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

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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.

John Holland 7/27/15
John Holland, UAT Quality Assurance Coordinator Date

Joette Steger 8/12/2015
Joette Steger, PPB Supervisor Date

Corey A Mosher for Jim Bowyer 8/12/15
Jim Bowyer, LAB Supervisor Date

Jim Bowyer for Donald Redmond 8/12/15
Donald D. Redmond, Jr., Ambient Monitoring Section Chief Date

Table of Contents

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:

2.25.4.1.3.1 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)

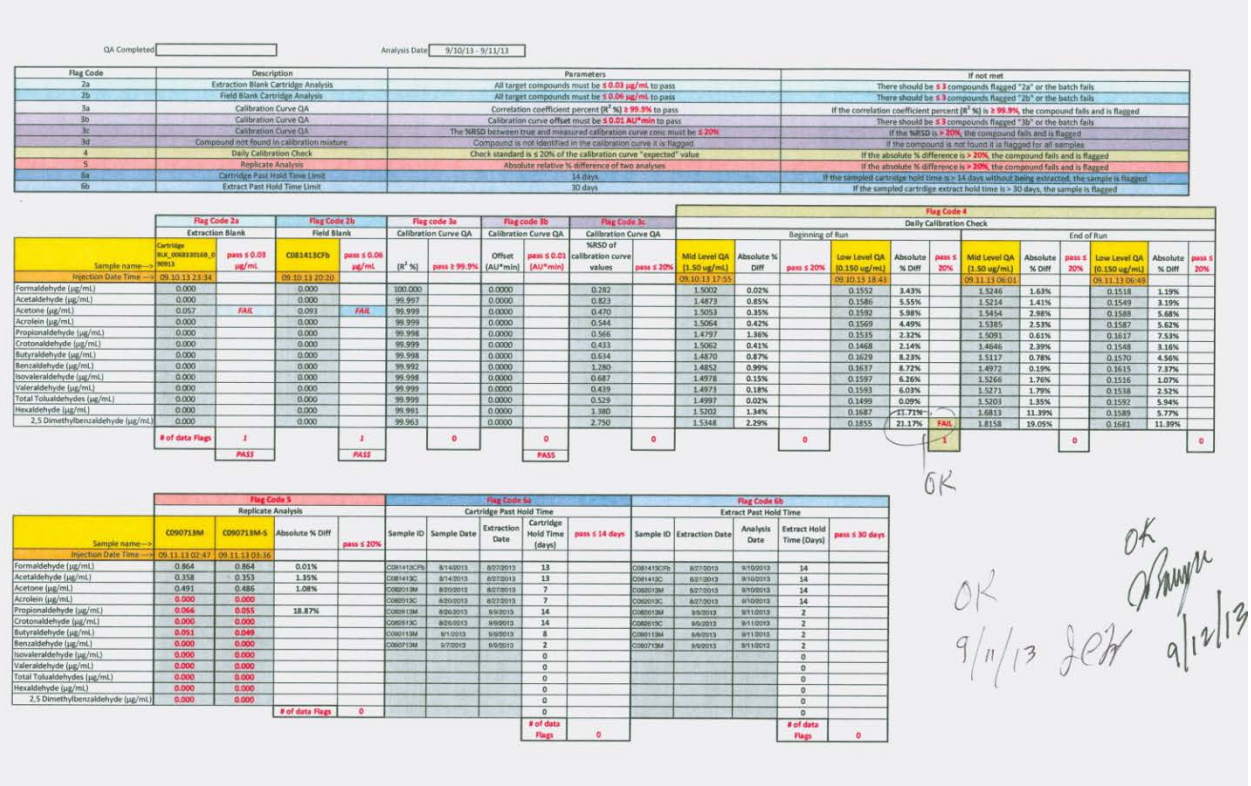
2.52.4.1.3.2 Check the dates (the "*Injection Date/Time:*" on the aldehyde sample chromatograms) and make sure that all the reports are in chronological order. If there are any missing time periods, check the Sequence printout and ask the UHPLC Laboratory Analyst. Each aldehyde sample chromatogram should 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.



QA Completed: _____ Analysis Date: 9/10/13 - 9/11/13

Flag Code	Description	Parameters	If not met
2a	Extraction Blank Cartridge Analysis	All target compounds must be ≤ 0.03 µg/mL to pass	There should be ≤ 3 compounds flagged "2a" or the batch fails
2b	Field Blank Cartridge Analysis	All target compounds must be ≤ 0.06 µg/mL to pass	There should be ≤ 3 compounds flagged "2b" or the batch fails
3a	Calibration Curve QA	Correlation coefficient percent (R^2 %) ≥ 99.9% to pass	If the correlation coefficient percent (R^2 %) is ≥ 99.9%, the compound fails and is flagged
3b	Calibration Curve QA	Calibration curve offset must be ≤ 0.01 AU*min to pass	There should be ≤ 3 compounds flagged "3b" or the batch fails
3c	Calibration Curve QA	The %RSD between true and measured calibration curve conc must be ≤ 20%	If the %RSD is > 20%, the compound fails and is flagged
3d	Compound not found in calibration mixture	Compound is not identified in the calibration curve it is flagged.	If the compound is not found it is flagged for all samples
4	Daily Calibration Check	Check standard is ≤ 20% of the calibration curve "expected" value	If the absolute % difference is > 20%, the compound fails and is flagged
5	Replicate Analysis	Absolute relative % difference of two analyses	If the absolute % difference is > 20%, the compound fails and is flagged
6a	Cartridge Past Hold Time Limit	14 days	If the sampled cartridge hold time is > 14 days without being extracted, the sample is flagged
6b	Extract Past Hold Time Limit	30 days	If the sampled cartridge extract hold time is > 30 days, the sample is flagged

Sample name-->	Flag Code 2a: Extraction Blank: Cartridge BLK_0068330160_0 0013	Flag Code 2a: pass ≤ 0.03 µg/mL	Flag Code 2b: Field Blank: C081413CFb	Flag Code 2b: pass ≤ 0.06 µg/mL	Flag Code 3a: Calibration Curve QA: (R^2 %)	Flag Code 3a: pass ≥ 99.9%	Flag Code 3b: Calibration Curve QA: Offset (AU*min)	Flag Code 3b: pass ≤ 0.01 (AU*min)	Flag Code 3c: Calibration Curve QA: %RSD of calibration curve values	Flag Code 3c: pass ≤ 20%	Flag Code 4: Daily Calibration Check: Beginning of Run: Mid Level QA (1.50 ug/mL)	Beginning of Run: Absolute % Diff	Beginning of Run: pass ≤ 20%	Beginning of Run: Low Level QA (0.150 ug/mL)	Beginning of Run: Absolute % Diff	Beginning of Run: pass ≤ 20%	End of Run: Mid Level QA (1.50 ug/mL)	End of Run: Absolute % Diff	End of Run: pass ≤ 20%	End of Run: Low Level QA (0.150 ug/mL)	End of Run: Absolute % Diff	End of Run: pass ≤ 20%
Injection Date Time-->	09.10.13 23:34		09.10.13 20:20								09.10.13 17:55			09.10.13 18:43			09.11.13 06:01			09.11.13 06:49		
Formaldehyde (µg/mL)	0.000		0.000		100.000		0.0000		0.282		1.5002	0.02%		0.1552	3.43%		1.5246	1.63%		0.1518	1.19%	
Acetaldehyde (µg/mL)	0.000		0.000		99.997		0.0000		0.823		1.4873	0.85%		0.1586	5.55%		1.5214	1.41%		0.1549	3.19%	
Acetone (µg/mL)	0.057	FAIL	0.093	FAIL	99.999		0.0000		0.470		1.5053	0.35%		0.1592	5.98%		1.5454	2.98%		0.1588	5.68%	
Acrolein (µg/mL)	0.000		0.000		99.999		0.0000		0.544		1.5064	0.42%		0.1569	4.49%		1.5385	2.53%		0.1587	5.62%	
Propionaldehyde (µg/mL)	0.000		0.000		99.998		0.0000		0.566		1.4797	1.36%		0.1535	2.32%		1.5091	0.61%		0.1617	7.53%	
Crotonaldehyde (µg/mL)	0.000		0.000		99.999		0.0000		0.433		1.5062	0.41%		0.1468	2.14%		1.4646	2.39%		0.1548	3.16%	
Butyraldehyde (µg/mL)	0.000		0.000		99.998		0.0000		0.634		1.4870	0.87%		0.1629	8.23%		1.5117	0.78%		0.1570	4.56%	
Benzaldehyde (µg/mL)	0.000		0.000		99.992		0.0000		1.280		1.4852	0.99%		0.1637	8.72%		1.4972	0.19%		0.1615	7.37%	
Isovaleraldehyde (µg/mL)	0.000		0.000		99.998		0.0000		0.687		1.4978	0.15%		0.1597	6.26%		1.5266	1.76%		0.1516	1.07%	
Valeraldehyde (µg/mL)	0.000		0.000		99.999		0.0000		0.439		1.4973	0.18%		0.1593	6.03%		1.5271	1.79%		0.1538	2.52%	
Total Tolualdehydes (µg/mL)	0.000		0.000		99.998		0.0000		0.529		1.4997	0.02%		0.1499	0.09%		1.5203	1.35%		0.1592	5.94%	
Hexaldehyde (µg/mL)	0.000		0.000		99.991		0.0000		1.380		1.5202	1.34%		0.1687	11.71%		1.6813	11.39%		0.1589	5.77%	
2,5 Dimethylbenzaldehyde (µg/mL)	0.000		0.000		99.963		0.0000		2.750		1.5348	2.29%		0.1855	21.17%	FAIL	1.8158	19.05%		0.1683	11.39%	
	# of data Flags	1		1		0		0		0			0			1			0			0
		FAIL		FAIL				PASS								OK						

Sample name-->	Flag Code 5: Replicate Analysis: C090713M	C090713M-S	Absolute % Diff	pass ≤ 20%	Flag Code 6a: Cartridge Past Hold Time: Sample ID	Sample Date	Extraction Date	Cartridge Hold Time (days)	pass ≤ 14 days	Flag Code 6b: Extract Past Hold Time: Sample ID	Extraction Date	Analysis Date	Extract Hold Time (Days)	pass ≤ 30 days
Injection Date Time-->	09.11.13 02:47	09.11.13 03:16												
Formaldehyde (µg/mL)	0.864	0.864	0.01%		C081413CFb	8/14/2013	8/27/2013	13		C081413CFb	8/27/2013	9/10/2013	14	
Acetaldehyde (µg/mL)	0.358	0.353	1.35%		C081413C	8/14/2013	8/27/2013	13		C081413C	8/27/2013	9/10/2013	14	
Acetone (µg/mL)	0.491	0.486	1.08%		C082013M	8/20/2013	8/27/2013	7		C082013M	8/27/2013	9/10/2013	14	
Acrolein (µg/mL)	0.000	0.000			C082013C	8/20/2013	8/27/2013	7		C082013C	8/27/2013	9/10/2013	14	
Propionaldehyde (µg/mL)	0.066	0.055	18.87%		C082813M	8/26/2013	9/9/2013	14		C082813M	9/9/2013	9/11/2013	2	
Crotonaldehyde (µg/mL)	0.000	0.000			C082813C	8/26/2013	9/9/2013	14		C082813C	9/6/2013	9/11/2013	2	
Butyraldehyde (µg/mL)	0.051	0.049			C090113M	9/1/2013	9/9/2013	8		C090113M	9/6/2013	9/11/2013	2	
Benzaldehyde (µg/mL)	0.000	0.000			C090713M	9/7/2013	9/9/2013	2		C090713M	9/9/2013	9/11/2013	2	
Isovaleraldehyde (µg/mL)	0.000	0.000						0					0	
Valeraldehyde (µg/mL)	0.000	0.000						0					0	
Total Tolualdehydes (µg/mL)	0.000	0.000						0					0	
Hexaldehyde (µg/mL)	0.000	0.000						0					0	
2,5 Dimethylbenzaldehyde (µg/mL)	0.000	0.000						0					0	
			# of data Flags	0				# of data Flags	0				# of data Flags	0

OK
9/11/13 JCH
OK
9/12/13

Figure 1. QA/QC Spreadsheet

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 ≥ 0.5 µg/mL that has not been flagged.
- Mark with an arrow any value ≥ 1 µg/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.

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

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: