

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-AES**
Method: **EPA Method 200.7, Rev. 4.4 (1994)**

Equipment and Reagents:

Inductively Coupled Plasma Emission Spectrometer	Drying Oven at 180 ± 5 °C	Argon gas supply - High purity grade (99.99%).
Analytical Balance	Mortar and Pestle, ceramic or nonmetallic material	All acids used for this method must be of ultra high-purity grade or equivalent. Suitable acids are available from a number of manufacturers. Redistilled acids prepared by sub-boiling distillation are acceptable.
Temperature adjustable hot plate or hot block capable of maintaining a temperature of 95 °C	Polypropylene sieve, 5-mesh (4 mm opening)	

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	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 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 SOP documents.
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	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]			Per November 7, 2007 EPA letter, additional metals that are not in Table 1.1 may be analyzed as long as QC is acceptable.
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. Two pH measurements would be required to demonstrate that the pH was less than 2 for at least 24 consecutive hours.
9	Are aqueous samples (except Silica and boron) <u>verified and documented</u> 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.

				Rules: Sample preservation shall be verified and documented.
10	Are Silica samples iced to above freezing but $\leq 6^{\circ}\text{C}$ 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 $\leq 6^{\circ}\text{C}$ during storage? [40 CFR 136.3 Table II]			
12	For dissolved metals, are the samples filtered through a $0.45\ \mu\text{m}$ filter within 15 minutes of collection and before preservation with HNO_3 to $\text{pH} < 2$ S.U.? [40 CFR 136.3 Table II Footnote 7]			For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.
	PROCEDURE – Interferences	L A B	S O P	EXPLANATION
13	Is glassware cleaned according to section 6.10 of the method? [EPA Method 200.7, Rev. 4.4 (1994), Section 6.10]			Several procedures found to provide clean labware include washing with a detergent solution, rinsing with tap water, soaking for four hours or more in 20% (v/v) nitric acid or a mixture of HNO_3 and HCl (1+2+9), rinsing with reagent water and storing clean. Chromic acid cleaning solutions must be avoided because chromium is an analyte.
14	Are spectral overlaps avoided by using an alternate wavelength or by interelement corrections? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.1.2]			This is allowable but not required. Some potential on-line spectral interferences observed for the recommended wavelengths are given in Table 2. These interferences will produce false-positive determinations and be reported as analyte concentrations unless corrected.
15	When interelement corrections are applied, are their accuracies verified by analyzing Spectral Interference Check (SIC) solutions? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.1.3 and 7.13]			When interelement corrections are applied, SIC solutions are needed containing concentrations of the interfering elements at levels that will provide an adequate test of the correction factors as described in Section 7.13. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating plus the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
16	When using the recommended wavelengths in Table 1, are the effects from the known interferences given in Table 2 determined, documented, and automatically corrected by the computer? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.1.4]			
17	If a wavelength other than the recommended wavelength is used, are the on-line and off-line spectral interference effects for all analytes determined, documented, and automatically corrected by the computer? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.1.4]			
18	If the interelement interference accounts for 10% or more of the analyte concentration, is an alternate wavelength free of interference or another approved procedure used? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.1.5]			
19	How are physical interferences mitigated? Check all that apply. <input type="checkbox"/> high-solids nebulizer <input type="checkbox"/> diluting the sample <input type="checkbox"/> using an internal standard element <input type="checkbox"/> wetting the argon prior to nebulization			Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-solids nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element.

	<input type="checkbox"/> using a tip washer [EPA Method 200.7, Rev. 4.4 (1994), Section 4.2]			Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-solids nebulizer, wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rates, especially for the nebulizer, improves instrument stability and precision; this is accomplished with the use of mass flow controllers
20	When observed, are chemical interferences minimized by careful selection of operating conditions (such as incident power and observation height), by buffering of the sample, by matrix matching, and by standard addition procedures? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.3]			Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. Normally, these effects are not significant with the ICP-AES technique. Chemical interferences are highly dependent on matrix type and the specific analyte element.
21	Is a rinse blank used to flush the instrument uptake system and nebulizer between standards, check solutions, and samples to reduce memory interferences? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.4 and 7.10]			A rinse blank is used to flush the instrument uptake system and nebulizer between standards, check solutions, and samples to reduce memory interferences. Memory effects can result from sample deposition on the uptake tubing to the nebulizer, and from the buildup of sample material in the plasma torch and spray chamber.
22	Are rinse times determined by aspirating a standard containing elements corresponding to either their LDR or a concentration ten times those usually encountered, followed by analysis of the rinse blank at designated time intervals? [EPA Method 200.7, Rev. 4.4 (1994), Section 4.4]			The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to either their LDR or a concentration ten times those usually encountered. The aspiration time should be the same as 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 two of the method detection limit, should be noted.
23	How is this documented? [15A NCAC 02H .0805 (a) (7) (E)] ANSWER:			All analytical records, including original observations and information necessary to facilitate historical reconstruction of the calculated results, shall be maintained for five years.
	PROCEDURE – Sample Preparation	L A B	S O P	EXPLANATION
24	Are samples digested/extracted prior to analysis? [EPA Method 200.7, Rev. 4.4 (1994), Section 1.5]			
25	Are samples containing suspended or particulate material $\geq 1\%$ (w/v) extracted as a solid-type sample? [EPA Method 200.7, Rev. 4.4 (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.
26	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 200.7, Rev. 4.4 (1994), Section 1.7]			This is a method recommendation.
27	For the analysis of tin in solid samples, are they prepared using aliquots <1 g when sample concentrations exceed 1%? [EPA Method 200.7, Rev. 4.4 (1994), Section 1.7]			This is a method recommendation.
28	When barium is analyzed in samples having varying or unknown concentrations of sulfate, is analysis completed as soon as possible after sample preparation? [EPA Method 200.7, Rev. 4.4 (1994), Section 1.8]			This is a method recommendation.
	PROCEDURE – Sample Preparation – Dissolved Metals	L A B	S O P	EXPLANATION
29	Is a ≥ 20 mL aliquot of a filtered and acid preserved sample pipetted into a 50 mL polypropylene centrifuge tube? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.1.1]			
30	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 200.7, Rev. 4.4 (1994), Section 11.1.1]			See method sections 11.2.2 through 11.2.7.
31	Is an appropriate volume of (1+1) nitric acid added to adjust the acid concentration of the aliquot to approximate			For example, add 0.4 mL (1+1) HNO ₃ to a 20 mL aliquot of

	a 1% (v/v) nitric acid solution? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.1.1]			sample.
32	Is the tube capped and mixed prior to sample analysis? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.1.1]			The sample is now ready for analysis.
	PROCEDURE – Sample Preparation – Total Metals (aqueous)	L A B	S O P	EXPLANATION
33	What initial volumes of sample are used? ANSWER:			
34	Is a well-mixed acid preserved sample transferred to an appropriate digestion vessel? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.2.2]			Although the method calls for 100 mL, smaller sample aliquot volumes may be used under method flexibility allowances.
35	Is 2 mL of (1+1) nitric acid and 1.0 mL of (1+1) hydrochloric acid (per 100 mL sample) added to the beaker? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.2.3]			If a smaller sample volume is used, proper ratios of acids must be maintained.
36	Is the beaker placed on a hot plate at 85 °C in a fume hood and covered with an elevated watch glass or other steps taken to prevent contamination? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.2.3]			Once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95 °C.
37	Is the sample volume reduced to about 20 mL by gentle heating at 85 °C (not boiling)? [EPA Method 200.7, Rev. 4.4 (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.
38	Is the beaker then covered with a watch glass to prevent additional evaporation and gently refluxed for 30 minutes? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.2.5]			
39	After cooling, what volume are samples brought to after digestion? [EPA Method 200.7, Rev. 4.4 (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.
40	Is undissolved material allowed to settle overnight or centrifuged? [EPA Method 200.7, Rev. 4.4 (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.
	PROCEDURE – Sample Preparation – Total Metals (non-aqueous)	L A B	S O P	EXPLANATION
41	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 200.7, Rev. 4.4 (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 = $\frac{\text{Dried Weight}}{\text{Wet Weight}} \times 100$
42	Is the dried sample sieved using a 5-mesh polypropylene sieve and ground in a mortar and pestle? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.3.2]			The sieve, mortar, and pestle should be cleaned between samples.
43	Is 1.0 ± 0.01 g of the dried, ground material transferred to a 250 mL Phillips beaker? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.3.2]			
44	Is 4 mL of (1+1) HNO ₃ and 10 mL of (1+4) HCL added to the beaker? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.3.3]			
45	Is the beaker placed on a hot plate at 85 °C in a fume hood, covered with an elevated watch glass, and gently refluxed for 30 minutes? [EPA Method 200.7, Rev. 4.4 (1994), Sections 11.3.3 & 11.3.4]			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).
46	After cooling, is the extract diluted to 100 mL in a volumetric flask and mixed well? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.3.5]			
47	Is the solution allowed to stand overnight to separate insoluble material or centrifuged? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.3.6]			The sample extract 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.
	PROCEDURE – Sample Preparation – Microwave Digestion	L A B	S O P	EXPLANATION
48	Is this digestion procedure only used for Al, Sb, As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Ni, P, Se, and Zn? [40 CFR 136.3 Table IB, Footnote 36]			
49	Is the instrument power check performed prior to sample digestion? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.1 and 8.2.1; Appendix 1 and Appendix 2.]			This procedure checks to ensure the microwave is heating properly. There is a check at 75% power for 1-6 samples and at 100% power for 7-12 samples.
50	Are 3 mL of concentrated HNO ₃ and 2 mL of concentrated HCl added to 50.0 mL of acid preserved sample in a Teflon PFA vessel? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.2 and 8.2.2]			
51	Is the safety pressure relief valve added and the vessel capped at 16.3 N.m (12 ft/lbs) torque using the capping station? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.2 and 8.2.2]			
52	Is the vessel weighed? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.2 and 8.2.2]			Record weight
53	Is the vessel placed in the microwave instrument turntable with a venting tube attached? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.2 and 8.2.2]			
54	If a full set of either 6 or 12 vessels are not used, are the remaining vessels filled with 50 mL of reagent water and 3 mL of concentrated HNO ₃ and 2 mL of concentrated HCl? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.3 and 8.2.3]			It is critical to the digestion that the total volume of solutions equals 330 mL for 1-6 samples and 660 mL for 7-12 samples. This is necessary to ensure uniform heating of all vessel solutions.
55	Is the exhaust set to maximum fan speed and the turntable activated so that it is rotating 360° continuously in one direction or alternating? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.4 and 8.2.4]			
56	Is the microwave set to operate at the correct power for the correct amount of time? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.5 and 8.2.5]			These heating parameters will allow the samples to reach a maximum temperature of 165 ± 5 °C.
	1-6 samples: 364-420 W at 75% power for 30 minutes			
	7-12 samples: 575-635 W at 100% power for 50 minutes			
	7-12 samples: 635-700 W at 100% power for 30 minutes			
57	If the instrument does not deliver the wattages at the listed power, is it adjusted to deliver the appropriate power? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.5 and 8.2.5]			
58	At the end of the digestion period, are the sample solutions removed from the instrument and allowed to cool to room temperature? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.6 and 8.2.6]			
59	Are the vessels shaken and vented to allow gas pressure to escape? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.6 and 8.2.6]			
60	Is the vent tubing removed and the vessels weighed after cooling to room temperature? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.7 and 8.2.7]			

61	If any vessel has a weight loss between 0.5-2.5 g, is an amount of reagent water equal to the weight loss added? [Closed Vessel Microwave Digestion of Wastewater Samples For Determination of Metals. April 16, 1992. Sections 8.1.7 and 8.2.7]			An analysis with a sample weight loss greater than 2.5 g should be repeated to determine if any analyte was lost. Samples containing large amounts of organics may experience excessive loss of liquid (greater than 10%)
62	If necessary, are silicates and other insoluble material removed by:			In this step, do not rinse or dilute the digested sample. Sample is now ready for analysis.
	a) Gravimetric filtration using a pyrex funnel with Whatman no. 41 filter paper?			
	b) Decanting after wither centrifuging or overnight settling of the insoluble material by gravity?			
PROCEDURE – Instrument Calibration		L A B	S O P	EXPLANATION
63	Are plasma operating conditions optimized prior to using this method? [EPA Method 200.7, Rev. 4.4 (1994), Section 10.2]			The purpose of plasma optimization is to provide a maximum signal to background ratio for the least sensitive element in the analytical array. Detailed instructions are provided in Section 10.2. and may be described in the instrument manual.
64	Prior to daily calibration, is the sample introduction system inspected and cleaned when needed? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.1]			
65	Is the instrument system configured to the selected power and operating conditions prior to calibration? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.2]			See sections 10.1 and 10.2.
66	Is the instrument allowed to become thermally stable before calibration? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.3]			30-60 minute warm up time.
67	After warmup, is any required optical profiling or alignment particular to the instrument performed? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.3]			
68	Is the instrument calibrated according to manufacturer's instructions? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.4]			The calibration line should consist of a minimum of a calibration blank and a high standard. Replicates of the blank and high standard provide an optimal distribution of calibration standards to minimize the confidence band for a straight line calibration in a response region with uniform variance.
	How many non-zero standards are used? ANSWER:			
69	Is a peristaltic pump used to introduce all solutions to the nebulizer? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.4]			
70	Are all solutions aspirated for 30 seconds after reaching the plasma before beginning integration of the background corrected signal to accumulate data? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.4]			
71	Is the system flushed with a rinse blank for a minimum of 60 seconds between each standard? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.4]			
PROCEDURE – Sample Analysis		L A B	S O P	EXPLANATION
72	Are samples analyzed in the same operational manner used in the calibration routine? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.5]			
73	If the sample is diluted, is the detection limit raised accordingly? [EPA Method 200.7, Rev. 4.4 (1994), Section 11.4.7]			
74	For dissolved aqueous analytes, are the results reported directly from the instrument with allowance for sample dilution? [EPA Method 200.7, Rev. 4.4 (1994), Section 12.2]			
75	For total recoverable aqueous analytes, is the instrument result multiplied by the dilution factor of 0.5 when a 100 mL aliquot is used to produce the 50 mL final solution with allowance for sample dilution? [EPA Method 200.7, Rev. 4.4 (1994), Section 12.3]			
76	If a different volume is used for sample preparation, is the			

	dilution factor adjusted properly? [EPA Method 200.7, Rev. 4.4 (1994), Section 12.3]			
77	For analytes with MDLs ≥ 0.01 mg/L, are results rounded to the nearest hundredth place and reported to three significant figures? [EPA Method 200.7, Rev. 4.4 (1994), Section 12.4]			
78	For total recoverable analytes in solid samples, are the mg/L results rounded as above and reported to three significant figures as mg/kg dry-weight basis? [EPA Method 200.7, Rev. 4.4 (1994), Section 12.5]			$\text{Sample Conc. (mg/kg) = } \frac{C \times V \times D}{W}$ Dry-weight basis C= concentration in extract (mg/L) V= volume of extract (L) D= dilution factor (undiluted = 1) W=weight of sample aliquot extracted (g x 0.001 = kg)
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
79	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 200.7, Rev. 4.4 (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.
80	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.
81	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.
82	Is a known second-source standard analyzed after initial calibration? [15A NCAC 2H .0805 (a) (7) (H) (ii) and EPA Method 200.7, Rev. 4.4 (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 7.12 and 9.2.3 of the method.
83	What is the acceptance criterion of the second source standard? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.2.3] ANSWER:			To verify the calibration standards the determined mean concentrations from three analyses of the QCS must be within $\pm 5\%$ of the stated values. If the calibration standard cannot be verified, performance of the determinative step of the method is unacceptable. 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.
84	What corrective action does the laboratory take if the second source standard does not meet the acceptance criterion? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.2.3] ANSWER:			If the calibration standard cannot be verified, performance of the determinative step of the method is unacceptable. 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.
85	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.
86	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.
87	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.

88	Is at least one Laboratory Reagent Blank (LRB) analyzed with each batch of 20 or fewer of samples of the same matrix? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.3.1]		The laboratory must analyze at least one LRB (Section 7.10.2) with each batch of 20 or fewer samples of the same matrix. LRB data are used to assess contamination from the laboratory environment.
89	What is the acceptance criterion of the LRB? [15A NCAC 2H .0805 (a) (7) (H) (i) or EPA Method 200.7, Rev. 4.4 (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.
90	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. LRB values that exceed the MDL indicate laboratory or reagent contamination should be suspected. When LRB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained.
91	Is at least one Laboratory Fortified Blank (LFB) analyzed with each batch of samples? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.3.2]		The laboratory must analyze at least one LFB (Section 7.10.3) with each batch of samples. The laboratory fortified blank (LFB) is prepared by fortifying an aliquot of the laboratory reagent blank with all analytes to a suitable concentration using the following recommended criteria: Ag ≤ 0.1 mg/L, K ≥ 5.0 mg/L and all other analytes 0.2 mg/L or a concentration approximately 100 times their respective MDL, whichever is greater. The LFB must be carried through the same entire preparation scheme as the samples including sample digestion, when applicable.
92	What is the acceptance criterion of the LFB? [EPA Method 200.7, Rev. 4.4 (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.
93	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.
94	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 200.7, Rev. 4.4 (1994), Section 9.3.4]		For all determinations the laboratory must analyze the IPC solution (Section 7.11) 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.
95	What is the acceptance criterion of the calibration blank? [15A NCAC 2H .0805 (a) (7) (H) (i) or EPA Method 200.7, Rev. 4.4 (1994), Section 9.3.4] 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. Analysis of the calibration blank should always be $<$ the analyte IDL, but greater than the lower 3-sigma control limit of the calibration blank.
96	What corrective action does the laboratory take if the calibration blank does not meet the acceptance criterion? [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

	ANSWER:		corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. Recalibrate/re-verify the curve.
97	What is the acceptance criterion of the calibration standard? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.3.4] ANSWER:		Analysis of the IPC solution immediately following calibration must verify that the instrument is within $\pm 5\%$ of calibration with a relative standard deviation $< 3\%$ from replicate integrations ≥ 4 . Subsequent analyses of the IPC solution must be within $\pm 10\%$ of calibration.
98	What corrective action does the laboratory take if the calibration standard does not meet the acceptance criterion? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.3.4] ANSWER:		If the calibration cannot be verified within the specified limits, reanalyze either or both the IPC solution and the calibration blank. If the second analysis of the IPC solution or the calibration blank confirm calibration to be outside the limits, sample analysis must be discontinued, the cause determined, corrected and/or the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.
99	Is a Laboratory Fortified Matrix (LFM) analyzed at a minimum frequency of 10% of samples? [EPA Method 200.7, Rev. 4.4 (1994), Section 9.4.2]		The laboratory must add a known amount of each analyte to a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determinations added prior to sample preparation. For water samples, the added analyte concentration must be the same as that used in the laboratory fortified blank (Section 7.10.3). For solid samples, however, the concentration added should be expressed as mg/kg and is calculated for a one gram aliquot by multiplying the added analyte concentration (mg/L) in solution by the conversion factor $100 \text{ (mg/L} \times 0.1\text{L}/0.001\text{kg} = 100$, Section 12.5). (For notes on Ag, Ba, and Sn see Sections 1.7 and 1.8.) Over time, samples from all routine sample sources should be fortified.
100	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.
101	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.
102	What is the acceptance criterion of the LFM? [EPA Method 200.7, Rev. 4.4 (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% or a 3-sigma recovery range calculated from the regression equations given in Table 9. NC rule requires acceptance criterion to be established.
103	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.
104	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 must be set by the laboratory.
105	What corrective action does the laboratory take if duplicates do not meet the acceptance criterion? [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,

	ANSWER:		and any samples involved shall be reanalyzed, if possible.
106	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)]		<p>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.</p> <p>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.</p>

Additional Comments:

Inspector: _____ Date: _____