NC DEQ/DWR 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: **Ammonia Nitrogen**

Method: EPA Method 350.1, Rev. 2.0, 1993 (Hach Method 10205 TNT)

Hach Method 10205 is not approved in 40 CFR Part 136; however, EPA has acknowledged that it is as an allowable method modification of EPA Method 350.1 per 40 CFR Part 136.6. This means you can follow the Hach 10205 TNT method procedure, using the Hach instrument and TNT reagents. However, you must follow the QC requirements of EPA 350.1, Rev. 2.0, 1993 since modified methods must meet the QC requirements of the reference method. Also, if your samples require distillation (as determined by your distillation study), you must follow the distillation requirements in SM 4500 NH3 B-2011, which is required by the Hach 10205 method.

EQUIPMENT:

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|  | Spectrophotometer  **Model:** |
|  | pH Meter or Short-Range pH Paper |
|  | All Borosilcate Glass Distillation Equipment |

DISTILATION REAGENTS:

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|  | Borate Buffer |
|  | Boric Acid Solution, 20 g/L |

ANALYSIS REAGENTS:

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|  | Ammonia free water |
|  | Hach Company TNTplus™ Ammonia Kits (TNTplus 830, 0.015 – 2.000 mg NH3-N/L) |
|  | Hach Company TNTplus™ Ammonia Kits (TNTplus 831, 1 - 12 mg NH3-N/L) |
|  | Hach Company TNTplus™ Ammonia Kits (TNTplus 832, 2 – 47 mg/L NH3-N) |
|  | NaOH, 1N |
|  | *Dechlorinating reagent:* Dissolve 3.5 g sodium thiosulfate (Na2S2O3 · 5H2O) in water and dilute to 1 L. Prepare fresh  weekly. Use 1 mL reagent to remove 1 g/L residual chlorine in 500-mL sample. |
|  | *Sulfuric Acid. 0.04N:*  *Dilute 1.0 mL conc. H2SO4 to 1L.* |
|  | Sulfuric Acid, conc. |
|  | Sodium hypochlorite solution |
|  | Sodium phenolate |
|  | Sodium nitroprusside |
|  | EDTA buffer, 5% |

**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.

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|  | **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. |

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| 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 checked for Residual Chlorine at the time of collection and prior to pH preservation adjustment? [SM 4500 NH3 B-2011 (4) (b)]  If no, skip to question 7. |  |  | SM 4500 NH3 B-2011 requires that residual chlorine be checked and mitigated at sample collection, however, guidance from EPA Region IV confirms that the residual chlorine removal is not required to be performed at collection. It may be removed prior to distillation, or analysis if distillation is not required. Removal is not required at all if the permittee does not use chlorine for  disinfection. |
| 5 | What action is taken if chlorine is present? [SM 4500 NH3 B-2011  (4) (b)]  **Answer:** |  |  | Remove residual chlorine by adding, at the time of collection, dechlorinating agent equivalent to the chlorine residual. |
| 6 | Is the residual chlorine check and any necessary mitigation documented? [NC WW/GW LC Policy] |  |  | Dechlorinating agents used at the time of sampling must be documented to have been effective (either by the sample collector or the receiving laboratory) by verifying a chlorine residual <0.5 mg/L at a neutral pH. If measuring chlorine concentration in an acidified sample, pour off a small portion of the sample and neutralize the pH prior to testing. Use sufficiently strong base to not dilute the sample. Discard that portion after  testing. |
| 7 | Are samples preserved at time of collection with H2SO4 to pH of <2 S.U.? [40 CFR 136.3 Table II] |  |  | Preservation not required if analyzed within 15 minutes. |
| 8 | Are samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136.3 Table II and footnote 18] |  |  |  |
| 9 | Is pH checked and documented to be <2 S.U. upon receipt in the laboratory? [40 CFR 136.3 Table II] |  |  |  |
| 10 | What action is taken if pH is > 2 S.U.? [15A NCAC 02H .0805 (a) (7) (M)]  **Answer:** |  |  | Sample preservation shall be verified and documented. 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. |
| 11 | Are samples refrigerated above freezing to 6°C during storage? [40 CFR 136.3 Table II and footnote 18] |  |  |  |
| 12 | Are samples analyzed within 28 days of collection? [40 CFR 136.3 Table II] |  |  |  |
|  | **PROCEDURE – Sample Preparation** | **L A**  **B** | **S O**  **P** | **EXPLANATION** |

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| 13 | If residual chlorine is not checked prior to acidification in the field, is a portion of the preserved sample neutralized in the laboratory and checked for residual chlorine prior to distillation or analysis if distillation is not required? [SM 4500 NH3 B-2011 (4) (b)]  [Hach 10205 TNT, Rev. 2.0, Section 2.3] |  |  | Remove residual chlorine by adding, at the time of collection, dechlorinating agent equivalent to the chlorine residual.  Note: Guidance from EPA Region IV confirms that the residual chlorine removal is not required to be performed at collection. It may be removed prior to distillation, or analysis if distillation is not required. Removal is not  required at all if the permittee does not use chlorine for disinfection |
| 14 | Is the residual chlorine check documented? [15A NCAC 02H .0805  (a) (7) (M)] |  |  | Sample preservation shall be verified and documented. 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. |
| 15 | If the sample is determined to contain chlorine at a level above 0.5 mg/L, is it removed prior to distillation and/or analysis by adding dechlorinating agent equivalent to the chlorine residual? [EPA Method 350.1, Rev. 2.0 (1993), Section 11.2] [SM 4500 NH3 B-2011  (4) (b)] |  |  | Remove the residual chorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. |
|  | **PROCEDURE – Distillation** | **L A**  **B** | **S O**  **P** | **EXPLANATION** |
| 16 | If manual distillation is not performed on all samples, is a distillation comparison study on file? [NC WW/GW LC Policy]  **NOTE: This question may not be applicable to Commercial laboratories.**  **Attach a copy of the study to this checklist.**  **If not distilling samples, skip to question # 30** |  |  | Manual distillation may not be required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. A comparison study may be performed in-house or contracted to another certified laboratory. Permittees that do not perform the analyses in-house, and contract the analyses or the distillation study to a NC WW/GW LC certified commercial laboratory must obtain a copy of the initial comparison data and all subsequent comparison data, keep it on file at their facility and make these records available to the Department upon request.  The following frequencies are required:  Initially, compare a minimum of 9 samples, spiked in duplicate, both with and without the distillation step (a total of 36 samples), to evaluate the need for distillation.  Per Hach 10205 Section 2.3, SM 4500 NH3 B must be followed for distillation. |
| 17 | How is the distillation equipment cleaned? [SM 4500 NH3 B-2011 (4) (a)]  **Answer:** |  |  | Equipment preparation: Add 500 mL water and 20 mL borate buffer, adjust pH to 9.5 with 6N NaOH solution, and add to a distillation flask. Add a few glass beads or boiling chips and use this mixture to steam out the distillation apparatus until distillate shows no traces of  ammonia. |

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| 18 | What sample volume is distilled? [SM 4500 NH3 B-2011 (4) (b)]  **Answer:** |  |  | Use 500 mL dechlorinated sample or a known portion diluted to 500 mL with water. When NH3-N concentration is less than 100 µg/L,  use a sample volume of 1000 mL. |
| 19 | Is the sample volume to be distilled documented? [15A NCAC 02H .0805 (a) (7) (F) (xviii)] |  |  | All laboratories shall use printable laboratory benchsheets. Certified Data shall be traceable to the associated sample analyses and shall consist of: any other data needed to  reconstruct the final calculated result. |
| 20 | Is the sample pH adjusted to approximately 7 S.U. with dilute acid or base using a pH meter? [SM 4500 NH3 B-2011 (4) (b)] |  |  |  |
| 21 | Is 25ml of borate buffer solution added to the sample? [SM 4500 NH3 B-2011 (4) (b)] |  |  | Add 25 mL borate buffer solution and adjust to pH 9.5 S.U. with 6N NaOH using a pH meter. |
| 22 | Is the sample pH adjusted to 9.5 S.U. with 6*N* NaOH using a pH meter? [SM 4500 NH3 B-2011 (4) (b)] |  |  | Add 25 mL borate buffer solution and adjust  to pH 9.5 S.U. with 6*N* NaOH using a pH meter. |
| 23 | Is the distillate collected in 50 ml of 0.04N H2SO4?? [SM 4500 NH3 B-2011 (4) (c)] |  |  | Collect distillate in a 500-mL erlenmeyer flask  containing 50 mL indicating boric acid solution for titrimetric method. |
| 24 | Is the condenser outlet tip submerged below the surface of the receiving acid solution? [SM 4500 NH3 B-2011 (4) (c)] |  |  | Distill at a rate of 6 to 10 mL/min with the tip of the delivery tube below the surface of acid  receiving solution. |
| 25 | What volume of distillate is collected? [SM 4500 NH3 B-2011 (4) (c)]  **Answer:** |  |  | Collect at least 200 mL distillate. |
| 26 | Is the distillation receiver lowered for the last minute or two of distillation? [SM 4500 NH3 B-2011 (4) (c)] |  |  | Lower distillation receiver so that the end of the delivery tube is free of contact with the liquid and continue distillation during the last minute or two to cleanse condenser and  delivery tube |
| 27 | Is the distillate diluted to the original volume distilled? [SM 4500 NH3 B-2011 (4) (c)] |  |  | Dilute to 500 mL with water. |
| 28 | Is the final volume documented? [15A NCAC 02H .0805 (a) (7) (F) (xviii)] |  |  | All laboratories shall use printable laboratory benchsheets. Certified Data shall be traceable to the associated sample analyses and shall  consist of: any other data needed to reconstruct the final calculated result. |
| 29 | Is the distillate neutralized with 1*N* NaOH solution? [SM 4500 NH3 B-2011 (4) (c)] |  |  |  |
|  | **PROCEDURE – Calibration** | **L**  **A B** | **S**  **O P** | **EXPLANATION** |
| 30 | Is a stored calibration curve used? [Hach 10205 TNT, Rev. 2.0, Section 10.1]  If no, skip to question # 33 |  |  | The Hach DR series spectrophotometers have a built-in calibration that is automatically used when the TNTplus Ammonia sample vial is placed in the cell holder of the instrument. No further initial calibration is required. However, the instruments have the capability of developing a user-calibration.  See manufacturer’s manual for instructions. |
| 31 | Is the stored calibration curve verified across the range of use annually with a series of five or more non-zero standards? [15A NCAC 02H .0805 (a) (7) (H) (v)]  **Date of last verification:** |  |  | For colorimetric analyses, a series of five or more non-zero standards for a curve prepared every 12 months or three or more non-zero standards for curves established each day, or standards as set forth in the analytical procedure, shall be analyzed to establish a calibration curve. A manufacturer's factory-set calibration (internal curve) shall be verified with  the same number of standards and frequency as a prepared curve. |
| 32 | What is the acceptance criterion for each verified standard? [15A NCAC 02H .0805 (a) (7) (A)]  **List each standard and its acceptance criterion:** |  |  | Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control  analyses. Each laboratory shall calculate and |

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|  |  |  |  | document the precision and accuracy of all quality control analyses with each sample set. When the method of choice specifies performance acceptance criteria for precision and accuracy, and the laboratory chooses to develop laboratory-specific limits, the laboratory-specific limits shall not be less  stringent than the criteria stated in the approved method. |
| 33 | Is a user-generated calibration curve prepared and for each range used? [Hach 10205 TNT, Rev. 2.0, Section 10.1]  If no, skip to question # 37. |  |  | The Hach DR series spectrophotometers have a built-in calibration that is automatically used when the TNTplus Ammonia sample vial is placed in the cell holder of the instrument. No further initial calibration is required. However, the instruments have the capability of developing a user-calibration.  See manufacturer’s manual for instructions. |
| 34 | Does the calibration curve consist of a blank and at least 5 non-zero standards? [15A NCAC 02H .0805 (a) (7) (H) (v)].  **List values of standards used for Standard Curve:** |  |  | For colorimetric analyses, a series of five or more non-zero standards for a curve prepared every 12 months or three or more non-zero standards for curves established each day, or standards as set forth in the  analytical procedure, shall be analyzed to establish a calibration curve. |
| 35 | Does the calibration curve have a correlation coefficient ≥0.995? [NC WW/GW LC Policy] |  |  | When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of  determination, r2, of 0.99) is required. |
| 36 | Is each initial calibration verified before sample analysis with a second source standard? [15A NCAC 02H .0805 (a) (7) (H) (ii)] [EPA Method 350.1, Rev. 2.0 (1993), Section 10.7]  What is the concentration of the second source standard used for verification?  **Answer:** |  |  | Rule: Laboratories shall analyze one known second source standard to verify the accuracy of standard preparation if an initial calibration is performed and in accordance with the referenced method requirements thereafter.  350.1: After the calibration has been established, it must be verified by the analysis of a suitable QCS. The QCS is obtained from a source external to the laboratory and different from  the source of calibration standards. |
|  | **PROCEDURE – Analysis** | **L**  **A B** | **S**  **O P** | **EXPLANATION** |
| 37 | Are samples analyzed at a neutral pH? [Hach 10205, Section 8.2] |  |  | Neutralize preserved samples before analysis. This is interpreted to mean that the  samples must be adjusted to 6.51-7.49 S.U. |
| 38 | If the volume used to neutralize samples is >1%, are the results corrected for the additional volume? [Hach 10205, Section 8.2] |  |  | Correct the test result for volume additions. |
| 39 | What volume of sample is added to the TNT vials? [Hach 10205 TNT, Rev. 2.0, Section 11.2.2]  **Answer:**  **TNT 830:**  **TNT 831:**  **TNT 832:** |  |  | Pipet 5.0 mL of sample into the vial for TNT830, 0.5 mL of sample for TNT831, or 0.2 mL of sample for TNT832. |

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| 40 | Is the DosiCap Zip lid flipped over so that the reagent side faces the  vial and cap tightly screwed down? [Hach 10205 TNT, Rev. 2.0, Section 11.2.3] |  |  | Flip the DosiCap Zip over so that the reagent  side faces the vial. Screw the cap tightly onto the vial. |
| 41 | Are the reagents verified to be dissolved after being shaken 2-3 times by looking down through the open end of the DosiCap Zip? [Hach 10205 TNT, Rev. 2.0, Section 11.2.4] |  |  | Shake the capped vial 2-3 times to dissolve the reagent in the cap. Verify that the reagent has dissolved by looking down through the  open end of the DosiCap Zip. |
| 42 | Are samples allowed to react for at least 15 minutes but not more than 30 minutes? [Hach 10205 TNT, Rev. 2.0, Sections 11.2.5 and  11.2.6] |  |  | React for 15 minutes. |
| 43 | After 15 minutes, are sample vials inverted an additional 2-3 times to mix? [Hach 10205 TNT, Rev. 2.0, Section 11.2.6] |  |  | After 15 minutes, invert the sample an  additional 2-3 times to mix. The color remains stable for 15 minutes. |
| 44 | Is the outside of sample vials thoroughly cleaned before inserting into the instrument? [Hach 10205 TNT, Rev. 2.0, Section 11.3.1 and 11.3.2] |  |  | Thoroughly clean the outside of the vial. Insert the prepared vial into the cell holder. The instrument reads the barcode, then selects and performs the correct test. Note:  No instrument zero is required. |
| 45 | Are results reported as mg/L NH3-N? [Hach 10205 TNT, Rev. 2.0, Section 11.3.2] |  |  | Results are in mg/L NH3-N. |
|  | **QUALITY ASSURANCE** | **L**  **A B** | **S**  **O P** | **EXPLANATION** |
| 46 | Has an MDL been established according to 40 CFR 136 Appendix B? [Method 350.1, Rev. 2.0 (1993), Section 9.2.4] [Hach 10205 TNT,  Rev. 2.0, Section 9.2.1] |  |  | Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three  batches on three separate calendar dates and analyzed on three separate calendar dates. |
| 47 | Is ongoing MDL data being collected quarterly? [Procedure for the Determination of the Method Detection Limit, Rev. 2, (3) (a)] |  |  | During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked samples on each instrument, in  separate batches, using the same spiking concentration used in Section 2. |
| 48 | Are MDL values verified at least every 13 months according to the ongoing MDL determination requirements and updated if necessary?  [Procedure for the Determination of the Method Detection Limit, Rev. 2, (4) (a)] |  |  | At least once every thirteen months, re- calculate MDLs and MDLb from the collected  spiked samples and method blank results using the equations in Section 2. |
| 49 | If a user generated curve is established, what is the acceptance criterion of the second source standard (QCS)? [EPA Method 350.1, Rev. 2.0 (1993), Section 10.7]  **Answer:** |  |  | Must not exceed ±10% of the established QCS value. |
| 50 | What corrective action is taken if the second source standard varies by greater than 10%? [EPA Method 350.1, Rev. 2.0 (1993), Section  10.7]  **Answer:** |  |  | If measurements exceed ±10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check. |
| 51 | Is a laboratory reagent blank (LRB) analyzed with each batch of samples? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.1] |  |  | The laboratory must analyze at least one LRB with each batch of samples.  Definition of LRB (Section3.6): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in  the laboratory environment, the reagents, or the apparatus. |
| 52 |  |  |  | The concentration of reagent, method, and calibration blanks shall not exceed 50 percent |

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|  | What is the acceptance criterion of the LRB and calibration blanks? [15A NCAC 02H .0805 (a) (7) (H) (i)]  **Answer:** |  |  | of the lowest reporting concentration or as otherwise specified by the reference method. |
| 53 | What corrective action is taken if the blanks do not meet the acceptance criterion? [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. |
| 54 | Is a mid-range Instrument Performance Check (IPC) standard and calibration blank analyzed prior to sample analysis, after every 10th sample, and at the end of sample analysis? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4] |  |  | For all determinations the laboratory must analyze the IPC (instrument performance check) (a mid-range check standard) and a calibration blank immediately following daily calibration, after every 10th sample (or more frequently, if required) and at the end of the sample run. Although the meter may not be  calibrated daily, the IPC must be analyzed daily prior to sample analysis. |
| 55 | What is the acceptance criterion for the mid-range IPC standard? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4]  **Answer:** |  |  | Analysis of the IPC solution immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. |
| 56 | What corrective action is taken if the mid-range IPC standard recovery is not within specified limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.4]  **Answer:** |  |  | If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution  must be reanalyzed. |
| 57 | Is a laboratory fortified blank (LFB) analyzed with each batch of samples? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2] |  |  | Definition of LFB (Section 3.4): An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the  laboratory is capable of making accurate and precise measurements. |
| 58 | What is the acceptance criterion for the LFB? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2]  **Answer:** |  |  | Recovery within 90%- 110% |
| 59 | What corrective action is taken if the LFB does not meet specified limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.3.2]  **Answer:** |  |  | If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. |
| 60 | Does the laboratory analyze duplicate samples at a rate of 5%? [15A NCAC 02H .0805 (a) (7) (C)] |  |  | Except where otherwise specified in an analytical method, laboratories shall analyze five percent of all samples in duplicate to document precision. Laboratories analyzing fewer than 20 samples per month shall analyze one duplicate during each month that  samples are analyzed. **NOTE: A Laboratory Fortified Matrix Duplicate (LFMD/MSD) can** |

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|  |  |  |  | **satisfy our Rule requirement for a sample duplicate** |
| 61 | What is the acceptance criterion for duplicates? [15A NCAC 02H .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. |
| 62 | What corrective action does the laboratory take if the duplicate samples results are outside of established control limits or method precision 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. |
| 63 | At what frequency is a Matrix Spike (MS) analyzed? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.1]  **Answer:** |  |  | Also called Laboratory Fortified Matrix (LFM). The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. |
| 64 | How is the MS prepared? [NC WW/GW LC Matrix Spike Technical Assistance.]  **Answer:** |  |  | See Matrix Spike Technical Assistance document. |
| 65 | What is the acceptance criterion for MS recovery? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.2]  **Answer:** |  |  | designated LFM recovery range 90-110% |
| 66 | What corrective action does the laboratory take if the MS results are outside of established control limits? [EPA Method 350.1, Rev. 2.0 (1993), Section 9.4.3]  **Answer:** |  |  | If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related. |
| 67 | Is a lower reporting limit standard analyzed or back-calculated each day samples are analyzed? [15A NCAC 02H .0805 (a) (7) (H)] |  |  | Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day  samples are analyzed. |
| 68 | What is the acceptance criterion for the lower reporting limit standard? [15A NCAC 02H .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. |
| 69 | What corrective action does the laboratory take if the lower reporting limit standard does not meet the acceptance criterion? [15A NCAC 02H .0805 (a) (7) (B)]  **Answer:** |  |  | Recalibrate/re-verify the curve |
| 70 | 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. |

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|  |  |  |  | If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with  sufficient information to determine the usability of the qualified data. |

Additional Comments:

Stock Standard – Dissolve 3.819 g anhydrous NH4CL (dried at 1000 C) in water and dilute to 1000 mL. 1.00mL = 1.00 mg N = 1.22 mg NH3.

NOTE: Data is reported as NH3 –N, that is Ammonia as Nitrogen, so 1.00 mL of stock standard equals 1 mg of

Ammonia nitrogen. That solution equals a 1000 mg/L concentration of Ammonia as Nitrogen. The difference between the 1000 and 1220 can be calculated from the molecular weights, N = 14 and NH3 = 17. So 17÷14 = 1.22. That is where you get a concentration of 1.0 mg/L for Ammonia as Nitrogen (N) and 1.22 mg/L for Ammonia (NH3)

Additional Comments:

Inspector: Date: