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EPA 1664B– Oil and Grease – N- Hexane Extractable Material

Access to this SOP shall be available within the laboratory for reference purposes; the official copy of this SOP resides on the official Georgia EPD website at https://epd.georgia.gov/about-us/epd-laboratory-operations. Printed copies of this SOP will contain a watermark indicating the copy is an uncontrolled copy.

1 Scope and Application

1.1 This method covers the determination of n-hexane extractable material in surface and saline waters and industrial wastes. Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials. This method is based on prior Environmental Protection Agency methods for determination of "oil and grease: and "total petroleum hydrocarbons".

1.2 This procedure is restricted to use by an analyst experienced in the operation of a Buchi Rotavapor. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Plan (see SOP reference 13.2) for Quality Control Definitions.

2.2 Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3 Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4 Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5 Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method Blank (MBLK), Method Detection Limit Blank (MDLB) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.6 MDLS (Method Detection Limit Spike) – MDLB spiked with analytes at the lowest calibration level to be used for the determination of MDL.

2.7 LCS (Laboratory Control Sample) and LCSD (Laboratory Control Sample Duplicate) are prepared by spiking laboratory reagent water, Ottawa sand or air sampling device with the target analyte or compound. They are used to validate

the analytical batch with respect to accuracy and precision.

3 Interferences

3.1 Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts that affect results. Specific selection of reagents and purification of solvents may be required.

3.2 All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running laboratory blanks.

3.3 Glassware is cleaned by washing in hot water containing detergent, rinsing with tap and distilled water, and rinsing with solvent or baking. Boiling flasks that will contain the extracted residue are dried in an oven at 105 –115ºC.

3.4 Sodium sulfate and silica gel fines have the potential to inflate results for HEM and SGT-HEM by passing through the filter paper. If the filter paper specified in this method is inadequate for removal of these fines, use of a 0.45-micron filter is recommended.

3.5 Interferences extracted from samples will vary considerably from source to source, depending upon the diversity of the site being sampled. For those instances in which samples are thought to consist of complex matrices containing substances (such as particulates or detergents) that may interfere with the extraction procedure, a smaller sample may need to be collected for analysis.

4 Safety

4.1 Refer to the EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision. (See SOP reference 13.8)

5 Apparatus and Equipment

5.1 Sample Container: 1-Liter wide – mouth amber bottle.

5.2 Desiccator, provided with desiccant containing a color indicator for moisture

5.3 Analytical Balance, capable of accurately weighing to the nearest 0.0001 g

5.4 Buchi rotavapor with heating water bath capable of maintaining a temperature of at least 85ºC.

5.5 Oven

5.6 2000 mL glass separatory funnels, with PTFE stopcocks

5.7 250 ml evaporating flasks with flask stands

5.8 500 ml receiving flask

5.9 Glass wool

5.10 Whatman 125 mm filter paper – Fisher Catalog No. 09-805F or equivalent

5.11 Fume Hood

5.12 Glassware -- Class “A” volumetric flasks, graduated cylinders, and pipettes.

5.13 Funnels

5.14 Laboratory vacuum system

5.15 Tongs

5.16 Low range pH paper - Fisherbrand™ Paper pH Strips Catalog No. 13-640-511 or equivalent.

5.17 Laboratory cold water tap

5.18 Boiling Chips - Chemware™ PTFE Boiling Stones Fisher Catalog No.09-191-20 or equivalent.

5.19 Drying columns – special ordered from Hammett - 32 mm special wall 12" long with short tip- or equivalent.
5.20 Glass stirring rod

6 Reagents

6.1 Reagent Water:
6.1.1 Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest (Deionized, HPLC, Milli-Q water, or equivalent. Milli-Q water has a resistivity of 18.2[MΩ.cm] @ 25ºC and a TOC of 50 ug/L or less).n-Hexane-85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L (0.0001% max.)
6.2 Sodium Sulfate (Na_2SO_4): anhydrous, granular, ACS grade:
6.2.1 Dry at 200-250 degrees Celsius for 24 hours minimum and store in desiccator.
6.3 Hexadecane/stearic acid 40 mg/L HEM spiking solution (20mg of hexadecane and 20mg of stearic acid):
6.3.1 A commercially prepared Hexadecane/stearic acid (1:1) spiking solution is purchased from Environmental Express and used.
6.3.1.1 Environmental Express Catalog No. G3025 - SPE - O/G Standard, 10mL tubes, 40mg HEM or equivalent is used.
6.3.2 This standard is used for spiking the LCS/LCSD and MS/MSD.
6.3.3 Store at room temperature.
6.4 HCl solution (HCl 1:1):
6.4.1 In a 200 ml volumetric flask, add 100 ml of reagent water and then add 100 ml of concentrated HCl. Mix thoroughly.
6.4.2 Solution is stable for one year.
6.5 ICV Standard Solution (ICV/SS):
6.5.1 Must be different from the source for the hexadecane and stearic acid spiking solution.
6.5.2 The ICV should be analyzed with each batch.
6.5.3 US-QCI-770 ordered from Fisher Scientific may be used or Environmental Express Catalog No.: G3020 - SPE - O/G, 9X30mL Bottles, HEM at 40mg (w/v) or equivalent.
6.5.4 Prepare the ICV at levels at or above the Hexadecane/stearic acid 40 mg/L spiking solution level following the manufacturer's instructions. If the ICV standard solution concentration is below 40 mg/L, more than one bottle of standard may be added to reach the required concentration.
6.5.5 Store at room temperature.
6.6 Laboratory Control Sample and Laboratory Control Sample (LCS/LCSD) 40 mg/L concentration:
6.6.1 Prepare an LCS and LCSD using the Hexadecane/stearic acid 40 mg/L spiking solution (20mg of hexadecane and 20mg of stearic acid) (Sec. 6.3).
6.6.2 Snip and pour the contents of one 10 ml SPE-O/G HEM 40 mg Standard PTFE tube (Environmental Express Catalog# G3025) into 950-1050 mL of reagent water. Acidify contents of container with 5.0 ml of 1:1 HCl Solution (Sec. 6.4). Shake to mix thoroughly. Record lot # of sample container on reagent list.
6.6.3 LCS's are prepared at the 40.0 mg/l concentration.
6.7 Matrix spike/Matrix spike Duplicate (40 mg/L concentration):

6.7.1 Prepare an MS and MSD using the Hexadecane/stearic acid 40 mg/L spiking solution (20mg of hexadecane and 20mg of stearic acid)(Sec. 6.3).
6.7.2 Snip and pour the contents of one 10 ml SPE-O/G HEM 40 mg Standard PTFE tube (Environmental Express Catalog# G3025) into each 950-1050 mL matrix spike and matrix spike duplicate sample bottle (provided by collector). Shake to mix thoroughly.
6.7.3 MS and MSDs are prepared at the 40.0 mg/l concentration.
6.7.4 The MS and MSD samples are provided by the sample collector.
6.7.4.1 If the MS and MSD samples are not provided, initiate a corrective action form stating that insufficient sample provided by collector to analyze the MS and MSD and comment on O&G1664B test code. Remove QC test codes associated with the Matrix spike and matrix spike duplicate.
6.7.4.2 All QC tests associated with MS/MSD will need to be deleted.
6.7.5 Spike a minimum of 5% of all samples.
6.8 MDLB/ MBLK:
6.8.1 Transfer 950 ml of reagent water to a clean sample collection container. Acidify using 5.0 ml 1:1 HCl. Shake to mix thoroughly and fill to top with reagent water. Record lot # of sample container on reagent list.
6.9 MDLS (Method Detection Limit Spike) 5.0 mg/L standard:
6.9.1 Snip and pour the contents of one 10 ml SPE - O/G- 5 mg MDL Standard PTFE tube (Environmental Express Catalog# G3024) into 950-1050 mL of reagent water. Acidify contents of container with 5.0 ml of 1:1 HCl Solution (Sec. 6.4). Shake to mix thoroughly. Record lot # of sample container on reagent list.

7 Sample Collection

7.1 Samples are collected in a 1L wide mouthed amber glass bottles containing 5 ml of 1:1 HCl to preserve the sample to a pH of < 2 at the time of collection.
7.2 Samples are cooled and stored at 0-6° C (not frozen).
7.3 Sample holding time is 28 days.
7.4 Sample preservation is checked in the receiving lab at time of receipt.

8 Calibration

8.1 Analytical balances are serviced and calibrated once per year by an independent technician.
8.2 The balance used for this analysis must have the calibration verified before and after each day of use with certified Class 1 weights that bracket the expected weight range of the analysis.
8.3 Calibration Curve
8.3.1 Not applicable.
8.4 Calibration Verification
8.4.1 Verify calibration of the balance before and after each analytical batch per Section 8.2.
8.4.2 If calibration is not verified before and after each day or after measurement of the analytical batch, recalibrate the balance and reweigh the batch.

9 Quality Control

9.1 Refer to Table 14.1 for Reporting Limits (RLs), Appendix A for Quality Assurance criteria and Table 14.2 for a summary of Quality Control procedures

associated with this method.

9.2 A method detection limit study must be performed twice per year. Refer to SOP reference 13.6.

9.3 Refer to SOP references 13.3 for training and certification procedures.

9.4 Refer to SOP reference 13.4 for control charting procedures.

9.5 Default control limits for recovery and precision are based on Table 1 in Section 17.0 of EPA Method 1664B. See SOP reference 13.1. Precision limit defaults are set by the method for IDCs and matrix spikes, and by the EPD Laboratory for laboratory control spikes. In-house limits based on control charts may never exceed the default limits. The control limit defaults below are presented to assist in defining control limits established with control charts and are not used as batch acceptance criteria.

9.5.1 SOP reference 13.7 requires that LCS (and therefore LCSD) QC ranges be adjusted periodically based on control charts. Method 1664B requires that matrix spikes be charted with ranges based on 2 standard deviations from the mean. Therefore, all ranges for EPA Method 1664B are based on 2 standard deviations rather than 3 standard deviations per the control chart SOP.

9.5.2 LCS control limits are used to monitor LCSD recovery. LCSD recovery is not used to validate batch data.

Table 9.5.2.1 Default QC Limits for Method EPA 1664B

QC Type	Analyte	Accuracy(%R) LCL		UCL	Precision (%RPD)
IDC*	Oil and Grease	83	-	101	11
LCS/LCSD	Oil and Grease	78	-	114	15**
MS/MSD	Oil and Grease	78	-	114	18

*Initial Demonstration of Capability. See reference 13.3

**EPA Method 1664 does not address LCS/LCSD precision; therefore, the EPD Lab sets a default limit of 15% for LCS/LCSD %RPD

9.6 MDL (method detection limit) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

9.6.1 The actual MDL varies depending on instrument and matrix.

9.6.2 The MDL must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.6.3 The Method Detection Limit Study for all analytes must be performed initially on a new instrument and performed after major instrument repairs or changes to procedures. There are two ways to perform the MDL. The first is with 7 samples and 7 blanks over 3 separate days. The second preferred way the MDL is run is as a continuous format.

9.6.4 The 7 MDL samples study is performed by preparing 7 spiked vials, MDLSpike, spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDLBlank. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there may or may not be a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blank.

9.6.5 A continuous format MDL study is performed where one vial is spiked as an MDLSpike, at the lowest point of the calibration curve and analyzed with every batch of samples along with the method blank vial as an MDLBlank.

9.6.6 The results of the MDLBlank will be entered into Labworks using the Method Blank test code, B_O&G-1664B. The MDLSpike result will be entered using the ML O&G-1664B. The MDL Spiked Amount will be entered into the test code MA O&G-1664B. The instrument used for the MDL and Blank analysis will be selected using the test code INSTR- O&G-1664B.

9.6.7 MDL study must be performed every six months and before the MDL for the instrument expires.

9.6.8 Data for the MDL study is pulled from a two year period.

10 Procedure

10.1 Preparation of the analytical batch

10.1.1 Create a batch consisting of an ICV, MDLB, LCS/LCSD pair, MDLS, and MS/MSD pair (if provided by collector), and up to 20 field samples extracted in a 12-hour shift. If greater than 20 samples are to be extracted in a 12-hour shift, the samples must be separated into analytical batches of 20 or fewer samples.

10.1.2 Remove sample bottles, standards and reagents from cold storage and allow equilibration to room temperature prior to sample preparation and/or analysis.

10.1.3 Prepare the Blank per Section 6.8.

10.1.4 Prepare the LCS/LCSD per Section 6.6.

10.1.5 Mark the sample bottles (including provided MS/MSD pair) at the water meniscus for later determination of sample volume.

10.1.6 Perform all work under a hood. Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

10.2 pH Verification:

10.2.1 Verify the pH of each sample including the MBLK/MDLB, LCS/LCSD, MS/MSD and ICV by using a clean stirring rod to transfer a drop of sample to narrow range pH paper. The desired pH is < 2. Rinse the rod with hexane over the sample container to prevent any loss of oil and grease. If the pH is > 2, adjust the pH to < 2 by adding 5-mL of HCl solution (Section 6.4) to 1-L sample. If the sample is at high pH, use a proportionately larger amount of HCl solution.

10.2.2 Cap the container and shake the sample to mix thoroughly. Check the pH of the sample using the procedure in Section 10.2.1. If necessary, add more acid to the sample and retest.

10.3 Extraction

10.3.1 Tare and assign a clean boiling flask containing 3-5 boiling chips as follows for each sample, MDLB, LCS, LCSD, MS, MSD and QCS.

10.3.1.1 Place the flask containing the chips in an oven at 105-115ºC for a minimum of 2 hours to dry the flask and chips.

10.3.1.2 Remove from the oven and immediately transfer to a desiccator to cool to room temperature.

10.3.1.3 When cool, remove from desiccator with tongs and weigh immediately on a calibrated balance (Section 8). Repeat the cycle of desiccating and weighing

until the weight loss of the flask is less than 4% of the previous weight or less than 0.5 mg, whichever is less. Record weights on bench sheet. Verify calibration of the balance before and after each analytical batch.

10.3.2 Transfer the sample into a 1 L separatory funnel.

10.3.3 Add 30 mL of n-hexane to the sample bottle and seal the bottle with the original bottle cap. 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.

10.3.4 Extract the sample by shaking the separatory funnel vigorously for 2 minutes with periodic venting into a hood to release the excess pressure.

10.3.5 Allow the organic phase to separate from the aqueous phase for a minimum of 10 minutes. 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 may include stirring or filtration through glass wool.

10.3.6 Drain the aqueous layer (lower layer) into the original sample container. Drain a small amount of the organic layer into the sample container to minimize the amount of water remaining in the separatory funnel. Note: The amount of water remaining with the n-hexane must be minimized to prevent dissolution or clumping of the sodium sulfate in the extract drying process.

10.3.7 Place a filter paper in a filter funnel, add approximately 10 grams of dried anhydrous Na_2SO_4 (See section 6.2), and rinse with a small portion of n-hexane. Discard the rinsate.

10.3.8 Drain the n-hexane layer (upper layer) from the separatory funnel through the Na_2SO_4 into the pre-weighed boiling flask containing boiling chips (Section 10.3.1). Note: It is important that water be removed in this step. Water allowed to filter through the Na_2SO_4 will dissolve some of the Na_2SO_4 and carry it into the boiling flask compromising the determination.

10.3.9 Repeat the extraction (Section 10.3.3-10.3.6 and 10.3.8) twice more with fresh 30-mL portions of n-hexane, combining the extracts in the boiling flask.

10.3.10 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 rinsate in the flask.

10.3.11 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 10.3.7 and 10.3.8. Rinse the glassware and sodium sulfate with small portions of n-hexane to affect a quantitative transfer.

10.4 Solvent distillation

10.4.1 Fill the Buchi R-100 water bath two-thirds full of reagent water.

10.4.2 Turn the rotation speed knob to 0. Plug the power cord into the hood receptacle and turn the main switch on. The switch is located on the right side of water bath.

10.4.3 Adjust temperature to 95°C by using the up and down arrows. Record the temperature of the thermometer and thermometer # on the extraction sheet. The temperature must be greater than 85°C.

10.4.4 Place the receiving flask on the bottom of the condenser. Secure the flask with the clamp. Then place round bottom flask (evaporating flask) on the vapor duct. Make sure to push all the way up.

10.4.5 Lower the slide clip over the lip of the evaporating flask. Screw the combi-clip

clockwise to engage the clip and lock flask in place. Make sure to only make it hand-tight.

10.4.6 Turn the glass stopcock handle towards the front of hood. This is the sealed position.

10.4.7 Open the cap on top of condenser to allow venting in case the vacuum system goes down.

10.4.8 Make sure water drain for the condenser is in drain. Turn the hood's water valve about one-fourth of a turn counterclockwise. The position should be marked. Slowly lift the drain tube to make sure a stream of water is coming out. Place tube back in drain.

10.4.9 Turn hood's vacuum one-half turn counterclockwise. The position should already be marked.

10.4.10 Turn the rotation speed to 5 and slowly lower the flask in water bath by pressing handle. Make sure the bath is pushed as far to the left as possible to prevent flask from contacting water bath. Evaporate until there is only a few mL of solvent remaining. Do not evaporate to dryness. 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 flasks. If crystals are observed, re-dissolve the extract in n-hexane, quantitatively transfer through a filter into another tarred boiling flask, and repeat distillation procedure.

10.4.11 Raise the flask out of bath by pressing lever. Turn rotation speed to 0. Remove flask with tongs. Rinse outside of flask with D.I. Water. Sweep the flask with a stream of air with tube attached to air outlet on hood. Wipe the outside with paper towel. Do not handle with bare hands since oil from hands can add weight. Dry flasks overnight in hood.

10.4.12 The next day, place flasks in a desiccator for at least 30 minutes. Verify balance per Section 8.4. Verify calibration of the balance before and after each analytical batch. Remove with tongs and weigh immediately. Record weight on bench sheet. 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. Record all weights on bench sheet.

10.4.13 Determine the HEM ("oil and grease") by subtracting the tare weight (Section 10.3.1) from the total weight of the flask. See equation (Section 11.1)

10.4.14 Determine the original sample volume in liters by filling the sample bottle to the mark with water and measuring the volume of water in a 1-2 L graduated cylinder.

10.4.15 Always verify the excel calculation of one sample on the Excel spreadsheet, by hand calculating the result and writing it at the bottom of the spreadsheet.

11 Calculations

11.1 (Weight of flask (g) + HEM (g)) – Weight of flask (g) = HEM (g) in sample

11.2 HEM (g) x 1000 = HEM (mg)

11.3 To calculate sample concentration of HEM as mg/L:

$$\text{Sample Concentration HEM } {}^{mg}/_{L} = \frac{\text{HEM } (mg) * 1000 \ {}^{ml}/_{L}}{\text{Sample Volume } (ml)}$$

11.4 Relative Percent Difference (%RPD or RPD):

$$\%\text{RPD} = \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} * 100$$

11.4.1 Where:

$|X_1 - X_2|$ = Absolute difference between two values

$\frac{(X_1 + X_2)}{2}$ = Average of two values

11.5 Percent Drift, %Drift:

$$\%\text{Drift} = \frac{(\text{Concentration}_{\text{Calculated}} - \text{Concentration}_{\text{Expected}})}{\text{Concentration}_{\text{Expected}}} * 100$$

11.5.1 Where:

$\text{Concentration}_{\text{Calculated}}$ = Concentration calculated from result

$\text{Concentration}_{\text{Expected}}$ = Theoretical concentration of the standard

11.6 Percent Recovery:

11.6.1 LCS/LCSD:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.6.1.1 Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.6.2 MS/MSD:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}} - \text{Conc}_{\text{unspiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.6.2.1 Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{unspiked}}$ = Concentration found in unspiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.7 Mean ($\bar{X}$):

$$\overline{X} = \frac{X_1 + X_2 + \cdots X_n}{n}$$

11.7.1 Where:

$X_1 + X_2 + \cdots X_n$ = The sum of a set of values X_i, i = 1 to n

n = The number of values in the set

11.8 Standard Deviation (n – 1) (σ_{n-1}):

$$\sigma_{n-1} = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{n-1}}$$

11.8.1 Where:

$\overline{X}$ = Mean of the values

X_i = Individual values 1 through i

n = Number of values

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures, See SOP reference 13.5.

13 References

13.1 Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry. February 2010. U.S. Environmental Protection Agency, Office of Water (4303T). Washington, DC. EPA-821-R-10-001.

13.2 EPD Laboratory Quality Assurance Plan, online revision.

13.3 GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, online revision or Continuing Demonstration of Capability SOP 6-002, online revision.

13.4 GA EPD Laboratory SOP-EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.

13.5 GA EPD Laboratory SOP-EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.6 GA EPD Laboratory SOP – Determination of Method Detection Limit, Method Detection Limit SOP 6-007, online revision.

13.7 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; Final Rule. EPA-HQ-OW-2010-0192-0328. Docket ID: EPA-HQ-OW-2010-0192. Environmental Protection Agency. Federal Register Volume 77, Number 97 (Friday, May 18, 2012).

13.8 GA EPD Laboratory Safety Plan – EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1 RLs for Method EPA 1664B			
Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
EPA 1664B	Oil and Grease	5.0	mg/L

Table 14.2 Summary of Calibration and QC Procedures for Method EPA 1664B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
EPA 1664B	Oil and Grease	Second source calibration verification (ICV)	Once per analytical run	Recovery must be within 78-114% of expected value.	Correct problem then reanalyze QCS and all samples in affected batch.	If unable to reanalyze, flag with a "J"
		Method Blank (MBLK/MDLB)	One per analytical batch	Oil and Grease value must be < 5.0 mg/L.	Correct problem then analyze method blank and all samples processed with the contaminated blank	If unable to reanalyze, flag with a "B"
		Laboratory Control Sample (LCS/LCSD)	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table SOP 3-020 A.1, Appendix A	Correct problem then reanalyze LCS/LCSD and all samples in affected batch.	If unable to reanalyze, flag with a "J"
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analyses of a QC check sample, a method blank and a blind sample.	Once per analyst	QC Acceptance Criteria Table, SOP 3-020 A.1, Appendix A and Initial Demonstration of Capability SOP (Reference 13.3)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Every 6 months	QC Acceptance Criteria Table, SOP 3-020 A.1, Appendix A and Continuing Demonstration of Capability SOP (Reference 13.3)	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	

Table 14.2 Summary of Calibration and QC Procedures for Method EPA 1664B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
EPA 1664B	Oil and Grease	Matrix spike (MS/MSD)	Minimum Frequency of 5% of samples	QC Acceptance Criteria Table SOP 3-020 A.1, Appendix A	Evaluate out of control event, reanalyze or flag data	Comment on sample if collector fails to provide sample bottles for MS/MSD
		MDL Study	Every six months or after major maintenance of the instrument	All Spiked MDLs must have a value greater than 0. Minimum Detection Limits established shall be < the RLs in Table 14.1	Re-do MDL Study	
		MDL analysis	Once per batch or as needed to acquire data points per SOP 6-007, online revision	All Spiked MDLs must have a value greater than 0. All other QC in the MDL blank and MDL sample (i.e., Surrogate Spike or Internal Standard, etc. if included) must meet established criteria	Correct problem and re-run the MDL sample or MDL blank once and initiate a corrective action. If the re-run fails a second time, do not use MDL data. Update corrective action, and use associated sample data	
		MDL Low level Spike (MDLS) 5.0 mg/L	Once per analytical batch	All batch QC must be valid	Correct problem then reanalyze the affected batch	

Appendix A – Quality Assurance Criteria for Method EPA 1664B*

Table A.1 Quality Assurance Criteria for Method EPA 1664B

QC Type	Analyte	Accuracy(%R) LCL		UCL	Precision (%RPD)
IDC*	Oil and Grease	83	-	101	11
LCS/LCSD**	Oil and Grease	78	-	114	15
MS/MSD***	Oil and Grease	78	-	114	18

Control chart data generated twice annually for trend monitoring purposes only.
Control chart data generated from 01/01/2019 - 01/01/2021

Updates to Previous Version:
Section 2
Section 6
Section 9
Section 10
Table A.1
Table 14.2