NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION

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

Parameter: **Total Petroleum Hydrocarbons (TPH) – Gasoline Range Organics (GRO)**

Method: **SW846 – 8015 C (Aqueous & Non-Aqueous)**

Prep Method: **SW846 – 5030 B**

Prep Method: **SW846 – 5035 B**

**To promote consistency with the use of SW-846 methods and to assure generation of data of known quality, the minimum recommended quality control benchmarks in the methods will be considered the minimum QA/QC requirements.**

Equipment:

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| --- | --- | --- | --- | --- | --- |
|  |  | List GC instrument make and model below: | | List Column(s) and Trap(s) Used Below | |
|  | Analytical balance -- 160-g capacity, capable of measuring to 0.0001 g. |  | |  | |
|  | Microsyringes - 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL. |  | Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device. |  | Two 5-mL glass hypodermic syringes with Luer-Lok tip (other sizes are acceptable depending on sample volume used). |
|  | Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers. |  | Vials - 2-mL, for GC autosampler. |  | Purge-and-trap device. |

Reagents and Standards:

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| --- | --- | --- | --- | --- | --- |
|  | Organic-free reagent water |  | Alkane standard -- A standard containing a homologous series of  n-alkanes (C6-C10 for diesel). |  | Methanol, pesticide quality or equivalent -- Store away from other  solvents. |

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| **PLEASE COMPLETE CHECKLIST IN INDELIBLE INK**  **Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice**  **and in the column labeled SOP to indicate whether it is addressed in the SOP.** | | | | |
|  | **GENERAL** | **LAB** | **SOP** | **EXPLANATION** |
| 1 | Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 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. |
| 2 | 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. |
| 3 | Is there North Carolina data available for review? |  |  | If not, review PT data. |
|  | **PRESERVATION and STORAGE** | **LAB** | **SOP** | **EXPLANATION** |
| 4 | Are aqueous samples collected in 3 x 40­mL vials with PTFE­lined septum caps? [SW-846 Chapter 4, Table 4-1] |  |  |  |
| 5 | Are aqueous samples, with no residual chlorine present, cooled to 0 ­ 6°C and adjusted to pH less than 2 with H2SO4, HCl, or solid NaHSO4? [SW-846 Chapter 4, Table 4-1] |  |  | Except for 2-chloroethyl vinyl ether (no acid preservation, 7 day holding time) |
| 6 | Are aqueous samples, WITH residual chlorine present, collected in a 125­mL container which has been pre­preserved with 4 drops of 10% sodium thiosulfate solution, gently swirled to mix sample and then transferred to 3 x 40­mL vials with PTFE­lined septum caps? [SW-846 Chapter 4, Table 4-1] |  |  |  |
| 7 | Is the chlorine removal documented to have been effective? [NC WW/GW LC Policy] |  |  | Dechlorinating agents used at the time of sampling must be documented to have been effective (either by the sample collector or the receiving laboratory) by verifying a chlorine residual <0.5 mg/L at a neutral pH. |
| 8 | Are non-aqueous (solid) samples collected in a 250-mL wide mouth glass container with PTFE -lined lid? [SW-846 Chapter 4, Table 4-1] |  |  |  |
| 9 | Are aqueous samples collected and stored in capped bottles with minimal headspace so that when cooled, any bubble caused by degassing does not exceed 5 - 6 mm? [SW-846 Method 5030 B, Section 8.1] |  |  | Samples should be stored in capped bottles, with minimum headspace, at 4°C or less in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling the sample should not exceed 5 - 6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at time of sampling. Is there any evidence of leakage? If the sample was improperly sealed, the sample should be discarded. |
| 10 | Are samples shipped and stored at 0 – 6 °C? [SW-846 Chapter 4, Table 4-1] |  |  | Sealed ice packs are not to be used. |
| 11 | Are samples (aqueous and non-aqueous) analyzed within 14 days? [SW-846 Chapter 4, Table 4-1] [SW-846 Method 5030 B, Section 6.2] |  |  | All samples should be analyzed within 14 days of collection. Samples not analyzed within this period must be noted and data are considered minimum values. |
| 12 | What action (s) is taken if sample do not meet preservation and hold time requirements? [15A NCAC 2H .0805 (a) (7) (N)]  **ANSWER:** |  |  | Anytime a laboratory receives samples which do not meet sample collection, holding time, or preservation requirements, the laboratory must notify the sample collector or client and secure another sample if possible. If another sample cannot be secured, the original sample may be analyzed but the results reported must be qualified with the nature of the infraction(s) and the laboratory must notify the State Laboratory about the infraction(s). The notification must include a statement indicating corrective actions taken to prevent the problem for future samples. |
|  | **PROCEDURE – Non-Aqueous Sample Prep by SW-846 5035** | **LAB** | **SOP** | **EXPLANATION** |
| 13 | Are samples with expected low soil concentrations (generally between 0.5 to 200 µg/kg) sampled into a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis? [SW-846 Method 5035, Section 1.2 and 2.1] |  |  | The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.  Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg.  Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to a laboratory or appropriate analysis site. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40EC and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method. |
| 14 | Are non-aqueous samples that contain oily materials that are not soluble in water-miscible solvents prepared according to Method 3585? [SW-846 Method 5035, Section 2.3.2] |  |  | 2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585. |
| 15 | How are non-oily, non-aqueous samples extracted prior to being introduced to the purge and trap system? [SW-846 Method 5035, Section 2.2.1] |  |  | 2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to 5 mL of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling. |
| 16 | Is 5 ml of the extracted sample combined with 5 ml of reagent water in the purge tube and the extraction continued according to Method 5030 B? [SW-846 Method 5035, Section 2.3.1] |  |  | After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method. |
|  | **PROCEDURE – Aqueous Sample Prep by SW-846 5030 B** | **LAB** | **SOP** | **EXPLANATION** |
| 17 | Does the gaseous headspace between the water column and the trap have a total volume of less than 15 mL? [SW-846 Method 5030 B, Section 4.6.1] |  |  | The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. |
| 18 | Is the purge gas introduced no more than 5 mm from the base of the water column? [SW-846 Method 5030 B, Section 4.6.1] |  |  | The purge gas must be introduced no more than 5 mm from the base of the water column. |
| 19 | Does the purge gas pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin? [SW-846 Method 5030 B, Section 4.6.1] |  |  | The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. |
| 20 | What is the trap packed with? [SW-846 Method 5030 B, Section 4.6.5, 4.6.5.5 and 4.6.5.5.1]  **ANSWER:** |  |  | If only compounds boiling above 35°C (GRO corresponds to the range of alkanes from C6 to C10 and a boiling point range of approximately 60°C - 170°C) are to be analyzed, both the silica gel and charcoal can be eliminated, and the polymer increased to fill the entire trap.  See List of Acceptable Trap Packing Materials at the bottom of this Checklist. |
| 21 | Before initial use, is the trap vented to a fume hood (not to the analytical column) and conditioned overnight at 180°C in purge mode or by backflushing with an inert gas flow of at least 20 mL/min. [SW-846 Method 5030 B, Section 4.6.2 and 7.2.1.1] |  |  | Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the hood, not to the analytical column.  Section 7.2.1.1 says to condition the Tenax trap overnight at 180°C (condition other traps at the manufacturers recommended temperature) in the purge mode with an inert gas flow of at least 20 mL/min. |
| 22 | Prior to daily use, is the trap conditioned for 10 min at 180°C with backflushing? [SW-846 Method 5030 B, Section 4.6.2 and 7.2.1.1] |  |  | Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. |
| 23 | If the trap is vented to the analytical column during daily conditioning, is the column run through the temperature program prior to analysis of samples. [SW-846 Method 5030 B, Section 4.6.2 and 7.2.1.1] |  |  | The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples or the column temperature set at 220°C. |
| 24 | Is the desorber capable of rapidly heating the trap to 180°C for desorption? [SW-846 Method 5030 B, Section 4.6.3] |  |  | The desorber must be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. |
|  | **PROCEDURE – Calibration** | **LAB** | **SOP** | **EXPLANATION** |
| 25 | Are a series of at least five calibration standards at different concentrations prepared in organic-free reagent water from the secondary dilution of the stock standards? [SW-846 Method 8015 C, Section 7.9] |  |  | Prepare calibration standards at a minimum of five different concentrations in organic-free reagent water (for purge-and-trap, direct aqueous injection, azeotropic distillation, or vacuum distillation) or in methylene chloride (for solvent injection) from the secondary dilution of the stock standards. |
| 26 | If standards are not stored in sealed vials with zero headspace, are they discarded after 1 hour? [SW-846 Method 8015 C, Section 7.9.7] |  |  | Aqueous standards used for purge-and-trap analyses (Method 5030) are not stable and should be discarded after 1 hr, unless held in sealed vials with zero headspace. If so stored, they may be held for up to 24 hrs. |
| 27 | If standards are stored in sealed vials with zero headspace, are they discarded after 24 hours? [SW-846 Method 8015 C, Section 7.9.7] |  |  |  |
| 28 | Is one of the calibration standards at or below a concentration equivalent to the minimum reporting limit? [SW-846 Method 8015 C, Section 11.3.3.1] [15A NCAC 2H .0805 (a) (7) (H)] |  |  | One of the standards should be at a concentration at or below the lower limit of quantitation necessary for the project (based on the concentration in the final volume described in the preparation method, with no dilutions). |
| 29 | Do the remaining standard concentrations correspond to the expected range of concentrations found in real samples? [SW-846 Method 8015 C, Section 11.3.3.1] [15A NCAC 2H .0805 (a) (7) (H)] |  |  | The concentrations of the other standards should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. |
| 30 | Is each calibration standard introduced into the GC using the same technique used to introduce the actual samples into the GC? [SW-846 Method 8015 C, Section 11.3.3.2] |  |  | Introduce each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. |
| 31 | Are sample concentrations determined using the average calibration factor (a.k.a. Response Factor)? [SW-846 Method 8015 C, Section 11.3.4.1] |  |  | If the percent relative standard deviation (%RSD) of the calibration factors is less than 20% over the working range, then linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve. |
| 32 | If so, how is the calibration factor (CF) calculated? [SW-846 Method 8015 C, Section 11.3.3.2]  **ANSWER:** |  |  | Calculate the calibration factor (CF) for each fuel type as shown below: |
| 33 | If a linear calibration is used, is the correlation coefficient of the calibration curve at least ≥ 0.995? [NC WW/GW LC Policy] |  |  | When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of determination, r2, of 0.99) is required. Ref: NC WW/GW LC Policy. |
| 34 | How is the retention time range for GRO determined? [SW-846 Method 8015 C, Section 11.4.2] [SW-846 Method 8000 D, Section 11.6]  **ANSWER:** |  |  | The retention time range for GRO is defined during initial calibration. Two specific gasoline components are used to establish the range, 2-methylpentane and 1,2,4-trimethylbenzene. Use the procedure described in Method 8000 D Section 11.6 to establish the retention time windows for these two components. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component. |
| 35 | Is the initial calibration and retention time window verified at the beginning of each 12-hr work shift, at a minimum? [SW-846 Method 8015 C, Section 11.5.1] |  |  | The initial calibration and retention times need to be verified at the beginning of each 12-hr work shift, at a minimum. When petroleum hydrocarbons are being analyzed, verification is accomplished by the measurement of the fuel standard and the hydrocarbon retention time standard. Additional analyses of the verification standard(s) throughout a 12-hr shift are strongly recommended, especially for samples that contain visible concentrations of oily material. See Method 8000 for more detailed information on calibration verification. |
| 36 | If calibration factors are used for quantitation, are they verified to be within 20% of those determined in the initial calibration? [SW-846 Method 8000 Section 11.7] |  |  | If the % Difference (when using average RF calibration) or % Drift (for all other types of  calibration) of an analyte is within ±20% of the expected concentration or amount based on the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the calibration curve to quantitate sample results. |
| 37 | If not within 20%, what corrective action is performed? [SW-846 Method 8015 C, Section 11.5.2] |  |  | If the response for any analyte varies from the predicted response by more than ±20%, corrective action must be taken to restore the system, or a new calibration curve must be prepared for that compound. |
| 38 | Are solvent blanks and any method blanks analyzed with calibration verification analyses to confirm that laboratory contamination does not cause false positive results. [SW-846 Method 8015 C, Section 11.5.4] |  |  | Solvent blanks and any method blanks should be run with calibration verification analyses to confirm that laboratory contamination does not cause false positive results. |
|  | **PROCEDURE – Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
| 39 | How are sample areas determined? [SW-846 Method 8015 C, Section 11.11.3]  **ANSWER:** |  |  | For the analysis of GRO, sum the areas of all peaks eluting between 2-methylpentane and 1,2,4-trimethylbenzene. This area is used to calculate the GRO concentration, using the equations in Method 8000. Column bleed subtraction is not generally necessary for GRO analysis. |
| 40 | When, and how, are manual integrations performed? [NC WW/GW LC 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. |
| 41 | Are non-aqueous samples reported on a dry-weight basis? [Division Memo, May 24, 1994] [SW-846 Method 5035 B, Section 6.2.1.6] |  |  | The results for all of the soil sample analyses, performed to meet the requirements of the Division of Environmental Management, must be reported on a dry weight basis.  An unpreserved aliquot must be used for the dry weight determination. |
|  | **QUALITY ASSURANCE** | **LAB** | **SOP** | **EXPLANATION** |
| 42 | Has the laboratory completed an initial demonstration of proficiency by analyzing at least four replicate aliquots of the well-mixed reference samples by the same procedures used to analyze actual samples? [SW-846 Method 8015 C, Section 9.4 and Method 8000 D, Section 9.3.4.2] |  |  | From 8015C - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions.  From 8000D - Prepare and analyze at least four replicate aliquots of the well-mixed reference samples by the same procedures used to analyze actual samples (procedure section for each of the methods). This will include a combination of the sample preparation method and the determinative method. |
| 43 | Is the demonstration of proficiency repeated whenever new staff members are trained or significant changes in instrumentation are made? [SW-846 Method 8015 C, Section 9.4] |  |  | The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made. |
| 44 | How is acceptability of the demonstration of proficiency determined? [SW-846 Method 8000 D Section 9.3.4.3 and 9.3.4.4]  **ANSWER:** |  |  | Calculate the mean recovery and the standard deviation of the recovery for each analyte of interest using the four results.  Compare the mean recovery and the standard deviation of the recovery for each analyte with the corresponding performance data for precision and bias given in the performance table at the end of the determinative method. If the mean recovery and the standard deviation of the recovery for all analytes of interest meet the appropriate acceptance criteria, then the system performance is acceptable and analysis of actual samples can begin. If any individual standard deviation value exceeds the precision limit or any mean recovery value falls outside the range for bias, then the system performance may be unacceptable for that analyte. Once sufficient data points are available, each laboratory is strongly encouraged to develop in-house control limits. |
| 45 | How are laboratory control limits established and how frequently are they updated? [SW-846 Method 8000 D, Section 9.6.5]  **ANSWER:** |  |  | Once established, control limits should be reviewed regularly and updated on a routine basis as established by the laboratory’s quality management plan. |
| 46 | Is a Method Blank (MB) included in every batch? [SW-846 Method 8000 D Section 9.5] |  |  | Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank should be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the laboratory should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. |
| 47 | Is a calibration blank analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group? [15A NCAC 02H .0805 (a) (7) (H)] |  |  | A calibration blank and calibration verification standard shall be analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group |
| 48 | Are all blank values ≤ ½ the reporting limit? [15A NCAC 02H .0805 (a) (7) (H) (i)] |  |  | For analyses requiring a calibration curve, the concentration of reagent, method and calibration blanks must not exceed 50% of the reporting limit or as otherwise specified by the reference method. |
| 49 | Is a second-source standard analyzed to verify the calibration? [15A NCAC 02H .0805 (a) (7) (H) (ii)] |  |  | When a standard curve is manually prepared (as opposed to a factory-set calibration), it is required to analyze one known standard in addition to calibration standards each day samples are analyzed to document accuracy. This standard must be prepared from materials obtained from a source independent from the one used for preparing the calibration standards (often referred to as a second source standard or external reference standard). |
| 50 | What is the acceptance criterion for the second-source standard? [15A NCAC 02H .0805 (a) (7) (A)]  **ANSWER:** |  |  | Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses. Each laboratory shall calculate and document the precision and accuracy of all quality control analyses with each sample set. When the method of choice specifies performance acceptance criteria for precision and accuracy, and the laboratory chooses to develop laboratory-specific limits, the laboratory-specific limits shall not be less stringent than the criteria stated in the approved method. |
| 51 | Does the laboratory analyze either a MS and one duplicate un-spiked sample or a MS/MSD with each extraction batch of 20 or fewer samples? [15A NCAC 02H .0805 (a) (7) (D)] [SW-846 Method 8015 C, Section 9.6.1] |  |  | Rule: Unless the referenced method states a greater frequency or the parameter is not amenable to spiking, laboratories shall spike five percent of samples monthly. Laboratories analyzing fewer than 20 samples per month shall analyze one Matrix Spike during each month that samples are analyzed.  SW846 8015 C - Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair. Consult Method 8000 for information on developing acceptance criteria for the MS/MSD. |
| 52 | What are the laboratory’s established control limits for MS/MSD results? [15A NCAC 02H .0805 (a) (7) (A)] [SW-846 Method 8000 D, Section 9.4.4]  **ANSWER:** |  |  | Ideally, the acceptance criteria for MS/MSD recovery and/or duplicate relative % difference will be established for the field samples through the DQOs contained in a written QAPP. These criteria should be established with consideration given to performance data provided in the reference method and/or by the laboratory in order to avoid overly conservative expectations. In the absence of site- or project-specific acceptance criteria for matrix spike and duplicate QC samples, these criteria should be based on in-house performance data generated by the laboratory or on the performance data in the reference method. |
| 53 | What action is taken if MS results are out of control? [15A NCAC 02H .0805 (a) (7) (B)]  **ANSWER:** |  |  | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. 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. |
| 54 | Does the volume of the spike solution used in the MS constitute ≤ 5 % of the total MS volume? [NC WW/GW LC Policy] |  |  | The volume of spike solution used in MS preparation must in all cases be ≤ 5 % of the total MS volume. |
| 55 | If the volume of the spike solution used in the MS constitutes ≤ 10% but > 1% of the total MS volume, then is the sample volume adjusted by calculation? [NC WW/GW LC Policy] |  |  | It is preferable that the spike solution constitutes ≤ 1% of the total MS volume so that the MS can be considered a whole volume sample with no adjustment (i.e., volume correction) by calculation necessary. If the spike solution volume constitutes >1% of the total sample volume, the sample concentration or spike concentration must be adjusted by calculation. |
| 56 | What are the control limits for duplicate RPD results? [15A NCAC 02H .0805 (a) (7) (A)] [SW-846 Method 8000 D, Section 9.4.4]  **ANSWER:** |  |  | Ideally, the acceptance criteria for MS/MSD recovery and/or duplicate relative % difference will be established for the field samples through the DQOs contained in a written QAPP. These criteria should be established with consideration given to performance data provided in the reference method and/or by the laboratory in order to avoid overly conservative expectations. In the absence of site- or project-specific acceptance criteria for matrix spike and duplicate QC samples, these criteria should be based on in-house performance data generated by the laboratory or on the performance data in the reference method. |
| 57 | Does the laboratory analyze at least one LCS with each batch of 20 or fewer samples? [SW-846 Method 8015 C, Section 9.6.2] |  |  | A Laboratory Control Sample (LCS) should be included with each analytical batch. |
| 58 | Does the LCS consist of an aliquot of clean matrix similar to the sample matrix and of the same weight or volume? [SW-846 Method 8015 C, Section 9.6.2] |  |  | The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. |
| 59 | Is the LCS spiked with the same analytes at the same concentrations as the matrix spike? [SW-846 Method 8015 C, Section 9.6.2] |  |  | The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS. |
| 60 | What is the acceptance criterion for the LCS recovery? [SW-846 Method 8000 D, Section 9.4.4]  **ANSWER:** |  |  | Many methods may not contain recommended acceptance criteria for LCS results. The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed (Sec. 9.6). Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix. |
| 61 | Is a calibration verification standard analyzed at the beginning and end of the analytical sequence? [SW-846 Method 8015 C, Section 11.6.1] |  |  | Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with calibration verification followed by sample extract analyses. Additional analyses of the verification standard(s) throughout a 12-hr shift are strongly recommended, especially for samples that contain visible concentrations of oily material. A verification standard is also necessary at the end of a set (unless internal standard calibration is used). The sequence ends when the set of samples has been injected or when retention time and/or % difference QC criteria are exceeded. |
| 62 | Is the calibration verification standard for any analyte within ±20% of the response obtained during the initial calibration? [SW-846 Method 8015 C, Section 11.5.2]  **ANSWER:** |  |  | Calculate the % difference as detailed in Method 8000. If the response for any analyte is within ±20% of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the mean  CF or RF values from the initial calibration to quantitate sample results. |
| 63 | What corrective action is taken if the calibration verification standard recovery is outside the acceptance criterion? [SW-846 Method 8015 C, Section 11.5.2]  **ANSWER:** |  |  | If the response for any analyte varies from the predicted response by more than ±20%, corrective action must be taken to restore the system or a new calibration curve must be prepared for that compound. |
| 64 | Is each sample, standard, and blank spiked with one or two surrogate compounds which are not affected by method interferences? [SW-846 Method 8015 C, Section 7.11] |  |  | Whenever possible, the analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix, by spiking each sample, standard, and blank with one or two surrogate compounds which are not affected by method interferences. |
| 65 | What are the surrogate compounds? [SW-846 Method 8015 C, Section 7.11]  **ANSWER:** |  |  | Whenever possible, the analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each 8015C sample matrix, by spiking each sample, standard, and blank with one or two surrogate compounds which are not affected by method interferences. |
| 66 | What is the acceptance criterion for the surrogate compound? [SW-846 Method 8015 C, Section 11.6.1]  **ANSWER:** |  |  | From in-house performance criteria and control charting.  See [SW-846 Method 8000 D, Section 9.6] |
| 67 | What corrective action is taken if the calibration verification standard recovery is outside the acceptance criterion? [SW-846 Method 8015 C, Section 11.6.1]  **ANSWER:** |  |  | All sample analyses performed using external standard calibration need to be bracketed with acceptable data quality analyses (e.g., calibration and retention time criteria). Therefore, all samples that fall between the standard that failed to meet the acceptance criteria and the preceding standard that met the acceptance criteria need to be reanalyzed. |

Additional Comments:

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Trap Packing Materials:

2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

Silica gel - 35/60 mesh, Davison, grade 15 or equivalent

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Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or

equivalent, by crushing through 26 mesh screen