

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: **Alkalinity**

Method: **Standard Methods 2320 B-2021 (Aqueous)**

**This checklist does not address determination of Alkalinity by titration curve**

Equipment:

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

Reagents:

Sodium carbonate solution, 0.05N
NaOH, 0.1N
H <sub>2</sub> SO <sub>4</sub> or HCl, 0.1N
NaOH, 0.02N
H <sub>2</sub> SO <sub>4</sub> or HCl, 0.02N
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	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)]  <b>Date:</b>			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 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 iced to above freezing but ≤6 °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 but ≤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	L A B	S O P	EXPLANATION
7	Is 0.1 N H <sub>2</sub> SO <sub>4</sub> (or HCl) standardized against 40.00 mL of standard 0.05 N sodium carbonate solution? [SM 2320 B-2021 (3) (b)]			Prepare acid solution of approximately 0.1N and determine exact N as follows. Standardize against 40.00 mL 0.05 N Na <sub>2</sub> CO <sub>3</sub> 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
8	Is 0.02 N H <sub>2</sub> SO <sub>4</sub> (or HCl) standardized against 15.00 mL of standard 0.05 N sodium carbonate? [SM 2320 B-2021 (3) (c)]		Standardize by potentiometric titration of 15.00 mL 0.05N Na <sub>2</sub> CO <sub>3</sub> according to the procedure of ¶ 3b.  Note: purchased reagents do not have to be standardized initially, only monthly
9	How long is the standard 0.05N sodium carbonate solution stored? [SM 2320 B-2021 (3) (a)]  <b>Answer:</b>		Do not store longer than a week
10	Is standard sodium carbonate dried 4h @ 250 °C and cooled in a desiccator prior to preparing the solution? [SM 2320 B-2021 (3) (a)]		
11	How is normality of H <sub>2</sub> SO <sub>4</sub> titrant calculated? [SM 2320 B-2021 (3) (b)]  <b>Answer:</b>		Normality, $N = \frac{A \times B}{53.00 \times C}$  where: A= g Na <sub>2</sub> CO <sub>3</sub> weighted into 1-L flask B= mL Na <sub>2</sub> CO <sub>3</sub> solution taken for titration, and C= mL acid used.  <u>If using Tris or THAM instead of Na<sub>2</sub>CO<sub>3</sub></u>  $N \text{ of H}_2\text{SO}_4 = \frac{(\text{Tris g}) (1000)}{(\text{mL H}_2\text{SO}_4) (\text{eq. wt. Tris})}$  121.1 = eq. wt. Tris, (Tris = THAM)
12	Is the calculated normality used to determine Alkalinity? [SM 2320 B-2021 (3) (b)]		
13	If the calculated normality is not used, is the titrant normality then adjusted to 0.1000 N? [SM 2320 B-2021 (3) (b)]		1 mL 0.1000 N solution = 5.00 mg CaCO <sub>3</sub> .
14	How is sample size and normality of titrant determined? [SM 2320 B -2021 (1) (e)]  <b>Answer:</b>		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 <sub>2</sub> SO <sub>4</sub> ) 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 <sub>2</sub> SO <sub>4</sub> 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 <sub>3</sub> )/L, select a volume with less than 50 mg CaCO <sub>3</sub> equivalent acidity and titrate with 0.02N sodium hydroxide (NaOH). For acidities greater than about 1000 mg as CaCO <sub>3</sub> /L, use a portion containing acidity equivalent to less than 250 mg CaCO <sub>3</sub> and titrate with 0.1N NaOH. If necessary, make a preliminary titration to determine optimum sample size and/or normality of titrant.
15	Is sample filtered, diluted, concentrated, or altered? [SM 2320 B-2021 (1) (c)]		Do not filter, dilute, concentrate, or alter sample.
16	Is sample titrated at room temperature? [SM 2320 B-2021 (1) (d)]		Titrate at room temperature.
17	What method is used to determine alkalinity? [SM 2320 B-2021 (4)] <input type="checkbox"/> Potentiometric titration to preselected pH <input type="checkbox"/> 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.
18	If using color indicators is an indicator blank titrated? [SM 2320 B-2021 (1) (d)]		If using color indicators, prepare and titrate an indicator blank.

19	If using color indicators, is the sample checked for free residual chlorine, and if present, treated with Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> ? [SM 2320 B-2021 (4) (a)]		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 <sub>2</sub> S <sub>2</sub> O <sub>3</sub> solution, or destroy with ultraviolet radiation.
20	Is the pH meter or electric titrator calibrated before use? [SM 2320 B-2021 (4)]		
21	Is the pH meter or electric titrator readable to 0.05 pH units? [SM 2320 B-2021 (2)]		
22	If titrating to preselected pH, what endpoint is used? [SM 2320 B-2021 (1) (b)]  <b>State endpoint(s) used:</b>		Table 2320:1 includes suggested endpoint pH values. 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 routine or automated or if industrial waste or complex system.
23	What calculation is used to determine total alkalinity? [SM 2320 B-2021 (5) (a)]  <b>Answer:</b>		Alkalinity, mg CaCO <sub>3</sub> /L = $\frac{(A \times N \times 50,000)}{\text{mL sample}}$  where: A = mL standard acid used, and N = normality of standard acid  or Alkalinity, mg CaCO <sub>3</sub> /L = $\frac{(A \times t \times 1000)}{\text{mL sample}}$  where: t=titer of standard acid, mg/mL CaCO <sub>3</sub>
24	Is the low alkalinity method used for alkalinities less than 20 mg CaCO <sub>3</sub> /L? [SM 2320-2021 B (1) (d)]		Report alkalinity less than 20 mg/L CaCO <sub>3</sub> only if it has been determined by the low-alkalinity method of 2320 B.4d.
25	What sample volume is used for low alkalinity determination? [SM 2320 B-2021 (4) (d)]  <b>Answer:</b>		100 – 200 mL
26	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-2021 (4) (d)]		
27	Is additional titrant then added to reduce the pH exactly 0.30 pH unit and volume recorded? [SM 2320 B-2021 (4) (d)]		
28	What calculation is used for low alkalinity determination? [SM 2320 B-2021 (5) (b)]  <b>Answer:</b>		Alkalinity, mg/L CaCO <sub>3</sub> =  $\frac{(2B - C) \times N \times 50,000}{\text{mL sample}}$  where: B = mL titrant to first recorded pH, C = total mL titrant to reach pH 0.3 unit lower, N = normality of acid, and 50 000 = equivalent weight of CaCO <sub>3</sub> (mg).
<b>QUALITY ASSURANCE</b>			<b>EXPLANATION</b>
29	Is the purchased or prepared titration reagent re-standardized monthly? [SM 2020 B-2021 (2) (b)]		Re-standardized reagents once a month or when 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 prepare and standardized fresh titration reagent as needed.
30	Does the laboratory analyze a laboratory-fortified blank (LFB) at least daily or per batch of 20 or fewer samples? [SM 2020 B-2021 (2) (e)]		Table 2020:2 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.

31	What is the value and acceptance criterion for the LFB? [SM 2020 B-2021 (2) (e)]  <b>Answer:</b>		Calculate percent recovery, plot control charts, and determine control limits for these measurements to demonstrate ongoing capability.
32	What corrective action is taken if the LFB does not meet the acceptance criterion? [15A NCAC 02H .0805 (a) (7) (F)] [SM 2020 B-2021 (2) (e)]  <b>Answer:</b>		Our Rule requires corrective action any time quality control results indicate a problem. <b>SM states:</b> Establish corrective actions to take if the LFB does not satisfy acceptance criteria.
33	Is a duplicate sample analyzed daily? [SM 2020 B-2021 (2) (f)]		When appropriate (Table 2020:2), 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.
34	What is the acceptance criterion for the duplicates? [SM 2020 B-2021 (2) (f)]  <b>Answer:</b>		Calculate control limits for duplicates when method-specific limits are not provided.
35	What corrective action does the laboratory take if duplicates do not meet the acceptance criterion? [15A NCAC 02H .0805 (a) (7) (B)]  <b>Answer:</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 an Initial Demonstration of Capability (IDC) on file for each analyst? [SM 2020 B-2021 (1) (a)]		Analysts must demonstrate their capability to use a method before analyzing any samples for the first time using that method. For methods in which bias is applicable (see Table 2020:1), run an 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.
37	Are results qualified to indicate quality control failures or sample anomalies when reporting results? [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.

Additional Comments:

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