NC DEQ/DWR LABORATORY CERTIFICATION

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

Parameter: **Mercury**
Cold Vapor Atomic Absorption Spectrometry, Manual (Aqueous)
Method: **EPA Method 245.1, Rev. 3.0, 1994**

EQUIPMENT:

	Atomic Absorption Cold Vapor System: (AA Spectrophotometer, Mercury Hollow Cathode Lamp, Absorption Cell, Aeration Tubing, Air Pump, Drying Tube, Recorder)
	Or a Mercury Analyzer:
	Flowmeter – capable of measuring flow of 1 L/min
	Water Bath – able to maintain 2-3 in. water at 95°C
	Heating block – able to maintain 95°C with polypropylene digestion tubes
	Analytical balance – capable of measuring to 0.1 mg
	Labware: Glassware, BOD bottles or suitable closed containers, assorted pipettes
	Stir plate and stir bar

REAGENTS & STANDARDS:

	Reagent Water, ASTM Type II		Potassium Persulfate, 5%
	Nitric Acid		Sodium chloride-hydroxylammonium chloride
	Sulfuric Acid		or Sodium chloride – hydroxylamine sulfate
	Mercury Stock Standard		Potassium Permanganate, 5%
	Carrier gas:		Stannous chloride

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 2H .0805 (a) (7)] **ANSWER:**			Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made. Verify proper method reference. During review notate deviations from the approved method and SOP.
2	Are all revision dates and actions tracked and documented? [15A NCAC 2H .0805 (a) (7)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents.
3	Is there North Carolina data available for review?			If not, review PT data
	PRESERVATION and STORAGE	**LAB**	**SOP**	**EXPLANATION**
4	Are samples collected in polyethylene, fluoropolymer (e.g., PTFE or Teflon®) or glass bottles? [40 CFR 136.3 Table II and footnote 1]			
5	Are samples preserved with HNO_3 to pH of <2 s.u.? [40 CFR 136.3 Table II and footnotes 2 and 3]			Normally, 3 mL of (1+1) nitric acid per liter of sample is sufficient for most samples.
6	After acidification, is the sample mixed and held for 16 hours and the pH verified to be <2 S.U. just prior to processing? [EPA Method 245.1, Rev. 3.0, 1994, Section 8.2]			Following acidification, the sample should be mixed, held for 16 hours, and then verified to be pH <2 just prior to withdrawing an aliquot for processing.

				Certification Note - Even though the initial method directions say that it **should** be held for 16 hours, it later goes on to say that if the pH is >2 immediately prior to processing, that additional acid **must** be added and held for an additional 16 hours.
7	If the pH is >2 S.U., then is more acid added and the sample held for an additional 16 hours? [EPA Method 245.1, Rev. 3.0, 1994, Section 8.2]			If for some reason such as high alkalinity the sample pH is verified to be >2, more acid must be added and the sample held for additional 16 hours until verified to be pH <2.
8	Does the sample documentation verify that the pH was <2 S.U. for 16 hours prior to analysis? [15A NCAC 2H .0805 (a) (7) (L)]			A record of sample collection date, sample collection time, sample collector, and the use of proper preservatives and preservation techniques shall be maintained. Bottom line is that the sample must be verified to have been at a pH <2 for at least 16 hours prior to beginning the digestion.
9	Are samples analyzed within 28 days of collection? [40 CFR 136.3 Table II]			
	PROCEDURE – Instrument Preparation	**LAB**	**SOP**	**EXPLANATION**
10	If the monochromator is adjustable, then is it set to 253.65 nm? [EPA Method 245.1, Rev. 3.0, 1994, Section 10.1] If using a Mercury Analyzer, skip to Question 13.			If adjustable, the monochromator should be set to 253.65 nm.
11	Is the air flow optimized prior to analysis? [EPA Method 245.1, Rev. 3.0, 1994, Section 10.1]			Prior to the use of this method the air flow should be optimized. The recommended air flow rate through the system is 1 L/min.
12	Is the instrument allowed to warm up for at least 15 minutes prior to analysis? [EPA Method 245.1, Rev. 3.0, 1994, Section 10.1]			For all determinations allow an instrument and hollow cathode lamp warm up period of not less than 15 minutes.
13	Are the manufacturer's instructions followed for Mercury Analyzers? [EPA Method 245.1, Rev. 3.0, 1994, Section 10.1]			When an instrument; designed specifically for the determination of mercury by the cold vapor technique, is being utilized, the analyst should follow the instructions provided by the manufacturer.
	PROCEDURE – Calibration Standard and Sample Preparation	**LAB**	**SOP**	**EXPLANATION**
14	Is a 100-mL sample/standard aliquot utilized for analysis (or a smaller aliquot diluted to 100 mL)? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.1] **If using reduced volume, note volume used:**			Transfer 100 mL of the water sample [or an aliquot diluted with reagent water (Section 7.2) to 100 mL] into a sample container. Note: For reduced volume analysis, adjust sample and reagent volumes to maintain the required sample to reagent ratios for the following: H_2SO_4 $K_2S_2O_8$ HNO_3 $NaCl$-(NH_2OH)•H_2SO_4 (or alt) $KMnO_4$ $SnCl_2$
15	Is 5 mL of H_2SO_4 and 2.5 mL HNO_3 added to each standard/sample? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.2]			Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to the container.
16	Is 15 mL of $KMnO_4$ solution added to each standard/sample? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.3]			To each container add 15 mL $KMnO_4$ solution (Section 7.7).
17	Are the standards/samples shaken until the purple color persists for at least 15 minutes (adding more $KMnO_4$, if necessary)? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.3]			Shake and add additional portions of $KMnO_4$ solution, if necessary (may be needed for sewage or industrial wastewaters), until the purple color persists for at least 15 minutes.
18	Is 8 mL of $K_2S_2O_8$ solution added to each standard/sample? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.3]			Add 8 mL of $K_2S_2O_8$ solution (Section 7.8) to each container.
19	Are the standards/samples mixed thoroughly and covered appropriately? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.3]			Mix thoroughly, cap and cover the top of the sample container (if required) with aluminum foil or other appropriate cover.

20	Are the **samples** heated for two hours at 95°C? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.3]			Heat samples for two hours in a water bath at 95°C. (**Do not heat standards**). Note: Section 11.2.2 states to process calibration standards as in 11.1.3 without heating. EPA Region IV has given guidance that the reason this method does not include heating is due to the fact that the standards only include the inorganic form of Hg. Heating is meant to dissociate organic or bound Hg and is therefore unnecessary. As such, EPA did not recommend combining 245.1 with 7470A since 7470A states that the standards are heated like the samples. Laboratories may combine methods but would have to follow the EPA 245.1 requirements.
21	Are the samples removed from heat and allowed to cool to room temperature? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.4]			Remove the sample containers from heat and cool to room temperature. (During the cool down period proceed with instrument warm up and calibration.)
22	Is 6 mL of sodium chloride-hydroxylamine sulfate or sodium chloride-hydroxylammonium chloride solution added to each standard/sample to reduce excess permanganate? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.1.5] Circle which solution is used			When the samples are at room temperature, to each container, add 6 mL of sodium chloride-hydroxylamine sulfate ($NaCl$-$(NH_2OH)_2$•H_2SO_4) solution (Section 7.9) to reduce the excess permanganate. Alternatively, 6 mL sodium chloride-hydroxylammonium chloride may be used per Section 7.9.
	PROCEDURE – Calibration Standard and Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
23	Is the $SnCl_2$ solution stirred continuously during use, as recommended? [EPA Method 245.1, Rev. 3.0, 1994, Section 7.10]			Stannous chloride solution - Add 25 g of $SnCl_2$ • $2H_2O$ to 250 mL of 0.5 N H_2SO_4 (Section 7.4.1). This mixture is a suspension and should be stirred continuously during use.
24	Is 5 mL of $SnCl_2$ solution added to each **standard** individually, and immediately attached to the aeration apparatus? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.3]			Treating each standard solution container individually, add 5 mL of $SnCl_2$ solution (Section 7.10) and immediately attach the container to the aeration apparatus.
25	Is the absorbance allowed to reach its maximum response, approximately 30 seconds to one minute, before the bypass valve is opened and the response allowed to return to its minimum value? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.3]			The absorbance, as exhibited either on the instrument or recording device, will increase and reach maximum within 30 sec. As soon as the maximum response is obtained, approximately one minute, open the bypass valve (or optionally remove aspirator from the sample container if it is vented under the hood) and continue aeration until the absorbance returns to its minimum value.
26	Is the bypass valve closed and the standard removed from the aspirator? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.4]			Close the by-pass value, remove the aspirator from the standard solution container and continue aeration. Repeat (Section 11.2.3) until data from all standards have been collected.
27	Is the calibration blank used to auto-zero the instrument or is the calibration curve (including the calibration blank) forced through the origin? [EPA Method 245.1 Rev. 3.0 (1994), Section 3.1]			The calibration blank is a zero standard and is used to auto-zero the instrument. Forcing through the origin essentially auto-zeroes the instrument with the calibration blank, which is what you want to do.
28	Is a standard curve consisting of an undigested calibration blank and 5, **undigested**, non-zero standards analyzed daily? [EPA Method 245.1 Rev. 3.0 (1994), Section 11.2.2] **List values of standards used:**			Prepare calibration standards by transferring 0.5, 1.0, 2.0, 5.0, and 10 mL aliquots of the 0.1 µg/mL CAL (Section 7.6) to a series of sample containers (Section 6.5.2). Dilute the standard aliquots to 100 mL with reagent water (Section 7.2) and process as described in Sections 11.1.2, 11.1.3 (**without heating**),

				and 11.1.5. These solutions contain 0.05-1.0 µg of Hg. (Other appropriate calibration standards, volumes, and ranges may also be used.)
29	Does each standard curve have a correlation coefficient of ≥0.995? [NC WW/GW LC Policy]			When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of determination, r^2, of 0.99) is required.
30	How is the standard curve constructed? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.5] **ANSWER:**			Construct a standard curve by plotting peak height, area or maximum response obtained from each standard solution, versus micrograms of mercury in the container. The standard curve must comply with Section 9.2.2. Calibration using computer or calculator based regression curve fitting techniques on concentration/response data is acceptable.
31	For **samples**, is the aspirator placed inside the container but above the liquid to purge the head space to remove possible gaseous interference? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.6]			However, prior to the addition of the SnCl2 solution, place the aspirator inside the container above the liquid, and purge the head space (20-30 seconds) to remove possible gaseous interference.
32	Following the purge of head space, are the digested samples analyzed in the same manner as the standards? [EPA Method 245.1, Rev. 3.0, 1994, Section 11.2.6]			Following calibration, the digested samples are analyzed in the same manner as the standard solutions described in Section 11.2.3.
	QUALITY ASSURANCE	**LAB**	**SOP**	**EXPLANATION**
33	Is the undigested, second-source Quality Control Sample (QCS) analyzed after each initial calibration, prior to sample analysis? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.2.3] [15A NCAC 2H .0805 (a) (7) (H) (ii)] **ANSWER:**			Rule: Laboratories shall analyze one known second source standard to verify the accuracy of standard preparation if an initial calibration is performed and in accordance with the referenced method requirements thereafter. Method: When beginning the use of this method, on a quarterly basis, after the preparation of stock or calibration standard solutions or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. The QCS must be obtained from an outside source different from the standard stock solution, but prepared in the same manner as the calibration solutions (i.e., not digested). The concentration of the mercury in the QCS solution should be such that the resulting solution will provide an absorbance reading near the midpoint of the calibration curve.
34	Is the acceptance range for the QCS standard ±10% of the stated value? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.2.3]			To verify the calibration standards, the determined concentration of the QCS must be within ±10% of the stated value.
35	What action is taken when the QCS does not meet the established acceptance criteria? [EPA Method 245.1 Rev. 3.0 (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 ongoing analyses.

36	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.
37	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.
38	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.
39	Is the MDL established following the requirements of the 40 CFR Appendix B?			
40	How often is the MDL determined? [40 CFR 136 Appendix B]			Ongoing data accumulation and annual verification is required with the 2017 MUR that was made effective September 27, 2017
41	Does the laboratory digest and analyze at least one Laboratory Reagent Blank (LRB) with each digestion batch of 20 or fewer samples? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.1]			EPA Method - The laboratory must analyze at least one LRB (Section 7.11.2) with each batch of 20 or fewer samples of the same matrix.
42	Is the LRB prepared in the same manner as the calibration blank and then carried through the entire sample preparation scheme and treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples? [EPA Method 245.1 Rev. 3.0 (1994), Section 3.8 and 7.11.2]			7.11.1 - The calibration blank must contain all reagents in the same concentrations and in the same volume as used in preparing the calibration solutions. The LRB is then one calibration blank carried through the rest of the sample preparation In Section 3.8, the LRB is defined as an aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus. This includes chemical preservatives.
43	Is the LRB acceptance criterion ≤ 50% of the reporting limit? [15A NCAC 2H .0805 (a) (7) (H) (i)]			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.
44	If the NC WW/GW LC LRB acceptance criterion of ≤ 50% RL is not used, is the LRB acceptance criterion <10% of the analyte level determined for any associated samples or <2.2 times the analyte MDL, whichever is greater? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.1] [15A NCAC 2H .0805 (a) (7) (H) (i)]			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. Rule: states that the ≤50% RL or method referenced criterion may be used.
45	What corrective action is taken if the LRB exceeds the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B)] [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.1]			Rule - 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

	ANSWER:			resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. EPA Method - 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.
46	Is a calibration blank analyzed immediately following each calibration, after every 10th sample and at the end of the sample run? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.4]			EPA Method – For all determinations the laboratory must analyze the IPC solution and a calibration blank immediately following each calibration, after every 10th sample (or more frequently, if required) and at the end of the sample run. The instrument is not to be zeroed with each analysis of the calibration blank.
47	Is the acceptance criterion for the calibration blanks ≤ 50% of the reporting limit? [15A NCAC 2H .0805 (a) (7) (H) (i)]			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.
48	What corrective action is taken if the calibration blank is greater than 50% of the reporting limit? [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.
49	Does the lab analyze an undigested, mid-range Instrument Performance Check (IPC) standard immediately after calibration, after every ten samples and at the end of the sample run? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.4]			For all determinations the laboratory must analyze the IPC solution (Section 7.12) and a calibration blank immediately following each calibration, after every 10th sample (or more frequently, if required) and at the end of the sample run. 7.12 - It **must** contain all reagents in the same concentration as the calibration solutions and mercury at an appropriate **concentration to approximate the midpoint** of the calibration curve. The IPC solution should be prepared from the same CAL standard (Section 7.6) as used to prepare the calibration solutions.
50	Is the acceptance criterion for the IPC standard immediately following calibration ±5% of the true value? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.4]			Analysis of the IPC solution immediately following calibration must verify that the instrument is within ±5% of calibration.
51	Is the acceptance criterion for subsequent analyses of the IPC standard ±10% of the true value? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.4]			Subsequent analyses of the IPC solution must be within ±10 % of calibration.
52	What action is taken if the IPC does not meet the established acceptance criteria? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.4] **ANSWER:**			If the calibration cannot be verified within the specified limits, 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. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.
53	Is at least one Laboratory Fortified Blank (LFB) digested and analyzed with each digestion batch of samples? [EPA Method 245.1 Rev. 3.0			The laboratory must analyze at least one LFB (Section 7.11.3) with each batch of samples.

	(1994), Section 3.6 and 9.3.2]			Section 3.6 defines an LFB as an aliquot of LRB to which a known quantity of the method analyte is 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.
54	Is the LFB prepared by fortifying a volume of laboratory reagent blank solution with mercury between >10X MDL and < the midpoint of the standard concentrations and carried through the entire sample preparation scheme? [EPA Method 245.1 Rev. 3.0 (1994), Section 7.11.3] **State concentration:**			The laboratory fortified blank (LFB) is prepared by fortifying a sample size volume of laboratory reagent blank solution with mercury to a suitable 245.1-7 concentration of >10X the MDL, but less than the midpoint concentration of the calibration curve. The LFB must be carried through the entire sample preparation scheme.
55	How is the LFB recovery calculated? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.2] **ANSWER:**			Calculate accuracy as percent recovery using the following equation: $R = \frac{LFB - LRB}{s} \times 100$ where: R = percent recovery LFB = laboratory fortified blank LRB = laboratory reagent blank s = concentration equivalent of mercury added to fortify the LRB solution. Note: the LRB is subtracted
56	What is the acceptance range of the LFB? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.3] **ANSWER:**			The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 85-115%. Optional control limits may be calculated, but must be equal to or better than the required 85-115%.
57	What corrective action is performed if the LFB falls outside the control limits? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.3.2] **ANSWER:**			If the recovery of mercury falls outside the required control limits of 85-115%, the analysis is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
58	Is a known amount of mercury added to at least 10% of samples or one sample per set, whichever is greater? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.4.2]			The laboratory must add a known amount of mercury to a minimum of 10% of samples or one sample per sample set, whichever is greater. In each case the Laboratory Fortified Matrix (LFM) (i.e., matrix spike) aliquot must be a duplicate of the aliquot used for sample analysis. Select a sample with a low mercury background that is representative of the type of water samples being analyzed. It is recommended that this sample be analyzed prior to fortification. The concentration of mercury added may vary based on the nature of samples being analyzed. When possible, the concentration should be the same as that added to the LRB, but should not exceed the midpoint concentration of the calibration curve. Over time, samples from all routine sample sources should be fortified.
59	Does the volume of spike solution used constitute ≤5% of the total MS volume? [NC WW/GW LC policy]			The volume of spike solution used in MS preparation must in all cases be ⩽ 5% of the total MS volume. 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. If the sample concentration is below the reporting limit, use zero for amount of target in the unspiked sample.
60	How is the MS prepared? [NC WW/GW LC Matrix Spike Technical Assistance.] **ANSWER:**			See Matrix Spike Technical Assistance document.
61	How is the percent recovery of the MS calculated? [NC WW/GW LC Matrix Spike Technical Assistance] **ANSWER:**			See Matrix Spike Technical Assistance document. If the spike solution volume constitutes >1% of the total sample volume, the sample concentration or spike concentration must be adjusted by calculation.
62	Is the established control limit for the MS ±30%? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.4.3]			Calculate the percent recovery, corrected for background concentration measured in the unfortified sample aliquot, and compare these values to the control limits to the designated LFM recovery range of 70-130%.
63	What action is taken when MS recovery falls outside of the designated range? [EPA Method 245.1 Rev. 3.0 (1994), Section 9.4.4] **ANSWER:**			If mercury recovery falls outside the designated range, and the laboratory performance is shown to be in control (i.e., LRB/LFB/IPC acceptable), the recovery problem encountered with the fortified water sample is judged to be matrix related, not system related. The result for mercury in the unfortified sample must be labelled to inform the data user that the results are suspect due to matrix effects.
64	Does the laboratory analyze duplicate samples at a rate of 5%? [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.
65	What is the acceptance criterion for duplicates? [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. Establish laboratory control limits.
66	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits? [15A NCAC 2H .0805 (a) (7) (B)] **ANSWER:**			If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
67	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.
68	Are sample values calculated by comparing the sample response with the standard curve? [EPA Method 245.1 Rev. 3.0 (1994), Section 12.1]			From the prepared calibration curve (Section 11.2.4) compute sample values by comparing response with the standard curve.
69	How are concentrations of the sample calculated? [EPA Method 245.1			Calculate the mercury concentration in the

	Rev. 3.0 (1994), Section 12.2] **ANSWER:**			sample by the formula: $\mu g\ Hg/L = \left(\frac{\mu g\ Hg\ in}{aliquot}\right)\left(\frac{1{,}000}{mL\ of\ aliquot}\right)$
70	How are the results reported with regard to units and significant figures? [EPA Method 245.1 Rev. 3.0 (1994), Section 12.3] **ANSWER:**			Report mercury concentrations to the proper significant figures in mg/L, µg/L or ng/L as required.

Additional Comments:
_____
_____
_____
Inspector:_____Date:_____