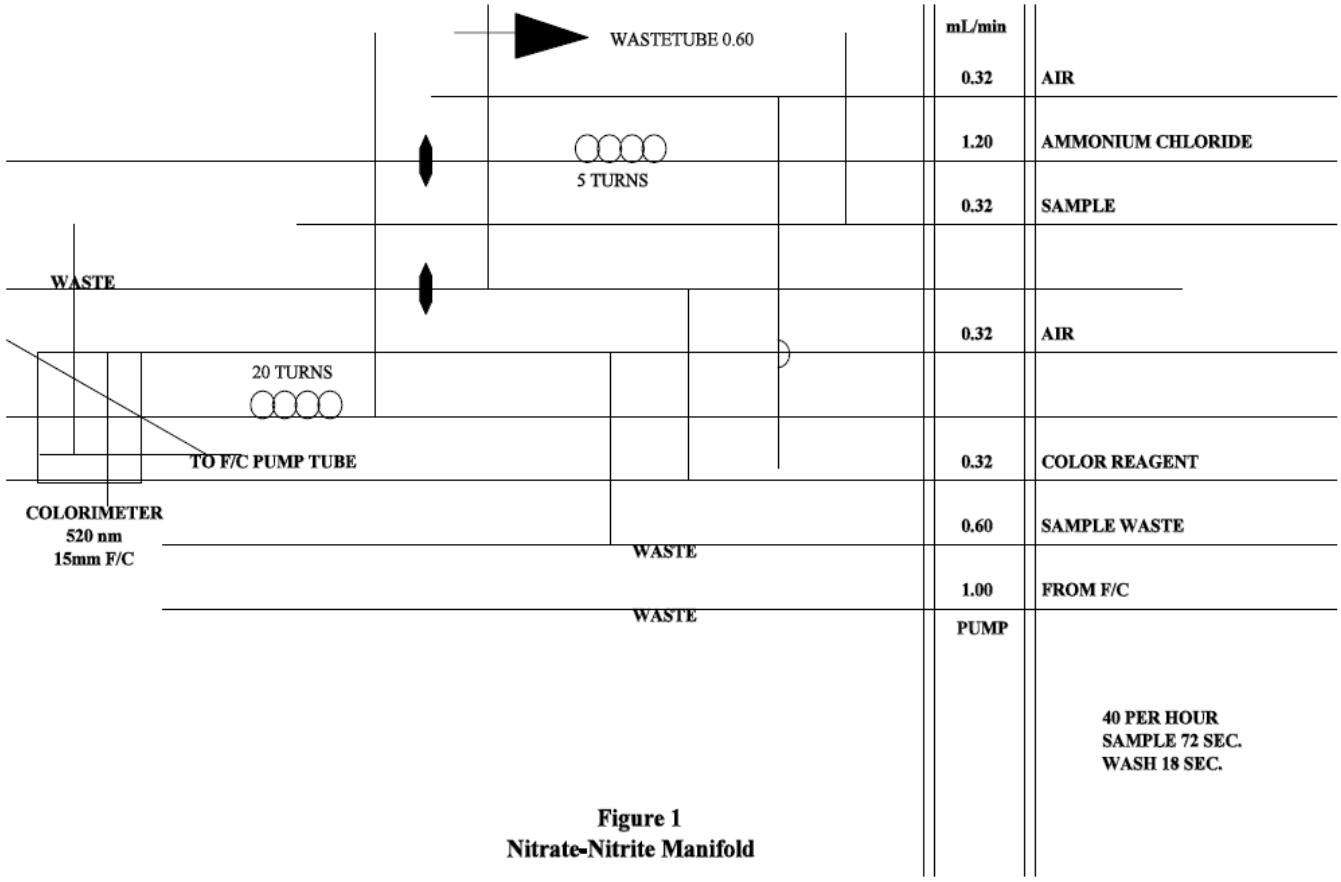

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Figure 1
Nitrate-Nitrite Manifold