BNA Group Data Review/Authorization Checklist

The supervisor (or designate) must affix his/her signature and the date of review to all data packs. The signature indicates that the appropriate data review checks outlined below was performed on the date indicated.

1. Sample Preparation: The primary responsible party is the group supervisor, but the supervisor (or designate) must also review the preparation logs to provide a secondary check.

1.1 Review sample preparation records and verify that the correct preparation method was used. If not, re-prepare all affected samples using the appropriate preparation method. Verify that all preparation information is documented correctly.

1.2 Verify that the appropriate surrogate spike, matrix spike and internal standard solutions and spike levels were used. Check the expiration date of all working solutions. **If any of the working solutions have expired, the samples must be re-prepared by using new working solutions or the data must be flagged with the qualifier "J" and a comment must be made in the report.**

1.3 Verify that the preparation holding times were met. **If not, flag the data with the qualifier "Q" for each analyte reported beyond the holding time.**

1.4 Note any observations made by the sample preparation group such as unusual odors, colors, or reactions during sample extraction. Consider these observations when making any determinations, and evaluate potential ramifications to the final results.

1.5 Review the job folder for special instructions or conditions noted during sample receipt and log-in. Consider special instructions when making any determinations. Consult the section administrator or customer if the instructions are not legible or well understood.

1.6 Ensure that the appropriate QC samples were analyzed for the analysis batch including extraction blank, calibration checks, laboratory fortified blanks, and duplicate spike samples. Refer to the BNA standard operating procedures and the corresponding EPA methods for details on the analysis requirements. **If there are problems with the QC, re-prepare the samples. If the samples are expired, continue with the re-analysis, but flag the data with the "Q" qualifier.**

1.7 Check and review if the following things are done properly. The % solids calculations and results entry, all the preparation volumes extracted and made are correct, if more than one bottle available, then the bottle number used in the preparation, lot numbers of reagents and solvents used, and bath temperature for extracts concentration.

2. Instrumental Analysis: The primary responsible party is the analyst. The supervisor (or designate) must provide a duplicate check of the sample analysis. The following items must be checked for every analytical batch of samples.

2.1 Hardcopies of the analysis results sheet, run sequence log, preparation log sheets, sample dilutions record, QC statistical result (for Laboratory Fortified Blanks and matrix spikes) sheet, sample and standards Quantitation data and chromatograms must be compiled into a sample data folder. Also include in the folder, hardcopies of the tune check results, calibration curve standards sheet, Quantitation data for all of the calibration standards (if the curve is built on the same day), calibration check standards, extraction blanks, laboratory fortified blanks and matrix spikes.

2.2 All computer file names, folder file names, as well as marks or comments on the data hardcopies must conform to the conventions used in the laboratory. These conventions should be stipulated in the method SOPs.

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2.3 Check the tune of the mass spectrometer (with the tuning compound DFTPP every 12 hours) and ensure that the mass spectrometer meets all of the tuning requirements. If not, re-analyze the samples analyzed during the time period when the instrument was not tuning properly. If any of the samples have exceeded the analysis holding time, report all positives with the qualifier "J". Include a comment in the report explaining the use of this qualifier.

2.4 Ensure that a valid instrument calibration curve is on file and complies with method specifications for all parameters analyzed. If not, re-analyze the sample batch after establishing a valid instrument calibration curve.

2.5 Ensure that the calibration check standards pass for all parameters reported. The calibration check standards must be analyzed every 12 hours and must comply with method specifications. Verify the MDLs are met for the failing CCVs, otherwise, analyze a check standard at lower levels to meet this requirement. If not, re-analyze the affected samples with valid calibration check standards.

2.6 Check the accuracy and precision for surrogates, laboratory fortified blanks, and matrix spikes. Ensure that all values are within the current QC acceptance ranges. A comment must be included in the report for any compounds outside of the acceptance ranges and the data affected must be qualified with "J" accordingly.

2.7 Check the internal standard area counts and confirm that they comply with method specifications. Ensure that any retention time shift is within method specifications. If not, re-analyze the affected samples.

2.8 Check the results of all field duplicates for reproducibility. If the degree of replication is not within the acceptance limits, a comment must be made in the report.

2.9 Check the laboratory extraction blanks, trip blanks, field blanks and equipment blanks for evidence of contamination. If laboratory contamination is suspected, determine whether the contamination occurred during sample preparation or analysis. Reanalyze the associated samples if the contamination is from the laboratory and can be eliminated. If an analyte is detected in both sample and laboratory extraction blank, and 1) the concentration detected in blank is lower than one half of current MDL, report the sample based on current MDL; 2) if the concentration detected in blank is higher than one half of current MDL, elevate the MDL to a level equal to twice of the detected concentration in blank, and report the results based on new MDL.

2.10 For all positive analyte results, check the integration of the peak to ensure that the baseline was drawn appropriately. For certain types of matrix interference, it may be necessary to use background subtraction to alleviate the spectral interference from the mass spectrum of the target analyte. Ensure that this technique was utilized appropriately. If an analyte cannot be accurately quantified due to matrix interference, the result for the affected analyte must be flagged as an estimate (i.e., append the qualifier "J" to the final result). Include a comment in report explaining the use of this qualifier.

2.11 Check for carryover contamination by checking all samples analyzed after a highly contaminated sample. Rerun all samples with suspected or potential carryover contamination.

2.12 Two criteria must be met to verify identification of an analyte: 1) the analyte in the sample eluted at the same relative retention time as the analyte in standard and 2) correspondence of the mass spectrum for the analyte. ALWAYS manually assess each chromatogram and the mass spectra for each analyte.

2.13 Ensure that the proper dilution levels were used for sample analysis. Dilute and re-analyzed any samples which contain analytes at concentrations higher than the calibration range. If the sample extract has exceeded its holding time, report the result and append the qualifier "J". If the MDL and PQL are elevated after sample dilution, a comment must be made in the report.

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2.14 Review all sample calculations to ensure dilution factors and dry weight corrections (if applicable) were properly applied. Make corrections as needed.

2.15 If a sample with QC or process errors is not (or cannot be) re-analyzed, then report the analytes detected with the qualifier "J" and add a comment to the report explaining the use of the qualifier.

2.16 Closely evaluate the Total Ion Chromatogram and associated single ion chromatograms for all reported analytes. Do not rely on the instrument-generated reports without reviewing the chromatograms and mass spectra.

2.17 For spectral library search, exercise caution when utilizing the search results to report tentatively identified compounds, and make sure to examine the returned spectra against the sample spectra. Background subtraction may be needed. Remember: Do not rely on the library search program over your own scientific judgment. Examine the spectra; you may have additional information about the sample that will lead you to a different conclusion than what the search program concluded.

2.18 Ensure appropriate qualifiers are used. Refer to the *Department of Environmental Protection: Standard Operating Procedures for Laboratory Operations and Sample Collection Activities* (DEP QA-001/92) for details on the use and the reporting priority of these qualifiers.

2.19 Evaluate analytical results against any field information and make certain that the transcribed information is correct and consistent with the laboratory findings. The final results should not be posted until all inconsistencies are accounted for. If a library search for non-target analytes was performed, then review the guidelines in the BNA SOP (SV-006). Ensure that these guidelines were properly followed.

2.20 Check field preservation records (e.g., log-in details and sample prep records) for any indication that the samples were improperly preserved at collection. If samples were received improperly preserved, all data must be flagged with a “Y”.

3. Uploaded LIMS Data: The primary responsible parties are the analyst and supervisor. The Environmental Manager of the organic section provides a secondary check for the items noted by an asterisk.

3.1 When samples with the same field identification are analyzed by both BNA and VOC (or GC) groups, then a check for compounds evaluated by both groups must be made and confirmed when detected. *

3.2 Compare effluent samples with background samples, the effluent concentrations are usually higher than background concentrations. *

3.3 Compare results with any information related to the site history. *

3.4 Confirm the sample preparation dates and the analysis dates. The collection time must come before sample preparation, which must come before analysis.*

3.5 Make sure the final analytical results are reported with the correct number of significant figures.*

3.6 Ensure appropriate qualifiers are used and comments are entered when needed.*

3.7 Verify that sample identifier information in the LIMS matches that in the reported data package.

3.8 Evaluate the analytical results against any field information. If anything looks suspicious, further research is necessary before posting results. *

3.9 In data review/calculations do calculations and check that surrogate, method blanks and CCV data were entered correctly for each analyte.

***For additional details, refer to BNA SOPs (SV-006, -007, -009, -010 and 013) and LB-027.**

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4. Authorization of Samples: The primary responsible party is the BNA group supervisor or designate.

4.1 After completion of the above data review process, the supervisor (or designate) authorizes samples and affixes his/her signature and date of review to the data package. This signature and date indicate that the above checks were performed on the date indicated. If any check results in a corrective action(s), the supervisor (or designate) must indicate the corrective action(s) performed and affix his/her signature and date, indicating that the specified corrective action was initiated and completed satisfactorily.

5. REFERENCES:

5.1 DEP QA-001/92. Department of Environmental Protection: Standard Operating Procedures for Laboratory Operations and Sample Collection Activities

5.2 DEP SOP SV-006. Analysis of Base/Neutral/Acid Extractables by Gas Chromatography/Mass Spectrometry

5.3 DEP SOP SV-007. Analysis of Petroleum-Range Organics by GC-FID (FL-PRO Method) and Analysis of Extractable Petroleum Hydrocarbons (EPH) by GC-FID (The Massachusetts Method)

5.4 DEP SOP SV-009. BNA Standard Preparation.

5.5 DEP SOP SV-010. Quality Assurance/Quality Control in the Semi-Volatile Organics Laboratory (BNA-GC/MS).

5.5 DEP SOP SV-013. Analysis of Extractable Polycyclic Aromatic Hydrocarbons (PAHs) In Polyurethane Foam (PUF) Traps by Gas Chromatography/Mass Spectrometry

5.6 DEP SOP LB-027. Standard Operating Procedure for Reporting Qualified Data.

Appendix of Changes

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