

NC DENR/DWQ LABORATORY CERTIFICATION

LABORATORY NAME:		CERT #:	
PRIMARY ANALYST:		DATE:	
NAME OF PERSON COMPLETING CHECKLIST (PRINT):			
SIGNATURE OF PERSON COMPLETING CHECKLIST:			

Parameter: **Total Suspended Solids (Non-filterable Residue)**
 Method: Gravimetric (**Guidance Document**)

METHOD OF ANALYSIS:

	Standard Methods 18th <input type="checkbox"/> , 19th <input type="checkbox"/> , 20th <input type="checkbox"/> , 21st <input type="checkbox"/> Edition: 2540 D
	Standard Methods Online 2540 D-97 (SM 20 th) <input type="checkbox"/>

EQUIPMENT:

	Glass fiber filter disks without organic binder. Type:
	Filtration apparatus suitable for the filter disk selected (circle apparatus used below):
	1) Membrane filter funnel. 2) Gooch crucible with Gooch crucible adapter. 3) Filtration apparatus with reservoir and coarse fritted disk as a filter support
	Suction Flask, of sufficient capacity for sample size selected.
	Drying oven, for operation at 103 to 105 °C
	Desiccator (w/color indicating desiccant or an instrumental indicator)
	Wide-bore pipettes (Kimble Nos. 37005 or 37034B or equivalent)
	Analytical Balance capable of weighing to 0.1 mg (0.0001g)
	Type-III Water
	Graduated Cylinder
	Aluminum or stainless steel weighing pans (planchet)
	Flat tipped forceps or metal tongs

PLEASE COMPLETE CHECKLIST IN INDELIBLE INK		Y	N	EXPLANATION
PRESERVATION and STORAGE				
1	What type of container is being used to store samples? [40 CFR 136 Table II]			Use resistant glass or plastic bottles; provided that the material in suspension does not adhere to container walls.
2	Are samples iced above freezing but ≤ 6 °C during shipment? [40 CFR 136 Table II]			Document downward trend for short transport samples.
3	Are samples refrigerated above freezing but ≤ 6 °C during storage? [40 CFR 136 Table II]			
4	Are samples analyzed within 7 days of collection? [40 CFR 136 Table II]			Preferably do not hold samples more than 24 hours. In no case more than 7 days.
PROCEDURE				
5	Are pre-prepared (i.e., commercially pre-weighed) filters being used? [SM 2540 D. (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.
6	Is the glass fiber filter being placed in the filter funnel or Gooch crucible wrinkle side up during filter preparation? [SM 2540 D. (3) (a).]			Insert disk with wrinkled side up in filtration apparatus or Gooch crucible.
7	Is the laboratory washing the filter with 3 consecutive washings using at least 20 mL of reagent grade Type-III water? [SM 2540 D. (3) (a).]			Apply vacuum and wash with 3 successive 20 mL portions of reagent grade water.
8	Is filter suctioned to remove all traces of water? [SM 2540 D. (3) (a).]			Continue suction to remove all traces of water, turn off vacuum and discard washings.
9	At what temperature is the prepared filter and holder being dried? [SM 2540 D. (3) (a).]			Dry in an oven at 103 to 105 °C for 1 hr. Cool in desiccator to ambient temperature prior to weighing.

10	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? [Ref: SM 18 th , 19 th , 20 th & 21 st 2540 D. (3) (a).]		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.
11	If the filters are not weighed to a constant weight, 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. Reweigh must be within 4% of the previous weighing or 0.5 mg (0.0005 g); whichever is less. It is recommended pre-weighed filters be checked, at a minimum, per lot. This fulfills the initial drying cycle verification required by the method.
12	Are dried filters being stored in a desiccator? [SM 2540 D. (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. Loss of CO ₂ will result in conversion of bicarbonate to carbonate.
13	Are samples well mixed prior to analysis? [SM 2540 D. (3) (c).]		Stir sample with a magnetic stirrer (SM 20 th & 21 st states to stir sample at a rate of speed to shear larger particles if practical to obtain a more uniform (preferably homogeneous) particle size. SM 21 st states... Centrifugal force may separate particles 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. Use of graduated cylinders is acceptable with adequate mixing of sample.
14	How is the sample volume measured? [SM 2540 D. (2).]		Wide-bore pipet or graduated cylinder. SM 2540 D. (2) refers to 2540 B. (2) which includes graduated cylinders in the apparatus section.
15	Did sample volume yield the required residue: 2.5 to 200 mg [NC WW/GW LC Policy per SM 20 th , 21 st 2540 D. (3) (b).]		If volume filtered fails to meet minimum yield, increase sample up to 1 L. EPA indicated the 10 to 200 mg mentioned in 18 th , 19 th SM was in error.
16	What is the maximum filtration time allowed to filter samples? [SM 2540 D. (3) (b).]		If complete filtration takes more than 10 minutes, increase filter diameter or decrease sample volume.
17	Is filter placed with wrinkled side up during sample filtration? [SM 2540 D. (3) (b).]		Insert disk with wrinkled side up in filtration apparatus.
18	Is filter being seated with distilled water prior to filtering sample? [SM 2540 D. (3) (c).]		Assemble filtering apparatus and begin suction. Wet filter with a small volume of reagent-grade water to seat it.
19	Are sample filters being washed after sample transfer? [SM 2540 D. (3) (c).]		Wash with 3 successive 10 ml volumes of reagent grade water.
20	Are samples allowed to drain completely between washings? [SM 2540 D. (3) (c).]		Allow complete drainage between washings and continue suction for about 3 min after filtration is complete. Samples with high dissolved solids may require additional washings.
21	How are samples transferred to the drying oven? [SM 2540 D. (3) (c).]		Remove filter from filtration apparatus and transfer to an inert weighing dish. If a Gooch crucible is being used remove crucible and filter combination.
22	At what temperature is the sample being dried? [SM 2540 D. (3) (c).]		Dry in an oven at 103 to 105 °C for 1 hr.

23	Are samples being cooled in a desiccator after evaporation until they reach ambient temperature? [SM 2540 D. (3) (c).]		Cool sample and dish in desiccator until they reach ambient temperature and weigh.
24	If an instrumental indicator (humidity gauge) is being used, does the desiccator also have an indicating desiccant? [SM 2540 D. (2).]		It is recommended that the laboratory include color indicating desiccant as a backup to the humidity indicator that is currently being used in the desiccator.
25	Is the laboratory using a balance that is capable of weighing at least 0.1 mg (i.e., 0.0001 g)? [SM 2540 D. (2).]		
26	Is the analytical balance being serviced every 12 months by a qualified vendor/technician? [NC WW/GW LC Policy]		Laboratory analytical balance(s) must be serviced by a qualified vendor/technician at a minimum every 12 months to ensure that the balance is functioning within required tolerances.
27	Does the laboratory have documentation to verify that the balance has been serviced? [NC WW/GW LC Policy]		Documentation that this service has been performed must be available for review upon request. If the operational capabilities of the balance are in question at any time during the 12-month period, corrective action must be taken.
28	Is the laboratory using Class S or ASTM Class 1 weights? [NC WW/GW LC Policy]		ASTM Class 1 and Class 2 or NIST Class S weights must be used in determining all mass determinations.
29	Are the weights being verified every 5 years? [NC WW/GW LC Policy]		ASTM Class 1 and Class 2 weights must be verified at least every 5 years. ASTM Class 1 (20 g to 25 kg) and Class 2 (10 g to 1 mg) weights are equivalent to the NBS Class S weights specified in 15A NCAC 2H .0805 (a) (7) (K). Verification may be accomplished by: <ol style="list-style-type: none"> 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.
30	Does the laboratory have documentation indicating that the weights were verified? [NC WW/GW LC Policy]		<u>Date Verified:</u> 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.
31	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. (3) (c).]		Repeat sample of drying, cooling, desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg (0.0005), whichever is less.
32	If not, when was the last annual drying time study performed? [NC WW/GW LC Policy]		<u>Date:</u> Samples representing each matrix type encountered by the laboratory must be included in the annual study. Verify minimum daily drying time is \geq 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 ASSURANCE			
33	What corrective actions are taken when interferences are observed? [SM 2540 A. (2). and 15A NCAC 2H .0805 (a) (7) and (a) (7) (F).]		<p>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.</p> <p>Large floating particles or submerged agglomerates may affect final result and should be excluded if it is determined that their inclusion is not representative.</p> <p>High in dissolved solids - the filters must be thoroughly washed to ensure the removal of dissolved material.</p> <p>Sample adheres to the wall of the container - qualify sample results.</p> <p>Magnetic particles- Avoid using a magnetic stirrer with samples that contain.</p> <p>Residues high in oil & grease may be questionable because of the difficulty of drying to constant weight in a reasonable time; therefore, samples should be analyzed in duplicate.</p> <p>Multiphase samples - Sampling, subsampling and pipetting two-phase or three phase samples may introduce serious errors. Make and keep such samples homogeneous during transfer by mixing with a magnetic stirrer.</p>
34	At what frequency are duplicate samples analyzed? [SM 20 th and 21 st 2540 D. (3) (c). or 15A NCAC 2H .0805 (a) (7) (C).]		SM 20 th and 21 st 2540 D (3) (c). specify 10% frequency. 15A NCAC 2H .0805 (a) (7) (C) requires 5% frequency. Frequency is determined by the method referenced.
35	What are the acceptance criteria for duplicates? [15A NCAC 2H .0805 (a) (7) (F).]		SM 2540 D. (3) (c). suggests... Duplicate determinations should agree within 5% of their average weight (RPD). The laboratory must establish or adopt an acceptance criterion to assess precision.
36	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) (F).]		
37	Is a check standard being analyzed quarterly? [15A NCAC 2H .0805 (a) (7) (B).]		A check standard must be analyzed quarterly.
38	What type of standard is being used? [15A NCAC 2H .0805 (a) (7) (B).]		An ash-type residue standard can be used and weighed as a QC, or a commercially prepared QC sample may be used.
39	What acceptance criterion is used? [15A NCAC 2H .0805 (a) (7).]		Establish acceptance criteria for standards prepared in-house or use the manufacturer's limits for purchased standards.
40	Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [NC WW/GW LC policy based upon 15A NCAC 2B .0506 (b) (3) (J).]		All documented results (e.g., benchsheets, reports and DMRs) must indicate appropriate qualifications.
41	What is the reporting limit (PQL)? [NC WW/GW LC Policy based upon Standard Methods, 20th and 21st Editions - Method 2540 D. (3) (b).]		The minimum reporting value is established at 2.5 mg/L when 1000 mL of sample is analyzed. The minimum weight gain allowed by any approved method is 2.5 mg. 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. For example, if 500 mL of sample is analyzed and < 2.5 mg of dried residue is obtained, the value reported would be < 5 mg/L.
42	What is the most recent review/revision date of the SOP? [15A NCAC 2H .0805 (a) (7).]		Date: Recommend an annual review. Update any time changes are made to procedure.

