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, Manual
Method: **SW-846 Method 7470A (Aqueous)**

NOTE: To promote consistency with the use of SW-846 methods and to assure generation of data of known quality, the minimum recommended quality control benchmarks in the methods will be considered the minimum QA/QC requirements (**i.e., when the method says "should", we consider that to mean "must"**).

EQUIPMENT:

	Atomic Absorption Cold Vapor System: (AA Spectrophotometer, Mercury Hollow Cathode Lamp, 10-cm Absorption Cell, Aeration Tubing, Air Pump, Drying Tube, Recorder)
	Or a Mercury Analyzer:
	Flowmeter – capable of measuring air flow of 1 L/min
	Water Bath or hot plate – able to maintain a temperature of 90-95°C
	Analytical balance – capable of measuring to 0.1 mg
	Labware: Glassware, BOD bottles or suitable closed containers, assorted pipettes

REAGENTS & STANDARDS:

	Reagent Water, interference free		Potassium Persulfate, 5% solution (w/v)
	Nitric Acid, conc.		Sodium chloride-hydroxylamine sulfate (or sodium chloride-hydroxylamine hydrochloride)
	Sulfuric Acid, 0.5 *N*		Stannous sulfate (or stannous chloride)
	Mercury Stock Standard		Potassium Permanganate, mercury-free, 5% solution (w/v)
	0.15% Nitric Acid, for working standards		

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 plastic, fluoropolymer or glass bottles? [SW-846 Method 7470A Section 6.2]			Plastic and glass containers are both suitable.

5	Are sample containers prewashed with detergents, acids, and reagent water? [SW-846 Method 7470A Section 6.2]			All sample containers must be prewashed with detergents, acids, and reagent water.
6	Are samples preserved at time of collection with HNO_3 to pH of <2 S.U.? [SW-846 Method 7470A Section 6.3]			Aqueous samples must be acidified to a pH <2 with HNO_3.
7	Is pH checked to document pH <2 S.U. upon receipt? [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.
8	After acidification, is the sample mixed and held for 16 hours and the pH verified <2 S.U. just prior to processing? **Recommendation only for 7470 A**			This requirement is not found in SW-846 Method 7470A but is recommended. EPA Method 245.1 Rev. 3.0, 1994, Section 8.2 requires: 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 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.
9	If the pH is >2 S.U., then is more acid added and the sample held for an additional 16 hours? **Recommendation only for 7470 A**			This requirement is not found in SW-846 Method 7470A but is recommended. EPA Method 245.1 Rev. 3.0, 1994, Section 8.2 requires: 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.
10	Are samples analyzed within 28 days of collection? [SW-846 Method 7470A Section 6.3]			SW-846 Method 7470A: The suggested maximum holding times for mercury is 28 days.
	PROCEDURE – Instrument Preparation	**LAB**	**SOP**	**EXPLANATION**
11	Is the instrument allowed to warm up according to manufacturer's instructions prior to analysis?			For all determinations allow an instrument and hollow cathode lamp warm up period of not less than 15 minutes. EPA Method 245.1, Rev. 3.0, 1994, Section 10.1 says to allow the instrument to warm up at least 15 minutes prior to analysis
12	If the monochromator is adjustable, then is it set to 253.7 nm? [SW-846 Method 7470A Section 2.2] **If using a Mercury Analyzer, skip to question #14.**			Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7 nm by mercury vapor.
13	Is the circulating pump adjusted to a rate of 1 liter/min? [SW-846 Method 7470A Section 7.3]			The circulating pump, which has previously been adjusted to a rate of 1 liter/min, is allowed to run continuously.
14	Are the manufacturer's instructions followed for Mercury Analyzers?			
	PROCEDURE – Calibration Standard and Sample Preparation	**LAB**	**SOP**	**EXPLANATION**
15	How often are the calibration standards prepared? [SW-846 Method 7000B Section 10.1.1] **ANSWER:**			Calibration standards can be prepared fresh each time a batch of samples is analyzed. If the ICV solution is prepared daily and the ICV is analyzed within the acceptance criteria, calibration standards do not need to be prepared daily and may be prepared and stored for as long as the calibration standard viability can be verified through the use of the ICV. If the ICV is outside of the acceptance criteria, the calibration standards must be prepared fresh and the instrument recalibrated.

16	Is a 100-mL sample/standard aliquot utilized for analysis (or a smaller aliquot diluted to 100 mL)? [SW-846 Method 7470A Section 7.1]			**Sample:** Transfer 100 mL, or an aliquot diluted to 100 mL, containing <1.0 g of mercury, to a 300-mL BOD bottle or equivalent. Note: For reduced volume analysis, adjust sample and reagent volumes to maintain the required sample to reagent ratios. **Standard:** Transfer 0, 0.5, 1.0, 2.0, 5.0, and 10.0 mL aliquots of the mercury working standard, containing 0-1.0 µg of mercury, to a series of 300-mL BOD bottles. Add enough reagent water to each bottle to make a total volume of 100 mL.
17	Is 5 mL of H_2SO_4 and 2.5 mL conc. HNO_3 added to each standard/sample and mixed after each addition? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Add 5 mL of H_2SO_4 and 2.5 mL of concentrated HNO_3, mixing after each addition. **Standard:** Mix thoroughly and add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to each bottle.
18	Is 15 mL of $KMnO_4$ solution added to each standard/sample? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Add 15 mL $KMnO_4$ solution to each sample bottle. Sewage [or industry wastewater] samples may require additional permanganate. Ensure equal amounts of permanganate are added to standards and blanks. **Standard:** Add 15 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 min.
19	Are the standards/samples shaken until the purple color persists for at least 15 minutes (adding more $KMnO_4$ if necessary)? [SW-846 Method 7470 A Section 7.1]			**Sample:** Shake and add additional portions of $KMnO_4$ solution, if necessary, until the purple color persists for at least 15 minutes. **Standard:** Add 15 mL of $KMnO_4$ solution to each bottle and allow to stand at least 15 min.
20	Is 8 mL of $K_2S_2O_8$ solution added to each standard/sample? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Add 8 mL of potassium persulfate [$K_2S_2O_8$] solution to each bottle and heat for 2 hr in a water bath maintained at 95°C. **Standard:** Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C.
21	Are the standards/samples heated for two hours in a water bath at 95°C? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Add 8 mL of potassium persulfate [$K_2S_2O_8$] solution to each bottle and heat for 2 hr in a water bath maintained at 95°C. **Standard:** Add 8 mL of potassium persulfate to each bottle and heat for 2 hr in a water bath maintained at 95°C. (**NOTE: When combining SW-846 7470A analyses with EPA Method 245.1, a comparative study showing acceptable results with both heated and unheated standards must be kept on file and available for inspection to demonstrate equivalent performance with undigested standards. EPA 245.1 requires that you do not heat standards).**
22	Are the standards/samples removed from the water bath and allowed to cool? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. **Standard:** Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate.
23	Is 6 mL of sodium chloride-hydroxylamine sulfate solution added to each standard/sample to reduce excess permanganate? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. **Standard:** Cool and add 6 mL of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. **NOTE:** Sodium chloride-hydroxylamine hydrochloride may be substituted for sodium chloride-hydroxylamine sulfate. [SW-846 Method 7470 A Section 5.6]

24	After waiting at least 30 sec, is 5 mL of stannous sulfate added to each standard/sample? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			**Sample:** After a delay of at least 30 sec. add 5 mL of stannous sulfate, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3. **Standard:** When the solution has been decolorized, wait 30 sec. add 5 mL of the stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3. **NOTE:** Stannous chloride may be substituted for stannous sulfate. If so, this mixture is a suspension and should be stirred continuously during use. [SW-846 Method 7470 A Section 5.5]
25	After stannous sulfate addition, are standard/sample bottles immediately attached to the aeration apparatus for analysis? [SW-846 Method 7470 A Section 7.1 and Section 7.2]			Sample: After a delay of at least 30 sec. add 5 mL of stannous sulfate, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3. Standard: When the solution has been decolorized, wait 30 sec. add 5 mL of the stannous sulfate solution, immediately attach the bottle to the aeration apparatus, and continue as described in Paragraph 7.3.
	PROCEDURE – Calibration Standard and Sample Analysis	**LAB**	**SOP**	**EXPLANATION**
26	If not using a mercury analyzer, is the absorbance allowed to reach its maximum response, within approximately 30 seconds, before the bypass valve is opened and the response allowed to return to its minimum value? [SW-846 Method 7470A Section 7.3]			The absorbance will increase and reach a maximum within 30 sec. As soon as the recorder pen levels off (approximately 1 min), open the bypass valve and continue the aeration until the absorbance returns to its minimum value.
27	If not using a mercury analyzer, is the bypass valve closed and the standard removed from the aspirator when the absorbance returns to the minimum value? [SW-846 Method 7470A Section 7.3]			Close the by-pass value, remove the stopper and frit from the BOD bottle, ad continue the aeration. Because of instrument variation refer to the manufacturers recommended operating conditions when using this method.
28	Are metal concentrations calculated by the method of standard additions or from a calibration curve? [SW-846 Method 7470A Section 7.5] **Circle one:** MSA or Calibration Curve			Calculate metal concentrations (1) by the method of standard additions, or (2) from a calibration curve. All dilution or concentration factors must be taken into account.
	QUALITY ASSURANCE	**LAB**	**SOP**	**EXPLANATION**
29	Is a standard curve consisting of a blank and at least 3 non-zero standards analyzed? [SW-846 Method 7000B Section 10.1.1 and 15A NCAC 2H .0805 (a) (7) (H) (iv)**List values of standards used:**			SW-846 7000 B: Prepare a blank and at least three calibration standards in graduated amounts in the appropriate range of the linear part of the curve. Rule: For metals analyses, a series of three or more non-zero standards or standards as set forth in the analytical procedure shall be analyzed with each sample set.
30	Is the standard curve analyzed daily? [SW-846 Method 7000B Section 10.2]			A calibration curve must be prepared each day with a minimum of a calibration blank and three standards.
31	Does each standard curve have a correlation coefficient of ≥0.995? [SW-846 Method 7000B Section 10.2 and NC WW/GW LC Policy]			SW-846 7000B: A calibration curve must be prepared each day with a minimum of a calibration blank and three standards. The curve must be linear and have a correlation coefficient of at least 0.995. Policy: When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation 3coefficient is not specified, then a minimum value of 0.995 (or a coefficient of

				determination, r^2, of 0.99) is required.
32	Are calibration standards and quality control standards analyzed (injected) at least twice and an average value determined? [SW-846 Method 7000B Sections 10.1.3 and 10.3] **Recommended for quality control standards.**			10.1.3: Beginning with the calibration blank and working toward the highest standard, aspirate the solutions and record the readings. Repeat the operation with both the calibration standards and the samples a sufficient number of times to secure an average reading for each solution. Calibration curves are always required. 10.3: It is **recommended** that each standard should be analyzed (injected) twice and an average value determined. Replicate standard values should be within ±10% RPD.
33	At what frequency is the Laboratory Control Sample (LCS) analyzed? [SW-846 7000B Section 9.6] **ANSWER:**			For each batch of samples processed, at least one LCS must be carried throughout the entire sample preparation and analytical process as described in Chapter One.
34	How is the LCS prepared? [SW-846 7000B Section 9.6] **ANSWER:**			The laboratory control samples should be spiked with each analyte of interest at the project-specific action level or, when lacking project-specific action levels, at approximately mid-point of the linear dynamic range. **Concurrent analyses of reference materials (SRMs) containing known amounts of analytes in the media of interest are recommended and may be used as an LCS.** For solid SRMs, 80 - 120% accuracy may not be achievable and the manufacturer's established acceptance criterion should be used for soil SRMs.
35	What acceptance criterion is used for the LCS standard? [SW-846 7000B Section 9.6] **ANSWER:**			Acceptance criteria should either be defined in the project-specific planning documents or set at a laboratory derived limit developed through the use of historical analyses. In the absence of project-specific or historical data generated criteria, this limit should be set at ± 20% of the spiked value. Acceptance limits derived from historical data should be no wider that ± 20%.
36	What action is taken when the LCS does not meet the established acceptance criteria? [SW-846 7000B Section 9.6] **ANSWER:**			If the laboratory control sample is not acceptable, then the laboratory control sample should be re-run once and, if still unacceptable, all samples after the last acceptable laboratory control sample should be reprepared and reanalyzed.
37	Is a second source Initial Calibration Verification (ICV) standard analyzed each day samples are analyzed? [SW-846 Method 7000B Section 10.2.1 and 15A NCAC 2H .0805 (a) (7) (H) (ii)]			7000B: After initial calibration, the calibration curve must be verified by use of an initial calibration blank (ICB) and an initial calibration verification (ICV) standard. The ICV standard must be made from an independent (second source) material at or near mid-range. 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.
38	What acceptance criterion is used for the second source ICV			7000B: The acceptance criteria for the ICV standard must be ±10% of its true value and

	standard? [SW-846 Method 7000B Section 10.2.] **ANSWER:**			the ICB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid.
39	What corrective action is taken if the second source ICV standard is not within the acceptance criterion? [SW-846 Method 7000B Section 10.2.1] **ANSWER:**			7000B: If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed. The analysis data for the ICV must be kept on file with the sample analysis data.
40	Does the laboratory analyze at least one method blank (MB) with each batch of samples processed? [SW-846 Method 7000B Section 9.5]			For each batch of samples processed, at least one method blank must be carried throughout the entire sample preparation and analytical process, as described in Chapter One.
41	Is the MB prepared in the same manner as the calibration blank except the MB must be carried through the entire sample preparation scheme? [SW-846 Method 7000B Section 9.5]			A method blank is prepared by using a volume or weight of reagent water at the volume or weight specified in the preparation method, and then carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering, and analysis.
42	What is the acceptance criterion for the MB? [15A NCAC 2H .0805 (a) (7) (H) (i)and SW-846 Method 7000B Section 9.5] **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. 7000B: In the absence of project-specific DQOs, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable.
43	What corrective action is taken if the MB exceeds the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B] **ANSWER:**			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 resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. 7000B: If the method blank cannot be considered acceptable, the method blank should be re-run once, and if still unacceptable, then all samples after the last acceptable method blank should be reprepared and reanalyzed along with the other appropriate batch QC samples. These blanks will be useful in determining if samples are being contaminated. If the method blank exceeds the criteria, but the samples are all either below the reporting level or below the applicable action level or other DQOs, then the sample data may be used despite the contamination of the method blank. Refer to Chapter One for the proper protocol when analyzing blanks.
44	Is an initial calibration blank (ICB) analyzed immediately following each calibration? [SW-846 Method 7000B Section 10.2.1]			After initial calibration, the calibration curve must be verified by use of an initial calibration blank (ICB) and an initial calibration verification (ICV) standard.
45				The concentration of reagent, method, and

	What is the acceptance criterion for the ICB? [15A NCAC 2H .0805 (a) (7) (H) (i)and SW-846 Method 7000B Section 10.2.1] **ANSWER:**			calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method.7000B: The acceptance criteria for the ICV standard must be ±10% of its true value and the ICB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid.
46	What corrective action is taken if the ICB exceeds the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B) and SW-846 Method 7000B Section 10.2.1] **ANSWER:**			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 resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. 7000B: If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed. The analysis data for the ICV must be kept on file with the sample analysis data.
47	Are the CCB and CCV analyzed after every 10 samples and at the end of the analytical batch? [SW-846 Method 7000B Section 10.2.2 and 15A NCAC 2H .0805 (a) (7) (H)]			10.2.2: The calibration curve must also be verified at the end of each analysis batch and/or after every 10 samples by use of a continuing calibration blank (CCB) and a continuing calibration verification (CCV) standard. The CCV standard should be made from the same material as the initial calibration standards at or near midrange. Rule: A calibration blank and calibration verification standard shall be analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group, unless otherwise specified by the method, to check for carryover and calibration drift.
48	What is the acceptance criterion for the CCB? [15A NCAC 2H .0805 (a) (7) (H) (i)and SW-846 Method 7000B Section 10.2.2] **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.7000B: The acceptance criteria for the CCV standard must be ±10% of its true value and the CCB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid.
49	What corrective action is taken if the CCB exceeds the acceptance criterion? [SW-846 Method 7000B Section 10.2.2 and 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.7000B: If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable CCV/CCB must be reanalyzed. The analysis data for the CCV/CCB must be kept on file with the sample analysis data.
50	What acceptance criterion is used for the CCV standard? [SW-846 Method 7000B Section 10.2.2] **ANSWER:**			7000B: The acceptance criteria for the CCV standard must be ±10% of its true value and the CCB must not contain target analytes at or above the lowest limit of quantitation for the curve to be considered valid.

51	What corrective action is taken if the CCV standard is not within the acceptance criterion? [SW-846 Method 7000B Section 10.2.2 and 15A NCAC 2H .0805 (a) (7) (B)] **ANSWER:**			7000B: If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable CCV/CCB must be reanalyzed. The analysis data for the CCV/CCB must be kept on file with the sample analysis data. Rule: 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.
52	Is a matrix spike (MS) analyzed at a frequency of at least 5%? [15A NCAC 2H .0805 (a) (7) (D)]			Rule: Unless the referenced method states a greater frequency or the parameter is not amenable to spiking, laboratories shall spike five percent of samples monthly. Laboratories analyzing fewer than 20 samples per month shall analyze one Matrix Spike during each month that samples are analyzed. Recommendation SW-846 Method 7000B Section 9.7: For each batch of samples processed, at least one MS/Sample Dup or MS/MSD sample set should be carried throughout the entire sample preparation and analytical process as described in Chapter One. MS/MSDs are intralaboratory split samples spiked with identical concentrations of each analyte of interest. The spiking occurs prior to sample preparation and analysis. An MS/Dup or MS/MSD is used to document the bias and precision of a method in a given sample matrix.
53	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 $\leqslant$ 5% of the total MS volume. It is preferable that the spike solution constitutes $\leqslant$ 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.
54	How is the MS prepared? [NC WW/GW LC Matrix Spike Technical Assistance.] **ANSWER:**			See Matrix Spike Technical Assistance document. 7000B: MS/MSD samples should be spiked at the same level, and with the same spiking material, as the corresponding laboratory control sample that is at the project-specification level or, when lacking project-specific action levels, at approximately mid-point of the linear dynamic range.
55	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.

56	What is the established control limit for the MS? [SW-846 Method 7000B Section 9.7] **ANSWER:**			Acceptance criteria should either be defined in the project-specific planning documents or set at a laboratory-derived limit developed through the use of historical analyses per matrix type analyzed. In the absence of project-specific or historical data generated criteria, these limits should be set at ± 25% of the spiked value for accuracy and 20 relative percent difference (RPD) for precision. Acceptance limits derived from historical data should be no wider that ± 25% for accuracy and 20% for precision. Refer to Chapter One for additional guidance.
57	What action is taken when MS recovery falls outside of the designated range? [SW-846 Method 7000B Section 9.7] **ANSWER:**			If the bias and precision indicators are outside the laboratory control limits, if the percent recovery is less than 75% or greater than 125%, or if the relative percent difference is greater than 20%, then the interference test discussed in Sec. 9.8 should be conducted.
58	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.
59	What is the acceptance criterion for the lower reporting limit standard? [15A NCAC 2H .0805 (a) (7) (A)]			Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses.
60	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
61	Does the laboratory analyze duplicate samples at a rate of 5%? [15A NCAC 2H .0805 (a) (7) (C)]			Analyze five percent of all samples in duplicate to document precision. Laboratories analyzing less than 20 samples per month much analyze at least one duplicate each month samples are analyzed. If the lab chooses to analyze a matrix spike duplicate that will satisfy the duplicate requirement.
62	Does the laboratory analyze a matrix spike duplicate (MSD) with each matrix spike? [SW-846 Method 7000B Section 9.7]			For each batch of samples processed, at least one MS/Dup or MS/MSD sample set should be carried throughout the entire sample preparation and analytical process as described in Chapter One.
63	What is the acceptance criterion for duplicates? [NC WW/GW LC Policy and SW-846 Method 7000B Section 9.7] **ANSWER:**			Establish laboratory control limits. Acceptance criteria should either be defined in the project-specific planning documents or set at a laboratory-derived limit developed through the use of historical analyses per matrix type analyzed. In the absence of project-specific or historical data generated criteria, these limits should be set at ± 25% of the spiked value for accuracy and 20 relative percent difference (RPD) for precision. Acceptance limits derived from historical data should be no wider that ± 25% for accuracy and 20% for precision. Refer to Chapter One

				for additional guidance.
64	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits? [15A NCAC 2H .0805 (a) (7) (F)] **ANSWER:**			Rule - Any time quality control results indicate an analytical problem, the problem must be resolved and any samples involved must be rerun if the holding time has not expired.
65	If less than acceptable accuracy and precision data are generated, are post-digestion spikes and/or dilution tests performed? [SW-846 Method 7000B Section 9.8] **Not required – the method says "should".**			If less than acceptable accuracy and precision data are generated, the following additional quality control tests are recommended prior to reporting concentration data for the elements in this method. At a minimum these tests, outlined in Secs. 9.8.1 and 9.8.2, should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests will then serve to ensure that neither positive nor negative interferences are affecting the measurement of any of the elements or distorting the accuracy of the reported values. If matrix effects are confirmed, the laboratory should consult with the data user when feasible for possible corrective actions which may include the use of alternative or modified test procedures or possibly the method of standard additions so that the analysis is not impacted by the same interference.
66	How is the post-digestion spike prepared? [SW-846 Method 7000B Section 9.8.1] **ANSWER:**			The same sample from which the MS/MSD aliquots were prepared (assuming the MS/MSD recoveries are unacceptable) should also be spiked with a post digestion spike. Otherwise another sample from the same preparation should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation.
67	What corrective action is taken if the post digestion spike recovery falls outside 80 to 120% of the known value? [SW-846 Method 7000B Section 9.8.1] **ANSWER:**			If this spike fails, then the dilution test should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.
68	If the dilution test is performed, does analysis of a 1:5 dilution agree within ±10% of the original determination? [SW-846 Method 7000B Section 9.8.2]			If the analyte concentration is sufficiently high (minimally, a factor of 10 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ±10% of the original determination.
69	What corrective action is taken if analysis of a 1:5 dilution does not agree within ±10% of the original determination? [SW-846 Method 7000B Section 9.8.2]			If not, then a chemical or physical interference effect should be suspected. For both a failed post digestion spike or an unacceptable dilution test agreement result,

	ANSWER:			the method of standard additions should be used as the primary means to quantitate all samples in the associated preparation batch.
70	If the Method of Standard Additions (MSA) is used, is a single-addition or multiple-addition method used? [SW-846 Method 7000B Sections 9.10.1 and 9.10.2] **Circle one:** Single-addition or Multiple-addition			9.10.1: The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume VS of reagent water. The analytical signals of A and B are measured and corrected for non-analyte signals. 9.10.2: Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the indigenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1. A linear regression program may be used to obtain the intercept concentration.
71	Are the apparent concentrations from the calibration curve linear (0.995 or greater) over the concentration range of concern? [SW-846 Method 7000B Section 9.10.3]			The apparent concentrations from the calibration curve must be linear (0.995 or greater) over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve.
72	Does the effect of the interference vary as the ratio of analyte concentration to sample matrix changes? [SW-846 Method 7000B Section 9.10.3]			The effect of the interference should <u>not</u> vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
73	Is the determination free of spectral interference and corrected for nonspecific background interference? [SW-846 Method 7000B Section 9.10.3]			The determination must be free of spectral interference and corrected for nonspecific background interference.
74	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.

The typical detection limit for this method is 0.0002 mg/L.

Reagent Preparation:
Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter.
Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5N H_2SO_4. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)
Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)
Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water.
Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of reagent water.
Stock mercury solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated HNO_3 and adjust the volume to 100.0 mL (1 mL = 1 mg Hg). Stock solutions may also be purchased.
Maintain acidity of working standards at 0.15% nitric acid.

Additional Comments:

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Inspector:_____Date:_____