Data Review/Authorization Checklist

Work Group(s): GC Pesticides and associated Organic Sample Preparation

DEPARTMENT OF ENVIRONMENTAL PROTECTION
CHEMISTRY SECTION
TALLAHASSEE FLORIDA

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The supervisor (or designate) must affix their signature and date of review to all data packs. The signature of the supervisor indicates that the appropriate data review checks indicated below were performed on the date indicated.

I. **Sample Preparation** (primary responsible party: GC group supervisor, but the analyst involved in the data processing must also review to provide a secondary check)

- Review sample preparation records and verify that the correct preparation was used for the samples. If not, re-prepare using correct method.

- Verify appropriate matrix spike and surrogate solutions were used (were correct levels/solutions used? check expiration date of solutions). **If solutions have expired, samples must be re-prepared or data must be flagged appropriately with a "J".**

- Verify preparation holding times were met - **if not, flag data appropriately (i.e., LIMS will append "Q" qualifier to all reported data)**.

- Note any observations made by the sample prep team (e.g., unusual odors, colors, reaction during prep?); consider these in making any determinations.

- Ensure that appropriate QC samples were prepared/analyzed for the sample batch (e.g., blank, LFBs and duplicate matrix spikes per batch of 20, etc.)- **if not, re-prepare samples if there is sufficient time remaining prior to sample expiration. If there is insufficient time prior to expiration, continue with analysis but flag data with the "J" qualifier.**

II. **Instrumental Analysis** (primary responsible party: analyst. The supervisor of the work group, or another senior analyst provides a duplicate check of the same items).

- Ensure that a valid initial calibration for all parameters analyzed is on file and complies with method specifications- **If not, re-analyze sample batch after generating a valid initial calibration curve. If samples have already passed the analysis holding time of 40 days, report data flagged with the qualifier "J". Include comment in final report explaining the use of this qualifier.**

- Ensure that passing continuing calibration verification (CCV) standards for all parameters reported has been analyzed every analysis run sequence and

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complies with method specifications-. If CCVs do not meet method requirements but the associated analytical results are below the detection limit or below the quantifiable threshold (PQL) and no systematic failure is observed (i.e., the failure is deduced to be due to a statistical outlier in a multi-component analysis where results from other CCV components and LFB results do not indicate a systematic bias in the analysis or detector response), an appropriate test comment and result qualifier (J) shall be provided to indicate the CCVs are outside control limits. However, when a systematic or system failure is detected or the affected analytical data are above the quantifiable threshold, the analysis shall be re-run if possible. If re-analysis is not an option, the analytical results shall be qualified as estimates.

- Check if the tune check report for the GC/MS/MS analysis was performed and whether it passed.

- Check surrogates, lab fortified blanks and matrix spikes for recoveries/precision (if applicable). Ensure that all are within the current QC acceptance range. If they are outside acceptance ranges, a comment must be included in the report and data is qualified accordingly **(i.e., append "J" qualifier to all reported positive hits. Include comment in final report explaining the use of this qualifier). See also**[7] DEP SOP LB-027. Standard Operating Procedure for Reporting Qualified Data.**.**

- Check internal standard (ISTD) area counts- confirm that they comply with method specifications. **If ISTD response is outside of acceptance limits, re-analyze sample to determine if the sample injection was faulty. If it is determined that the injection was acceptable and that the ISTD response failed due to the inappropriate addition of the standard to the sample, use the external calibration mode and include comment. If the method was not validated for external calibration mode flag the positive hits with qualifier "J" and include comment.**

- Ensure that any retention time drifts are within method specifications**; if not, re-analyze sample batch after performing any necessary instrument maintenance (e.g., check for leaks, check injector function). If samples have already passed the analysis holding time of 40 days, report data flagged with the qualifier "N". Include comment in final report explaining the use of this qualifier.**

- Check Endrin and DDT-p,p' breakdown reports for GC-ECD analysis. Ensure that all are within the method specifications. No analysis run sequence should proceed if the initial Endrin/DDT-p,p' decomposition exceed 15%. Clean the instrument and re-run if needed. **If samples are expired, flag the positive hits for these two parameters and their decomposition products with qualifier "J" and include the appropriate comment.**

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- Check tailing factor /DDT-p,p' decomposition reports for for GC/MS/MS and GC/MS SIM analysis. Tailing factor for Pentachlorophenol may not be above 2.0 and the DDT-p,p' degradation cannot exceed 20%. **Clean the instrument and rerun the samples if failures observed. If samples are expired, flag the positive hits with qualifier "J" and include the appropriate comment**

- Check field duplicates/replicates: is the degree of precision within accepted margins? **-If not, re-analyze duplicates/replicates samples. Include the appropriate comment in the final report if precision is outside accepted limits.**

- Check lab, trip, field and/or equipment blanks for evidence of laboratory or field contamination. If lab contamination is suspected, delineate whether the contamination occurred at prep or analysis- it may be possible to re-analyze or re-prep batch, if samples have not exceeded their holding time. **If a sample batch must be reported that has an associated blank contamination,** the affected samples' results will be reported with a "V" qualifier up to ten times the amount found in the blank.

- For positive hits, check the integration of the peak to ensure that the baseline was drawn appropriately. Use manual integration to correct the baseline if needed. Ensure that this technique was utilized appropriately based on lab accepted protocol (See Lab SOP CM-018-1.). **If a parameter cannot be accurately quantified due to matrix interferences, the result for the affected parameter must be flagged as an estimate (i.e., append the qualifier "J" to the final result). Include comment in final report explaining the use of this qualifier.**

- Two criteria must be satisfied to verify identification of a compound by GC_ECD or GC-NPD: 1.) elution of the sample component at the same GC retention time window as the standard component: and 2.) agreement between the amounts found in the primary and confirmatory columns.

- Two criteria must be satisfied to verify identification of a compound by GC/MS/MS or GC/MS SIM or GC/MS NCI: 1.) elution of the sample component at the same GC retention time window as the standard component: and 2.) the relative ratios of qualifier to quantifier ions has to pass the method criteria.

  If any samples contain parameters at concentrations higher than the calibration range, ensure that they were properly diluted and re-analyzed. If sample was not diluted within the calibration range, prepare the appropriate dilution and re-analyze. **If the sample extract has exceeded its holding time, report**

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result but append the qualifier "J".

- Review all sample calculations- ensure any dilution factors and dry weight corrections were properly applied. **Make corrections as needed**.

- Ensure appropriate qualifiers are used. Refer to the DEP SOP LB-027. Standard Operating Procedure for Reporting Qualified Data. for details on the use and the reporting priority of these qualifiers.

- Check all information in the Analysis Summary Report Header (SOP number, Instrument ID, Column ID) to be right. **Make correction as needed. Also verify that all run sequences, including reruns, are included in the analysis file.**

- Evaluate data against any field information. Do the results make sense, given the field information? For example, does the trip blank contain several hits while an effluent sample does not? If anything looks suspicious, further research is necessary before posting results. Ensure that these guidelines were properly followed.

- Check NCR report in the Laboratory Information Management System (LIMS) Data Review/Authorize for field preservation records (e.g., log-in details and sample prep records) for any indication that samples were improperly preserved at collection. **If samples were received improperly preserved (i.e., for 608/8081/8082 sample held at temperatures < 4 deg. C), all data must be flagged with a "Y".**

III. **Uploaded LIMS Data** (primary responsible party: analyst, work group supervisor. The Environmental Administrator of the work group provides a secondary check for the items noted by *****)

- If samples were also analyzed by the BNA group, check if any positive hits in the GC analysis are confirmed in the BNA analysis *****

- Compare effluents with backgrounds - usually effluent concentrations will be higher than background concentrations *****

- Compare results with any information known about the site, i.e., are the reported hits consistent with what is known about the site history? *****

- Confirm sample prep dates and analysis dates are correct (e.g., does prep come BEFORE analysis?) *****

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- Make sure the result has the correct amount of significant figures. *****
- Ensure appropriate qualifiers are used and that comments are entered when needed (i.e. confirmation of blank contamination etc.). *****
- Verify that sample identifier information in the LIMS matches that in the reported data package.
- Evaluate data against any field information. Do the results make sense, given the field information? For example, does the trip blank contain several hits while an effluent sample does not? If anything looks suspicious, further research is necessary before posting results. *****

For additional details, refer to internal GC SOPs.

IV. Authorization of Data Review (primary responsible party: work group supervisor or designate)

After completion of the above data review process, the supervisor (or designate) must affix their 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 their signature and date, indicating that the specified corrective action was initiated and completed satisfactorily.

Appendix of Changes

07/20/2015 Requirements specific to GC/MS/MS analysis were added.

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