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: **Extractable Petroleum Hydrocarbons (EPH)**

Method: **Massachusetts Department of Environmental Protection EPH, December 2019, Rev. 2.1 (Aqueous & Non-Aqueous)**

Equipment:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Syringes |  | Water or steam bath |  | Kuderna Danish Concentrator or equivalent |
|  | Pipets |  | Fractionation Cartridges |  |  |
|  | GC with Flame Ionization Detector  Model: |  | Capillary Column (Restek RTX-5 30m x 0.32mm ID, 0.25µm film thickness or equivalent. Type: | | |

Reagents:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Reagent water, organic free |  | Methylene Chloride – Pesticide grade or equivalent |  | Fractionation Cartridges |
|  | Acetone – Pesticide Grade or Equivalent |  | Sodium Sulfate – ACS grade, anhydrous |  |  |
|  | Hexane – Pesticide grade or equivalent |  | Sand – EPH free |  |  |

Standards:

|  |  |  |  |
| --- | --- | --- | --- |
|  | Stock Aromatic Hydrocarbon Standard: List Vendor/Lot# |  | Stock Aliphatic Hydrocarbon Standard: List Vendor/Lot# |
|  | Stock EPH Surrogate Standards: List Vendor/Lot# |  | Stock Fractionation Surrogate Standard: List Vendor/Lot# |
|  | Second Source Aliphatic Hydrocarbon Standard: Vendor/Lot# |  | Second Source Aromatic Hydrocarbon Standard: Vendor/Lot# |
|  | Matrix Spiking Solution – Second Source |  | Fractionation Check Standard |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **PLEASE COMPLETE CHECKLIST IN INDELIBLE INK**  **Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice**  **and in the column labeled SOP to indicate whether it is addressed in the SOP.** | | | | | |
|  | **GENERAL** | **LAB** | **SOP** | **EXPLANATION** |
|  | 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. |
|  | Are all revision dates and actions 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 SOP documents. |
|  | Is there North Carolina data available for review? |  |  |  |
|  | Is the method used to report individual target analytes as well as hydrocarbon ranges? |  |  | Per UST section: Target analytes must be analyzed by SW-846 8270 E or EPA 625.1 |
|  | **PRESERVATION and STORAGE** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are aqueous samples preserved to pH <2 S.U. with 1:1 HCl at the time of collection in 1 Liter Amber glass? [MADEP EPH Rev. 2.1 (Dec 2019) Section 8.1.2] |  |  |  |
|  | Are non-aqueous samples collected in 4 oz. amber glass jars? [MADEP EPH Rev. 2.1 (Dec 2019) Section 8.2.1] |  |  |  |
|  | Are samples cooled to 0-6°C immediately after collection? [MADEP EPH Rev. 2.1 (Dec 2019) Section 8.1.4 and 8.2.2] |  |  |  |
|  | What corrective action is taken if samples do not meet the thermal and/or chemical preservation requirements? [15A NCAC 02H .0805 (a) (7) (M)] |  |  |  |
|  | Are samples extracted within 14 days of collection and analyzed within 40 days of extraction? [MADEP EPH rev. 2.1, (Dec 2019) Section 8.1.5 and 8.2.5] |  |  |  |
|  | **PROCEDURE – Reagents and Standards** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are stock and working standards stored ≤ 6°C or as recommended by the manufacturer? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.2.3] |  |  | Store the vials (protected from light) at ≤ 6°C or as recommended by the standard manufacturer. |
|  | Are stock and working standards disposed of within 6 months of opening or by the manufacturer’s expiration date, whichever comes first? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.2.3] [15A NCAC 02H .0805 (a) (7) (K)] |  |  | **Method:** Stock standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.  **Rule:** Chemicals and reagents exceeding the expiration date shall not be used.  NOTE: working standard expiration dates may not exceed the stock standard expiration date. |
|  | Is the sodium sulfate baked at 400° for at least 4 hours to eliminate interferences? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.1.3] |  |  |  |
|  | Are the surrogate OTP and fractionation surrogates included in the Aromatic Hydrocarbon calibration standard? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.3] |  |  | The surrogate OTP and the fractionation surrogates are included in the Aromatic Hydrocarbon calibration standard; |
|  | Are the surrogate COD, naphthalene, and  2-methylnaphthalene included in the Aliphatic Hydrocarbon calibration standard? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.3] |  |  | The surrogate COD, naphthalene, and 2-methylnaphthalene are included in the Aliphatic Hydrocarbon calibration standard.  Note: naphthalene and 2-methylnaphthalene are incorporated into the aliphatic hydrocarbon standard per Section 7.2.2, but the laboratory may choose to use a smaller concentration for these two compounds in the Aliphatic standard in order to more accurately determine the breakthrough value. |
|  | **PROCEDURE – Instrument Calibration** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is internal standard (IS) calibration used? [MADEP EPH Rev. 2.1 (Dec 2019) Section 7.7.1]  **List, if used:** |  |  | For the EPH method, ISs are only utilized when GC/MS is utilized for quantification. |
|  | Are a minimum of 5 calibration points prepared? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.7.2.4]  **List Calibration Points:** |  |  | The lowest concentration determines the reporting limit (RL). |
|  | Are the aliphatic and aromatic ranges defined per the method? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.6.6] |  |  | **9.6.6:** EPH Rt windows are defined as beginning 0.1 minutes before the Rt of the beginning marker compound and ending 0.1 minutes after the Rt of the ending marker compound, except for n-C19, which is both a beginning and ending marker compound for two different ranges.  Summary: The C9-C18 Aliphatic Range begins 0.1 minutes before the Rt of n-nonane, and ends 0.1 minutes before the elution of n-nonadecane. The C19-C36 Aliphatic Range begins 0.1 minutes before the elution of n-nonadecane and ends 0.1 minutes after the elution of hexatriacontane. The C11-C22 Aromatic Range begins 0.1 minutes before the elution of naphthalene and ends 0.1 minutes after the elution of benzo(g,h,i)perylene. |
|  | Are retention time windows established over the course of a 72-hour period for each GC and any time a new column is installed? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.6.1 and 9.6.5] |  |  | Make 3 injections of the Aromatic and Aliphatic Hydrocarbon standards over the course of a 72 hour period (injections over shorter time spans may result in windows that are too restrictive). |
|  | Are the retention time windows based on 3 times the standard deviation of the absolute retention times of each component, or set to the default of 0.1 minutes if the standard deviation is close to zero? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.6.3 and 9.6.4]. |  |  | **9.6.3**: The Rt window is defined as plus or minus three times the standard deviation of the absolute Rt for each compound in the Aliphatic and Aromatic Hydrocarbon Standards.  **9.6.4:** In those cases where the standard deviation for a particular standard is close to zero, the default value of 0.1 minutes should be used. |
|  | Is nonane adequately resolved from the solvent peak? [MADEP EPH Rev. 2.1 (Dec 2019) Section 10.2.1.1]. |  |  | The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run. |
|  | Is the calibration factor (CF) method used? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.7.2.3]  If no, skip to Question 26. |  |  |  |
|  | Is the %RSD of the calibration factor ≤ 25% [MADEP EPH Rev. 2.1 (Dec. 2019) Section 9.7.2.13] |  |  | If the %RSD is ≤25% for Target PAH Analytes, the surrogates, and hydrocarbon ranges, linearity can be assumed and the average CF can be used for quantitation in lieu of a calibration curve. |
|  | Is the area for the surrogates subtracted from the corresponding range before calculating the CF? [MADEP EPH Rev. 2.1 (Dec. 2019) Section 9.7.2.8 and 9.7.2.9 and 9.7.2.10] |  |  | Do not include the area of any surrogate standard in calculating a hydrocarbon range CF. |
|  | Are the areas for naphthalene and 2-methylnaphthalene in the aliphatic range standard subtracted from the uncorrected C9-C18 Aliphatic range prior to calculating the CF? [MADEP EPH Rev. 2.1 (Dec. 2019) Section 9.7.2.8] |  |  | Do not include the areas of any surrogate standard or naphthalene and 2-methylnaphthalene in calculating a hydrocarbon range CF. |
|  | Alternatively, is a linear regression model used? [MADEP EPH Rev. 2.1 (Dec. 2019) Section 9.7.2.14]  Skip to Question 31 if not applicable. |  |  | In order for the linear regression model to be used for quantitative purposes, the correlation coefficient (r) must be ≥0.99. |
|  | Is the lowest calibration standard back-calculated using the linear regression, with recoveries of 70-130% for all hydrocarbon ranges? [MADEP EPH Rev. 2.1 (Dec. 2019) Section 9.7.2.14] |  |  | In order for the linear regression model to be used for quantitative purposes, the correlation coefficient (r) must be ≥0.99. In addition, the resulting calibration curve from the linear regression must be verified by recalculating concentrations of the Target PAH Analytes and hydrocarbon ranges in the lowest calibration standard using the final calibration curve. Recoveries must be 70-130%.  Note: The use of zero as a calibration point is not allowed. However, forcing the regression through the origin is not considered a calibration point and is permissible. |
|  | Is the calibration curve verified with a second source calibration standard? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.7.2.16] |  |  | Should be the midpoint. Recovery is 70-130% |
|  | Is the calibration curve verified before sample analysis, after every 20 samples and at the end of the analytical sequence with a primary source mid-level standard? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.7.3.1] |  |  | At a minimum, the calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance. Not required prior to analysis if a second source curve verification was analyzed. |
|  | Is a new calibration performed if the percent difference for any analyte varies by more than ±25% from the expected response for hydrocarbon ranges? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.7.3.5] |  |  | If more than one Target PAH Analyte or hydrocarbon range fails to meet the applicable criterion, the instrument must be recalibrated. Otherwise, sample analysis may proceed. |
|  | **PROCEDURE – Sample Preparation** | **LAB** | **SOP** | **EXPLANATION** |
|  | What extraction methods are used to prepare samples? [MADEP EPH Rev. 2.1 (Dec 2019) Section 9.1]  **Answer:** |  |  | Acceptable extraction methods are listed in the method, Section 9, Table 4 from SW-846. The auditor will review the laboratory’s SOP prior to inspection to ensure that acceptable preparation methods are being followed.  UST Section sent a letter dated 12/6/2005 that includes confirmation that SW-846 3550 is an acceptable preparation method. This letter can be found on the NC WW/GW LCB website:  https://www.deq.nc.gov/about/divisions/water-resources/water-sciences/chemistry-laboratory/laboratory-certification-branch/epa-and-deq-memos-and-guidance |
|  | What volume of sample used for aqueous samples? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1]  **Answer:** |  |  | NOTE: For optimum performance, the sample volumes/weights, solvent volumes, and final extract volumes cited in Sections 9.1.1 and 9.1.2 are recommended. Alternate volumes can be used as long as comparable RLs are achieved. |
|  | Are matrix spiking and surrogate spiking solutions added directly to the sample after the sample is transferred to the separatory funnel or liquid-liquid extractor? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1.1.1] |  |  | For all samples, LMBs, LCSs, LCSDs and matrix spikes add the specified volume of the surrogate spiking solution (see Section 7.5) directly to the separatory funnel. For samples selected for matrix spikes, also add the specified volume of the matrix spiking solution (see Section 7.8). |
|  | Is the pH of the sample checked with wide range pH paper, and if necessary adjusted to pH <2 S.U.? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1] |  |  | Check the pH of the sample with wide-range pH paper. Note the pH in the laboratory notebook. The pH of the sample must be adjusted to pH <2. |
|  | Are aqueous samples extracted according to the appropriate extraction method in SW-846 with methylene chloride? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1.1.2] |  |  | Samples are extracted using methylene chloride and solvent-exchanged into hexane. |
|  | What weight of non-aqueous samples is used?  **Answer:** |  |  | This may vary according to preparation method and/or manufacturer’s directions. |
|  | What extraction method is used for non-aqueous samples? [MADEP EPH Rev. 2.1 (December 2019) Table 5]  **Answer:** |  |  | Note: NC DEQ UST allows sonication as an acceptable extraction method. |
|  | Are samples spiked with surrogate and matrix spike solution (if applicable) prior to extraction? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1.2.1] |  |  | Consult extraction method if following one of those listed in Table 5. |
|  | Are aqueous and non-aqueous sample extracts dried by passing through sodium sulfate prior to solvent exchange? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1.1.9 and 9.1.2.5] |  |  | **9.1.1.9:** Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate or other suitable drying agent.  **9.1.2.5:** Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate or other suitable drying agent. |
| 1. n | Are the sample extracts exchanged with hexane and concentrated? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1] |  |  | Samples are extracted using methylene chloride and solvent-exchanged into hexane. |
|  | What is the final volume?  **Answer:** |  |  |  |
|  | **PROCEDURE – Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
|  | When and how is manual integration performed? [NC WW/GW LCB Manual Integration Policy]  **Answer:** |  |  | 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. |
|  | For hydrocarbon fraction ranges or TPH, is integration from the baseline, and does this integration include complex “hump” mixtures? [MADEP EPH Rev. 2.1 (December 2019) Section 9.8.6] |  |  | In samples, collective peak area integration for the hydrocarbon ranges, or TPH, must be from baseline (i.e., must include the unresolved complex mixture "hump" areas). |
|  | Are samples diluted if any non-target peak exceeds the peak height documented for the highest calibration standard or if a saturated chromatographic peak (flat topped) is encountered? [MADEP EPH Rev. 2.1 (December 2019) Section 9.8.10] |  |  |  |
|  | **PROCEDURE – Sample Fractionation** |  |  |  |
|  | Is the proper elution volume determined for each lot of cartridges using the Fractionation Check Standard? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.3.4 and Appendix 5, Section 5.0]. |  |  | **9.2.3.4:** The Fractionation Check Solution described in Section 7.9 must be used to evaluate each new lot of silica gel /cartridges to re-establish the optimum volume of hexane elutriate. See Appendix 5, Section 5.0 for optimization specifications. |
|  | Is 1 mL of sample extract available for fractionation? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.3.2]. |  |  | Load 1.0 mL of the combined sample extract and fractionation surrogate solution onto the column. |
|  | Are unused fractionation cartridges stored in a desiccator until use? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.2]. |  |  | Unsealed silica gel/cartridges must be stored in a properly maintained desiccator to avoid inadvertent adsorption of ambient moisture. Silica gel that has been exposed to moisture may perform erratically resulting in poor performance manifested by naphthalene/2-methylnaphthalene and fractionation surrogate breakthrough. |
|  | Are the fractionation cartridges rinsed with hexane prior to use? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.3.1] |  |  | Rinsing with methylene chloride is not required. If rinsing with methylene chloride, follow up with a rinse of 60 mL hexane; otherwise, rinse with 30 mL hexane. |
|  | Is elution controlled with the use of a stopcock, so that the hexane level is stopped at a point where the meniscus is just above the frit of the cartridge? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.3.1 and 9.2.3.3] |  |  | **9.2.3.1:** Exposure of the silica gel to air/moisture by fully draining the hexane may adversely affect the performance of the column.  **9.2.3.3:** The use of a stopcock is mandatory. |
|  | What fractionation surrogate(s) are used? [MADEP EPH Rev. 2.1 (December 2019) Section 7.6.2]  **Answer:** |  |  | 2-Bromonaphthalene is recommended, though 2-Fluorobiphenyl is acceptable with a demonstration of performance. |
|  | How much fractionation surrogate is added to the sample extract? [MADEP EPH Rev. 2.1 (December 2019) Section 9.1.1.13] |  |  | 1 mL is stated by the method. Alternatively, 20-50 ng of each surrogate may be added, to not exceed 10 µL additional volume. |
|  | Is the hexane fraction eluted before addition of methylene chloride? [MADEP EPH Rev. 2.1 (December 2019) Section 9.2.3.3] |  |  | Just prior to exposure of the column frit to the air, elute the column with an additional 19 mL of hexane, so that a total of approximately 20 mL of hexane is passed through the column. |
|  | Is the final volume of the fractions 1 mL? [MADEP EPH Rev. 2.1 (December 2019) Section 9.3.1] |  |  | Concentrate each of the extracts to a final volume of 1 mL under a gentle stream of air or nitrogen. |
|  | Are the fractions monitored to ensure that the volume is not reduced below 1 mL? [MADEP EPH Rev. 2.1 (December 2019) Section 9.3.1 Analytical Note] |  |  | Due caution must be exercised during blowdown to avoid losses of the more volatile (C9 through C12) EPH components. The fractionation extract (or any extract) volume should never be reduced below 1 mL in this or any other step to minimize volatilization losses.  Reduction below 1 mL may result in the loss of lighter components, Extract final volume should not be reduced below 1 mL. |
|  | Does the laboratory evaluate breakthrough for the batch LCS and LCSD? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.11] |  |  | If the calculated breakthrough exceeds 5% for either component, the entire batch must be re-fractionated |
|  | What corrective action is taken if the fractionation surrogate recovery exceeds the acceptable limits? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.11] |  |  | If the fractionation surrogate recovery exceeds the acceptable limits, the affected sample(s) must be refractionated. |
|  | **QUALITY ASSURANCE** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are IDLCs on file and updated when personnel are trained or when significant changes are made to the instrument or sample preparation? [MADEP EPH Rev. 2.1 (December 2019) Sections 10.1.1 and 10.5] |  |  | **Section 10.1.1:** Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an Initial Demonstration of Laboratory Capability (IDLC) and an ongoing analysis of prepared QC samples to evaluate and document the quality of data. The laboratory must maintain records to document the quality of the data produced.  **Section 10.5:** The QC procedures described in Appendix 5 and described in SW-846 Method 8000D, Section 9.3 must be conducted, successfully completed and documented as an IDLC, prior to the analysis of any samples by the EPH Method. |
|  | For the Initial Demonstration of Accuracy (IDA) and Initial Demonstration of Precision (IDP), does the laboratory prepare and analyze seven replicate Laboratory Control Samples (LCSs) according to the method? [MADEP EPH Rev. 2.1 December 2019) Appendix 5 Section 3.0] |  |  | **Appendix 5, Section 3.0**: Prepare and analyze seven (7) replicate Laboratory Control Samples (LCSs) fortified at a concentration of 50% of the highest calibration curve standard concentrations. |
|  | Are the LCS replicates prepared at a concentration half of the highest standard? [MADEP EPH Rev. 2.1 (December 2019) Appendix 5, Section 3.0] |  |  | See above |
|  | Is the acceptance criterion for accuracy of the IDLC average % recovery within 40-140%? [MADEP EPH Rev. 2.1 (December 2019) Appendix 5, Section 3.0] |  |  | The value derived for Cmean must be within 40-140% of the true value. |
|  | Is the acceptance criterion for precision of the IDLC ±25%? [MADEP EPH Rev. 2.1 (December 2019) Appendix 5, Section 4.0] |  |  | Using the results calculated from Section 3.0 above, calculate the percent relative standard deviation (%RSD) of the seven (7) replicate LCS analyses for Target PAH Analytes and hydrocarbon ranges, as indicated below. The %RSD must be ≤25 for both aqueous and soil/sediment samples. |
|  | Is a laboratory method blank (LMB) prepared and analyzed with each batch of up to 20 samples? [MADEP EPH Rev. 1.1 (December 2019) Section 10.1.3.2] |  |  |  |
|  | What is the acceptance criterion for the LMB? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.4] [15A NCAC 02 .0805 (a) (7) (H) (i)]  **Answer:** |  |  | **Method:** Peaks must not be detected above the RL within the Rt window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent applicable MCP cleanup standard for soil/sediment samples and 50% of the most stringent applicable MCP cleanup standards for aqueous samples.  **Rules:** The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method.  The laboratory may choose which criterion to use. |
|  | What corrective action is taken if the LMB exceeds the acceptance criterion? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.4]  **Answer:** |  |  | Peaks detected within the Rt window of any analyte or range of interest above the RL must be noted on the data report form. Re-extraction of all associated samples may be warranted. |
|  | Is a Continuing Calibration Standard (CCS) analyzed prior to sample analysis, after every 20 samples (or every 24 hours), and at the end of the run? [MADEP EPH Rev. 2.1 (December 2019) Section 9.7.3.1] |  |  | A Continuing Calibration Standard must be analyzed daily prior to sample analysis, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence.  Note: the CCS is equivalent to a CCV. |
|  | Is the CCS primary source? [MADEP EPH Rev. 2.1 (December 2019) Section 3.8] |  |  | The continuing calibration standard is prepared from the same stock standard solution as initial calibration standards, and is generally one of the mid-level range calibration standard dilutions. |
|  | Is the concentration of the CCS mid-range? [MADEP EPH Rev. 2.1 (December 2019) Section 9.7.3.2] |  |  | The concentration of the EPH Continuing Calibration Standard must be near the midpoint of the calibration curve. |
|  | What is the acceptance criterion of the CCS? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.6.2]  **Answer:** |  |  | For Target PAH Analytes, surrogates, and hydrocarbon ranges, the %D or Percent Drift must be ≤25. If more than one Target PAH Analyte or hydrocarbon range fails to meet this criterion, the instrument must be recalibrated. Otherwise, sample analysis may proceed. For the closing continuing calibration standard (analyzed after every 20 samples, every 24 hours, or at end of analytical sequence), four compounds may exhibit %Ds or Percent Drifts greater than 25% but less than 40%. |
|  | Is the response ratio of C28 to C20 ≥ 0.85 in the CCS? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.10] |  |  | In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory narrative. The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination. |
|  | Is a Laboratory Control Standard (LCS) prepared and analyzed with each analytical batch of up to 20 samples? [MADEP EPH Rev. 2.1 (December 2019) Section 10.1.3.2] |  |  | At a minimum, for each extraction batch (up to 20 samples of similar matrix), an LMB, LCS, and an LCS Duplicate must also be prepared and results analyzed as part of the laboratory’s continuing QC program. |
|  | What is the acceptance criterion for the accuracy of the LCS? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.7]  **Answer:** |  |  | The spike recoveries for the Target PAH Analytes and the hydrocarbon ranges must be between 40-140%. RPD <25 for hydrocarbon ranges, RPD <20 for target PAH waters, <30% for target PAH soils |
|  | What corrective action is taken if the LCS exceeds the acceptance criterion for accuracy? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.7]  **Answer:** |  |  | If the recoveries are low and outside of the acceptance limits, re-extract and reanalyze the LCS and associated samples. If still outside of the acceptance limits, recalibrate.  If the recoveries are high and outside of the acceptance limits and the affected compound was detected in the associated samples, re-extract and reanalyze the LCS and the associated samples. If recoveries are still outside of the acceptance limits, recalibrate.  If the recoveries are high and sample results were nondetect, data can be reported without qualification; however, the high recoveries should be noted in the laboratory narrative. |
|  | Is a Laboratory Control Standard Duplicate (LCSD) prepared and analyzed with each analytical batch of up to 20 samples? [MADEP EPH Rev. 2.1 (December 2019) Section 10.1.3.2] |  |  | See above |
|  | What is the acceptance criterion for the precision of the LCSD? [MADEP EPH Rev. 1.1 (December 2019) Section 10.2.8]  **Answer:** |  |  | The analytical batch precision is determined from the relative percent difference (RPD) of the concentrations (not recoveries) or the LCS/LCSD pair. The RPD for Target PAH Analytes and aliphatic and aromatic hydrocarbon range concentrations must be <25. |
|  | What action is taken if the surrogate recoveries are <40% or >140%? [MADEP EPH Rev. 2.1 (December 2019) Section 10.2.9] |  |  | Check spiking solutions for degradation and, for fractionated samples, examine recoveries of unfractionated samples to see whether recovery may be affected by fractionation. |
|  | Are non-aqueous samples reported on a dry-weight basis? [MADEP EPH Rev. 2.1 (December 2019) Section 9.4.1 and 9.9.3] |  |  | **9.4.1:** Soil and sediment results must be reported on a dry-weight basis.  **9.9.3:**  **Equation 10: Percent Moisture**    **Equation 11: Percent Solids**    **Equation 12: Dry Weight of Sample** |
|  | Do laboratory reports include the required information in Appendix 3? [MADEP EPH Rev. 2.1 (December 2019) Section 1.9] |  |  | Data reports produced using this method must contain all of the information presented in Appendix 3. The format of these reports is left to the discretion of the individual laboratories |
|  | Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 02H .0805 (e) (5)] |  |  | Reported data with quality control failures, improper sample collection, holding time exceedances, or improper preservation shall be qualified as such. |

Additional Comments:

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