

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: **Residue, Dissolved 180 C (Aqueous)**
 Method: **Standard Methods 2540 C-2011**

Total Dissolved 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: One of the following 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 [Gelman No. 4201 or equivalent]	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 Other that gives demonstrably equivalent results (list):
Suction flask, of sufficient capacity for sample size selected	Desiccator, provided with desiccant containing a color indicator of moisture concentration or an instrumental indicator.
Evaporating Dishes (100 mL capacity, 90 mm porcelain, platinum, or high silica glass)	Wide-bore pipets (Kimble Nos. 37005 or 37034B or equivalent) (not required)
Reagent grade water	Steam bath (not required)
Drying oven(s), for operation at ≈ 98 °C and 180 ± 2 °C	Magnetic stirrer with TFE stirring bar. (not required)
Analytical balance, capable of weighing 0.1 mg (0.0001 g)	

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 note 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 ≤ 6 ° 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 ° 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 - Sample Preparation	L A B	S O P	EXPLANATION
7	Are pre-prepared (i.e., commercially pre-weighed) filters being used? [SM 2540 C-2011 (3) (a)] If YES, skip to question #11			If pre-prepared (i.e., commercially pre-weighed) glass fiber filters disks are used, eliminate washing filters.
8	Is the glass fiber filter being placed in the filter funnel or Gooch crucible wrinkle side up during filter preparation? [SM 2540 C-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 C-2011 (3) (a)]			Apply vacuum and wash with 3 successive 20 mL portions of reagent grade water
10	Is filter suctioned to remove all traces of water? [SM 2540 C-2011 (3) (a)]			Continue suction to remove all traces of water, turn vacuum off and discard washings. Filter is now ready to use for sample filtering. Does not have to be dried in oven. Many labs may use a TSS filter that has been properly washed, heated, and stored in desiccator – either way OK
11	Are evaporating dishes cleaned and dried for one hour at 180 ± 2 °C prior to use? [SM 2540 C-2011 (3) (a)]			Heat clean dish to 180 ± 2 °C for 1 hr in an oven.
12	Are evaporating dishes stored in a desiccator until needed? [SM 2540 C-2011 (3) (a)]			Store in desiccator until needed.
13	Are evaporating dishes weighed immediately before use? [SM 2540 C-2011 (3) (a)]			Weigh immediately before use.
	PROCEDURE - Sample Analysis	L A B	S O P	EXPLANATION
14	Is filter placed with wrinkled side up? [SM 2540 C-2011 (3) (a)]			Insert disk with wrinkled side up in filtration apparatus.
15	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. Not cited in method for TDS – required by TSS method - can only recommend – but needs to be done
16	Are samples well mixed prior to analysis? [SM 2540 C-2011 (3) (d)]			Samples may be mixed by shaking in sample bottle or stirring with magnetic stirrer.
17	How is the sample volume measured? [SM 2540 C-2011 (3) (d)] 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. Method states: Stir sample with a magnetic stirrer and pipet a measured volume onto a glass fiber filter with applied vacuum.
18	What is the maximum filtration time allowed to filter samples? [SM 2540 C-2011 (3) (c)] ANSWER:			If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume.
19	Are sample filters being washed after sample transfer? [SM 2540 C-2011 (3) (d)]			Wash with 3 successive 10 ml volumes of reagent grade water.
20	Are samples allowed to drain completely between washings? [SM 2540 C-2011 (3) (d)]			Continue suction for about 3 min after filtration is complete.
21	Is sample and filtrate transferred to the weighed evaporating dish? [SM 2540 C-2011 (3) (d)]			Transfer total filtrate (with washings) to a weighed evaporating dish and evaporate to dryness on a steam bath or in a drying oven.
22	If minimum required weight gain of 2.5 mg is not achieved, is more filtered sample added to the same dish? [SM 2540 C-2011 (3) (c)]			If necessary, add successive volumes of sample (up to a total maximum of one

	and (3) (d)] ANSWER:		liter) to the same dish and repeat evaporating, drying, and weighing until weight gain is achieved.
23	If the sample volume yields more than 200 mg , is the test repeated with a lesser volume? [SM 2540 C-2011 (3) (c) and (3) (d)] ANSWER:		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.
24	What is the reporting limit (PQL)? [SM 2540 C-2011 (3) (c)] ANSWER:		The method-defined reporting limit for Dissolved 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 insure accuracy and appropriate adjustment by LIMS or lab personnel.
25	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 2H .0805 (a) (7) (M)] ANSWER:		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.
26	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)] ANSWER:		See above.
27	Are samples evaporated to dryness in a steam bath or drying oven? [SM 2540 C-2011 (3) (d)] ANSWER:		Evaporate to dryness on a steam bath or in a drying oven. When evaporating in a drying oven, lower temperature to approximately 2 °C below boiling to prevent splattering. Must not place dish with water directly into 180 °C ± 2 °C oven.
28	Are samples dried at 180 °C ± 2 °C for at least 1 hour after sample has evaporated? [SM 2540 C (3) (d)-2011]		Dry evaporated sample for at least 1 hour in an oven at 180 °C ± 2 °C.
29	Are the start/end times of the 180 °C ± 2 °C drying documented? [15A NCAC 2H .0805 (a) (7) (E)]		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 is usually not adequate. Must document actual time heated at proper temperature. This is considered pertinent information.

30	Are samples being cooled in a desiccator after evaporation until they reach ambient temperature? [SM 2540 C-2011 (3) (d)]			Cool sample and dish in desiccator until they reach balance temperature and weigh.
31	Is the desiccator equipped with an instrumental indicator (humidity gauge), color indicating desiccant, or both? [SM 2540 C-2011 (2)]			Desiccator must contain a color indicator of moisture content or an instrument indicator. 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 a balance that is capable of weighing at least 0.1 mg (0.0001 g)? [SM 2540 C-2011 (2)]			Use an analytical balance capable of weighing 0.1 mg (0.0001 g)
33	Is the analytical balanced being serviced every 12 months by a qualified vendor/technician? [15ANCAC 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 I tolerances (see table below) - 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	Where is this documented? [15A NCAC 2H .0805 (a) (7) (J)] ANSWER:			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)]			The analytical balance shall be verified monthly with three ASTM Type 1, Class 1 or 2, or equivalent standard weights across

	List weights:			the range of use.
41	Where is this documented? [15A NCAC 2H .0805 (a) (7) (J)] ANSWER:			The values obtained shall be recorded, dated, and initialed.
42	After sample analysis, is the laboratory drying, cooling, desiccating and weighing sample dishes until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg? [SM 2540 C-2011 (3) (d)]			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 g), 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 to substitute for drying and weighing to a constant weight. All 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 drying time.
	QUALITY CONTROL	L A B	S O P	EXPLANATION
44	What corrective actions are taken when interferences are observed? [SM 2540 C-2011 (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. Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg residue.
45	Does the laboratory analyze duplicate samples at a rate of at least 10% daily? [SM 2540 C-2011 (3) (d)]			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.
46	What are the acceptance criteria for duplicates? [15A NCAC 2H .0805 (a) (7) (F) and SM 2540 C-2011 (3) (d)] 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 \pm mg/L criterion.
47	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits or method accuracy limits? [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. 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.
48	Is a check standard being analyzed monthly? [15A NCAC 2H .0805 (a) (7) (G)]			If certified for total suspended residue, total dissolved residue, or total residue,

			laboratories shall analyze one standard monthly during each month samples are analyzed.
49	What type of standard is being used? [] ANSWER:		A variety of materials that dissolve in water can be used and weighed as a QC, or a commercially prepared QC sample may be used.
50	What acceptance criterion is used? [15A NCAC 2H .0805 (a) (7) (A)] ANSWER:		Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses. Establish acceptance criteria for standards prepared in-house or use the manufacturer's limits for purchased standards.
51	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., benchsheets, reports and DMRs) must indicate appropriate qualifications.

Calculations: mg total dissolved 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

Note: sample volume is volume of sample only, does not include volume of filter washings

Additional Comments:

Inspector:

_____ Date: _____