

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: **Total Kjeldahl Nitrogen**
 Method: **Hach 10242, Rev. 1.2, Rev. 1.2 March 2022 (Aqueous)**
Also known as the Hach TNTplus 880 Method 10242, Rev. 1.2

EQUIPMENT:

	Block digester capable of maintaining a temperature of 120°C for 30 minutes.		Spectrophotometer (Model):

REAGENTS:

	Reagent water – Water in which TKN is not detected at or above the method level of this method. Bottled distilled water, or water prepared by passage of tap water through ion exchange and activated carbon have been shown to be acceptable sources of reagent water.		TNTplus Simplified TKN (s-TKN™) Reagent - Hach Catalog Number TNT880.
	Sulfuric Acid, ACS - Hach catalog Number 97949, or Equivalent.		Sodium Hydroxide, 5N - Hach Catalog Number 245053, or equivalent
	Method Detection Limit Ammonia Standard Solution – 1.0 mg/L as NH ₃ -N, Hach Catalog Number 189149, or equivalent.		Initial Precision and Recovery Ammonia Standard Solution – 1000 mg/L as NH ₃ -N, Hach Catalog Number 2354153, or equivalent
	Sulfamic Acid (if needed for interference mitigation)		

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	L A B	S O P	EXPLANATION
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 02H .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 02H .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	L A B	S O P	EXPLANATION
4	Are samples collected and stored in polyethylene, Teflon®, or glass containers? [40 CFR 136 Table II]			
5	Are samples preserved at time of collection with H ₂ SO ₄ to pH of <2 S.U.? [40 CFR 136 Table II]			
6	Are samples iced to above freezing but ≤ 6 ° C during shipment? [40 CFR 136 Table II]			
7	Are samples refrigerated above freezing but ≤ 6 ° C during storage? [40 CFR 136 Table II]			
8	Are samples analyzed within 28 days of collection? [40 CFR 136 Table II]			

	INTERFERENCES	L A B	S O P	EXPLANATION
9	Are samples containing Nitrites in excess of 2.0 mg/L treated with 50 mg of sulfamic acid per 5.0 mL of sample, and allowed to sit for 10 minutes after the sulfamic acid has dissolved? [Hach 10242, Rev. 1.2, Section 3.2]			Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid to 5.0 mL of sample, dissolve, and wait for 10 minutes. Analyze the prepared sample as described in Section 11. High levels of oxidizable organic substances (COD) affect the reagent color and give high results.
	ANALYTICAL PROCEDURE	L A B	S O P	EXPLANATION
10	Is 1.3 mL of sample, 1.3 mL of Solution A and 1 Reagent B tablet added in quick succession to a dry 20-mm reaction tube? [Hach 10242, Rev. 1.2, Section 11.3.1]			Add 1.3 mL of sample, 1.3 mL of Solution A and 1 Reagent B tablet in quick succession to a dry 20-mm reaction tube. Close the reaction tube immediately.
11	After combining the sample and reagents, is care taken not to invert the reaction tube? [Hach 10242, Rev. 1.2, Section 11.3.1]			Do not invert tube.
12	Are reaction tubes placed in the 120 °C block digester and heated for 30 minutes? [Hach 10242, Rev. 1.2, Section 11.3.2]			Insert the reaction tube in the reactor and heat for 30 minutes.
13	After 30 minutes, are the tubes removed from the heating block and cooled to room temperature? [Hach 10242, Rev. 1.2, Section 11.3.3]			After 30 minutes, remove the reaction tube from the heating block and cool to room temperature (15-20 °C).
14	Is 0.5 mL of the digested sample pipetted from the reaction tube into a Test Vial 1 (red label)? [Hach 10242, Rev. 1.2, Section 11.3.4]			Pipette 0.5 mL of the digested sample from the reaction tube into a Test Vial 1 (red label).
15	Is 0.2 mL of Solution D pipetted into the test vial 1? [Hach 10242, Rev. 1.2, Section 11.3.5]			Pipette 0.2 mL of Solution D into the test vial.
16	Is the vial quickly capped and inverted 2-3 times until no more streaks can be seen in the vial solution? [Hach 10242, Rev. 1.2, Section 11.3.5]			Quickly cap and invert the test vial 2-3 times until no more streaks can be seen in the vial solution.
17	Is 1.0 mL of undigested sample pipetted into a Test Vial 2 (green label)? [Hach 10242, Rev. 1.2, Section 11.3.6]			Pipette 1.0 mL of undigested sample into a Test Vial 2 (green label).
18	Is 0.2 mL of Solution D pipetted into the test vial 2? [Hach 10242, Rev. 1.2, Section 11.3.7]			Pipette 0.2 mL of Solution D into the test vial.
19	Is the vial quickly capped and inverted 2-3 times until no more streaks can be seen in the vial solution? [Hach 10242, Rev. 1.2, Section 11.3.7]			Quickly cap and invert the test vial 2-3 times until no more streaks can be seen in the vial solution and let react for 15 minutes.
20	Is the sample then allowed to react for 15 minutes? [Hach 10242, Rev. 1.2, Section 11.3.7]			See above.
21	After 15 minutes, is Test Vial 1 wiped clean and inserted into the cell holder of the spectrophotometer and a display reading of "E1" observed? [Hach 10242, Rev. 1.2, Section 11.3.8]			After 15 minutes, wipe the Test Vial 1 with a clean tissue or cloth and insert the prepared vial into the cell holder of the spectrophotometer. The instrument will read the barcode on the Test Vial 1 and display E1. Remove the vial and proceed immediately to step 11.3.9.
22	Is Test Vial 1 then immediately removed and a wiped-clean Test Vial 2 inserted? [Hach 10242, Rev. 1.2, Section 11.3.9]			Wipe the Test Vial 2 with a clean tissue or cloth and insert the prepared vial into the cell holder of the spectrophotometer. The instrument will read the barcode on the Test Vial 2.
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
23	Has an initial MDL study been performed according to 40 CFR 136, Appendix B? [Hach 10242, Rev. 1.2, Section 9.2.1]			Method Detection Limit (MDL) - To establish the ability to detect nitrate the analyst shall determine the MDL per the procedure in 40 CFR 136, Appendix B using the apparatus, reagents, and standards used in the practice of this method.
24	Was the determined MDL \leq 0.08 mg/L NH ₃ -N as TKN? [Hach 10242, Rev. 1.2, Section 9.2.1] List current MDL:			
25	Is the Minimum Level calculated per Section 9.2.1 of the method shown to be \leq 1 mg/L NH ₃ -N as TKN? [Hach 10242, Rev. 1.2, Section 9.2.1] List calculated Minimum Level:			The analyst also shall calculate the Minimum Level (ML) of quantitation by multiplying the MDL by 3.18 and rounding to the number nearest to (1,2 or 5) x 10 ⁿ , where n is a positive or negative integer. The calculated MDL should be less than or equal to the MDL

			<p>in Section 13.0 prior to the practice of this method. Similarly, the calculated ML should be less than or equal to the ML in Section 13.0</p> <p>Note that the ML is not the reporting limit. The reporting limit is base lowest calibration concentration.</p>
26	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.
27	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 Procedure, Rev. 2, (4)]		The verified MDL is the greater of the MDL _s or MDL _b . If the verified MDL is within 0.5 to 2.0 times the existing MDL, and fewer than 3% of the method blank results (for the individual analyte) have numerical results above the existing MDL, then the existing MDL may optionally be left unchanged. Otherwise, adjust the MDL to the new verification MDL. (The range of 0.5 to 2.0 approximates the 95 th percentile confidence interval for the initial MDL determination with six degrees of freedom.)
28	<p>Were 4 replicate Initial Precision and Recovery standards analyzed and the average percent recovery and RSD calculated per 9.2.2.3 prior to use of the method? [Hach 10242, Rev. 1.2, Section 9.2.2 (9.2.2.1 through 9.2.2.4)]</p> <p>IPR RSD:</p> <p>IPR recovery:</p>		
29	If the average percent recovery and RSD of the replicate IPR standards do not meet method acceptance criteria, is the problem corrected and the test repeated? [Hach 10242, Rev. 1.2, Section 9.2.2.5]		If, however, the RSD exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable. In this event correct the problem and repeat the test.
30	<p>Is the factory-set calibration curve verified with a series of five or more non-zero standards at least every twelve months? [15A NCAC 02H .0805 (a) (7) (H) (v)]</p> <p>List Standard concentrations:</p> <p>List Reporting Limit:</p>		For colorimetric analyses, a series of five or more non-zero standards for a curve prepared every twelve months or three or more non-zero standards for curves established each day, or standards as set forth in the analytical procedure, shall be analyzed to establish a calibration curve. <u>A manufacturer's factory-set calibration (internal curve) shall be verified with the same number of standards and frequency as a prepared curve.</u>
31	<p>Skip this question if the answer to the previous question was, yes.</p> <p>Is the factory-set calibration curve verified with a series of three or more non-zero standards each day? [15A NCAC 02H .0805 (a) (7) (H) (v)]</p> <p>List Standard concentrations:</p> <p>List Reporting Limit:</p>		
32	Is a 1.0 mg/L (or reporting limit standard if lower than 1.0 mg/L) and 10.0 mg/L NH ₃ -N standard analyzed each day? [Hach 10242, Rev. 1.2, Section 10.2.1] [15A NCAC 02H .0805 (a) (7) (H)]		<p>To verify that the instrument is measuring TKN properly, analyze a 1.0 mg/L and 10.0 mg/L NH₃-N standard.</p> <p>Perform this calibration verification daily while instrument is in use.</p> <p>Rule: Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day samples are analyzed.</p>
33	Is a calibration blank and calibration verification standard analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group? [15A NCAC 02H .0805 (a) (7) (H)]		A calibration blank and calibration verification standard shall be analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group, unless otherwise specified by the method, to check for carryover and calibration drift.
34	Do the calibration verification standards recover between 90-110%? [Hach 10242, Rev. 1.2, Section 10.2.1]		
35	What corrective action is taken if daily calibration verification standards vary by greater than 10% from the true value? [15A NCAC 02H .0805 (a) (7) (B)]		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.
36	Is at least one LRB analyzed with each batch of samples? [Hach 10242, Rev. 1.2, Section 9.3]		The laboratory reagent blank (LRB) is an aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment and reagents that are used with other samples. The laboratory must analyze at least one LRB with each batch of samples.
37	Are the values of all blanks $\leq \frac{1}{2}$ the reporting limit? [15A NCAC 02H .0805 (a) (7) (H) (i)]		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. They may also use the requirement set by the method (only choose one requirement to follow and specify in the SOP).
38	What corrective action is taken if the LRB or calibration blanks do not meet the established criteria? [15A NCAC 02H .0805 (a) (7) (B)]		Check for contamination, check viability of low standard, re-analyze blank, re-digest and reanalyze entire batch or qualify the data.
39	Is an Ongoing Precision and Recovery (OPR) standard at a concentration of 5 mg/L NH ₃ -N analyzed at the end of each analytical batch? [Hach 10242, Rev. 1.2, Section 9.4.1]		Prepare a precision and recovery standard following the procedure in Section 9.2.2 and analyze at the end of each analytical batch according to the procedure in Section 11.
40	If the OPR standard recovery is not within the acceptable range of 90 -110%, is the problem corrected and the batch re-analyzed, including repeating the ongoing precision and recovery standard analysis? [Hach 10242, Rev. 1.2, Section 9.4.1.1]		If the recovery is within the acceptable range of 90 -110%, measurement process is in control and analysis of samples may proceed. If, however, the recovery is not in the acceptable range, the analytical process is not in control. In this event, correct the problem, re-analyze analytical batch, repeating the ongoing precision and recovery test.
41	Are at least 5% of the samples from each analytical batch spiked in duplicate? [Hach 10242, Rev. 1.2, Section 9.5]		Matrix Spike and Matrix Spike Duplicate Precision and Recovery (MS/MSD) – The laboratory must, on an ongoing basis, spike at least 5% of the samples from each analytical batch as defined in Section 9.4. An analytical batch is a set of samples processed during a contiguous 8-hour period, not to exceed 20 samples.
42	Are matrix spike recoveries within the limits of 90 – 100%? [Hach 10242, Rev. 1.2, Sections 9.5.5, 9.5.5.1 and 17, Table 3]		Calculate each percent recovery (P) as $100 (A-B) \% / T$, where A is the concentration of TKN in the spiked samples and T is the known true value of the spike. Compare the percent recovery (P) TKN with the corresponding QC acceptance criteria found in Section 17, Table 3.
43	What corrective action does the laboratory take if the spike recoveries are outside of established acceptance limits? [15A NCAC 02H .0805 (a) (7) (B)]		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.
44	Is the RPD between the spike and spike duplicate within the limit of 20%? [Hach 10242, Rev. 1.2, Sections 9.5.6, 9.5.6.1 and 17, Table 3]		Calculate the relative percent difference (RPD) between two sample results using the following equation: $RPD = \frac{ D_1 - D_2 }{(D_1 + D_2) / 2} \times 100$ Where, D1 = Concentration of analyte in the MS, D2 = Concentration of analyte in the MSD. Compare the calculated RPD with the corresponding QC acceptance criteria found in Section 17, Table 3.
45	What corrective action does the laboratory take if the RPD is outside of acceptance criterion? [15A NCAC 02H .0805 (a) (7)(B)]		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.

46	Are results qualified to indicate quality control failures or sample anomalies? [15A NCAC 02H .0805 (e) (5)]		Reported data associated with Quality Control failures, improper sample collection, holding time exceedances, or improper preservation shall be qualified as such.
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Additional Comments:

Inspector: _____ Date: _____