NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

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

Parameter: **Metals by ICP-MS**
Method: **EPA Method 200.8, Rev. 5.4 (1994) (Aqueous and Non-aqueous)**

Note: Per November 7, 2007 EPA letter, additional metals that are not in Table 1.1 may be analyzed as long as QC is acceptable

Equipment:

	Inductively coupled plasma mass spectrometer		Analytical balance, capable to measure 0.1 mg		Temperature adjustable hot plate capable of maintaining 95 °C
	Drying oven capable of maintaining 105 ± 5 °C		(Optional) Air displacement pipettor capable of delivering volumes from 0.1-2500 µL with an assortment of high-quality disposable pipet tips		(Optional) Temperature adjustable block digester capable of maintaining 95 °C equipped with 250 mL constricted digestion tubes
	Mortar and pestle, ceramic or nonmetallic material		Polypropylene sieve, 5-mesh (4 mm opening)		Labware

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
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 02H .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.
2	Are all review/revision dates and procedural edits tracked and documented? [15A NCAC 02H .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 Standard Operating Procedure documents.
3	Is there North Carolina data available for review?			If not, review PT data
	PRESERVATION and STORAGE	**LAB**	**SOP**	**EXPLANATION**
4	What type of sample container is used? [40 CFR 136.3 Table II] **Answer:**			Silica requires polyethylene or quartz. Boron requires polyethylene, fluoropolymer (PTFE, Teflon®), or quartz. All others require polyethylene, fluoropolymer (PTFE, Teflon®), or glass.
5	Are samples (except Silica) analyzed within 6 months of collection? [40 CFR 136.3 Table II]			
6	Are Silica samples analyzed within 28 days? [40 CFR 136.3 Table II]			
7	Are aqueous samples (except Silica) preserved with HNO_3 to pH <2 S.U. within 15 minutes of collection? [40 CFR 136.3 Table II]			
8	If not, are they (except for Boron and Silica) preserved with HNO_3 to pH <2 S.U. at least 24 hours prior to analysis? [40 CFR 136.3 Table II]			40 CFR does not make this allowance for Boron, so it must be preserved within 15 minutes.
9	Are aqueous samples (except Silica and boron) verified and documented to be at a pH <2 for at least 24 hours prior to the start of analyses? [40 CFR 136.3 Table II and Footnote 19] [15A NCAC 02H .0805 (a) (7) (M)]			Footnote 19: An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see

				footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.
10	Are Silica samples iced to above freezing but ≤ 6 º C during shipment? [40 CFR 136.3 Table II]			Silica samples are not acid preserved and must be refrigerated.
11	Are Silica samples refrigerated above freezing but ≤ 6 ºC during storage? [40 CFR 136.3 Table II]			
12	For dissolved metals, are the samples filtered through a 0.45 µm filter within 15 minutes of collection and before preservation with HNO_3 to pH <2 S.U.? [40 CFR 136.3 Table II Footnote 7]			
	PROCEDURE – Interferences	**LAB**	**SOP**	**EXPLANATION**
13	If alternative analytical isotopes (other than those in Table 4) having higher natural abundance are selected in order to achieve greater sensitivity are chosen, is the data corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest? [EPA Method 200.8, Rev. 5.4 (1994), Section 4.1.1]			Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table 4), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.
14	Is the spectrometer resolution adjusted to minimize wing overlap interferences? [EPA Method 200.8, Rev. 5.4 (1994), Section 4.1.2]			Abundance sensitivity - Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one.
15	When isobaric polyatomic ion interferences (see Table 2) cannot be avoided by the selection of alternative analytical isotopes, are corrections made to the data? [EPA Method 200.8, Rev. 5.4 (1994), Section 4.1.3]			Isobaric polyatomic ion interferences - Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Equations for the correction of data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common ^{82}Kr interference that affects the determination of both arsenic and selenium, can be greatly reduced with the use of high purity krypton free argon.
16	Are internal standards used to compensate for physical interference effects? [EPA Method 200.8, Rev. 5.4 (1994), Section 4.1.4]			Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not

				exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standards ideally should have similar analytical behavior to the elements being determined.
17	Are suitable rinse times used to minimize memory interferences? [EPA Method 200.8, Rev. 5.4 (1994), Section 4.1.5]			The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to 10 times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of 10 of the method detection limit, should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected, the sample should be reanalyzed after a long rinse period. In the determination of mercury, which suffers from severe memory effects, the addition of 100 µg/L gold will effectively rinse 5 µg/L mercury in approximately two minutes. Higher concentrations will require a longer rinse time.
18	Are all acids used ultra high-purity grade? [EPA Method 200.8, Rev. 5.4 (1994), Section 7.1]			Reagents may contain elemental impurities that might affect the integrity of analytical data. Owing to the high sensitivity of ICP-MS, high-purity reagents should be used whenever possible.
19	When hydrochloric acid is used, are corrections for the chloride polyatomic ion interferences applied to all data? [EPA Method 200.8, Rev. 5.4 (1994), Section 7.1]			Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid is used (Table 2), however, it should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver.
	PROCEDURE – Sample Preparation	**LAB**	**SOP**	**EXPLANATION**
20	Are samples digested/extracted prior to analysis? [EPA Method 200.8, Rev. 5.4 (1994), Section 1.5]			
21	Are samples containing suspended or particulate material ≥1% (w/v) extracted as a solid type sample? [EPA Method 200.8, Rev. 5.4 (1994), Section 1.5 and 11.2.2]			A well-mixed, acid preserved aliquot containing no more than 1 g particulate material should be cautiously evaporated to near 10 mL and extracted using the acid mixture procedure described in sections 11.3.3 through 11.3.6.
22	For the analysis of silver, are samples with concentrations >0.1 mg/L or >50 mg/kg, diluted to be below this concentration? [EPA Method 200.8, Rev. 5.4 (1994), Section 1.7]			
	PROCEDURE – Sample Preparation – Dissolved Metals	**LAB**	**SOP**	**EXPLANATION**
23	Is a ≥20 mL aliquot of a filtered and acid preserved sample pipetted into a 50 mL polypropylene centrifuge tube? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.1.1]			
24	If a precipitate is formed during acidification, transport, or storage, is the sample aliquot treated using the procedure for total recoverable metals prior to analysis? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.1.1]			See method sections 11.2.2 through 11.2.8.
25	Is an appropriate volume of (1+1) nitric acid added to adjust the acid concentration of the aliquot to approximate a 1% (v/v) nitric acid solution? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.1.1]			For example, add 0.4 mL (1+1) HNO_3 to a 20 mL aliquot of sample.

26	If the direct addition procedure (Method A, Section 10.3) is being used, are internal standards added? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.1.1]			
27	Is the tube capped and mixed prior to sample analysis? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.1.1]			The sample is now ready for analysis.
	PROCEDURE – Sample Preparation – Total Metals (aqueous)	**LAB**	**SOP**	**EXPLANATION**
28	What initial volumes of sample are used? **ANSWER:**			
29	Is a well-mixed acid preserved sample transferred to an appropriate digestion vessel? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.2]			Although the method calls for 100 mL, smaller sample aliquot volumes may be used under method flexibility allowances.
30	Is 2 mL of (1+1) nitric acid and 1.0 mL of (1+1) hydrochloric acid added to the beaker? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.3]			If a smaller sample volume is used, proper ratios of acids must be maintained.
31	Is the beaker placed on a hot plate at 85 °C in a fume hood and covered with an elevated watch glass to prevent contamination? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.3]			For proper heating adjust the temperature control of the hot plate such that an uncovered Griffin beaker containing 50 mL of water placed in the center of the hot plate can be maintained at a temperature approximately but no higher than 85 °C. Once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95 °C. Using a hotblock and digestion tubes is a method modification allowed under 40 CFR Part 136.6.
32	Is the sample volume reduced to about 20 mL by gentle heating at 85 °C (not boiling)? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.4]			This step takes about two hours for a 100 mL aliquot. The rate of evaporation rapidly increases as the sample volume approaches 20 mL.
33	Is the beaker then covered with a watch glass to prevent additional evaporation and gently refluxed for 30 minutes? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.5]			Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the HCl-H_2O azeotrope.
34	After cooling, what volume are samples brought to after digestion? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.6] **ANSWER:**			The method states to start with 100 mL and bring post-digestion volume to 50 mL. Smaller initial volumes may be used, and final volumes may be returned to the initial volume to avoid concentration calculations.
35	Is undissolved material allowed to settle overnight or centrifuged? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.7]			The sample is now ready for analysis. All analyses should be performed ASAP after preparation. If the sample still contains suspended solids that would clog the nebulizer, a portion of the sample may be filtered prior to analysis. However, care should be exercised to avoid contamination from filtration.
36	How is chloride interference mitigated? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.8] **ANSWER:**			Prior to analysis, adjust the chloride concentration by pipetting 20 mL of the prepared solution into a 50 mL volumetric flask, dilute to volume with reagent water and mix. If the dissolved solids in this solution are >0.2%, additional dilution may be required to prevent clogging of the extraction and/or skimmer cones. Note: some instruments (such as Perkin Elmer) have an internal method of eliminating chloride interference. When using this feature, dilute the digested solution back to the original predigested volume rather than performing this step. However, before employing this method, laboratories must perform a comparison of samples with and without the technology to show that it won't reduce sensitivity or cause a

				magnification of other interferences
37	If the direct addition procedure (Method A, Section 10.3) is being used, are internal standards added? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.2.8]			The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.
	PROCEDURE – Sample Preparation – Total Metals (non-aqueous)	**LAB**	**SOP**	**EXPLANATION**
38	Is a portion (>20 g) of mixed sample weighed, dried to a constant weight at 60 °C, and weighed again to determine the percent solids? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.1]			For samples <35% moisture, a 20 g portion is sufficient. For samples >35% moisture, a larger aliquot 50-100 g is required. %Solids = $\frac{\text{Dried Weight X100}}{\text{Wet Weight}}$
39	Is the dried sample sieved using a 5-mesh polypropylene sieve and ground in a mortar and pestle? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.2]			The sieve, mortar, and pestle should be cleaned between samples.
40	Is 1.0 ± 0.01 g of the dried, ground material transferred to a 250 mL Phillips beaker? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.2]			
41	Is 4 mL of (1+1) HNO_3 and 10 mL of (1+4) HCL added to the beaker? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.3]			
42	Is the beaker placed on a hot plate at 85 °C in a fume hood, covered with a watch glass, and gently refluxed for 30 minutes? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.4]			For proper heating adjust the temperature control of the hot plate such that an uncovered Griffin beaker containing 50 mL of water placed in the center of the hot plate can be maintained at a temperature approximately but no higher than 85°C. Once the beaker is covered with a watch glass the temperature will rise to approximately 95 °C. Very slight boiling may occur, however vigorous boiling must be avoided. Some evaporation will occur (3-4 mL). Also, a block digester capable of maintaining a temperature of 95°C and equipped with 250 mL constricted volumetric digestion tubes may be substituted for the hot plate and conical beakers in the extraction step.
43	After cooling, is the extract diluted to 100 mL in a volumetric flask and mixed well? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.5]			
44	Is the solution allowed to stand overnight to separate insoluble material or centrifuged? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.6]			If after centrifuging or standing overnight the extract solution contains suspended solids that would clog the nebulizer, a portion of the extract solution may be filtered for their removal prior to analysis. However, care should be exercised to avoid potential contamination from filtration.
45	How is chloride interference mitigated? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.7] **ANSWER:**			Prior to analysis, adjust the chloride concentration by pipetting 20 mL of the prepared solution into a 100 mL volumetric flask, dilute to volume with reagent water and mix. If the dissolved solids in this solution are >0.2%, additional dilution may be required to prevent clogging of the extraction and/or skimmer cones.
46	If the direct addition procedure (Method A, Section 10.3) is being used, are internal standards added? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.3.7]			The sample is now ready for analysis. Because the effects of various matrices on the stability of diluted samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation. Determine the percent solids in the sample for use in calculations and for reporting data on a dry weight basis.

	PROCEDURE – Instrument Calibration	LAB	SOP	EXPLANATION
47	Are the manufacturer recommended operating conditions followed? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.1]			Because of the diversity of instrument hardware, no detailed instrument operating conditions are provided. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements and to maintain quality control data verifying instrument performance and analytical results.
48	Is the precalibration routine completed prior to calibrating the instrument until such time that instrument stability can be demonstrated periodically by running the tuning solution (Section 7.7) a minimum of five times with resulting relative standard deviations of absolute signals for all analytes of less than 5%? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.2]			Initiate proper operating configuration of instrument and data system. Allow a period of not less than 30 minutes for the instrument to warm up. During this process conduct mass calibration and resolution checks using the tuning solution. Resolution at low mass is indicated by magnesium isotopes 24, 25, and 26. Resolution at high mass is indicated by lead isotopes 206, 207, and 208. For good performance adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass.
49	Is internal standardization used in all analyses to correct for instrument drift and physical interferences? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.3]			For full mass range scans, a minimum of three internal standards must be used. Procedures described in this method for general application, detail the use of five internal standards; scandium, yttrium, indium, terbium and bismuth. These were used to generate the precision and recovery data attached to this method. Internal standards must be present in all samples, standards and blanks at identical levels. This may be achieved by directly adding an aliquot of the internal standards to the CAL standard, blank or sample solution (Method A, Section 10.3), or alternatively by mixing with the solution prior to nebulization using a second channel of the peristaltic pump and a mixing coil (Method B, Section 10.3). The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Depending on the sensitivity of the instrument, a concentration range of 20-200 µg/L of each internal standard is recommended. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.
50	Are a minimum of three replicate integrations used for data acquisition? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.4]			
51	Is the average of the integrations used for instrument calibration and data reporting? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.4]			
52	Is the rinse blank used to flush the system between solution changes for blanks, standards and samples? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.5]			Allow sufficient rinse time to remove traces of the previous sample (Section 4.1.5). Solutions should be aspirated for 30 seconds prior to the acquisition of data to allow equilibrium to be established.
53	Is the instrument tuned and calibrated for all analytes of interest? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.4.2]			
	PROCEDURE – Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
54	For all sample analyses, is a minimum of three replicate integrations used for data acquisition? [EPA Method 200.8, Rev. 5.4 (1994),			

	Section 11.4.3]			
55	Is the average of the integrations used for data reporting? [EPA Method 200.8, Rev. 5.4 (1994), Section 11.4.3]			
56	Is the rinse blank used to flush the system between samples? [EPA Method 200.8, Rev. 5.4 (1994), Section 10.5 and 11.4.6]			The rinse blank should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of one minute (Section 4.1.5). Samples should be aspirated for 30 seconds prior to the collection of data.
57	For aqueous samples prepared by total recoverable procedure (Section 11.2), are solution concentrations multiplied by the dilution factor 1.25? [EPA Method 200.8, Rev. 5.4 (1994), Section 12.3]			If additional dilutions were made to any samples or an aqueous sample was prepared using the acid mixture procedure described in Section 11.3, the appropriate factor should be applied to the calculated sample concentrations.
58	For total recoverable analytes in solid samples, are sample results calculated and reported properly? [EPA Method 200.8, Rev. 5.4 (1994), Section 12.4]			Round the solution analyte concentrations (µg/L in the analysis solution) as instructed in Section 12.2. Multiply the µ/L concentrations in the analysis solution by the factor 0.005 to calculate the mg/L analyte concentration in the 100 mL extract solution. (If additional dilutions were made to any samples, the appropriate factor should be applied to calculate analyte concentrations in the extract solution.) Report the data up to three significant figures as mg/kg dry-weight basis unless specified otherwise by the program or data user. Calculate the concentration using the equation below: Sample conc. (mg/kg) = $\frac{C \times V}{W}$ Dry weight basis C = Concentration in the extract (mg/L) V = Volume of extract (L, 100 mL = 0.1L) W = Weight of sample aliquot extracted (g x 0.001 = kg) Do not report analyte data below the estimated solids MDL or an adjusted MDL because of additional dilutions required to complete the analysis.
59	Are data values corrected for instrument drift or sample matrix induced interferences by the application of internal standardization? [EPA Method 200.8, Rev. 5.4 (1994), Section 2.2 and 12.6]			Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix **must** be corrected for by the use of internal standards. Corrections for characterized spectral interferences should be applied to the data. Chloride interference corrections should be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.
	QUALITY ASSURANCE	**LAB**	**SOP**	**EXPLANATION**
60	Has an MDL been established for each element according to 40 CFR 136 Appendix B [a.k.a Procedure for the Determination of the Method Detection Limit, Rev. 2]? [EPA Method 200.8, Rev. 5.4 (1994), 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.
61	Is ongoing MDL data being collected quarterly? [40 CFR 136 Appendix B] [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.
62	Are MDL values verified at least every 13 months according to the ongoing MDL determination requirements and updated if necessary? [40 CFR 136 Appendix B] [Procedure for the Determination of the Method Detection Limit, Rev. 2, (4) (a)]			At least once every thirteen months, re-calculate MDL_s and MDL_b from the collected spiked samples and method blank results using the equations in Section 2.

63	Is a known second source standard analyzed after initial calibration? [15A NCAC 2H .0805 (a) (7) (H) (ii) and EPA Method 200.8, Rev. 5.4 (1994), Section 9.2.3]			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. The second source standard is equivalent to the QCS in sections 7.8 and 9.2.3 of the method.
64	What is the acceptance criterion of the second source standard? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.2.3] **ANSWER:**			To verify the calibration standards the determined mean concentration from three analyses of the QCS must be within ±10% of the stated QCS value. If the QCS is used for determining acceptable on-going instrument performance, analysis of the QCS prepared to a concentration of 100 µg/L must be within ±10% of the stated value or within the acceptance limits listed in Table 8, whichever is the greater. (If the QCS is not within the required limits, an immediate second analysis of the QCS is recommended to confirm unacceptable performance.) If the calibration standards and/or acceptable instrument performance cannot be verified, the source of the problem must be identified and corrected before either proceeding on with the initial determination of method detection limits or continuing with on-going analyses.
65	What corrective action does the laboratory take if the second source standard does not meet the acceptance criterion? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.2.3] **ANSWER:**			(If the QCS is not within the required limits, an immediate second analysis of the QCS is recommended to confirm unacceptable performance.) If the calibration standards and/or acceptable instrument performance cannot be verified, the source of the problem must be identified and corrected before either proceeding on with the initial determination of method detection limits or continuing with on-going analyses.
66	Is a lower reporting limit standard analyzed or back-calculated with each analysis? [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.
67	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.
68	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:**			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. Recalibrate/re-verify the curve.
69	Is at least one Laboratory Reagent Blank analyzed with each batch of 20 or fewer of samples of the same matrix? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.1]			The LRB must contain all the reagents in the same volumes as used in processing the samples. The LRB must be carried through the same entire preparation scheme as the samples including digestion, when applicable. If the direct addition procedure (Method A, Section 10.3) is being used, add internal standards to the solution after preparation is complete.
70	What is the acceptance criterion of the LRB? [15A NCAC 2H .0805 (a) (7) (H) (i) or EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.1] **ANSWER:**			Rule: 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.

				Method: Must not exceed 10% or more of the analyte level determined for a sample or 2.2 times the analyte MDL, whichever is greater.
71	What corrective action does the laboratory take if the LRB does not meet the acceptance criterion? [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. Recalibrate/re-verify the curve.
72	Is at least one Laboratory Fortified Blank (LFB) analyzed with each batch of samples? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.2]			To an aliquot of LRB, add aliquots from multielement stock standards A and B (Section 7.4) to prepared the LFB. Depending on the sensitivity of the instrument, the fortified concentration used should range from 40-100 µg/L for each analyte, except selenium and mercury. For selenium the concentration should range from 200-500 µg/L, while the concentration range mercury should be limited to 2-5 µg/L. The LFB must be carried through the same entire preparation scheme as the samples including sample digestion, when applicable. If the direct addition procedure (Method A, Section 10.3) is being used, add internal standards to this solution after preparation has been completed.
73	What is the acceptance criterion of the LFB? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.2] **ANSWER:**			If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
74	What corrective action does the laboratory take if the LFB does not meet the acceptance criterion? [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. Recalibrate/re-verify the curve.
75	To verify calibration, are the calibration blank and a calibration standard analyzed immediately following each calibration routine, after every ten analyses and at the end of the sample run? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.4]			Per Ray Terhune with EPA Region IV, it is acceptable to analyze only one calibration point and the calibration blank after every ten samples and at the end of the sample run.
76	What is the acceptance criterion of 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.
77	What corrective action does the laboratory take if the calibration blank does not meet the acceptance criterion? [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. Recalibrate/re-verify the curve.
78	What is the acceptance criterion of the calibration standard? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.3.4] [15A NCAC 2H .0805 (e) (5)] **ANSWER:**			If the calibration standard exceeds ±10%, the instrument must be recalibrated and verified. If that failing calibration standard exceeded ±15%, the previous 10 samples, spikes and duplicates MUST be reanalyzed after the recalibration. If the failing CCV was between ± 10-15%R, then the samples do NOT need to be reanalyzed. However, per our Rules, 15A NCAC 02H .0805 (e) (5), the samples that were not reanalyzed MUST be qualified if reported.

79	Is a Laboratory Fortified Matrix (LFM) analyzed at minimum frequency of 10% of samples? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.4.2]			For water samples, the added analyte concentration must be the same as that used in the laboratory fortified blank (Section 7.9). For solid samples, the concentration added should be 100 mg/kg equivalent (200 µg/L in the analysis solution) except silver which should be limited to 50 mg/kg (Section 1.8). Over time, samples from all routine sample sources should be fortified.
80	How is the LFM prepared? [NC WW/GW LCB Matrix Policy and Technical Assistance] **ANSWER:**			Volume of spiking solution must not exceed 5% of total volume.
81	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.
82	What is the acceptance criterion of the LFM? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.4.3] [15A NCAC 2H .0805 (a) (7) (A)] **ANSWER:**			Calculate the percent recovery for each analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range of 70-130%.
83	How are internal standards responses evaluated? [EPA Method 200.8, Rev. 5.4 (1994), Section 9.4.5] **ANSWER:**			The analyst is expected to monitor the responses from the internal standards throughout the sample set being analyzed. Ratios of the internal standards responses against each other should also be monitored routinely. This information may be used to detect potential problems caused by mass dependent drift, errors incurred in adding the internal standards or increases in the concentrations of individual internal standards caused by background contributions from the sample. The absolute response of any one internal standard must not deviate more than 60-125% of the original response in the calibration blank. If deviations greater than these are observed, flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor of two, add the internal standards and reanalyze. If after flushing the response of the internal standards in the calibration blank are out of limits, terminate the analysis and determine the cause of the drift. Possible causes of drift may be a partially blocked sampling cone or a change in the tuning condition of the instrument.
84	Are 5% of the samples analyzed in duplicate? [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. Analysis of a LFM duplicate may also fulfill this requirement.
85	What is the duplicate acceptance criterion? [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.

				Limits set by the laboratory
86	What corrective action does the laboratory take if duplicates do not meet the acceptance criterion? [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.
87	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. 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:

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Inspector: _____Date:_____