North Carolina Division of Air Quality

Quality Assurance Project Plan

2.52 Aldehydes by TO-11a

Section 4.1

Raleigh Central Office Responsibilities

Standard Operating Procedure for Performing a QA Review of an Aldehyde Data Batch

Submitted by:
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Approval Sign-Off Sheet

I certify that I have read and approve of the contents of this revision of the SOP with an effective date of 5/30/15.

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Addendum  Aldehyde Data QA Checklist
2.52.4.1 Standard Operating Procedure for Performing a QA Review of an Aldehyde Data Batch

2.52.4.1.1 Purpose: The purpose of this SOP is to describe the steps needed to review the Aldehyde analysis data from the UHPLC laboratory before it is transferred to the master Aldehyde spreadsheet.

2.52.4.1.2 Equipment Description: PC connected to the network drive group on 'air.ncdenr.net\dfs' – usually mapped to drive letter P.

2.52.4.1.3 Directions for reviewing the Aldehyde data from the UHPLC laboratory:

2.52.4.1.3.1 Open the file folder and check that all the documents on the list below are there. The check list (Aldehyde Data QA Checklist) at the end of this SOP may be used to document this step.
- QA/QC spreadsheet print out and file(s) e-mailed or placed on the network drive by the Aldehyde Laboratory Analyst (see Figure 1)
- Raw Data Conversion sheet
- Calibration Curve Reports
- Sequence printout listing all the samples analyzed
- Individual aldehyde sample chromatograms in chronological order
- Solvent blank(s)
- Initial calibration check(s)
- Cartridge blank
- Field blank(s)
- Replicate injection
- Final calibration check(s)

2.52.4.1.3.2 Check the dates (the “Injection Date/Time:” on the aldehyde sample chromatograms) and make sure that all the reports are in chronological order. If there are any missing time periods, check the Sequence printout and ask the UHPLC Laboratory Analyst. Each aldehyde sample chromatogram should take about 30-50 minutes and be on 1 page.

2.52.4.1.3.3 On the QA/QC spreadsheet (see Figure 1) note all the flagged compounds. If a compound of interest like formaldehyde or acetaldehyde is flagged, check the details of that flag and void it if possible. For example, if formaldehyde has a flag 3c and in checking the calibration data the $r^2$ value is 0.9985 and/or the %RSD is 20.1%, void the 3c flag (delete the flag from the QA/QC spreadsheet).

2.52.4.1.3.4 Match the initial and final calibration check values on the QA/QC spreadsheet with the actual Aldehyde Sample Chromatograms and write the date and time of each run on the Aldehyde Data QA Checklist. The first check should be before any samples are run and the final check should be after all samples have been run. These Aldehyde Sample Chromatograms will be labeled at the top of the page as “HL QA”
or “LL QA” and in the “Injection Type:” line as “unknown.” There should be at least one such Aldehyde Sample Chromatograms at the beginning of the batch and one at the end. If there are many flag 4’s or a flag 4 for a compound of interest like formaldehyde or acetaldehyde, see if using a different calibration check would improve the results so that one or more flags can be removed. Also, try comparing the initial and final checks with each other; if that produces a good result, consider removing a flag 4.

2.52.4.1.3.5 Following (or just before) the initial check standards there should be at least one solvent blank chromatogram. There may be other blank samples run at the end or anywhere in the batch. Check the cartridge and field blanks for agreement with the Flag 2 criteria.

2.52.4.1.3.6 Review the replicate sample analyses in the columns to the left on the lowest block on the spreadsheet. Correct the flag 5’s if needed.

2.52.4.1.3.7 Now review each Aldehyde Sample Chromatogram.

- Make sure there is a correct Sample ID at the top of the page. This will be matched up to the “Aldehyde Sample Log No.” in the master spreadsheet.
- Does the UHPLC graph look OK? Are there strange peaks or lumps? If so, mark it as a bad UHPLC run.
- Mark out with a single line the data for any compound that has a flag.
- Circle any value \( \geq 0.5 \) \( \mu \)g/mL that has not been flagged.
- Mark with an arrow any value \( \geq 1 \) \( \mu \)g/mL that has not been flagged.
- Compare collocated data with primary data and mark any significant differences.
Do the concentrations seem reasonable for ambient air at that site; this could indicate that the IDs have been inadvertently switched.

- Compare the patterns. If two Sample Reports appear very similar, they may have been the same sample but are mislabeled.

2.52.4.1.3.8 Send any questions that arose during the review to the UHPLC Laboratory Analyst. His or her name or initials should be in the “Created by:” space at the top of each Aldehyde Sample Chromatogram.

2.52.4.1.3.9 When all questions have been resolved, begin recording QA data in the appropriate spreadsheets, for example RepeatsCV2014.xls, DuplicatesCV2014.xls, and CollocatedCV2014.xls. These spreadsheets are located on the P:\ drive in the P:\Toxics\Urban Air Toxics\Aldehydes\ folder. Record formaldehyde and acrolein precision data. Other compounds may be added.

<table>
<thead>
<tr>
<th>Type of Data (at least formaldehyde and acrolein)</th>
<th>File to record the data in (where xxxx = year)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Data from collocated and primary samplers run on the same day at the same site (the collocated sample ID will match the primary sample but end in “D”)</td>
<td>CollocatedCVxxxx.xls</td>
</tr>
<tr>
<td>Data from two analyses of the same sample in the same batch (the second analysis should have an ID ending with “S”)</td>
<td>DuplicatesCVxxxx.xls</td>
</tr>
<tr>
<td>Data from samples that were analyzed in a previous batch (the ID will end in “R”)</td>
<td>RepeatsCVxxxx.xls</td>
</tr>
<tr>
<td>Each year create a new set of spreadsheets by copying last year’s and deleting only the data.</td>
<td></td>
</tr>
</tbody>
</table>

2.52.4.1.3.10 This completes the review. Initial and date the QA Checklist and QA/QC Spreadsheet at the bottom and give it to the LAB Supervisor for review.

2.52.4.1.3.11 When the LAB Supervisor has approved the analytical run and returns the folder, notify the UHPLC Laboratory Analyst, update the tracking chart located on the network drive and transfer the data to the master spreadsheet. (See SOP for Transferring Aldehyde Data to Master Spreadsheet).
Aldehyde Data QA Checklist

For batch run on _____________________________________

Before beginning the Aldehyde data review verify that all the documents are present in the data packet:

___ QA/QC spreadsheet print out, see Figure 1 in the “SOP for Performing a QA Review of a Aldehyde Data Run” (and electronic copy of the spreadsheet file placed on the network by the Aldehyde Laboratory Analyst)

___ Raw Data Conversion Sheet

___ Calibration Curves Report

___ Sequence Overview Printout (may be part of the QA/QC spreadsheet file)

___ Aldehyde Sample Reports matching the Sequence Overview in chronological order:

___ Solvent blank(s): ______________________________________________________

___ Initial calibration check(s) ____________________________________________

___ Cartridge blank: ______________________________________________________

___ Field blank(s): _______________________________________________________

___ Replicate injection(s): _______________________________________________

___ Final calibration check(s) _____________________________________________

Update Tracking Chart ________ _______

Date of Review: __________________________   Reviewer:  ______________

Comments: