NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

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| 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-2015**

**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:

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|  | 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 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  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, high silica glass, StableWeigh, Environmental Express, Charleston, SC, or equivalent.) |  | Wide-bore pipets (Vycor, product of Corning Glass Works, Corning, NY, or equivalent.) |
|  | Reagent grade water |  | Steam bath (not required) |
|  | Drying oven(s), for operation at ≈ 98 ºC and 180 ± 2 ºC |  | Magnetic stirrer with TFE stirring bar (not required) |
|  | Analytical balance, capable of weighing 0.1 mg (0.0001 g) |  | Anti-static Device |

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| **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** |
|  | 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)]  **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. |
|  | 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. |
|  | Is there North Carolina data available for review? |  |  | If not, review PT data |
|  | **PRESERVATION and STORAGE** | **LAB** | **SOP** | **EXPLANATION** |
|  | 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. |
|  | Are samples refrigerated above freezing but ≤ 6 ºC during storage? [40 CFR 136.3 Table II] |  |  |  |
|  | Are samples analyzed within 7 days of collection? [40 CFR 136.3 Table II] |  |  |  |
|  | **PROCEDURE - Sample Preparation** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are pre-prepared (i.e., commercially washed and pre-weighed) filters being used? [SM 2540 C-2015 (3) (a)] **If YES, skip to question #11** |  |  | If using commercially prepared glass-fiber filter disks, the washing step may be skipped if the manufacturer certifies that the filters meet this method’s requirements. |
|  | Is the glass fiber filter being placed in the filter funnel or Gooch crucible wrinkle side up during filter preparation? [SM 2540 C-2015 (3) (a)] |  |  | Insert disk with wrinkled side up in filtration apparatus. |
|  | Is the laboratory washing the filter with at least 3 successive ≥20 mL portions of reagent-grade water? [SM 2540 C-2015 (3) (a)] |  |  | Apply vacuum and wash disk with 3 successive portions of ≥20 mL reagent-grade water. |
|  | Is filter suctioned to remove all traces of water? [SM 2540 C-2015 (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** |
|  | Are StableWeigh™ or equivalent bags being used? **If not, skip to question #13.** |  |  |  |
|  | What type of anti-static device is being used in conjunction with the analytical balance? [NC WW/GW LCB Policy]  **ANSWER:** |  |  | 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 is important for achieving the method specified constant weight requirement of ± 0.5mg. |
|  | Are evaporating dishes cleaned and dried for at least one hour at 180 ± 2 ºC prior to use? [SM 2540 C-2015 (3) (b)] |  |  | Heat clean dish to 180 ± 2 ºC for ≥1 hr in an oven. |
|  | Are evaporating dishes stored in a desiccator until needed? [SM 2540 C-2015 (3) (b)] |  |  | Store in desiccator until needed. |
|  | Are evaporating dishes weighed before use? [SM 2540 C-2015 (3) (b)] |  |  | Cool dishes to ambient temperature and weigh. |
|  | **PROCEDURE - Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is filter placed with wrinkled side up? [SM 2540 C-2015 (3) (a)] |  |  | Insert disk with wrinkled side up in filtration apparatus. |
|  | 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. |
|  | Are samples well mixed prior to analysis? [SM 2540 C-2015 (3) (d)] |  |  | Samples may be mixed by shaking in sample bottle or stirring with magnetic stirrer. |
|  | How is the sample volume measured? [SM 2540 C-2015 (3) (d)]  **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. |
|  | What is the maximum filtration time allowed to filter samples? [SM 2540 C-2015 (3) (c)]  **ANSWER:** |  |  | If filtration will take >10 min to complete, then increase filter size or decrease sample volume. |
|  | Are sample filters being washed after sample transfer? [SM 2540 C-2015 (3) (d)] |  |  | Wash the entire exposed surface of filter with three successive volumes of ≥10 mL reagent-grade water. |
|  | Are samples allowed to drain completely between washings? [SM 2540 C-2015 (3) (d)] |  |  | Allow complete drainage between washings, and continue suction until all traces of water are removed. |
|  | Is the graduated cylinder or pipet being rinsed onto the filter? |  |  | This is not required by the method but is recommended. |
|  | Is sample and filtrate transferred to the weighed evaporating dish? [SM 2540 C-2015 (3) (d)] |  |  | Transfer **total filtrate (with washings)** to a weighed evaporating dish and evaporate to dryness on a steam bath, hot plate, or block, or in a drying oven. |
|  | If minimum required weight gain of 2.5 mg is not achieved, is more filtered sample added to the same dish? [SM 2540 C-2015 (3) (c) and (3) (d)] |  |  | If necessary, add successive volumes of sample (up to a total maximum of one liter) to the same dish after evaporation and repeat evaporating, drying, and weighing until weight gain is achieved. |
|  | Is the dried residue weight documented to show minimum weight gain was achieved? [SM 2540 C-2015 (3) (c)] [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. |
|  | If the sample volume yields more than **200 mg,** is the test repeated with a lesser volume? [SM 2540 C-2015 (3) (c)and (3) (b)  **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. |
|  | What is the reporting limit (PQL)? [SM 2540 A-2015 (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 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 ensure accuracy and appropriate adjustment by LIMS or lab personnel. |
|  | 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. |
|  | 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. |
|  | Are samples evaporated to dryness on a steam bath, hot plate, or block, or in a drying oven? [SM 2540 C-2015 (3) (d)] |  |  | Evaporate to dryness on a steam bath, hot plate, or block, or in a drying oven. When evaporating in a drying oven, it is recommended to lower temperature to approximately 2 ºC below boiling to prevent splattering. **Must not place dish with water directly into 180 ºC ± 2 ºC oven.** |
|  | Are samples dried at 180 ºC ± 2 ºC for at least 1 hour after sample has evaporated? [SM 2540 C-2015 (3) (d)] |  |  | Dry evaporated sample for at least 1 hour in an oven at 180 ºC ± 2 ºC. |
|  | Is the temperature verified and documented each time samples are placed into, and removed from, the oven? [NC WW/GW LCB Policy] |  |  | The date, time and temperature must be documented each time samples are placed into, and removed from, a drying oven. |
|  | Are the start/end times of each 180 ºC ± 2 ºC drying documented? [15A NCAC 2H .0805 (a) (7) (E)] [NC WW/GW LCB 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. |
|  | Are samples being cooled in a desiccator after drying until they reach ambient temperature? [SM 2540 C-2015 (3) (d)] |  |  | Cool sample and dish in desiccator until they reach ambient temperature and weigh. |
|  | **QUALITY CONTROL** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is the desiccator equipped with an instrumental indicator (humidity gauge), color indicating desiccant, or both? [SM 2540 C-2015 (2)] |  |  | Desiccator must contain a color indicator of moisture content or an instrument indicator. SM 2540 C. 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. |
|  | Is the laboratory using a balance that is capable of weighing at least 0.1 mg (0.0001 g)? [SM 2540 B-2015 (2) (j)] |  |  | Use an analytical balance capable of weighing 0.1 mg (0.0001 g) |
|  | 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. |
|  | 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. |
|  | 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. |
|  | 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. |
|  | 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. |
|  | 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. |
|  | Where is this documented? [15A NCAC 2H .0805 (a) (7) (J)]  **ANSWER:** |  |  | The values obtained shall be recorded, dated, and initialed. |
|  | 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. |
|  | Where is this documented? [15A NCAC 2H .0805 (a) (7) (J)]  **ANSWER:** |  |  | The values obtained shall be recorded, dated, and initialed. |
|  | Is the laboratory drying, cooling, desiccating and weighing sample dishes until weight change is <0.5 mg? [SM 2540 C-2015 (3) (d)] |  |  | Repeat drying cycle (drying for ≥1 h,, cooling, desiccating and weighing) until weight change is < 0.5 mg (0.0005 g). |
|  | Is there documentation demonstrating the analyst is aware that the constant weight requirement has been met? [15A NCAC 2H .0805 (a) (7) (F) (xv)] |  |  |  |
|  | What corrective actions are taken when interferences are observed? [SM 2540 C-2015 (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. Samples with high bi-carbonate concentrations require careful, possibly prolonged drying at 180 + 2°C to ensure that bicarbonate completely converts to carbonate.  Residues dried at 180 ± 2°C will lose almost all mechanically occluded water, but some water of crystallization may remain, especially if sulfates are present. Organic matter may volatilize and be lost, but not completely removed. CO2 loss occurs when bicarbonates convert to carbonates, and carbonates may be de-composed partially to oxides or basic salts. Some chloride and nitrate salts may be lost. In general, evaporating and drying water samples at 180 ± 2°C yields TDS values closer to those obtained by adding individually determined mineral species than the values obtained when drying at 103–105°C.  Because excessive residue in the dish may form a water-trapping crust, limit sample to **no more than 200 mg** residue. |
|  | Is a method blank (MB) being analyzed each day or with each batch of 20 samples, whichever is more frequent? [SM 2540 A-2015 (5)] |  |  | Analyze one method blank (MB) per batch of 20 samples for each method except settleable solids (2540F). |
|  | How is the MB prepared and analyzed? [NC WW/GW LCB Policy]  **ANSWER:** |  |  | Using the same containers and glassware normally in contact with samples, put 30 ml of DI water through the sample filter and proceed through the entire analytical process. |
|  | What is the acceptance criterion for the MB? [NC WW/GW LCB Policy]  **ANSWER:** |  |  | Acceptance criterion is <0.5 mg weight gain. |
|  | What corrective action does the laboratory take if the MB results are outside of established control 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. |
|  | Does the laboratory analyze a duplicate sample each day or with each batch of ≤20 samples, whichever is more frequent? [SM 2540 A-2015 (5)] |  |  | Analyze ≥5% of all samples in duplicate or at least one duplicate sample with each batch of ≤20 samples. |
|  | What are the acceptance criteria for duplicates? [15A NCAC 2H .0805 (a) (7) (F)] [SM 2540 A-2015 (5)]  **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. |
|  | 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. |
|  | Is a laboratory-fortified blank (LFB) check standard being analyzed each day or once per batch of ≤20 samples, whichever is more frequent? [SM 2540 A-2015 (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 fulfills the monthly standard (QC check) requirement in the Rules. |
|  | What type of standard is being used? [SM 2540 C-2015 (1) (a)]  **ANSWER:** |  |  | To meet the LFB requirement (2540A.5), analysts can create a TDS standard from an inorganic salt (e.g., NaCl) and weigh as a QC, or a commercially prepared QC sample may be used. |
|  | What acceptance criterion is used? [SM 2540 A-2015 (5)]  **ANSWER:** |  |  | Plot the percent recoveries on a control chart for laboratory evaluation. |
|  | 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=  *(A – B) x 1000*

*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:

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Inspector: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_Date:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_