NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION

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

Parameter: **Alkalinity**
Method: **Standard Methods 2320 B-2011 (Aqueous)**

Equipment:

	Burette
	Electrometric Titrator
	pH Meter with glass electrode
	Pipets, Volumetric
	Flasks, Volumetric
	Magnetic Stirrer

Reagents:

	Sodium carbonate solution, 0.05*N*
	NaOH, 0.1*N*
	H_2SO_4 or HCl, 0.1*N*
	NaOH, 0.02*N*
	H_2SO_4 or HCl, 0.02*N*
	Bromcresol green indicator solution, pH 4.5 indicator
	Mixed bromcresol green-methyl red indicator solution, either aqueous or alcoholic solution
	Phenolphthalein indicator solution, alcoholic, pH 8.3 indicator
	Metacresol purple indicator solution, pH 8.3 indicator

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 revision dates and actions 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 iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136.3 Table II and footnote 18]			40 CFR footnote 2 allows 15 minutes for sample preservation, including thermal. This means that if a sample is received in the lab within 15 minutes it is not required to be on ice. Document temperature downward trend for short transport samples.
5	Are samples refrigerated above freezing to 6°C during storage? [40 CFR 136.3 Table II and footnote 18]			
6	Are samples analyzed within 14 days of collection? [40 CFR 136.3 Table II]			

	PROCEDURE	LAB	SOP	EXPLANATION
7	Is a titration curve constructed for standardization of reagents? [SM 2320 B (1) (d)-2011]			
8	Is 0.1 *N* H_2SO_4 (or HCl) standardized against 40.00 mL of standard 0.05 *N* sodium carbonate solution? [SM 2320 B (3) (b)-2011]			Prepare acid solution of approximately 0.1N and determine exact *N* as follows. Standardize against 40.00 mL 0.05 *N* Na_2CO_3 solution, with about 60 mL water, in a beaker by titrating potentiometrically to pH of about 5. Lift out electrodes rinse into the same beaker, and boil gently for 3 to 5 min under a watch glass cover. Cool to room temperature, rinse cover glass into beaker, and finish titrating to the pH inflection point. Note: purchased reagents do not have to be standardized initially, only monthly
9	Is 0.02 *N* H_2SO_4 (or HCl) standardized against 15.00 mL of standard 0.05 *N* sodium carbonate? [SM 2320 B (3) (c)-2011]			Standardize by potentiometric titration of 15.00 mL 0.05N Na_2CO_3 according to the procedure of ¶ 3b. Note: purchased reagents do not have to be standardized initially, only monthly
10	How long is the standard 0.05N sodium carbonate solution stored? [SM 2320 B (3) (a)-2011] ANSWER:			Do not store longer than a week
11	Is standard sodium carbonate dried 4h @ 250 °C and cooled in a desiccator prior to preparing the solution? [SM 2320 B (3) (a)-2011]			
12	How is normality of H_2SO_4 titrant calculated? [SM 2320 B (3) (b) – 2011] ANSWER:			Normality, *N* = $\frac{A \times B}{53.00 \times C}$. where: A= g Na_2CO_3 weighted into 1-L flask B= mL Na_2CO_3 solution taken for titration, and C= mL acid used. If using Tris or THAM instead of Na_2CO_3 *N* of H_2SO_4 = $\frac{(\text{Tris g})\ (1000)}{(\text{mL } H_2SO_4)\ (\text{eq. wt. Tris})}$. 121.1 = eq. wt. Tris, (Tris = THAM)
13	Is the calculated normality used to determine Alkalinity?			
14	If the calculated normality is not used, is the titrant normality then adjusted to 0.1000 *N*? [SM 2320 B (3) (b)-2011]			1 mL 0.1000 *N* solution = 5.00 mg CaCO3.
15	How is sample size and normality of titrant determined? [SM 2320 B (1) (e)-2011] ANSWER:			SM 2320 B (1) (e): See Section 2310B.1e for selection of size sample to be titrated and normality of titrant, substituting 0.02N or 0.1N sulfuric (H_2SO_4) or hydrochloric (HCl) acid for the standard alkali of that method. For the low-alkalinity method, titrate a 200-mL sample with 0.02N H_2SO_4 from a 10-mL buret. SM 2310 B (1) (e): Use a sufficiently large volume of titrant (20 mL or more from a 50-mL buret) to obtain relatively good volumetric precision while keeping sample volume sufficiently small to permit sharp end points. For samples having acidities less than about 1000 mg as calcium

				carbonate ($CaCO_3$)/L, select a volume with less than 50 mg $CaCO_3$ equivalent acidity and titrate with 0.02N sodium hydroxide (NaOH). For acidities greater than about 1000 mg as $CaCO_3$/L, use a portion containing acidity equivalent to less than 250 mg $CaCO_3$ and titrate with 0.1N NaOH. If necessary, make a preliminary titration to determine optimum sample size and/or normality of titrant.
16	Is sample filtered, diluted, concentrated, or altered? [SM 2320 B (1) (c)-2011]			Do not filter, dilute, concentrate, or alter sample.
17	Is sample titrated at room temperature? [SM 2320 B (1) (d)-2011]			Titrate at room temperature.
18	What method is used to determine alkalinity? [SM 2320 B (4) -2011] ☐ Potentiometric titration curve ☐ Potentiometric titration to preselected pH ☐ color indicators			Titrate at room temperature with a properly calibrated pH meter or electrically operated titrator, or use color indicators. Color indicators may be used for routine and control titrations in the absence of interfering color and turbidity.
19	If using color indicators is an indicator blank titrated? [SM 2320 B (1) (d)-2011]			If using color indicators, prepare and titrate an indicator blank.
20	If using color indicators, is the sample checked for free residual chlorine, and if present, treated with $Na_2S_2O_3$? [SM 2320 B (4) (a)-2011]			SM 2320 B (4) (a): color change: see Section 2310B.4b SM 2310 B (4) (b) If free residual chlorine is present add 0.05 mL (1 drop) 0.1 *M* $Na_2S_2O_3$ solution, or destroy with ultraviolent radiation.
21	Is the pH meter or electric titrator calibrated before use? [SM 2320 B (4) -2011]			
22	Is the pH meter or electric titrator readable to 0.05 pH units? [SM 2320 B (2)-2011]			
23	If titrating to preselected pH, is the appropriate endpoint selected from Table 2320: I? [SM 2320 B (2) (c)-2011] **State endpoint(s) used:**			Total Alkalinity, endpoint is 4.9 if 30 mg/L alkalinity, 4.6 if 150 mg/L, or 4.3 if 500 mg/L. May be 4.5 if automated or if industrial waste or complex system.
24	What calculation is used to determine total alkalinity? [SM 2320 B (5) (a) -2011]			Alkalinity, mg/L $CaCO_3$ = $\frac{(A \times N \times 5000)}{mL\ sample}$ where: A = ml titrant and *N* = normality of titrant or Alkalinity, mg/L $CaCO_3$= $\frac{(A \times t \times 1000)}{mL\ sample}$ where: t=titer of standard acid, mg $CaCO_3$ / mL
25	Is the low alkalinity method used for alkalinities less than 20 mg $CaCO_3$/L? [SM 2320 B (1) (d)-2011]			Report alkalinity less than 20 mg $CaCO_3$/L only if it has been determined by the low-alkalinity method of 2320 B.4*d.*
26	What sample volume is used for low alkalinity determination? [SM 2320 B (4) (d) -2011] ANSWER:			100 – 200 mL
27	For low alkalinity determination, is titration stopped at a pH in the range 4.3 to 4.7 s.u. and the volume and exact pH recorded? [SM 2320 B (4) (d)-2011]			
28	Is additional titrant then added to reduce the pH exactly 0.30 pH unit			

	and volume recorded? [SM 2320 B (4) (d)-2011]			
29	What calculation is used for low alkalinity determination? [SM 2320 B (4) (d) -2011]			Alkalinity, mg/L $CaCO_3$ = $\frac{(2B - C) \times N \times 50\,000)}{\text{mL sample}}$ where: B = ml titrant to first recorded pH C= total mL titrant to reach pH 0.3 unit lower, and N= normality of acid
	QUALITY ASSURANCE			**EXPLANATION**
30	Is the purchased or prepared titration reagent re-standardized monthly? [SM 2020 B (2) (b)-2010]			Re-standardized reagents once a month or when the improper storage occurs. If the titration reagent's normality (titer value) has changed, then use the measured value, adjust the normality (titer value) as the procedure describes, or prepared and standardized fresh titration reagent as needed.
31	Does the laboratory analyze a laboratory-fortified blank (LFB) at least daily or per batch of 20 or fewer samples? [SM 2020 B (2) (e)-2010]			Table 2020:II indicates methods in part 2000 where use of LFB is considered appropriate. Include at least one LFB daily or per each batch of 20 or fewer samples.
32	What is the value and acceptance criterion for the LFB? [SM 2020 B (2) (e)-2010] ANSWER:			Calculate percent recovery, plot control charts, and determine control limits for these measurements to demonstrate ongoing capability.
33	What corrective action is taken if the LFB does not meet the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (F)] [SM 2020 B (2) (e)-2010] ANSWER:			Our Rule requires corrective action any time quality control results indicate a problem. **SM states**: Establish corrective actions to take if the LFB does not satisfy acceptance criteria.
34	Is a duplicate sample analyzed daily? [SM 2020 B (2) (f)-2010]			When appropriate (Table 2020:II), randomly select routine samples to be analyzed twice. Include at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
35	What is the acceptance criterion for the duplicates? [SM 2020 B (2) (f)-2010] ANSWER:			Calculate control limits for duplicates when method-specific limits are not provided.
36	What corrective action does the laboratory take if duplicates 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.
37	Is an Initial Demonstration of Capability (IDC) on file for each analyst? [SM 2020 B (1) (a)-2010]			Before new analysts run any samples, verify their capability with the method. Run a LFB, performance evaluation sample, or standard with a known or otherwise certifiable concentration at least four times and compare results to the limits listed in the method or those established by the laboratory. Current analysts grandfathered in.

38	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.

Additional Comments:

Inspector: _____ Date: _____