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, Suspended (Aqueous)**
Method: **Standard Methods 2540 D-2020**

Total Suspended 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:

	Filtration apparatus suitable for the filter disk selected. Circle type used: 1. Membrane filter funnel 2. Gooch crucible, 25 ml to 40 ml capacity, with Gooch crucible adapter 3. Filtration apparatus with reservoir and coarse (40- to 60- µm) fritted disk as filter support		Glass fiber filter disks with ≤ 2 µm nominal pore size without organic binder (Practical filter diameters are 2.2 to 12.5 cm). **Circle Type Used:** Whatman grade 934AH Gelman type A/E Millipore type AP40 Ahlstrom grade 161 Environmental Express Pro Weigh® pre-weighed Other that gives demonstrably equivalent results (**list**):
	Aluminum weighing dishes		Desiccator, provided with desiccant containing a color indicator of moisture concentration or an instrumental indicator
	Drying oven, for operation at 103 to 105 ºC		Suction flask, of sufficient capacity for sample size selected
	Reagent grade water		Analytical balance, capable of weighing 0.1 mg (0.0001 g)
	Graduated cylinder		Beakers
	Magnetic stirrer with TFE stirring bar (not required)		Wide-bore pipets (VYCOR®, product of Corning Glass Works, Corning, NY, or equivalent.)
	Flat tipped forceps (recommended)		Computer equipped with ProWeigh® software and bar code reader (optional)

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)] **Answer:**			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	**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 – Filter Preparation	LAB	SOP	EXPLANATION
7	Are pre-prepared (i.e., commercially washed and pre-weighed) filters being used? [SM 2540 D-2020 (3) (a)] **If YES, skip to question #13**			If using commercially prepared glass-fiber filters, the ignition, washing, and weighing steps may be eliminated if the manufacturer certifies that the prepared filters meet this method's requirements.
8	Is the glass fiber filter being placed wrinkle side up in the filtration apparatus? [SM 2540 D-2020 (3) (a)]			Insert disk with wrinkled side up in filtration apparatus.
9	Is the laboratory washing the filter with 3 successive ≥20 mL portions of reagent-grade water? [SM 2540 D-2020 (3) (a)]			Apply vacuum and wash disk with three successive portions of ≥20 mL reagent-grade water.
10	Is filter suctioned to remove all traces of water? [SM 2540 D-2020 (3) (a)]			Continue suction to remove all traces of water, turn vacuum off and discard washings.
11	At what temperature is the prepared filter, aluminum weighing dish or Gooch crucible being dried? [SM 2540 D-2020 (3) (a)] **Answer:**			Remove filter from filtration apparatus and transfer to an inert weighing dish. If a Gooch crucible is used, remove crucible and filter combination. Dry in a 103–105°C oven for ≥1 h. Cool in desiccator to ambient temperature and weigh. Store filters (on inert dishes or pans) in desiccator or 103–105°C oven until needed. Adequate filter preparation is demonstrated by negligible weight loss or gain for method blanks.
12	How are dried filters being stored? [SM 2540 D-2020 (3) (a)] **Answer:**			Store filters (on inert dishes or pans) in desiccator or 103–105°C oven until needed.
	PROCEDURE- Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
13	Are samples well mixed prior to analysis? [SM 2540 D-2020 (3) (c)]			Samples may be mixed by shaking in sample bottle or stirring with magnetic stirrer.
14	How is the sample volume measured? [SM 2540 D-2020 (3) (c)] **Answer:**			Stir or mix sample and use a pipet or graduated cylinder to transfer a measured volume onto a glass-fiber filter with applied vacuum.
15	Did sample volume yield the required residue: **2.5 to 200 mg**? [SM 2540 D-2020 (3) (b)]			Choose sample volume to yield between **2.5 and 200 mg** dried residue.
16	Is the dried residue weight calculated and documented to show compliance with the minimum and maximum weight gain specified by the method? [15A NCAC 02H .0805 (a) (7) (F) (xv) and (xviii)]			Certified Data shall be traceable to the associated sample analyses and shall consist of: (xv) all quality control assessments; (xviii) any other data needed to reconstruct the final calculated result.
17	If minimum weight gain of 2.5 mg is not achieved, is the filtration repeated with a larger sample volume (up to 1 L)? [SM 2540 D-2020 (3) (b)]			If volume filtered fails to meet minimum yield, increase sample volume up to 1 L. The range of measurement for Suspended Residue is determined by the optimum solids loading on the filter, which can be controlled by adjusting the volume of sample filtered. The method-defined reporting limit for Suspended Residue is 2.5 mg/L when filtering 1 L of sample. This sample volume may not be necessary to demonstrate compliance with regulatory limits; however, it is not acceptable to routinely report less-than results using a reporting limit greater than 2.5 mg/L.
18	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 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 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 to ensure accuracy and appropriate adjustment by LIMS or lab personnel.
19	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.
20	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.
21	What is the maximum filtration time allowed to filter samples? [SM 2540 D-2020 (3) (b)] **Answer:**			If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.
22	Is filter placed with wrinkled side up during sample filtration? [SM 2540 D-2020 (3) (a)]			Insert disk with <u>wrinkled side up</u> in filtration apparatus. This is so the filter sits flat on the funnel or crucible and the residue is retained on the filter. NOTE: Examine lock/sealing mechanism on filter funnels to insure there is no leakage or loss of sample residue under the filter.
23	Is filter being seated with reagent-grade water prior to filtering sample?			Assemble filtering apparatus and begin suction. Wet filter with a small volume of reagent-grade water to seat it. **Seating the filter is not required.** However, it is important to do and is recommended.
24	Is the graduated cylinder or pipet being rinsed onto the filter?			This is not required by the method, but is recommended.
25	Are sample filters being washed after sample transfer? [SM 2540 D-2020 (3) (c)]			Wash filter with at least three successive volumes of ≥ 10 mL reagent-grade water.
26	Are samples allowed to drain completely between washings? [SM 2540 D-2020 (3) (c)]			Allow complete drainage between washings, and continue suction until all traces of water are removed. When filtering samples with high dissolved solids concentrations, additional washings may be required to ensure that dissolved material is removed from all exposed filter surfaces.
27	How are samples transferred to the drying oven? [SM 2540 D-2020 (3) (c)] **Answer:**			Using forceps, carefully remove filter from filtration apparatus and transfer to an inert weighing dish or pan as a support. If using a Gooch crucible, remove crucible and filter combination from the crucible adapter.
28	At what temperature is the sample being dried? [SM 2540 D-2020 (3) (c)] **Answer:**			Dry for at least 1 hr at 103 to 105 ºC in an oven.
29	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.
30	Are the start/end times of each 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.

31	Are samples being cooled in a desiccator after drying, until they reach ambient temperature? [SM 2540 D-2020 (3) (c)]			Cool in a desiccator to ambient temperature, and weigh.
32	Are only the filters being weighed, unless using Gooch crucibles? [SM 2540 D-2020 (1) (b)]			Weigh only the filters, not the support pans or dishes, unless a Gooch crucible is used.
33	Is the laboratory drying, cooling, desiccating and weighing sample filters until the weight change is < 0.5 mg? [SM 2540 D-2020 (3) (c)]			Repeat successive cycles of drying, cooling, desiccating, and weighing until the weight change is less than 0.5 mg.(0.0005 g)
	QUALITY CONTROL	**LAB**	**SOP**	**EXPLANATION**
34	Is the desiccator equipped with an instrumental indicator (humidity gauge), color indicating desiccant, or both? [SM 2540 D-2020 (2)]			Desiccator must contain a color indicator of moisture content or an instrument indicator. SM 2540 D. 2. References 2540 B (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.
35	Is the laboratory using an analytical balance that is capable of weighing at least 0.1 mg (0.0001 g)? [SM 2540 D-2020 (2)]			Use an analytical balance capable of weighing 0.1 mg.
36	Is there documentation demonstrating the analyst is aware that the constant weight requirement has been met? [15A NCAC 02H .0805 (a) (7) (F) (xv)]			Certified Data shall be traceable to the associated sample analyses and shall consist of: all quality control assessments.
37	Is there documented traceability of consumables (e.g., filters)? [15A NCAC 02H .0805 (a) (7) (K)] [NC WW/GW LCB Traceability Documentation Requirements for Chemical, Reagents, Standards and Consumables Policy]			The laboratory shall have a documented system of traceability for the purchase, preparation, and use of all chemicals, reagents, standards, and consumables. Traceability documentation for any filters would include vendor, lot #, and date put into use. Vendor certificates for pre-weighed filters must be kept on file. For pre-weighed filters, the date put into use could be documented on the certificate supplied by the manufacturer.
38	What corrective actions are taken when interferences are observed? [SM 2540 D-2020 (1) (b)] **Answer:**			Unless representative of source, exclude large floating particles or submerged agglomerates of nonhomogeneous materials from sample. Limit sample size so it yields ≤ 200 mg residue, because excessive filter residue may form a water-entrapping crust.
39	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% of all samples in duplicate or at least one duplicate sample with each batch of ≤20 samples.
40	What is the acceptance criterion for duplicates? [SM-2540 A-2020 (5)] [15A NCAC 02H .0805 (a) (7) (A)] **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.
41	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.
42	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). This requirement does not appear in the original 2020 Table II but was corrected in the Errata.

43	How is the MB prepared and analyzed? [NC WW/GW LCB Method Blank Analysis Requirement for Suspended, Dissolved, and Total Residue Policy] **Answer:**			Using the same containers and glassware normally in contact with samples, put at least 30 mL of DI water through the sample filter and proceed through the entire analytical process.
44	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. Initial filter weights are verified if the method blank weight differs by less than ±0.5 mg.
45	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.
46	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). This requirement does not appear in the original 2020 Table II but was corrected in the Errata. This fulfills the monthly standard (QC check) requirement in the Rules.
47	What type of standard is being used? **Answer:**			A residue standard can be prepared from a variety of materials (e.g., Sigmacell® Cellulose Type 20 or Celite 545) and weighed as a QC, or a commercially prepared QC sample may be used.
48	What acceptance criterion is used? [SM 2540 A-2020 (5)] **Answer:**			Plot the percent recoveries on a control chart for laboratory evaluation
49	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 suspended solids/L= $\frac{(A - B) \times 1000}{\text{sample volume, mL}}$

Where:

A = weight of filter + dried residue, mg, and
B = weight of filter, mg.

Additional Comments:

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Inspector: _____ Date: _____