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: **Residue, Total (Aqueous)**
Method: **Standard Methods 2540 B-2020**

Total Residue is considered a method-defined parameter per the definition in the Code of Federal Regulations, Part 136.6, Section (a) (5). This means that the method may not be modified per Part 136.6, Section (b) (3).

EQUIPMENT:

	Evaporating Dishes 100 mL capacity, 90 mm diameter made of Porcelain, Platinum, high silica glass, StableWeigh, Environmental Express, Charleston, SC, or equivalent.		Wide-bore pipets, Class B in glass, mechanical, or electronic (not required)
	Drying oven, for operation at 103 to 105 ºC		Beaker (not required)
	Desiccator, provided with desiccant containing a color indicator of moisture concentration or an instrumental indicator		Steam bath (not required)
	Analytical balance, capable of weighing 0.1 mg (0.0001g)		Magnetic stirrer with TFE stirring bar (optional)
	Anti-static Device		Blender or homogenizer (optional).
	Graduated Cylinder, Class A		

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 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 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	**LAB**	**SOP**	**EXPLANATION**
4	Are samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136.3 Table II]			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 ºC during storage? [40 CFR 136.3 Table II]			
6	Are samples analyzed within 7 days of collection? [40 CFR 136.3 Table II]			
	PROCEDURE - Sample Preparation	**LAB**	**SOP**	**EXPLANATION**
7	Are StableWeigh™ or equivalent bags being used? If not, skip to question 9.			
8	What type of anti-static device is being used in conjunction with the analytical balance? [NC WW/GW LCB Anti-Static Device Requirement for Total and Total Dissolved Residue Policy]			Plastic type bags have been known to hold a static charge, which may cause weight fluctuations on an analytical balance. When using StableWeigh™, or equivalent bags for Total and/or Total Dissolved Residue, anti-static devices must be in place. This

	ANSWER:			is important for achieving the method specified constant weight requirement of ± 0.5mg.
9	Are evaporating dishes cleaned and dried for at least one hour at 103-105 ºC prior to use? [SM 2540 B-2020 (3) (a)]			If only measuring total solids, then heat clean dish at 103–105°C for ≥1 h.
10	Are evaporating dishes cooled and stored in a desiccator until needed? [SM 2540 B-2020 (3) (a)]			Cool dishes to ambient temperature and weigh. Store weighed dishes in desiccator or oven until needed.
11	Are evaporating dishes weighed before use? [SM 2540 B-2020 (3) (a)]			See above.
	PROCEDURE - Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
12	Are samples well mixed prior to analysis? [SM 2540 B-2020 (1) (a) and (3) (c)]			Samples may be mixed by shaking in sample bottle, blending in a mixer or stirring with magnetic stirrer.
13	How is the sample volume measured? [SM 2540 B-2020 (3) (c)] **Answer:**			Stir or mix sample and quantitatively transfer with a pipet or graduated cylinder to a pre-weighed dish.
14	Are evaporated samples dried for at least one hour in an oven at 103 to 105 ºC? [SM 2540 B-2020 (3) (c)]			Dry evaporated sample for ≥1 h in a 103 - 105 ºC oven. Make sure evaporation temperature is ≥2°C below boiling to prevent splattering
15	Are dried samples being cooled in a desiccator until they reach ambient temperature? [SM 2540 B-2020 (3) (c)]			Cool dish in desiccator to ambient temperature and weigh.
16	Is the dried residue weight documented to show compliance with the method? [SM 2540 B-2020 (3) (c)]			This value must be documented.
17	If minimum required weight gain of 2.5 mg is not achieved, is more sample added to the same dish? [SM 2540 B-2020 (3) (b)] **Answer:**			Choose sample volume to yield between 2.5 and 200 mg dried residue. If necessary, successive sample portions may be added to the same dish after evaporation. Do not use more than a total of 1 liter.
18	If the sample volume yields more than **200 mg,** is the test repeated with a lesser volume? [SM 2540 B-2020 (1) (b) and (3) (b)]			Choose sample volume to yield between **2.5 and 200 mg** dried residue. Because excessive residue in dish may form a water trapping crust, limit sample to no more than 200 mg residue.
19	Is the laboratory drying, cooling, desiccating and weighing samples until weight change is <0.5 mg? [SM 2540 B-2020 (3) (c)]			Repeat cycle (drying for ≥1 h, cooling, desiccating and weighing) until weight change is <0.5 mg (0.0005 g).
20	Is there documentation demonstrating the analyst is aware that the constant weight requirement has been met? [15A NCAC 2H .0805 (a) (7) (F) (xv)]			Certified Data shall be traceable to the associated sample analyses and shall consist of: all quality control assessments.
21	Is the oven temperature verified and documented each time samples are placed into, and removed from, the oven? [NC WW/GW LCB Residue Oven Temperature Documentation Policy]			The date, time and temperature must be documented each time samples are placed into, and removed from, a drying oven.
22	Are the start/end times of each 103 to 105 ºC drying cycle documented? [15A NCAC 02H .0805 (a) (7) (E)] [NC WW/GW LCB Residue Oven Temperature Documentation Policy]			Rule: The date and time that samples are placed into and removed from ovens, water baths, incubators and other equipment shall be documented if a time limit is required by the method. Time in and out of oven if oven is preheated to proper temperature. If oven is not at proper temperature when samples are put in, must document actual time heated at proper temperature. This is considered pertinent information.
23	What is the reporting limit (PQL)? [SM 2540 A-2020 (4)] **Answer:**			The analytical range for 2540B–D is 2.5 to 200 mg/L for a 1000-mL sample but may be extended by using a small sample volume for analysis. The method-defined reporting limit for Total Residue is 2.5 mg/L when using one liter of sample. The minimum reporting value is determined by a

				minimum weight gain requirement of 2.5 mg and the volume of sample analyzed. In instances where the required one liter of sample was not obtained and the residue weight gain is less than the required 2.5 mg, the value must be reported as less than the appropriate value based upon the volume used. Verify reporting limit on final reports or DMR/eDMR to ensure accuracy and appropriate adjustment by LIMS or lab personnel.
24	If the minimum weight gain of 2.5 mg is not achieved and less than one liter of sample was provided for analysis, is the sample result qualified? [15A NCAC 02H .0805 (a) (7) (M)]			Sample preservation shall be verified and 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.
25	If less than one liter of sample is provided for analysis and the minimum weight gain of 2.5 mg is not achieved, is the State Laboratory notified? [15A NCAC 02H .0805 (a) (7) (M)]			See above.
	QUALITY CONTROL	**LAB**	**SOP**	**EXPLANATION**
26	Is the desiccator equipped with an instrumental indicator (humidity gauge), color indicating desiccant, or both? [SM 2540 B-2020 (2) (i)]			Desiccator, which includes either a desiccant whose color changes in response to moisture concentration or an instrument for measuring moisture (e.g., a hygrometer). It is **recommended** that color indicating desiccant be used as a backup even when a humidity indicator is being used in the desiccator.
27	Is the laboratory using a balance that is capable of weighing at least 0.1 mg (0.0001 g)? [SM 2540 B-2020 (2) (j)]			Use an analytical balance capable of weighing 0.1 mg (0.0001g).
28	Is the analytical balanced being serviced every 12 months by a qualified vendor/technician? [15A NCAC 2H .0805 (a) (7) (J)]			Laboratory analytical balances shall be serviced by a metrology vendor or technician every 12 months to verify that the balance is functioning within manufacturer's specifications.
29	Does the laboratory have documentation to verify that the balance has been serviced? [15A NCAC 02H .0805 (a) (7) and (a) (7) (E)]			Supporting Records shall be maintained as evidence that these practices are implemented. All analytical data and records pertinent to each certified analysis shall be available for inspection upon request.
30	Is the laboratory using ASTM Type 1, Class 1 or 2, or equivalent weights? [15A NCAC 02H .0805 (a) (7) (J)]			The analytical balance shall be checked with one ASTM Type 1, Class 1 or 2, or equivalent standard weight each day used.
31	Are the weights being verified every 5 years? [15A NCAC 02H .0805 (a) (7) (J)]			These weights shall be verified every five years. Verification may be accomplished by: 1. Sending laboratory weights back to the manufacturer for recertification - reference weights shall be calibrated by a body that can provide traceability to ASTM specifications, or 2. Checking laboratory weights against certified reference weights (i.e., weights that have been recertified as above) and found to be within ASTM Type I tolerances (see table below) - often the balance service technician may provide this service.

32	Does the laboratory have documentation indicating that the weights were verified? [NC WW/GW LCB Verification of Analytical Balance Weights Policy] **Date Verified:**			Supporting Records shall be maintained as evidence that these practices are implemented. Documentation of weight verifications or recertification must be maintained for 5 years. If the condition of a weight(s) is in question at any time due to damage (e.g., corrosion, nicks, scratching, etc.), the laboratory must have that weight(s) re-verified as described above.
33	Is the balance checked with a weight each day of use? [15A NCAC 02H .0805 (a) (7) (J)] **List weight:**			The analytical balance shall be checked with one ASTM Type 1, Class 1 or 2, or equivalent standard weight each day used.
34	Where is this documented? [15A NCAC 02H .0805 (a) (7) (J)] **Answer:**			The values obtained shall be recorded, dated, and initialed.
35	Is the balance checked with at least three weights monthly? [15A NCAC 02H .0805 (a) (7) (J)]			The analytical balance shall be verified monthly with three ASTM Type 1, Class 1 or 2, or equivalent standard weights across the range of use.
36	Where is this documented? [15A NCAC 02H .0805 (a) (7) (J)] **Answer:**			The values obtained shall be recorded, dated, and initialed.
37	What corrective actions are taken when interferences are observed? [SM 2540 B-2020 (1) (b)] **Answer:**			**Highly mineralized water** with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing. **Exclude large floating particles or submerged agglomerates of nonhomogeneous materials** from the sample if it is determined that their inclusion is not desired in the final result. Optionally, disperse visible floating **oil and grease** with a blender or homogenizer before withdrawing a sample portion for analysis. If oil and grease sticks to blender sides and blades, thus potentially affecting sample composition, note this in the lab report. Residues dried at 103–105°C may retain both water of crystallization and some mechanically occluded water. There will be CO_2 loss when bicarbonate converts to carbonate during drying. Usually, very little organic matter will volatilize. It may take a long time to attain constant weight because occluded-water removal is marginal at this temperature. Because excessive residue in the dish may form a water-trapping crust, limit sample to **no more than 200 mg** residue
38	Is a method blank (MB) being analyzed each day or with each batch of 20 samples, whichever is more frequent? [SM 2540 A-2020 (5)]			Analyze one method blank (MB) per batch of 20 samples for each method except settleable solids (2540F).
39	How is the MB prepared and analyzed? [NC WW/GW LCB Method Blank Analysis Requirement for Suspended, Dissolved and Total Residue Policy] **Answer:**			No water is used. Take a prepared dish through the entire analytical process.

40	What is the acceptance criterion for the MB? [NC WW/GW LCB Method Blank Analysis Requirement for Suspended, Dissolved and Total Residue Policy] **Answer:**			Acceptance criterion is <0.5 mg weight gain.
41	What corrective action does the laboratory take if the MB results are outside of established control limits? [15A NCAC 02H .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. 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.
42	Does the laboratory analyze a duplicate sample each day or with each batch of ≤20 samples, whichever is more frequent? [SM 2540 A-2020 (5)]			Analyze 5% or more of all samples in duplicate or at least one duplicate sample with each batch of 20 or fewer samples.
43	What is the acceptance criterion for duplicates? [SM 2540 A-2020 (5)] [15A NCAC 2H .0805 (a) (7) (A)] 0 **Answer:**			The laboratory may plot duplicate determinations on a control chart for evaluation. Typically, the relative percent difference (RPD) of duplicates should not exceed 10%, but RPDs may vary considerably due to sample matrix and concentration. If lab is using %RPD for acceptance criterion, check low concentration samples for compliance. They may need to use a separate low-level acceptance criterion that may be based upon calculated recoveries or a ± mg/L criterion.
44	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits or method accuracy limits? [15A NCAC 02H .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.
45	Is a laboratory-fortified blank (LFB) being analyzed once per day or with each batch of ≤ 20 samples, whichever is more frequent? [SM 2540 A-2020 (5)]			Include one laboratory-fortified blank (LFB) per batch of 20 samples for all tests except settleable solids (2540F) and total, fixed, and volatile solids in solid and semisolid samples (2540G).
46	What type of standard is being used? [SM 2540 B-2020 (1) (a)] **Answer:**			To meet the LFB requirement (2540A.5), a total solids standard can be created as follows: Dry, grind, and sieve a soil for use as a working control. This control may or may not be mixed with other reagents (e.g., Celite 545 or Sigmacell Cellulose Type 20) and may have water added according to the laboratory's procedures. A commercially prepared QC sample may be used.
47	What acceptance criterion is used? [SM 2540 A-2020 (5)] [15A NCAC 02H .0805 (a) (7) (A)] **Answer:**			Plot the percent recoveries on a control chart for laboratory evaluation.
48	Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 02H .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. All documented results (e.g., benchsheets, reports and DMRs) must indicate appropriate qualifications.

Calculations: mg total solids/L= $\frac{(A - B) \times 1000}{\text{sample volume, mL}}$

Where:

A = weight of dried residue + dish, mg and
B = weight of dish, mg

Additional Comments:

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Inspector: _____Date:_____