

NC DEQ/DWR LABORATORY CERTIFICATION

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

Parameter: **Oil & Grease**
Method: **EPA Method 1664 Rev. B (Aqueous)**

Oil and Grease is considered a method-defined parameter per the definition in the Code of Federal Regulations, Part 136.6, Section (a) (5). Only modifications expressly allowed in method section 1.7.1 are permitted.

EQUIPMENT:

Glassware Cleaning	
	Laboratory sink with overhead fume hood
	Oven—Capable of maintaining a temperature within ± 2 °C in the range of 20–250 °C
Calibration	
	Analytical Balance—Capable of weighing 0.1 mg.
	Volumetric flask—Glass, 100-mL.
	Vials—Assorted sizes, with PTFE-lined screw caps.
	Volumetric pipette—Glass, 10-mL.
Sample Extraction	
	Balance (optional)—Top loading, capable of weighing 500–2000 g within $\pm 1\%$
	Glass stirring rod
	Separatory funnel—Glass, 2000-mL, with PTFE stopcock
	Funnel—Large, glass, for pouring sample into separatory funnel
	Centrifuge (optional)—Explosion proof, capable of spinning at least four 100-mL glass centrifuge tubes at 2400 rpm minimum
	Centrifuge tubes (optional)—100-mL glass
	Wash bottle (optional)—Fluoropolymer construction for hexane rinses
Solid Reagent Removal	
	Funnel—Analytical, glass
	Filter paper—Whatman No. 40 (or equivalent), to fit funnel

Distillation	
	Water bath or Steam bath—Explosion-proof, capable of maintaining a temperature of at least 85°C
	Flask—Boiling, 125-mL (Corning No. 4100 or equivalent)
	Distilling head—Claisen (VWR Scientific No. 26339-005, or equivalent), includes Claisen-type connecting tube and condenser
	Distilling adaptor (attached to the distilling head and to the distillate collection flask for recovery of solvent)
	Distillate collection flask (attached to the distilling adaptor for collection of the distilled solvent)
	Ice bath or recirculating chiller (to aid in the condensation and collection of the distilled solvent)
	Vacuum—Vacuum pump or other source of vacuum
	Tongs, for handling the boiling flask (Humboldt Manufacturing No. H-23442, or equivalent)
	Desiccator—Cabinet- or jar-type, capable of keeping the boiling flask (Section 6.6.2) dry during cooling
	Hood—Explosion-proof, capable of accommodating the equipment used for solvent distillation (Section 6.6.1–6.6.5)
Adsorbable Reagent Removal	
	Magnetic stirrer
	PTFE-coated magnetic stirring bars
	Graduated cylinder—500-mL, capable of measuring ± 5 mL
	Pipettes—Assorted sizes, calibrated to within ± 0.5 percent

REAGENTS & STANDARDS:

	Reagent water—Water in which HEM is not detected at or above the minimum level (ML) of this method.
	Hydrochloric acid or sulfuric acid—ACS. Mix equal volumes of concentrated HCl and reagent water or 1 part H ₂ SO ₄ and 3 parts reagent water to produce an approximately 6N solution
	n-Hexane—85% minimum purity, 99.0% min. saturated C ₆ isomers, residue less than 1 mg/L (0.0001% max.)
	Acetone—ACS, residue less than 1 mg/L (0.0001% max)
	Sodium sulfate—ACS, granular anhydrous. Dry at 200–250 °C for 24 h minimum and store in a tightly sealed container until use
	Boiling chips—Silicon carbide or fluoropolymer
	Silica gel—Anhydrous, 75–150 μ m, 30 Å pore size (Davisil Grade 923, or equivalent).
	Hexadecane—98% minimum purity
	Stearic acid—98% minimum purity

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 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
	SAMPLE COLLECTION, PRESERVATION and STORAGE	L A B	S O P	EXPLANATION
4	Are samples collected in glass bottles? [40 CFR 136.3 Table II and footnote 1]			
5	Are samples iced to above freezing but $\leq 6^{\circ}\text{C}$ during shipment? [40 CFR 136.3 Table II and footnote 18]			
6	Are samples preserved at time of collection with HCl or H_2SO_4 to pH of <2 S.U.? [40 CFR 136.3 Table II and footnotes 2 and 3]			Preservation not required if analyzed within 15 minutes.
7	If a sample is known or suspected of containing greater than 500 mg/L of extractable material or consists of a matrix containing substances that may interfere with the extraction procedure, then is a proportionally smaller volume of sample collected with a proportionally smaller amount of preservative? [EPA Method 1664, Rev. B, Section 8.1.2]			If a sample is known or suspected to contain greater than 500 mg/L of extractable material or consists of complex matrix containing substances (such as particulates or detergents) that may interfere with the extraction procedure, collect a proportionately smaller volume of sample (the volume required will depend upon the estimated amount of extractable material) in a glass bottle. Add a proportionately smaller amount of HCl or H_2SO_4 solution to the smaller sample for preservation as necessary.
8	Is an additional aliquot collected for each set of twenty samples or less to be used for the matrix spike? [EPA Method 1664, Rev. B, Section 8.2]			Collect an additional one or two aliquots (1 L, additional smaller volume, or both) of a sample for each set of twenty samples or less for the matrix spike and, if used, the matrix spike duplicate.
9	Are samples collected as grab samples? [EPA Method 1664, Rev. B, Section 8.3]			The high probability that extractable matter may adhere to sampling equipment and result in measurements that are biased low precludes the collection of composite samples for determination of oil and grease. Therefore, samples must be collected as grab samples.
10	If a composite sample is required, then are individual grab samples collected at the prescribed time intervals and subsequently composited in the lab? [EPA Method 1664, Rev. B, Section 8.3]			If a composite measurement is required, individual grab samples collected at prescribed time intervals must be analyzed separately and the concentrations averaged. Alternatively, samples can be collected in the field and composited in the laboratory. For example, collect four individual 250 mL samples over the course of a day. In the laboratory, pour each 250-mL sample into the separatory funnel, rinse each of the four bottles (and caps) sequentially with 30 mL of

				n-hexane, and use the 30 mL of n-hexane for the extraction (Section 11.3).
11	Are samples refrigerated to above freezing but <6°C during storage? [40 CFR 136.3 Table II and footnote 18]			
12	Are samples analyzed within 28 days of collection? [40 CFR 136.3 Table II]			
	Calibration - BALANCE	L A B	S O P	EXPLANATION
13	Is the balance calibration verified with 2 mg and 1000 mg weights prior to weighing each batch of samples? [EPA Method 1664, Rev. B, Section 10.1]			Calibrate the analytical balance at 2 mg and 1000 mg, using class “S” or ASTM E 617-1997 Class 1 weights. It is recommended that the balance should also be calibrated with an additional class “S” or ASTM E 617-1997 Class 1 weight that will bracket the final expected weighing value.
14	Is the analytical balance calibration verified with 2 mg and 1000 mg weights after weighing each analytical batch of samples? [EPA Method 1664, Rev. B, Section 9.5]			Verify calibration of the balance per Section 10 before and after each analytical batch. If calibration is not verified before and after each day or after measurement of the analytical batch, recalibrate the balance and reweigh the batch.
15	If the verification is not within $\pm 10\%$ at 2 mg and $\pm 0.5\%$ at 1000 mg, then is the balance recalibrated? [EPA Method 1664, Rev. B, Section 10.2]			Calibration shall be within $\pm 10\%$ (i.e. ± 0.2 mg) at 2 mg, $\pm 0.5\%$ (i.e., ± 5 mg) at 1000 mg, and if applicable, at the appropriate user-specified tolerance for Class 1 weights greater than 1000 mg. If values are not within these limits, recalibrate the balance.
	PROCEDURE – PREPARATION	L A B	S O P	EXPLANATION
16	Are the samples and QC brought to room temperature? [EPA Method, 1664 Rev. B, Section 11.1.1]			Bring the analytical batch of samples, including the sample aliquots for the MS (and MSD), to room temperature.
17	Is approximately 1000 mL of reagent water placed in clean sample bottle to serve as the laboratory blank? [EPA Method 1664, Rev. B, Section 11.1.2]			Place approximately 1000 mL (950–1050 mL) of reagent water (Section 7.1) in a clean sample bottle to serve as the laboratory blank.
18	Is the OPR prepared using the PAR standard? [EPA Method 1664, Rev. B, Section 11.1.3]			Prepare the OPR (Section 9.6) using the PAR standard (Section 7.11).
19	Are sample bottles marked at the water meniscus or are the sample bottles weighed for subsequent determinations of sample volumes? [EPA Method 1664, Rev. B, Section 11.1.4]			Either mark the sample bottle at the water meniscus or weigh the bottle for later determination of sample volume. Weighing will be more accurate. Mark or weigh the MS (and MSD).
20	Are the QC samples (blank, OPR, MS and MSD) adjusted to a pH of < 2? [EPA Method 1664, Rev. B, Section 11.2.4]			Add the appropriate amount of HCl or H ₂ SO ₄ solution to the blank, OPR, MS (and MSD) to adjust the pH of these solutions to <2.
21	Is sample preservation to pH of <2 S.U. verified prior to sample analysis? [EPA Method 1664, Rev. B, Sections 11.2.1.1, 11.2.1.2, 11.2.1.3]			Preservation should not be checked in sample receiving. It should be checked at the bench, just prior to analysis. See comments in next question.
22	Is the preservation to pH of <2 S.U. verified by dipping a glass stirring rod into the well mixed sample, allowing a drop of sample to touch pH paper, and rinsing the glass stirring rod with n-hexane into the separatory funnel? [EPA Method 1664, Rev. B, Sections 11.2.1.1, 11.2.1.2, 11.2.1.3]			NOTE: Do not dip the pH paper into the bottle or touch it to the sample on the lid. 11.2.1.1 Dip a glass stirring rod into the well mixed sample. 11.2.1.2 Withdraw the stirring rod and allow a drop of the sample to fall on or touch the pH paper. 11.2.1.3 Rinse the stirring rod with a small portion of n-hexane that will be used for extraction (to ensure that no extractable material is lost on the stirring rod). Collect the rinsate in the separatory funnel to be used for

				sample extraction.
23	Describe the action taken to adjust sample pH when the pH of the sample is >2 S.U. [EPA Method 1664, Rev. B, Section 11.2.2] ANSWER:			If possible, collect and analyze another sample. If resampling is not possible, notify NC WW/GW LC of improper preservation, qualify results on the eDMR and/or client report and follow the following procedure: If the sample is at neutral pH, add 5-6 mL of HCl or H ₂ SO ₄ solution (Section 7.2) to the 1-L sample. If the sample is at high pH, use a proportionately larger amount of HCl or H ₂ SO ₄ solution. If a smaller sample volume was collected, use a proportionately smaller amount of HCl or H ₂ SO ₄ solution.
24	If more acid is added to the sample to adjust pH, then is the pH re-verified after the sample is capped and thoroughly mixed? [EPA Method 1664, Rev. B, Section 11.2.3]			Replace the cap and shake the bottle to mix thoroughly. Check the pH of the sample using the procedure in Section 11.2.1. If necessary, add more acid to the sample and retest.
	PROCEDURE – EXTRACTION	L A B	S O P	EXPLANATION
25	Is one of the allowable alternative extraction procedures performed? If so, describe the extraction process. [EPA Method 1664, Rev. B, Section 1.7.1.1]			Alternate extraction techniques including continuous liquid-liquid extraction, solid-phase extraction, solid-phase-heated solvent extraction, solid-phase-Soxhlet extraction, and others. Acceptable solid-phase extraction (SPE) products include: Xenosep filters for EPA Method 1664, part no. 24547 and 25390H or equivalent; UCT LLC Oil and Grease cartridge part no. ECUNIOGXF, or equivalent; or other SPE products that meet all method performance criteria such as Horizon part no. 50-021-HT, Snip and Pour QC Standard or equivalent.
26	Is the flask containing boiling chips dried in an oven at 105–115 °C for a minimum of 2 hours and cooled to room temperature? [EPA Method 1664, Rev. B, Sections 11.3.1.1, 11.3.1.2, 11.3.1.3]			11.3.1.1 Place the flask containing the chips in an oven at 105–115 °C for a minimum of 2 h to dry the flask and chips. 11.3.1.2 Remove from the oven and immediately transfer to a desiccator to cool to room temperature.
27	When cool, are the flasks containing boiling chips weighed? [EPA Method 1664, Rev. B, Section 11.3.1.3]			11.3.1.3 When cool, remove from the desiccator with tongs and weigh immediately on a calibrated balance (Section 10).
28	Is the sample added to the separatory funnel first? [EPA Method 1664, Rev. B, Section 11.3.2]			Pour the sample into the separatory funnel.
29	Is 30 mL of n-hexane added to the sample bottle and sealed with the original bottle cap? [EPA Method 1664, Rev. B, Section 11.3.3]			Add 30 mL of n-hexane to the sample bottle and seal the bottle with the original bottle cap.
30	Is the bottle shaken to ensure that all interior surfaces of the bottle including the lid are rinsed before pouring the solvent into the separatory funnel? [EPA Method 1664, Rev. B, Section 11.3.3]			Shake the bottle to rinse all interior surfaces of the bottle, including the lid of the bottle cap. Pour the solvent into the separatory funnel.
31	Is the sample extracted by shaking the separatory funnel vigorously for 2 minutes with periodic venting into a hood? [EPA Method 1664, Rev. B, Section 11.3.4]			Extract the sample by shaking the separatory funnel vigorously for 2 minutes with periodic venting into a hood to release excess pressure.
32	Is the organic phase allowed to separate from the aqueous phase for a minimum of 10 minutes? [EPA Method 1664, Rev. B, Section 11.3.5]			Allow the organic phase to separate from the aqueous phase for a minimum of 10 minutes.
33	If an emulsion occurs that constitutes more than one third of the volume of the solvent layer, are emulsion breaking techniques employed? [EPA Method 1664, Rev. B, Section 11.3.5]			If an emulsion forms between the phases and the emulsion is greater than one-third the volume of the solvent layer, the laboratory must employ emulsion-breaking techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration

				through glass wool, use of solvent phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods. Alternatively, solid-phase extraction (SPE), continuous liquid-liquid extraction, or other extraction techniques may be used to prevent emulsion formation, provided that the requirements in Section 9.1.2 are met.
34	Is the aqueous layer drained into the original sample container? [EPA Method 1664, Rev. B, Section 11.3.6]			Drain the aqueous layer (lower layer) into the original sample container.
35	Is a small amount of the organic layer drained into the sample container to minimize the amount of water remaining in the separatory funnel? [EPA Method 1664, Rev. B, Section 11.3.6]			Drain a small amount of the organic layer into the sample container to minimize the amount of water remaining in the separatory funnel.
36	Is a filter paper containing approximately 10 g of anhydrous Na ₂ SO ₄ placed into a filter funnel? [EPA Method 1664, Rev. B, Section 11.3.7]			Place a filter paper (Section 6.5.2) in a filter funnel (Section 6.5.1), add approximately 10 g of anhydrous Na ₂ SO ₄ .
37	Is the filter rinsed with a small portion of n-hexane and the rinsate discarded? [EPA Method 1664, Rev. B, Section 11.3.7]			Rinse with a small portion of n-hexane. Discard the rinsate.
38	Is the hexane layer drained from the separatory funnel through the Na ₂ SO ₄ into a pre-weighed boiling flask containing the boiling chips? [EPA Method 1664, Rev. B, Section 11.3.8]			Drain the n-hexane layer (upper layer) from the separatory funnel through the Na ₂ SO ₄ into the pre-weighed boiling flask containing the boiling chips (Section 11.3.1.3).
39	Is the extraction process repeated twice more with fresh 30 mL portions of n-hexane and combining the extracts in the boiling flask? [EPA Method 1664, Rev. B, Section 11.3.9]			Repeat the extraction (Sections 11.3.3–11.3.6 and 11.3.8) twice more with fresh 30-mL portions of n-hexane, combining the extracts in the boiling flask.
40	Is the tip of the separatory funnel rinsed along with the filter paper and funnel with 2-3 small portions of n-hexane which are collected in the boiling flask? [EPA Method 1664, Rev. B, Section 11.3.10]			Rinse the tip of the separatory funnel, the filter paper, and the funnel with 2–3 small (3–5 mL) portions of n-hexane. Collect the rinsings in the flask.
41	If the extract is milky, is it allowed to stand for up to an hour and decanted through sodium sulfate, followed by rinsing the glassware with n-hexane? [EPA Method 1664, Rev. B, Section 11.3.11]			A milky extract indicates the presence of water. If the extract is milky, allow the solution to stand for up to one hour to allow the water to settle. Decant the solvent layer (upper layer) through sodium sulfate to remove any excess water as in Sections 11.3.7 and 11.3.8. Rinse the glassware and sodium sulfate with small portions of n-hexane to effect a quantitative transfer.
PROCEDURE – EXTRACT DISTILLATION		L A B	S O P	EXPLANATION
42	Is the boiling flask connected to a distillation head and the lower half immersed in either a water or steam bath? [EPA Method 1664, Rev. B, Section 11.4.1]			Connect the boiling flask to the distilling head apparatus and distill the solvent by immersing the lower half of the flask in a water bath or a steam bath. Adjust the water temperature as appropriate to complete the concentration.
43	When the distilling head reaches approximately 70°C or the flask appears almost dry, is the distillation head removed and the flask swept for 15 seconds with air to remove any solvent vapor? [EPA Method 1664, Rev. B, Section 11.4.2]			When the temperature in the distilling head reaches approximately 70°C or the flask appears almost dry, remove the distilling head. Sweep out the flask for 15 seconds with air to remove solvent vapor by inserting a glass tube connected to a vacuum source. The requirement to dry the boiling flask for 30-45 minutes in an oven maintained at 70 ± 2°C at Section 11.4.2.2 may be completely omitted or replaced with a user-defined lower temperature and/or shorter drying time.
44	Is the boiling flask immediately removed from the heat source with tongs while it still contains approximately 2 mL of residual liquid? [EPA Method 1664, Rev. B, Section 11.4.2.1]			Using tongs, immediately remove the flask from the heat source while it still contains approximately 2 mL of residual liquid.
45	Is the outside of the flask wiped dry then allowed to cool in a hood at room temperature until visible dryness is achieved? [EPA Method 1664, Rev. B, Section 11.4.2.1]			Wipe the outside surface dry to remove moisture and fingerprints and place the flask in a hood until visible dryness is achieved while cooling at room temperature.

46	If not, then is the flask placed in an oven at $68 \pm 2^{\circ}\text{C}$ for up to 45 minutes? [EPA Method 1664, Rev. B, Section 11.4.2.2]			If desired, the flask may be placed in an oven capable of maintaining a user defined temperature within $\pm 2^{\circ}\text{C}$ (up to 70°C max.) for a user defined period of time (up to 45 minutes max.).
47	If crystal formation is observed in the flask, is the extract re-dissolved in n-hexane, quantitatively transferred through a filter into another tared flask, and the distillation procedure repeated? [EPA Method 1664, Rev. B, Section 11.4.3]			Inspect the residue in the boiling flask for crystals. Crystal formation is an indication that sodium sulfate may have dissolved and passed into the boiling flask. This may happen if the drying capacity of the sodium sulfate is exceeded or if the sample is not adjusted to low pH. If crystals are observed, re-dissolve the extract in n-hexane, quantitatively transfer through a filter into another tared boiling flask, and repeat the distillation procedure (Sections 11.4.1–11.4.2).
48	Is the flask allowed to dry in a room temperature desiccator for at least an additional 30 minutes before weighing? [EPA Method 1664, Rev. B, Section 11.4.4]			Continue to dry and cool the flask to room temperature in a desiccator for 30-minutes minimum. Remove with tongs and weigh immediately.
49	Is the act of desiccation and weighing repeated until the weight loss of the flask and dried residue less than 4% of the previous weight or less than 0.5 mg, whichever is less? [EPA Method 1664, Rev. B, Section 11.4.4]			While at room temperature and without additional heating, repeat the cycle of desiccating and weighing until the weight loss of the flask and dried residue is less than 4 % of the previous weight or less than 0.5 mg, whichever is less. The final weighing should be used for determining the value for HEM or SGT-HEM as appropriate.
50	Is the HEM determined by subtracting the tare weight from the total weight of the flask? [EPA Method 1664, Rev. B, Section 11.4.4.1]			If the extract was from the HEM procedure, determine the HEM (W_h) by subtracting the tare weight (Section 11.3.1) from the total weight of the flask.
51	Is the original sample volume determined by filling the sample bottle to the mark with water and measuring the volume or mass of the water? [EPA Method 1664, Rev. B, Section 11.4.5]			Determine the original sample volume (V_s) in liters by filling the sample bottle to the mark with water and measuring the volume of water in a 1- to 2-L graduated cylinder. If the sample weight was used (Section 11.1.4), weigh the empty bottle and cap and determine V_s by difference, assuming a sample density of 1.00.
DATA CALCULATION AND REPORTING		L A B	S O P	EXPLANATION
52	How is HEM calculated? [EPA Method 1664, Rev. B, Section 12.1] ANSWER:			n-Hexane extractable material—Calculate the concentration of HEM (“oil and grease”) in the sample per the following equation: $HEM (mg/L) = \frac{W_h(mg)}{V_s(L)}$ where: W_h = Weight of extractable material from Section 11.4.4.1 (mg) V_s = Sample volume from Section 11.4.5 (L)
53	Are results reported to three significant figures for results above 10 mg/L and to two significant figures for results below? [EPA Method 1664, Rev. B, Section 12.3]			Report results to three significant figures for HEM and SGT-HEM found at or above 10 mg/L, and report results to two significant figures for HEM and SGT-HEM found below 10 mg/L.
54	Are samples with results found to be below the method prescribed 5.0 mg/L or a laboratory-defined reporting limit reported as a “less than” value? [EPA Method 1664, Rev. B, Section 12.3.1] List Your Reporting Limit:			Report results for HEM and SGT-HEM found below the ML as < 5.0 mg/L, or as required by the permitting authority or permit. Guidance by EPA Region IV states that establishing an ML less than 5.0 mg/L is an acceptable modification as long as an IPR test per section 9.2.2 of the method demonstrates equivalent or better performance and all other mandated

				determinative techniques are followed as written in the method.
55	When QC is unacceptable, are the associated samples collected again and reanalyzed if possible? [EPA Method 1664, Rev. B, Section 12.3.3]			<p>The method says: Results from tests performed with an analytical system that is not in control (Section 9) must not be reported or otherwise used for permitting or regulatory compliance purposes and do not relieve a discharger or permittee of timely reporting.</p> <p>However, even though this a method-defined parameter and it says you can't use the data for permitting or regulatory compliance purposes, the permittee is required by the 2B rules to report the data. So, they have no choice but to report the data with qualifiers even if they do resample. Commercial labs don't have control over whether a lab resamples or not. They're going to have to report the data with qualifiers and let the client decided what to do with it. The data receiver (the State) would then have to determine whether to use the data, or not.</p>
56	When QC is unacceptable and resampling is not possible, are sample results qualified with the QC failure? [15A NCAC 2H .0805 (a) (7) (B)]			<p>If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such.</p> <p>If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data.</p>
QUALITY ASSURANCE		L A B	S O P	EXPLANATION
57	Are IDOCs analyzed before sample analysis? [EPA Method 1664, Rev. B, Section 9.1.1]			The laboratory shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.
58	Has an MDL been established? [EPA Method 1664, Rev. B, Section 9.2.1]			Method Detection Limit (MDL)–To establish the ability to detect HEM and SGT-HEM, the laboratory shall determine the MDL per the procedure in 40 CFR 136, Appendix B using the apparatus, reagents, and standards that will be used in the practice of this method.
59	What is the MDL? [EPA Method 1664, Rev. B, Section 9.2.1] ANSWER:			An MDL less than or equal to the MDL in Section 1.6 (1.4 mg/L) or less than 1/3 the regulatory compliance limit must be achieved prior to the practice of this method.
60	Has an Initial Precision and Recovery (IPR) study been performed by analyzing four replicates of the PAR standard? [EPA Method 1664, Rev. B, Section 9.2.2. 9.2.2.1]			<p>9.2.2 Initial precision and recovery (IPR)–to establish the ability to generate acceptable precision and accuracy, the laboratory shall perform the following operations:</p> <p>9.2.2.1 Determine the concentration of HEM and/or SGT-HEM in four samples of the PAR standard (Section 7.11) according to the procedure beginning in Section 11.</p>
61	Was the IPR acceptable? [EPA Method 1664, Rev. B, Section 9.2.2.2 and 9.2.2.3]			Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation of the percent recovery (s) for HEM and for SGT-HEM (if determined). When determining SGT-HEM,

			<p>the true concentration (T) must be divided by 2 to reflect the concentration of hexadecane that remains after removal of stearic acid. Use the following equation for calculation of the standard deviation of the percent recovery:</p> $s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n - 1}}$ <p>where: n = Number of samples x = % recovery in each sample</p> <p>Compare s and X with the corresponding limits for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem and repeat the test.</p> <p>If s and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem and repeat the test.</p> <p>IPR acceptance criteria from Table 1: HEM precision (s): 11% HEM accuracy (x): 83-101% SGT-HEM precision (s): 28% SGT-HEM accuracy (x): 83-116%</p>
62	Are at least 5 % of samples analyzed as matrix spikes? [EPA Method 1664, Rev. B, Section 9.1.3, 9.3]		<p>9.1.3 Analysis of a matrix spike (MS) is required to demonstrate recovery and to monitor matrix interferences (interferences caused by the sample matrix). The procedure and QC criteria for spiking are described in Section 9.3.</p> <p>9.3 Matrix spikes–The laboratory must spike a minimum of 5 percent of all samples from a given sampling site or, if for compliance monitoring, from a given discharge/waste stream (matrix type). The sample aliquot shall be spiked with the hexadecane/stearic acid spiking solution (Section 7.10). A duplicate matrix spike (MSD) is recommended, but not required.</p>
63	Is the concentration of the matrix spike 1 to 5 times the background concentration or at the concentration of the OPR, whichever concentration is highest? [EPA Method 1664, Rev. B, Section 9.3.1, 9.3.1.1]		<p>9.3.1 The concentration of the spike in the sample shall be determined as follows:</p> <p>9.3.1.1 If, as in compliance monitoring, the concentration of HEM or SGT-HEM in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit, at 1 to 5 times the background concentration of the sample (determined in Section 9.3.2), or at the concentration of the OPR (Section 9.6), whichever concentration is highest.</p>
64	If the HEM concentration is expected to exceed 1000 mg/L, then is a smaller sample volume collected for the background measurement and MS so that the amount of HEM plus the amount spiked does not		If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory compliance limit or at 1 to 5

	exceed 1000 mg/L? [EPA Method 1664, Rev. B, Section 9.3.2.1]		times the background concentration (per Section 9.3.1). NOTE: Samples containing high concentrations (> 100 mg/L) of HEM will require a large volume of spiking solution (Section 7.10) for the MS (and MSD). If the concentration of HEM is expected to exceed 1000 mg/L, smaller sample volumes should be collected for the background measurement and MS (and MSD) so that the amount of HEM plus the amount spiked does not exceed 1000 mg/L.
65	How is the MS prepared? [EPA Method 1664, Rev. B, Section 9.3.2.2 and NC WW/GW LC Policy] ANSWER:		Spike the additional sample aliquot(s) with the spiking solution and analyze the aliquot(s) to determine the concentration after spiking (A). See MS technical assistance document for guidance.
66	What is the acceptance criterion for the MS? [EPA Method 1664, Rev. B, Section 9.3.4 and Table 1] ANSWER:		HEM recovery: 78-114 % HEM RPD: 18 % SGT-HEM recovery: 64-132 % SGT-HEM RPD: 34 %
67	If the matrix spike fails the acceptance criterion but the QC standard in the ongoing precision and recovery test is acceptable, then is it understood to mean there is a matrix interference present? [EPA Method 1664, Rev. B, Section 9.3.4.1]		If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test (Section 9.6) for the analytical batch is within the acceptance criteria in Table 1, an interference is present.
68	If there is matrix interference, is the potential cause for the interference assessed and corrected before being recollected/reanalyzed? [EPA Method 1664, Rev. B, Section 9.3.4.1]		In this case, the result may not be reported or used for regulatory compliance purposes and the laboratory must assess the potential cause for the interference.
69	If the interference is attributable to sampling, is the sample recollected and reanalyzed? [EPA Method 1664, Rev. B, Section 9.3.4.1]		If the interference is attributable to sampling, the site or discharge/waste stream should be resampled.
70	If the interference is attributable to a matrix interference, then is the method modified to account for the interference before the sample is recollected/reanalyzed? [EPA Method 1664, Rev. B, Section 9.3.4.1]		If the interference is attributable to a matrix problem, the laboratory must modify the method, repeat the tests required in Section 9.1.2, and repeat the analysis of the sample and the MS (and MSD, if performed). Most matrix interference problems are attributable to the formation of emulsions in the extraction. Section 11.3.5 provides suggestions for overcoming emulsion problems.
71	If both the spike and OPR fail acceptance criteria, then are the associated samples recollected and reanalyzed? [EPA Method 1664, Rev. B, Section 9.3.4.2]		If the results of both the spike and the ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected, and the sample batch reanalyzed.
72	Are all reported samples associated with a valid MS result? [EPA Method 1664, Rev. B, Section 9.3.4.2]		All samples must be associated with a valid MS (and MSD, if performed).
73	Are 5% of samples analyzed in duplicate? [15A NCAC 2H .0805 (a) (7) (C)]		Except where otherwise specified in an analytical method, laboratories shall analyze five percent of all samples in duplicate to document precision. Laboratories analyzing fewer than 20 samples per month shall analyze one duplicate during each month that samples are analyzed.
74	What is the duplicate acceptance criterion? [EPA Method 1664, Rev. B, Section 9.3.6] ANSWER:		The relative percent difference for duplicates shall meet the acceptance criteria in Table 1. HEM recovery: 78-114% HEM RPD: 18% SGT-HEM recovery: 64-132% SGT-HEM RPD: 34%

75	<p>If an MSD is performed (optional), what is the acceptance criteria?? [EPA Method 1664, Rev. B, Section 9.3.6]</p> <p>ANSWER:</p>		<p>The relative percent difference for duplicates shall meet the acceptance criteria in Table 1.</p> <p>HEM recovery: 78-114% HEM RPD: 18% SGT-HEM recovery: 64-132% SGT-HEM RPD: 34%</p>
76	<p>If the RPD acceptance criteria are not met, then are the associated samples recollected/reanalyzed? [EPA Method 1664, Rev. B, Section 9.3.6]</p>		<p>If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch reanalyzed.</p>
77	<p>Are laboratory blanks analyzed to demonstrate the analysis is free of contamination? [EPA Method 1664, Rev. B, Section 9.1.4]</p>		<p>Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for analysis of a blank are described in Section 9.4.</p>
78	<p>Is a reagent blank extracted and concentrated initially and with each analytical batch? [EPA Method 1664, Rev. B, Section 9.4.1]</p>		<p>Extract and concentrate a laboratory reagent water blank initially (i.e. with the tests in Section 9.2) and with each analytical batch.</p>
79	<p>Is the reagent blank subjected to the same procedural steps as a sample? [EPA Method 1664, Rev. B, Section 9.4.1]</p>		<p>The blank must be subjected to the same procedural steps as a sample.</p>
80	<p>If material is detected in the blank at a concentration greater than the minimum level (i.e., 5.0 mg/L), then is the analysis of samples halted until the source of contamination is eliminated and a blank shows no evidence of contamination? [EPA Method 1664, Rev. B, Section 9.4.2]</p>		<p>If material is detected in the blank at a concentration greater than the minimum level (Section 1.6), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination.</p>
81	<p>Are all samples reported for regulatory compliance purposes associated with an uncontaminated method blank? [EPA Method 166, Rev. B, Section 9.4.2]</p>		<p>All samples must be associated with an uncontaminated method blank before the results may be reported for regulatory compliance purposes.</p>
82	<p>Does the laboratory, on an ongoing basis, demonstrate that the analytical system is in control by analyzing an ongoing precision and recovery sample (OPR)? [EPA Method 1664, Rev. B, Section 9.1.5]</p>		<p>The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery (OPR) sample that the analysis system is in control. These procedures are described in Sections 9.5 and 9.6, respectively.</p>
83	<p>With every batch is the lab extracting and concentrating a precision and recovery standard? [EPA Method 1664, Rev. B, Section 9.6.1]</p>		<p>Extract and concentrate a precision and recovery standard (Section 7.11) with each analytical batch according to the procedure beginning in Section 11.</p>
84	<p>Is the recovery of the precision and recovery standard found to be within the limits detailed in Table 1 of the Method before the analysis of blanks and samples may proceed? [EPA Method 1664, Rev. B, Section 9.6.2]</p>		<p>Compare the recovery with the limits for ongoing precision and recovery in Table 1. If the recovery is in the range specified, the extraction, distillation, and weighing processes are in control and analysis of blanks and samples may proceed.</p> <p>HEM recovery 78-114% SGT-HEM recovery 64-132%</p>
85	<p>If not, then is the problem identified and corrected before the batch is re-extracted and the OPR test is repeated? [EPA Method 1664, Rev. B, Section 9.6.2]</p>		<p>If, however, the recovery is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-extract the analytical batch, and repeat the ongoing precision and recovery test.</p>
86	<p>Is a second source QCS standard analyzed monthly to verify the hexadecane and stearic acid routinely used? [15A NCAC 2H .0805 (a) (7) (H) (ii)] [EPA Method 1664, Rev. B, Section 9.7]</p>		<p>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.</p> <p>1664B: Quality control sample (QCS)—It is suggested that the laboratory obtain a QCS from a source different from the source for the hexadecane and stearic acid used routinely in this method (Sections 7.8 and</p>

				7.9), and that the QCS be used for verification of the concentrations of HEM and SGT-HEM using the procedure given in the note in Section 7.10.3. The QCS should be analyzed monthly by laboratories performing routine analyses and less frequently by laboratories performing these analyses intermittently.
87	Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)]			<p>If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such.</p> <p>If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data.</p>
88	Has the laboratory modified the method in any way? [EPA Method 1664, Rev. B, Section 9.1.2]			Alternate determinative techniques, such as immunoassay or infrared spectroscopy, and changes that degrade method performance or change the chemistry of the method including the use of extraction solvents other than n-hexane (85% minimum purity, 99.0% min. saturated C ₆ isomers, residue less than 1 mg/L – see Section 7.3) are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for HEM and/or SGT-HEM in the sample of interest.
	If the answer above was yes, then complete the rest of the checklist. If not, then you may stop here.			
89	Is the IPR test repeated to demonstrate that any modifications produce results equivalent or superior to results produced by this method each time a modification is made? [EPA Method 1664, Rev. B, Section 9.1.2.1]			Each time a modification is made to this method, the laboratory is required to repeat the IPR test in Section 9.2.2 to demonstrate that the modification produces results equivalent to or superior to results produced by this method.
90	If the MDL is affected by any modifications, then does the laboratory demonstrate that the MDL is less than or equal to the MDL in this method or one-third the regulatory compliance limit, whichever is higher, each time a modification is made? [EPA Method 1664, Rev. B, Section 9.1.2.1]			If the detection limit of the method will be affected by the modification, the laboratory must demonstrate that the MDL (40 CFR Part 136, Appendix B) is less than or equal to the MDL in this method or one-third the regulatory compliance limit, whichever is higher.
91	Anytime the method is modified does the laboratory perform a comparison study for the recovery of HEM on each specific discharge/waste stream? [EPA Method 1664, Rev. B, Section 9.1.2.1]			If the modified method is to be used for compliance monitoring, the discharger/generator must also demonstrate that the modified method recovers an amount of HEM and/or SGT-HEM equivalent to the amount recovered by this method on each specific discharge/waste stream (not required for modifications allowed under Section 1.7.1).
92	<p>If any modifications of this method have been made, then list all items which are recorded with regard to the modifications. [EPA Method 1664, Rev. B, Section 9.1.2.2.1]</p> <p>ANSWER:</p>			<p>9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification (not required for modifications allowed under Section 1.7.1).</p> <p>9.1.2.2.2 A listing of pollutant(s) measured (HEM and/or SGT-HEM)</p>

			<p>9.1.2.2.3 A narrative stating reason(s) for the modification</p> <p>9.1.2.2.4 Results from all quality control (QC) tests comparing the modified method to this method, including:</p> <ul style="list-style-type: none"> (a) Calibration (Section 10) (b) Calibration verification (Section 9.5) (c) Initial precision and recovery (Section 9.2.2) (d) Analysis of blanks (Section 9.4) (e) Accuracy assessment (Section 9.3) (f) Ongoing precision and recovery (Section 9.6) (g) Method detection limit (Section 9.2.1). <p>9.1.2.2.5 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight or other signal) to the final result. These data are to include:</p> <ul style="list-style-type: none"> (a) Sample numbers and other identifiers (b) Extraction dates (c) Analysis dates and times (d) Analysis sequence/run chronology (e) Sample weight or volume (Section 11.1.4) (f) Extract volume for SGT-HEM (Section 11.5.2) (g) Make and model of analytical balance and weights traceable to NIST (h) Copies of logbooks, printer tapes, and other recordings of raw data (i) Data system outputs, and other data to link the raw data to the results reported.
93	<p>What types of samples are analyzed to demonstrate method modification equivalency? [EPA Method 1664, Rev. B, Section 9.2.3]</p> <p>ANSWER:</p>		<p>9.2.3 Equivalency demonstration for application of a method modification to compliance monitoring - To establish the ability of a modification of this method to recover an amount of HEM and/or SGT-HEM equivalent to the amount recovered by this method from a specific discharge/waste stream (matrix types), proceed as follows:</p> <p>NOTE: Equivalency demonstration for specific discharge/waste stream types is not required for the modifications listed as acceptable in Section 1.7.1. For modifications not listed as acceptable at Section 1.7.1, the demonstration is needed only for an example of the specific discharge/ waste stream (matrix type) to be analyzed up to a maximum of the 9 matrix types identified below which would be required to validate the modified method for use with all samples. If validating the modified method for use with all samples, each example waste stream below (except POTW effluent) should be known or suspected to contain an average concentration of 5 to 1,000 mg/L of HEM and/or SGT-HEM to be included in the demonstration:</p> <ul style="list-style-type: none"> 1. One POTW effluent 2. A second POTW effluent from a different source 3. Saline water 4. Two representative examples of different treated or untreated wastewaters likely to contain petroleum-based substances (hydrocarbons similar in chemical

			composition to n-hexadecane) at significantly different concentrations 5. Two representative examples of different treated or untreated wastewaters likely to contain animal-based substances (animal fats similar in chemical composition to stearic acid) at significantly different concentrations 6. Two representative examples of different treated or untreated wastewaters likely to contain other hexane extractable materials of various chemical composition including but not limited to, vegetable oils, waxes, soaps, greases and other related materials
94	For the comparison study, are two sets of four aliquots of unspiked wastewater analyzed...one set by the Method and one set by the modified method? [EPA Method 1664, Rev. B, Section 9.2.3.1]		Collect, extract, concentrate, and weigh the HEM or SGT-HEM in two sets of four aliquots of unspiked wastewater. One set of four wastewater aliquots is analyzed according to the protocol in Section 11 of this method and the other set of four aliquots is analyzed using the modified method.
95	Is the average concentration of the modified method 78 -114 percent of the average concentration produced by the Method? [EPA Method 1664, Rev. B, Section 9.2.3.2]		Calculate the average concentration of HEM and SGT-HEM for the set of results from this method and for the set of results from the modified method. The average concentration using the modified method must be 78 to 114 percent of the average concentration produced by this method for HEM and 64 to 132 percent of the average concentration produced by this method for SGT-HEM. If not, the modified method may not be used.
96	If the average concentration of the eight results produced by both methods is below the minimum level, then are MSs used to determine equivalency? [EPA Method 1664, Rev. B, Section 9.2.3.2]		NOTE: If the average concentration of the four results produced using this method and the average concentration of the four results produced using the modified method are below the minimum level (Section 1.6), and if the equivalency test of the modified method is passed for spikes of reference standards into reagent water (Section 9.2.2), the modified method is deemed to be equivalent to this method for determining HEM and or SGT-HEM on that specific discharge/waste stream.

Additional Comments:

Inspector: _____ Date: _____