

NC DENR/DWQ WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION

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-2011**

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 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 E-D Scientific Specialties 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 °C	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 (Kimble Nos. 37005 or 37034B or equivalent) (not required)
Flat tipped forceps (recommended)	Computer equipped with ProWeigh® software and bar code reader

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 2H .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 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	L A B	S O P	EXPLANATION

4	Are samples iced to above freezing but $\leq 6^{\circ}\text{C}$ 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 $\leq 6^{\circ}\text{C}$ 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	L A B	S O P	EXPLANATION
7	Are pre-prepared (i.e., commercially pre-weighed) filters being used? [SM 2540 D-2011 (3) (a)] If YES, skip to question #15			If pre-prepared (i.e., commercially pre-weighed) glass fiber filters disks are used, they must show that they were dried and weighed twice in order to satisfy method requirements. These type filters may be used without additional washing, drying and weighing. Pre-weighed filters that are washed, dried and weighed only once, must be dried again to verify constant weight. This may be achieved by performing a dry filter blank.
8	Is the glass fiber filter being placed in the filter funnel or Gooch crucible wrinkle side up during filter preparation? [SM 2540 D-2011 (3) (a)]			Insert disk with wrinkled side up in filtration apparatus or Gooch crucible.
9	Is the laboratory washing the filter with at least 3 successive 20 mL portions of reagent grade water? [SM 2540 D-2011 (3) (a)]			Apply vacuum and wash with at least 3 successive 20 mL portions of reagent grade water.
10	Is filter suctioned to remove all traces of water? [SM 2540 D-2011 (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-2011 (3) (a)] ANSWER:			Remove filter from filtration apparatus and transfer to an inert aluminum weighing dish. If a Gooch crucible is used, remove crucible and filter combination. Dry in an oven at 103 to 105 $^{\circ}\text{C}$ for 1 h. Cool in desiccator to balance temperature and weigh.
12	Prior to sample analysis, is the laboratory drying, cooling, desiccating, and weighing filters until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg, whichever is less? [SM 2540 D-2011 (3) (a)]			This is part of the filter preparation before sample analysis. Repeat cycle of drying or igniting, cooling, desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5 mg (0.0005 g), whichever is less.
13	If the filters are not weighed to a constant weight prior to sample analysis, is a dried filter (i.e., filter blank) verified to constant weight each day samples are analyzed? [NC WW/GW LC Policy]			Do not filter water through this filter. Post analysis weight (weighed with samples after drying) must be within 4% of the previous weighing or 0.5 mg (0.0005 g); whichever is less. This fulfills the initial drying cycle verification required by the method.
14	Are dried filters being stored in a desiccator? [SM 2540 D-2011 (3) (a)]			Store prepared filters in an adequately sealed (i.e., grease, vacuum, seals in good shape) desiccator until needed for sample analysis. Each sample requires close attention to desiccation after drying. Minimize opening desiccator because moist air enters. Some samples may be stronger desiccants than those used in the

				desiccator and may take on water. Residues dried at 103 to 105 °C may retain not only water of crystallization but also some mechanically occluded water.
	PROCEDURE- Sample Analysis	L A B	S O P	EXPLANATION
15	Are samples well mixed prior to analysis? [SM 2540 D-2011 (3) (c)]			Samples may be mixed by shaking in sample bottle or stirring with magnetic stirrer.
16	How is the sample volume measured? [SM 2540 D-2011 (2)] ANSWER:			Wide-bore pipet or graduated cylinder. Use of a graduated cylinder is considered the practical means to measure sample volume. Multiple draws with a pipet are not considered practical. If using graduated cylinder to measure sample volume, good laboratory practice is to rinse the graduated cylinder with reagent grade water after filtering sample. Rinsings are then filtered. Method states: Stir sample with a magnetic stirrer at a speed to shear larger particles, if practical , to obtain a more uniform, (preferably homogenous) particle size. Centrifugal force may separate particle by size and density, resulting in poor precision when point of sample withdrawal is varied. While stirring, pipet a measured volume onto the seated glass fiber filter. For homogeneous samples, pipet from the approximate midpoint of container but not in vortex. Choose a point both mid-depth and midway between wall and vortex.
17	Did sample volume yield the required residue: 2.5 to 200 mg ? [SM 2540 D-2011 (3) (b)]			Choose sample volume to yield between 2.5 and 200 mg dried residue.
18	If minimum weight gain of 2.5 mg is not achieved, was the filtration repeated with a larger sample volume (up to 1 L)? [SM 2540 D-2011 (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.
19	What is the reporting limit (PQL)? [SM 2540 D-2011 (4)] ANSWER:			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 insure accuracy and appropriate adjustment by LIMS or lab personnel.
20	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			Sample preservation shall be verified and If a laboratory receives a sample subject

	qualified? [15A NCAC 2H .0805 (a) (7) (M)]			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.
21	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 2H .0805 (a) (7) (M)]			See above.
22	What is the maximum filtration time allowed to filter samples? [SM 2540 D-2011 (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.
23	Is filter placed with wrinkled side up during sample filtration? [SM 2540 D-2011 (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.
24	Is filter being seated with reagent grade water prior to filtering sample? [SM 2540 D-2011 (3) (c)]			Assemble filtering apparatus and begin suction. Wet filter with a small volume of reagent-grade water to seat it.
25	Are sample filters being washed after sample transfer? [SM 2540 D-2011 (3) (c)]			Wash filter with three successive 10-ml volumes of reagent-grade water.
26	Are samples allowed to drain completely between washings? [SM 2540 D-2011 (3) (c)]			Allow complete drainage between washings, and continue suction for about 3 min after filtration is complete. Samples with higher dissolved solids may require additional washings. Incomplete filtration may result in the wet filter sticking to the weighing dish during drying.
27	How are samples transferred to the drying oven? [SM 2540 D-2011 (3) (c)] ANSWER:			Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish as a support. Alternatively remove the crucible and filter combination from the crucible adapter if a Gooch crucible is used. Although the method does not indicate how to remove filter it is recommended that flat tip forceps be used.
28	At what temperature is the sample being dried? [SM 2540 D-2011 (3) (c)] ANSWER:			Dry for at least 1 hr at 103 to 105 °C in an oven.
29	Are the start/end times of the drying documented? [15A NCAC 2H .0805 (a) (7) (E)] ANSWER:			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.
30	Are samples being cooled in a desiccator after drying, until they reach ambient temperature? [SM 2540 D-2011 (3) (c)]		Cool in a desiccator to balance temperature, and weigh.
31	Is the desiccator equipped with an instrumental indicator (humidity gauge), color indicating desiccant, or both? [SM 2540 D-2011 (2)]		Desiccator must contain a color indicator of moisture content or an instrument indicator. SM 2540 D. 2. References 2540 B (2) (d). It is recommended that color indicating desiccant be used as a backup even when a humidity indicator is being used in the desiccator.
32	Is the laboratory using an analytical balance that is capable of weighing at least 0.1 mg (0.0001 g)? [SM 2540 D-2011 (2)]		Use an analytical balance capable of weighing 0.1 mg.
33	Is the analytical balance 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.
34	Does the laboratory have documentation to verify that the balance has been serviced? [15A NCAC 2H .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.
35	Is the laboratory using ASTM Type 1, Class 1 or 2, or equivalent weights? [15A NCAC 2H .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.
36	Are the weights being verified every 5 years? [15A NCAC 2H .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 1 tolerances (see Weight Verification policy) - often the balance service technician may provide this service.
37	Does the laboratory have documentation indicating that the weights were verified? [15A NCAC 2H .0805 (a) (7)] 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.
38	Is the balance checked with a weight each day of use? [15A NCAC 2H .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.
39	Is this documented? [15A NCAC 2H .0805 (a) (7) (J)]		The values obtained shall be recorded, dated, and initialed.

40	Is the balance checked with at least three weights monthly? [15A NCAC 2H .0805 (a) (7) (J)] List weights:			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.
41	Where is this documented? [15A NCAC 2H .0805 (a) (7) (J)]			The values obtained shall be recorded, dated, and initialed.
42	After sample analysis, is the laboratory drying, cooling, desiccating and weighing sample filters until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg? [SM 2540 D-2011 (3) (c)]			Repeat drying cycle of drying, cooling, desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weight or 0.5 mg (0.0005), whichever is less.
43	If not, when was the last annual drying time study performed? [NC WW/GW LC Policy] ANSWER:			Effective January 1, 2021, NC WW/GW LC will no longer allow drying studies or dry filter blanks to substitute for drying and weighing to a constant weight. Prior to filtering samples, all filters will have to be washed and dried to a constant weight (within 0.5 mg of the prior weight). All filters after filtering samples will also have to be dried and weighed to a constant weight (within 0.5 mg of the prior weight). Samples representing each matrix type encountered by the laboratory must be included in the annual study. Verify minimum daily drying time is equal to or greater than the time used for the initial verification study drying cycle. Subsequent drying cycles must be a minimum 1 hour for verification. Check documentation of time in/out of oven.
	QUALITY CONTROL	L A B	S O P	EXPLANATION
44	Is there documented traceability of consumables (e.g., filters)? [15A NCAC 2H .0805 (a) (7) (K) and NC WW/GW LC 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.
45	What corrective actions are taken when interferences are observed? [SM 2540 D (1) (b)-20111997 and 15A NCAC 2H .0805 (a) (7) (B)] ANSWER:			Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not representative. Because excessive residue on the filter may form a water entrapping crust, limit the sample size to that yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material
46	Does the laboratory analyze duplicate samples at a rate of at least 10% daily? [SM 2540 D-2011 (3) (c)]			2020 B requires a duplicate daily or per 20 samples, whichever is more frequent. The daily requirement still stands, however, the method tightens the frequency to 10% of samples each day.
47	What is the acceptance criterion for duplicates? [SM-2540 D-2011 (3) (c) and 15A NCAC 2H .0805 (a) (7) (A)] ANSWER:			Duplicate determinations should agree within 5% of their average weight. This is not a requirement – lab is required to set acceptance criterion. If lab is using %RPD for acceptance criterion check low concentration samples for compliance –

			may need to use a separate low-level acceptance criterion – may be based upon calculated recoveries or a ± mg/L criterion.
48	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits or method accuracy limits? [15ANCAC 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.
49	Is a check standard being analyzed monthly? [15A NCAC 2H .0805 (a) (7) (G)]		A check standard must be analyzed quarterly.
50	What type of standard is being used? [] ANSWER:		An ash-type residue standard can be used and weighed as a QC, or a commercially prepared QC sample may be used.
51	What acceptance criterion is used? [15A NCAC 2H .0805 (a) (7) (A)] ANSWER:		Establish acceptance criteria for standards prepared in-house or use the manufacturer's limits for purchased standards.
52	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. All documented results (e.g., benchesheets, 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:

Inspector: _____ Date: _____