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: **Total Kjeldahl Nitrogen**

Method: **Block Digestion and Semi-Automated Colorimetry**

 **EPA Method 351.2, Revision 2.0, 1993 (Aqueous)**

Equipment:

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|  | *Block digestor* capable of maintaining a temperature of 380°C for 2 h. |  | *Digestion tubes* capable of being heated to 380°C for 2 h and having a cover to prevent ammonia contamination and loss of sulfuric acid. |  | Automated Continuous Flow Analyzer with colorimetric detector and data acquisition system.**Make/Model:** |
|  | Analytical balance (if preparing reagents) |  | Teflon boiling chips |  |  |

Reagents:

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|  | Digestion solution: Dissolve 133 g of K2SO4 (CASRN 7778-80-5) in 700 mL of reagent water and 200 mL of conc. H2SO4 . Add 25 mL of mercuric sulfate solution and dilute to 1 L.Note 1: An alternate mercury-free digestion solution can be prepared by dissolving 134 g K2SO4 and 7.3 g CuSO4 in 800 mL reagent water and then adding 134 mL conc. H2SO4 and diluting to 1 L. Use 10 mL solution per 25 mL of sample. |  | Stock Buffer solution: Dissolve 134.0 g of sodium phosphate, dibasic (Na2HPO4) (CASRN 7558-79-4) in about 800 mL of reagent water. Add 20 g of sodium hydroxide and dilute to 1 L. |
|  | Mercuric sulfate: Dissolve 8 g red mercuric oxide (HgO) (CASRN 21908-53-2) in 50 mL of 1:4 sulfuric acid (10 mL conc. H2SO4: [CASRN 7664-93-9] 40 mL reagent water) and dilute to 100 mL with reagent water. |  | Working Buffer solution: Combine the reagents in the stated order, add 250 mL of stock sodium potassium tartrate solution to 200 mL of stock buffer solution and mix. Add xx mL sodium hydroxide solution and dilute to 1 L. See concentration ranges, Table 2 at the end of this checklist, for composition of working buffer. |
|  | Sulfuric Acid solution (4%): Add 40 mL of conc. sulfuric acid to 800 mL of reagent water, cool and dilute to 1 L. Note 2: If alternate mercury-free digestion solution is used, adjust the above solution to equal the acid concentration of the digested sample. |  | Sodium Salicylate/Sodium Nitroprusside solution: Dissolve 150 g of sodium salicylate (CASRN 54-21-7) and 0.3 g of sodium nitroprusside (CASRN 13755-38-9 or 14402-89-2) in about 600 mL of reagent water and dilute to 1 L. |
|  | Stock Sodium Hydroxide (20%, 5*N*): Dissolve 200 g of sodium hydroxide (CASRN 1310-73-2) in 900 mL of reagent water and dilute to 1 L. |  | Sodium Hypochlorite solution: Dilute 6.0 mL sodium hypochlorite solution (CASRN 7681-52-9) (Clorox) to 100 mL with reagent water. |
|  | Stock Sodium Potassium Tartrate solution (20%): Dissolve 200 g sodium potassium tartrate (CASRN 6381-59-5) in about 800 mL of reagent water and dilute to 1 L. |  | Ammonium chloride, stock solution: Dissolve 3.819 g NH4Cl (CASRN 12125-02-9) in reagent water and bring to volume in a 1 L volumetric flask. 1 mL = 1.0 mg NH 3 -N. |

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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 review/revision dates and procedural edits 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 collected and stored in polyethylene, fluoropolymer (e.g., Teflon®), or glass containers? [40 CFR 136 Table II] |  |  |  |
|  | Are samples preserved at time of collection with H2SO4 to pH of <2 S.U.? [40 CFR 136 Table II] |  |  |  |
|  | Are samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136 Table II] |  |  |  |
|  | Are samples refrigerated above freezing but ≤ 6 º C during storage? [40 CFR 136 Table II] |  |  |  |
|  | Are samples analyzed within 28 days of collection? [40 CFR 136 Table II]  |  |  |  |
|  | **PROCEDURE – Block Digestion** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are both standards and samples digested? [EPA Method 351.2, Rev. 2.0 (1993), Section 10.2 and 11.0] |  |  | Method says to carry both standards and samples through the digestion procedure.  |
|  | What volume of sample is digested? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.1]**Answer:** |  |  | Pipet 25.0 mL of sample, standard or blank in the digestor tube. |
|  | What volume of standard is digested? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.1]**Answer:** |  |  | Pipet 25.0 mL of sample, standard or blank in the digestor tube. |
|  | Is 5 mL of digestion solution added to samples and standards in the digestion tube and mixed with a vortex mixer? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.2] |  |  | Add 5 mL of digestion solution and mix with a vortex mixer.  |
|  | If alternate mercury-free digestion solution is used, is 10 mL added to samples and standards in the digestion tube and mixed with a vortex mixer? [EPA Method 351.2, Rev. 2.0 (1993), Sections 11.2 and 7.3, Note 1] |  |  | Note 1: An alternate mercury-free digestion solution can be prepared by dissolving 134 g K2SO4 and 7.3 g CuSO4 in 800 mL reagent water and then adding 134 mL conc. H2SO4 and diluting to 1 L. Use 10 mL solution per 25 mL of sample. |
|  | Are four to eight Teflon boiling chips added to each tube? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.3] |  |  | Add four to eight Teflon boiling chips CAUTION: An excess of Teflon chips may cause the sample to boil over. |
|  | Is the block digestor preheated to 160°C? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.4] |  |  | Place tubes in block digestor preheated to 160°C and maintain temperature for one hour. |
|  | Are samples digested at 160°C for 1 hour? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.4] |  |  | Place tubes in block digestor preheated to 160°C and maintain temperature for one hour. |
|  | After 1 hour, is the temperature increased to 380°C? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.5] |  |  | Reset temperature to 380°C and continue to heat for 1.5 hours. |
|  | Are samples digested for an additional 1 and a half hours at 380°C? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.5] |  |  | Reset temperature to 380°C and continue to heat for 1.5 hours. (380°C MUST BE MAINTAINED FOR 30 MINUTES) |
|  | Is the temperature of the block digester checked and documented to be 160°C and 380°C during digestion? [15A NCAC 2H .0805 (a) (7) (E)] |  |  | All analytical records, including original observations and information necessary to facilitate historical reconstruction of the calculated results, shall be maintained for five years. All analytical data and records pertinent to each certified analysis shall be available for inspection upon request. |
|  | Are samples allowed to cool before diluting? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.6] |  |  | Remove digestion tubes, cool and dilute to 25 mL with reagent water. |
|  | Are samples diluted to 25 mL with reagent water? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.6] |  |  | Remove digestion tubes, cool and dilute to 25 mL with reagent water. |
|  | **PROCEDURE – Calibration** | **LAB** | **SOP** | **EXPLANATION** |
|  | What is your laboratory’s reporting limit? [15A NCAC 2H .0805 (a) (7) (H)]**Answer:** |  |  | For analytical procedures requiring analysis of a series of standards, the concentrations of these standards shall bracket the range of the sample concentrations measured. One of the standards shall have a concentration equal to or less than the laboratory's lowest reporting concentration for the parameter involved. |
|  | Is the instrument calibrated with at least three digested standards and one blank each day samples are analyzed? [EPA Method 351.2, Rev. 2.0 (1993), Section 10.1 and 9.3.4] |  |  | Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solution with reagent water.9.3.4 states: For all determinations the laboratory must analyze the IPC (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. |
|  | Is the correlation coefficient (r) of the calibration curve ≥0.995 (or a coefficient of determination (r2) ≥0.99)? [NC WW/GW LCB 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. |
|  | Are standards placed in the sampler in order of decreasing concentration? [EPA Method 351.2, Rev. 2.0 (1993), Section 10.5] |  |  | Place appropriate standards in the sampler in order of decreasing concentration and perform analysis. |
|  | Is the calibration curve verified with a second-source QCS standard? [15A NCAC 2H .0805 (a) (7) (H) (ii) and EPA Method 351.2, Rev. 2.0 (1993), Section 10.7] |  |  | After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (QCS).  |
|  | What is the acceptance criterion for the second-source standard? [EPA Method 351.2, 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.  |
|  | What corrective action is taken if the second-source standard recovery is 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. |
|  | **PROCEDURE – Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is the sampler wash receptacle flushed with about 25 mL of 4% sulfuric acid? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.8] |  |  | Flush the sampler wash receptacle with about 25 mL of 4% sulfuric acid (Section 7.4) (See Note 2). |
|  | If alternate mercury-free digestion solution is used, is the sulfuric acid used to flush the sampler wash receptacle adjusted to equal the acid concentration of the digested samples? [EPA Method 351.2, Rev. 2.0 (1993), Section 7.4, Note 2] |  |  | Note 2: If alternate mercury-free digestion solution is used, adjust the above solution to equal the acid concentration of the digested sample (Section 11.6). |
|  | Are all reagents except the salicylate allowed to pump through the system for at least five minutes before beginning the flow of salicylate? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.9] |  |  | Excluding the salicylate line, place all reagent lines in their respective containers, connect the sample probe to the sampler and start the pump. When reagents have been pumping for at least five minutes, place the salicylate line in its respective container and allow the system to equilibrate. |
|  | Are the lines checked to ensure that no precipitate has formed after starting the flow of salicylate? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.9] |  |  | If a precipitate forms after the addition of salicylate, the pH is too low.Immediately stop the proportioning pump and flush the coils with water using a syringe. Before restarting the system, check the concentration of the sulfuric acid solutions and/or the working buffer solution. |
|  | Is analysis started only after a stable baseline has been obtained? [EPA Method 351.2, Rev. 2.0 (1993), Section 11.11]  |  |  |  |
|  | **QUALITY ASSURANCE** | **LAB** | **SOP** | **EXPLANATION** |
|  | Has an MDL been established according to 40 CFR 136 Appendix B? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.2.4] |  |  | 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. |
|  | 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. |
|  | 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. |
|  | Is at least one Laboratory Reagent Blank (LRB) analyzed with each batch of samples? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.1] |  |  | Definition (Sec 3.6)The LRB is 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 laboratory must analyze at least one LRB with each batch of samples.EPA defines an analytical batch: a group of samples, including quality control samples, which are processed together using the same method, the same lots of reagents, and at the same time or in continuous, sequential time periods. Samples in each batch should be of similar composition and share common internal quality control standards. |
|  | What is the acceptance criterion for the LRB? [15A NCAC 2H .0805 (a) (7) (H) (i)] or [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.4]**Answer:** |  |  | Rules state: The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method.Section 9.3.1 states: Values that exceed the MDL indicate laboratory or reagent contamination should suspected and corrective actions must be taken before continuing the analysis.May choose which criterion to follow. Must be established in the SOP. |
|  | What corrective action is taken if the LRB is not acceptable? [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 mid-range Instrument Performance Check Solution (IPC) analyzed immediately following daily calibration, after every 10th sample (or more frequently, if required), and at the end of the sample run? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.4] |  |  | For all determinations the laboratory must analyze the IPC (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. |
|  | What is the acceptance criterion for the IPC standard? [EPA Method 351.2, 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%. |
|  | What corrective action is taken if the IPC result exceeds ±10% of the true value? [EPA Method 351.2, 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. |
|  | Is a calibration blank analyzed initially, after every tenth sample and at the end of each sample group to check for carry over and calibration drift? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.4] |  |  | For all determinations the laboratory must analyze the IPC (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. |
|  | What is the acceptance criterion for the calibration blank? [15A NCAC 2H .0805 (a) (7) (H) (i)]**Answer:** |  |  | The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method. |
|  | What corrective action is taken if the calibration blank is not acceptable? [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 Laboratory Fortified Blank (LFB) with each batch of samples? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.2] |  |  | The laboratory must analyze at least one LFB with each batch of samples. Definition (Sec. 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 |
|  | What is the acceptance criterion for the LFB standard? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.3]**Answer:** |  |  | The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed. The optional control limits must be equal to or better than the required control limits of 90-110%. |
|  | What corrective action is taken if the LFB is not acceptable? [15A NCAC 2H .0805 (a) (7) (B)] [EPA Method 351.2, Rev. 2.0 (1993), Section 9.3.2]**Answer:** |  |  | Calculate accuracy as percent recovery (Section 9.4.2). 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.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 Matrix (LFM) analyzed at a frequency of 10% of samples? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.4.1] |  |  | The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. |
|  | How is the MS prepared? [NC WW/GW LCB Matrix Spiking Policy and Technical Assistance]**Answer:** |  |  | The volume of spike solution used in MS preparation must in all cases be ≤ 5% of the total MS volume.  |
|  | If the volume of spike solution added to the sample is greater than 1% of the total volume, is the recovery calculation adjusted? [NC WW/GW LCB Policy] |  |  | It is preferable that the spike solution constitutes < 1% of the total MS volume so that the MS can be considered a whole volume sample with no adjustment (i.e., volume correction) by calculation necessary. If the spike solution volume constitutes >1% of the total sample volume, the sample concentration must be adjusted by calculation. |
|  | What is the acceptance criterion for LFM recovery? [EPA Method 351.2, Rev. 2.0 (1993), Section 9.4.2]**Answer:** |  |  | Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%. |
|  | What corrective action does the laboratory take if the LFM results are outside of established control limits for **accuracy**? [15A NCAC 2H .0805 (a) (7) (B) and EPA Method 351.2, Rev. 2.0 (1993), Section 9.4.3]**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.Sec 9.4.3 states: 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. |
|  | Is a sample duplicate analyzed at a frequency of 5% of samples? [15A NCAC 2H .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) can satisfy our Rule requirement for a sample duplicate but should be analyzed at the same frequency as the LFM.** |
|  | If no sample duplicate is analyzed, is a Laboratory Fortified Matrix Duplicate (LFMD) analyzed at a frequency of 5% of samples? [15A NCAC 2H .0805 (a) (7) (C)]  |  |  | See Note above. |
|  | What is the acceptance criterion for precision between sample duplicates or LFM/LFMD (i.e., relative percent difference)? [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. Each laboratory shall calculate and document the precision and accuracy of all quality control analyses with each sample set. |
|  | What corrective action does the laboratory take if the duplicate/LFMD results are outside of established control limits for **precision**? [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 lower reporting limit standard analyzed or back-calculated each day? [15A NCAC 2H .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. |
|  | What is the acceptance criterion for the lower reporting limit standard? [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. |
|  | What corrective action does the laboratory take if the lower reporting limit standard does not meet the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B)]**Answer:** |  |  |  |
|  | 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 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. |

Additional Comments:

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