

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: **Purgeable Organics**
 Method: **SW-846 Method 8260D (Aqueous & Non-Aqueous)**

NOTE: Per the NC WW/GW LCB SW-846 Method Implementation Policy, 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"). Instances in the explanation are denoted as [must].

Apparatus:

Purge & Trap System - Model:	
Gas Chromatograph - Model:	
Mass Spectrometer - Model:	

Equipment:

Syringes
Syringe valves
Micro syringes
Bottles, 40-mL with TFE lined screw cap
Volumetric Flasks
Analytical Balance
Injection Port Liner

Primary Source
Surrogate
Internal
Carrier Gas

List Columns & Traps:

Reagents:

Organic-free reagent water
Methanol, purge-and-trap grade
Hydrochloric acid: HCl, 1 + 1
Ascorbic Acid

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. Recommend an annual review. Update SOPs any time changes are made to procedure and make a list or highlight any changes that were made to methodology.
2	Are all review/revision dates and procedural edits tracked and documented? [15A NCAC 02H .0805 (a) (7)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control, and Standard Operating Procedure documents.
3	Is there North Carolina data available for review?			If not, review PT data
	PRESERVATION and STORAGE	L A B	S O P	EXPLANATION
	Aqueous Samples			
4	Are samples verified to be free of residual chlorine and, if not, are they preserved with a dechlorinating reagent? [SW-846 Chapter 4, Section 4.1.3.3] [SW-846 Chapter Four, Table 4-1]			Aqueous samples containing free chlorine should also be preserved with a dechlorinating agent in order to minimize formation of trihalomethanes and other possible chemical reactions. Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial.
5	If samples are free of residual chlorine, then are samples collected in at least two 40-mL glass			Section 4.1.3.2: At least two replicate VOA vials should be collected and labeled immediately for each collected field sample.

	containers with PTFE-lined septum cap? [SW-846 Chapter Four, Section 4.1.3.2 & Table 4-1]		Table 4-1: 3 x 40-mL vials with PTFE-lined septum caps. Note: 2 vials are required, 3 vials are recommended.
6	Unless otherwise stated in the method, are samples preserved at time of collection to a pH of ≤2 S.U.? [SW-846 8260D, Section 1.3.3] [SW-846 Chapter Four, Section 4.1.3.3 and Table 4-1]		Chapter 4, Table 4-1: Adjust pH to less than 2 S.U. with H ₂ SO ₄ , HCl, or solid NaHSO ₄ 8260 D Section 1.3.3: Elevated sample temperatures may be necessary during purges as heated samples will exhibit better performance of these analytes. However, ethers such as diethyl ether and MTBE hydrolyze more readily when heated in acid-preserved water. Acid preservation is not recommended for analysis of these target analytes at elevated sample temperature. Chapter 4, Section 4.1.3.3: Chemical preservation may be inappropriate for highly reactive compounds (e.g., 2-chloroethyl vinyl ether, acrylamide, etc.), since it may accelerate loss by rapid chemical reaction. Aqueous samples containing free chlorine should also be preserved with a dechlorinating agent in order to minimize formation of trihalomethanes and other possible chemical reactions.
7	Are samples preserved at time of collection with ice to a temperature of 0 - 6°C? [SW-846 Chapter Four, Table 4-1]		Cool to 0 - 6°C
8	Are aqueous samples collected and stored with minimal or no headspace and non-aqueous samples collected in air-tight containers compatible with closed-system sample preparation and analysis techniques? [SW-846 8260D, Sections 8.2 & 8.3]		Aqueous samples should [must] be stored with minimal or no headspace to minimize the loss of highly volatile analytes. Solid and waste samples should [must] be collected in air-tight containers compatible with closed-system sample preparation and analysis techniques, if possible.
9	Are samples analyzed within 14 days of collection? [SW-846 Chapter Four, Table 4-1]		Table 4-1, footnote 3: A longer holding time may be appropriate if it can be demonstrated that the reported analyte concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.
10	Are samples not acidified and analyzed within 7 days if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used? [SW-846 Chapter Four, Table 4-1]		If carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples.
11	Is a second set of samples collected without acid preservation and analyzed within 7 days if compounds that readily degrade in acidified water (e.g., 2-chloroethyl vinyl ether ^b) are analytes of interest? [SW-846 Chapter Four, Table 4-1]		If compounds that readily degrade in acidified water (e.g., 2-chloroethyl vinyl ether ^b) are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
Non-Aqueous Samples			
12	Are soil samples collected and preserved as described in SW-846 Method 5035? [SW-846 Method 5035, Sections 6.1.1 – 6.1.4]		6.1.1 Low concentration soil samples The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035. Add a clean magnetic stirring bar to each clean vial. If the purge-and trap device (Sec. 4.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted. Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤2 S.U. Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes. Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer. Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label. Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is

			<p>opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.</p> <p>6.1.2 High concentration soil samples collected without a preservative When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 4.4).</p> <p>6.1.3 High concentration soil samples collected and preserved in the field The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030. Add 10 mL of methanol to each vial. Seal the vial with the screw-cap and septum seal. Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label. NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection. Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.</p> <p>6.1.4 Oily waste samples When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 6.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.</p>	
	PROCEDURE – Instrument Calibration	L A B	S O P	EXPLANATION
13	Are stock standard solutions prepared in-house replaced after one year or sooner if comparison with QC check samples indicates a problem? [SW-846 Method 8260D, Section 7.6.1]			Stock standard solutions prepared in-house must be replaced after one year, or sooner if comparison with QC check samples indicates a problem. When solutions are mixed together, regardless of the source, they must be replaced after the manufacturer's expiration date or one year (whichever occurs first) or sooner if problems are indicated. The assigned expiration date of the mixed standard should [must] correspond to that of the stock that expires the earliest.
14	Are Initial calibration standards (ICAL) mixed from fresh stock standards and dilution standards when generating an ICAL curve? [SW-846 Method 8260D, Section 7.11.1]			ICAL standards should [must] be mixed from fresh stock standards and dilution standards when generating an ICAL curve.
15	Are non-gaseous working standards replaced after 4 weeks? [SW-846 Method 8260D, Section 7.7]			Working standards – Using stock standard solutions, prepare working standards in methanol (or other appropriate solvent), containing the compounds of interest, either singly or mixed together. Working standards should [must] be stored with minimal headspace and should [must] be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards. Working standards for most compounds should [must] be replaced after four weeks unless the integrity of the standard is suspected of being compromised prior to that time.
16	Are working standards for gases replaced after one week if the acceptability of the standard cannot be documented? [SW-846 Method 8260D, Section 7.7]			Working standards for gases should [must] be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.
17	What surrogate standards are used? [SW-846 Method 8260D, Section 7.8]			Surrogate standards - The recommended surrogates are toluene-d ₈ , 4-bromofluorobenzene and 1,2-dichloroethane-d ₄ . Other compounds with physicochemical properties better resembling the analyte classes of interest may be used as surrogates (e.g., deuterated monitoring

	Answer:		compounds in the EPA Contract Laboratory Program's (CLP) current statement of work, which can be found in Reference 14 in Sec. 16), provided they can be unambiguously identified and meet any applicable acceptance criteria described in Sec. 11 for ICAL and continuing calibration verification (CCV).																
18	What internal standards are used? [SW-846 Method 8260D, Section 7.9] Answer:		Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d ₅ , and 1,4-dichlorobenzene-d ₄ . Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS.																
19	Is BFB used as the tuning standard? [SW-846 Method 8260D, Section 7.10]		4-Bromofluorobenzene (BFB) tune verification standard – A standard solution of BFB in methanol (or other appropriate solvent) may be prepared for direct injection. If BFB is used as a surrogate, the surrogate solution may be used for this purpose.																
20	IF BFB is not used as the tuning standard are other acceptable reference compounds used to meet manufacturer's tuning specifications? [SW-846 8260D, Section 11.3.1]		Acceptable system performance may also be demonstrated by meeting manufacturer specifications for mass resolution, mass accuracy, and sensitivity using the internal calibrant (e.g., perfluorotributylamine, also known as PFTBA). Other reference compounds may also be appropriate for demonstrating acceptable MS performance depending on the system or conditions used for analysis (e.g., octafluoronaphthalene for negative ion CI). Regardless of how MS performance is evaluated, system calibration must not begin until performance criteria are met, and calibration standards and samples must be analyzed under the same conditions, i.e., if the system is retuned a new calibration should [must] be performed. If CI, SIM or tandem MS is used, the manufacturer's MS tuning criteria or one of the alternative procedures listed above may be substituted for the BFB tune verification requirement.																
21	Is the BFB Tune check performed prior to the ICAL? [SW-846 Method 8260D, Section 11.4]		NOTE: Tune checks (Sec. 11.3.1) are only required prior to ICAL.																
22	What acceptance criteria is utilized for the BFB tune standard? [SW-846 Method 8260D, Section 11.3.1.2 & Table 3]		<p>Compare BFB mass intensities to the criteria in Table 3 (below). Alternatively, other documented ion ratio criteria may be used provided that method performance is not adversely affected. If hydrogen is used as a carrier gas, the Table 3 criterion for 96/95 m/z ratio of BFB will be difficult to achieve. A relative abundance of 5 to 15% for 96/95 m/z is acceptable due to interactions with the carrier gas and water vapor. The analyst is free to choose criteria that are tighter than those included in this method or to use other documented criteria provided they are used</p> <table border="1" data-bbox="797 1232 1539 1564"> <thead> <tr> <th><i>m/z</i></th> <th>Intensity (relative abundance)</th> </tr> </thead> <tbody> <tr> <td>95</td> <td>50 to 200% of mass 174</td> </tr> <tr> <td>96</td> <td>5 to 9% of <i>m/z</i> 95 (5 to 15% when using H₂ carrier)</td> </tr> <tr> <td>173</td> <td><2% of <i>m/z</i> 174</td> </tr> <tr> <td>174</td> <td>50 to 200% of mass 95</td> </tr> <tr> <td>175</td> <td>5 to 9% of <i>m/z</i> 174</td> </tr> <tr> <td>176</td> <td>95 to 105% of <i>m/z</i> 174</td> </tr> <tr> <td>177</td> <td>5 to 10% of <i>m/z</i> 176</td> </tr> </tbody> </table> <p>consistently throughout the ICAL, calibration verification, and sample analyses.</p>	<i>m/z</i>	Intensity (relative abundance)	95	50 to 200% of mass 174	96	5 to 9% of <i>m/z</i> 95 (5 to 15% when using H ₂ carrier)	173	<2% of <i>m/z</i> 174	174	50 to 200% of mass 95	175	5 to 9% of <i>m/z</i> 174	176	95 to 105% of <i>m/z</i> 174	177	5 to 10% of <i>m/z</i> 176
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23	For the ICAL, are there a minimum of five different concentrations in the calibration for average response factor (RF)/linear (first-order) calibration models or six different concentrations for a quadratic (second-order) model utilized? [SW-846 Method 8260D, Sections 7.11.1 & 11.3.5.1]		<p>ICAL standards must be prepared at a minimum of five different concentrations from the working dilution of stock standards or from premixed certified solutions. Prepare these solutions in organic-free reagent water or in a solvent appropriate for the specific sample preparation method used. Include a minimum of five different concentrations in the calibration for average response factor (RF) or linear (first-order) calibration models or six different concentrations for a quadratic (second-order) model, with the low standard at or below the LLOQ</p> <p>Note (Section 11.3.5.1): Forcing the calibration model through the origin (for analytes that are consistently detected in the laboratory reagent blanks) allows for a better estimate of the background level of blank contaminants. An accurate estimate of background contamination is</p>																

			necessary to set method reporting limits for method analytes when blank levels are problematic.
24	Is at least one of the calibration standards prepared at or below the concentration necessary to meet the data quality objectives? [15A NCAC 02H .0805 (a) (7) (H)] [SW-846 Method 8260D, Section 7.11.1]		Rules: One of the standards shall have a concentration equal to or less than the laboratory's lowest reporting concentration for the parameter involved. SW-846 8260 D: At least one of the calibration standards should [must] correspond to a sample concentration at or below that necessary to meet the DQOs of the project.
25	Do the remaining calibration standards bracket the concentrations of typical samples? [15A NCAC 02H .0805 (a) (7) (H)] [SW-846 Method 8260D, Section 7.11.1]		Rules: For analytical procedures requiring analysis of a series of standards, the concentrations of these standards shall bracket the range of the sample concentrations measured. SW-846 8260 D: The remaining standards should [must] correspond to the range of concentrations found in typical samples but should [must] not exceed the working range of the GC/MS. ICAL standards should [must] be mixed from fresh stock standards and dilution standards when generating an ICAL curve.
26	Are all calibration points recalculated (not reanalyzed) using the final calibration curve? [15A NCAC 02H .0805 (a) (7) (H)] [SW-846 Method 8260D, Section 11.3.5.4] List acceptance criteria for each standard:		Rules: Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day samples are analyzed. SW-846 8260 D: All calibration points, especially those equivalent to the LLOQ, should [must] be recalculated (not reanalyzed) using the final calibration curve in which this standard is used (i.e., re-fitting the response from the calibration standard back into the curve). See Method 8000 for additional details. The recalculated concentration of the calibration standard corresponding to the LLOQ, especially where linear regression fits are used, should be within $\pm 50\%$ of the standard's true concentration if it is the lowest point, and within $\pm 30\%$ for all others (i.e., Above the low standard). No refit criteria need be passed for calibration levels below the LLOQ. Alternate criteria may be applied depending on the needs of the project. However, those criteria should be clearly defined in a laboratory SOP or a project-specific QAPP. Analytes which do not meet the re-fitting criteria should be evaluated for corrective action. If a failure occurs in the low point and it is equivalent to the LLOQ, the analyte should be reported as estimated near that concentration or the LLOQ should be reestablished at a higher concentration (See Method 8000 Sec. 11.5.4 for calculations).
27	Is the ICAL verified using an initial calibration verification standard (ICV) prepared from a second source ? [SW-846 Method 8260D, Section 7.11.3]		Second source standards for ICV must be prepared using source materials from a second manufacturer or from a manufacturer's batch prepared independently from the batch used for calibration. A second lot number from the same manufacturer may be adequate to meet this requirement. Target analytes in the ICV are recommended to be prepared at concentrations near the mid-point of the calibration range. The standard must contain all calibrated target analytes that will be reported for the project, if readily available. See Secs. 9.3.2 and 11.3.6 for guidance and acceptance limits.
28	What is the acceptance criterion for the ICV? [SW-846 Method 8260D, Section 11.3.6] Answer:		Suggested acceptance criteria for the analyte concentrations in this standard are 70 - 130% of the expected analyte concentration(s). Alternative criteria may be appropriate based on project-specific DQOs
29	What corrective action is taken if the ICV exceeds the acceptance criterion? [SW-846 Method 8260D, Section 11.3.6] Answer:		Quantitative sample analyses should not proceed for those analytes that do not meet the ICAL verification criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values.
30	Does the ICAL and the ICV contain all calibrated target analytes that will be reported for the project? [SW-846 Method 8260D, Section 7.11.3]		The standard must contain all calibrated target analytes that will be reported for the project, if readily available.
31	Does each standard curve have a correlation coefficient of ≥ 0.995 ? [NC WW/GW LCB		When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not

	Correlation Coefficient for Linear Calibration Curves Policy]		specified, then a minimum value of 0.995 (or a coefficient of determination, r^2 , of 0.99) is required.
32	Are all standards prepared in methanol and stored with minimal headspace, protected from light, at ≤ 6 °C? [SW-846 Method 8260D, Section 7.13]		Great care must be taken to maintain the integrity of all standard solutions. It is recommended that standards be stored with minimal headspace, protected from light, at ≤ 6 °C, or as recommended by the standard manufacturer using screw-cap or crimp-top amber containers equipped with PTFE liners.
33	What method of sample introduction is utilized? [SW-846 Method 8260D, Sections 11.1.1 through 11.1.5] Answer:		-Direct injection -Purge-and-trap -Vacuum distillation -Automated static headspace -Cartridge desorption
34	Are the standards, field samples, and QC samples associated with this analysis using identical MS instrument conditions with the exception of SIM analysis? [SW-846 Method 8260D, Section 11.3.1.2]		All subsequent standards, field samples, and QC samples associated with this analysis must use identical MS instrument conditions with the exception of SIM analysis. BFB may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM.
35	For SIM and SRM analysis, are at least two ions for each target analyte used and is the mid-point of the calibration curve used to establish proper ion ratios for each compound? [SW-846 Method 8260D, Section 11.3.3]		Monitor at least two ions for each target analyte and use the mid-point of the calibration curve to establish proper ion ratios for each compound. The ratios of primary and secondary ions are the only qualitative tools available in SIM and SRM runs (other than RT), which increases their importance in proper identification. When interferences are expected or observed in a given matrix, acquiring multiple secondary ions may aid in qualitative identification.
36	How is the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the ICAL? [SW-846 Method 8260D, Section 11.3.4.1]		Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows: $SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$ $RSD = \frac{SD}{\overline{RF}} \times 100$ where: RF _i = RF for each of the calibration standards RF = mean RF for each compound from the initial calibration n = Number of calibration standards, e.g., 5 SD = Standard Deviation
37	Is the RSD acceptance criterion for ICALs set at $\leq 20\%$? [SW-846 Method 8260D, Section 11.3.4.2]		The RSD should be $\leq 20\%$ for each target analyte (see Sec. 11.3.5). Table 4 contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly.
38	If the average response factor is used for quantitation, is the RSD for all target analytes $\leq 20\%$? [SW-846 Method 8260D, Section 11.3.5]		Linearity of target analytes – If the RSD of any target analyte is $\leq 20\%$, then the RF is assumed to be constant over the calibration range, and the average RF may be used for quantitation (Sec. 11.7.2).
39	When the RSD of a compound's response factors is $\leq 20\%$, is the concentration of the extract determined using the average response factor (RF) from the initial calibration data? [SW-846 Method 8260D, Section 11.7.2]		If the RSD is 20% or less, then the RF calibration model is acceptable for the ICAL (Sec. 11.3.4). See Method 8000 for the equations describing IS calibration and either linear or non-linear calibrations.
40	What action is taken when more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard		If more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard error (RSE) $\leq 20\%$) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Correct the source of the problem; then repeat the calibration procedure beginning with Sec. 11.3.

	error (RSE) \leq 20%) for alternate curve fits? [SW-846 Method 8260D, Section 11.3.5.2]			If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated.
	PROCEDURE – Sample Preparation	L A B	S O P	EXPLANATION
41	Is Method 5035 used for Non-Aqueous samples? [SW-846 Method 8260D, Section 11.1.2]			Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge and trap from an aqueous matrix using Method 5030.
42	Is a method blank (MB) included with each preparation batch? [SW-846 8260D, Section 9.6.1]			A MB must be included with each preparation batch. MBs consist of an aliquot of clean (control) matrix similar to the sample and of a similar weight or volume.
43	Are all of the internal standards, surrogates, and matrix spiking compounds added to the samples before introduction into the GC/MS? [SW-846 Method 8260D, Section 11.1]			All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system.
44	If a heated purge is used are all calibration standards, field samples, and associated QC samples are purged at the same temperature? [SW-846 Method 8260D, Section 11.1.2.2]			Aqueous and soil/solid samples may also be purged at higher temperatures as long as all calibration standards, field samples, and associated QC samples are purged at the same temperature, and the laboratory demonstrates acceptable method performance for the project.
45	Is a Laboratory Control Sample (LCS) included with each preparation batch? [SW-846 Method 8260D, Section 9.6.2]			An LCS must be included with each preparation batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.
46	Is the Laboratory Control Sample (LCS) spiked with the same analytes at the same concentrations as the matrix spike? [SW-846 Method 8260D, Section 9.6.2] If they are not at the same concentration, give explanation:			The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
47	Do dilutions keep the response of major constituents (i.e., previously saturated peaks) near the middle of the calibration range of the curve? [SW-846 Method 8260D, Section 11.5.4]			Dilutions should be targeted so the response of the major constituents (previously saturated peaks) falls near the middle of the calibration range.
	PROCEDURE – Analysis	L A B	S O P	EXPLANATION
48	Are the retention times (RTs) of all compounds of interest within \pm 10 seconds of the RTs for this analyte in the midpoint ICAL standard or CCV standard analyzed at the beginning of the 12-hour period (delta RT 0.17 minute), or within \pm 10 seconds relative to the shift of the associated IS (delta RT of the IS \pm 10 seconds).? [SW-846 Method 8260D, Section 11.6.1.2]			The RT is within \pm 10 seconds of the RT for this analyte in the midpoint ICAL standard or CCV standard analyzed at the beginning of the 12-hour period (delta RT 0.17 minute), or within \pm 10 seconds relative to the shift of the associated IS (delta RT of the IS \pm 10 seconds). Chromatograms should be carefully inspected to minimize the occurrence of both false positive and false negative results. If the RT for the IS has shifted, the sample should be inspected for similar shifts for the associated target analytes. If RT drift is significant, relative retention time (RRT) may be useful as an alternative to delta retention times. See Section 11.4 of Method 8000 for additional information.
49	Are the acceptance criteria for the relative intensities of the secondary characteristic ions \pm 30% of the ions in the reference spectrum? [SW-846 Method 8260D, Section 11.6.1.3]			The relative intensities of the qualifier ion(s) (i.e., secondary characteristic ions or alternate MS/MS transitions) should agree within 30% of the relative intensities of these ions in the reference spectrum. For example, for a qualifier ion with a response of 50% of the quantitation ion in the reference spectrum, the corresponding qualifier ion ratio in a sample spectrum can range between 20% and 80%. The reference mass spectrum used for this comparison should be generated by the laboratory using the conditions of this method (typically a mid-level calibration standard). Qualitative identification of sample mass spectra not acquired in limited ion acquisition modes (i.e., SIM or SRM) may also be supported by comparison to a reference library as described in Sec. 11.6.2.
50	Are structural isomers that produce very similar mass spectra but have sufficiently different retention times identified as individual isomers? [SW-846 Method 8260D, Section 11.6.1.4]			Unresolved structural isomers with similar mass spectra are identified as isomeric pairs. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is \leq 50% of the average of the two peak heights, or $1 - [\text{valley height}] / [\text{average peak height}] \geq 50\%$). The resolution should be verified on the mid-point concentration of the ICAL as well as the laboratory-designated CCV level if closely eluting isomers are to be reported. It is important to check the separation of structural isomers in the ICV and the daily CCV check standards to verify if the instrument performance is

				adequate regarding separation of compounds of interest which are structural isomers.
51	Is analyte quantitation based on the integrated abundance from the EICP of the primary characteristic ion? [SW-846 Method 8260D, Section 11.7.1]			Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.
52	Is the internal standard used for analyte quantitation the one with the nearest retention time to that of the given analyte? [SW-846 Method 8260D, Section 11.7.1]			The internal standard used shall be the one nearest the retention time of that of a given analyte.
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
53	Does the lab perform IDOCs for new staff or when significant instrumentation changes are made? [SW-846 Method 8260D, Section 9.4]			Initial demonstration of proficiency (IDP) - Prior to implementation of a method, each laboratory must perform an IDP consisting of at least four replicate reference samples spiked into a clean matrix taken through the entire sample preparation and analysis. Whenever a significant change to instrumentation or procedure occurs, the laboratory must demonstrate that acceptable precision and bias can still be obtained. Also, whenever new staff members are trained, each analyst must perform an IDP for the method or portion of the method for which the analyst is responsible. Refer to Sec. 9.3 of Method 8000 for more information on how to perform an IDP.
54	Is a method blank (MB), carried through all stages of sample preparation, analyzed after calibration or at any other time during the analytical shift? [SW-846 Method 8260D, Sections 9.5.1 and 11.4.2]			MBs, trip blanks, and other field blanks must be carried through all stages of sample preparation and analysis. At least one MB must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples. Blank(s) analyzed after a high concentration calibration standard can also be used to estimate the extent of decontamination needed to reduce the signal to an acceptable level (Sec. 9.5.2) after analyzing a sample at a similar concentration. Section 11.4.2: A blank must also be analyzed after the CCV standard and prior to any samples in order to demonstrate that the total system (introduction device, transfer lines and GC/MS system) is free from contaminants. Analytes of interest for the project that did not meet the criteria should be identified to the data user and results qualified appropriately
55	What is the acceptance criterion of the blanks? [15A NCAC 02H .0805 (a) (7) (H) (i)] [SW-846 Method 8260D, Section 9.5.2]			Rules: For analyses requiring a calibration curve, the concentration of reagent, method, and calibration blanks shall not exceed 50% of the lowest reporting concentration, or as otherwise specified by the reference method. SW-846 8260 D: Blanks are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., target analytes are not present in samples or sample concentrations/responses are >10X the blank). The analyst (or laboratory) should document detected common laboratory contaminants and distinguish those from situations (e.g., carryover), where corrective action may be required. Other criteria may be used depending on the needs of the project.
56	What corrective action is taken if the blank exceeds the acceptance criterion? [SW-846 Method 8260D, Sections 9.5.1 and 11.4.2]			Section 11.4.2: If the blank indicates contamination, then it may be appropriate to analyze additional blanks to reduce any system contamination due to carryover from standards or samples. See Sec. 9.5 for MB performance criteria. See Method 8000 for information regarding MB performance criteria. Section 9.5.1: If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source and eliminate it, if possible.
57	Does the response of the primary m/z for any of the ISs in the field samples or associated QC samples vary more than a factor of two (50% - 200%) from that of the same IS in the mid-point ICAL standard, average of ICAL standards, or most recently analyzed CCV standard? [SW-846 Method 8260D, Section 11.5.6]			IS responses and RTs should be monitored in all field samples and associated QC samples in order to provide sample-specific QA of proper analyte introduction to the GC/MS system and to anticipate the need for system inspection and/or maintenance. If the response of the primary m/z for any of the ISs in the field samples or associated QC samples varies by more than a factor of two (50% - 200%) from that of the same IS in the mid-point ICAL standard, average of ICAL standards, or most

			recently analyzed CCV standard (as defined in the laboratory's SOP), corrective action should be taken. Any affected field samples and associated QC samples should be re-analyzed, or the associated data should be qualified.
58	If retention times for internal standards change by more than 30 seconds, from those in the mid-point standard from the most recent calibration sequence, is the chromatographic system inspected for malfunctions and corrections made before re-analysis of affected samples occurs? [SW-846 Method 8260D, Section 11.4.4]		Internal standard retention time - If the retention time for any internal standard changes by more than 30 seconds from those in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
59	Is the ICAL for each compound of interest verified once every twelve hours and at the beginning of each twelve-hour analytical period with a continuing calibration verification standard (CCV)? [SW-846 Method 8260D, Sections 11.4 & 11.4.1]		A CCV standard must be analyzed at the beginning of each twelve-hour analytical period prior to any sample analysis. The ICAL function (Sec. 11.3) for each compound of interest must be verified once every twelve hours prior to sample analysis, using the same introduction technique and conditions as used for analysis of ICAL standards and samples. This is accomplished by analyzing a CCV standard (containing all the compounds that will be reported) prepared from the same stock solutions or source materials used for ICAL standards and at a concentration near the midpoint of the ICAL range. The results must be compared against the most recent calibration curve and should meet the CCV acceptance criteria provided in Secs. 11.4.3-11.4.5. This QC check may be omitted if samples are analyzed within twelve hours of ICAL, and injection of the last ICAL standard may be used as the starting time reference for evaluation.
60	Is the CCV prepared from the same source as the ICAL? [SW-846 Method 8260D, Section 11.4.1]		This is accomplished by analyzing a CCV standard (containing all the compounds that will be reported) prepared from the same stock solutions or source materials used for ICAL standards and at a concentration near the midpoint of the ICAL range.
61	Is the CCV prepared at a concentration near the mid-point of the initial calibration curve range? [SW-846 Method 8260D, Section 11.4.1] List the concentration:		This is accomplished by analyzing a CCV standard (containing all the compounds that will be reported) prepared from the same stock solutions or source materials used for ICAL standards and at a concentration near the midpoint of the ICAL range.
62	Is the calculated concentration or amount of each analyte of interest in the CCV standard within $\pm 20\%$ of the expected value? [SW-846 Method 8260D, 11.4.3]		The calculated concentration or amount of each analyte of interest in the CCV standard should fall within $\pm 20\%$ of the expected value.
63	Does the laboratory analyze a Matrix Spike (MS) with each batch of 20 or fewer samples? [15A NCAC 02H .0805 (a) (7) (C)] [SW-846 Method 8260D, Section 9.6.3]		Rules: Unless the referenced method states a greater frequency or the parameter is not amenable to spiking, laboratories shall spike 5% of samples monthly. Laboratories analyzing fewer than 20 samples per month shall analyze one Matrix Spike (MS) during each month that samples are analyzed. SW846 8260 D: The laboratory must also have procedures for documenting the effect of the sample matrix on method performance (i.e., precision, bias, and method sensitivity). At a minimum, this must include the analysis of a MB and LCS, and, where practical, either a laboratory sample duplicate/matrix spike or matrix spike/matrix spike duplicate (provided sufficient material is made available to the laboratory for doing so) in each preparation batch of 20 or fewer samples, as well as monitoring the recovery of surrogates in all samples.
64	What is the acceptance criterion of the MS recovery? [SW-846 Method 8260D, 9.6.3] [SW-846 Method 8000 D, Section 9.6.1] Answer:		SW-846 8260 D: Consult Method 8000 for information on developing acceptance criteria for the matrix spike/laboratory sample duplicates or matrix spike/matrix spike duplicates. SW-846 8000 D: Once sufficient data have been acquired and the recovery and RPD calculated as in Secs. 9.4.3 and 9.5 for a given sample matrix, the following statistics should be used to calculate acceptance criteria. 9.6.1.1 Mean percent recovery (\bar{x}) and standard deviation (s) for: 1) Each added target compound in the MS/MSD samples;

65	What action is taken if MS results are out of control? [NC WW/GW LCB Matrix Spiking Policy] Answer:		If MS results are out of control, the unspiked sample result must be qualified or the laboratory must take corrective action to rectify the effect, use another method, or employ the method of standard additions.
66	Is the MS spiking solution prepared from the same source as the ICAL standards? [SW-846 Method 8260D, Section 7.12]		Matrix spikes and LCSs should [must] be prepared with target analytes from the same source as the ICAL standards to restrict the influence of accuracy on the determination of recovery throughout preparation and analysis.
67	Does the laboratory analyze either a sample duplicate or a Matrix Spike Duplicate (MSD) with each batch of 20 or fewer samples? [15A NCAC 02H .0805 (a) (7) (C)] [SW-846 Method 8260D, Section 9.6.3]		Rules: 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. SW-846 8260 D: The decision of whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples, and project goals.... If samples are expected to contain reportable levels of target analytes, then laboratories may use one matrix spike and a duplicate analysis of a non-spiked field sample. If samples are not expected to contain reportable levels of target analytes, laboratories may use a matrix spike and matrix spike duplicate pair.
68	What is the acceptance criterion used to evaluate precision? [15A NCAC 2H .0805 (a) (7) (A)] [SW-846 Method 8000 D, Sections 9.6.1 and 9.6.3] Answer:		SW-846 8260 D: Consult Method 8000 for information on developing acceptance criteria for the matrix spike/laboratory sample duplicates or matrix spike/matrix spike duplicates. SW-846 8000 D 9.6.1: Once sufficient data have been acquired and the recovery and RPD calculated as in Secs. 9.4.3 and 9.5 for a given sample matrix, the following statistics should [must] be used to calculate acceptance criteria.... Mean RPD and standard deviation for MS/MSD or duplicate QC samples. A minimum of 20 data points should [must] be used to generate meaningful criteria. Inclusion of additional data should result in more robust criteria that better describe variance in method performance and result in fewer outliers. If the lower limit of the acceptance range is calculated to be <10%, it should be set to 10%. However, an alternative lower acceptance limit may be established by the laboratory or at the project level through the DQOs in a QAPP. SW-846 8000 D 9.6.3: Calculate the upper control limit for the RPD for the MS/MSD using the mean RPD value + 3s of the RPDs of historical MS/MSD pairs. RPD should [must] be calculated based on the concentration or amount, not the spike recovery.
69	What corrective action does the laboratory take if duplicates do not meet the acceptance criterion? [15A NCAC 2H .0805 (a) (7) (B)] Answer:		If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
70	Does the laboratory analyze at least one LCS with each batch of 20 or fewer samples? [SW-846 Method 8260D, Section 9.6.2]		An LCS must be included with each preparation batch.
71	Does the LCS consist of an aliquot of clean matrix similar to the sample matrix and of the same weight or volume? [SW-846 Method 8260D, Section 9.6.2]		The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.
72	Is the LCS spiked with the same analytes at the same concentrations as the matrix spike? [SW-846 Method 8260D, Section 9.6.2]		The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
73	Is surrogate recovery data evaluated by comparing the individual samples to the surrogate control limits developed by the laboratory? [SW-846 Method 8260D, Section 9.7]		The laboratory should [must] evaluate surrogate recovery data from individual samples relative to the surrogate recovery acceptance criteria developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate recovery acceptance criteria. Suggested surrogate recovery limits for

			field samples are 70 to 130% until laboratory or project-specific criteria can be developed.
74	Is the lower limit of quantitation (LLOQ) verified at least annually and whenever significant changes are made to the preparation and/or analytical procedure? [SW-846 Method 8260D, Section 9.9]		The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve. The laboratory shall establish the LLOQ at concentrations where both quantitative and qualitative criteria can consistently be met (see Sec. 11.6). The laboratory shall verify the LLOQ at least annually and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the preparation and/or analysis of an LCS (or matrix spike) at 0.5 - 2 times the established LLOQ.
75	Is a new initial calibration prepared and analyzed when the analyte responses of the CCV verification are not within 20%? [SW-846 Method 8000D, Section 11.7]		If the calibration does not meet the acceptance criteria, perform any necessary instrument maintenance, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not $\pm 20\%$, then a new initial calibration may be necessary.
76	When and how is manual integration performed? [NC WW/GW LCB Manual Integration Policy]		When manual integration is employed, the laboratory must clearly identify manually integrated compounds, document the reason the manual integration was performed, the date performed and who completed the work. A flag or qualifier code may suffice for simple manual integrations. In addition, a hardcopy printout of the data displaying the manual integration shall be included in the raw data package (i.e., both the original and manually integrated chromatograms, of similar scale, must be present in the data package). All information necessary for the historical reconstruction of data must be maintained by the lab. Additionally, the laboratory must employ a systematic data validation procedure to check manual integrations to assure integrations are technically sound and representative of the response.
77	Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)]		If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such. If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data.

Additional Comments:

Inspector: _____ Date: _____