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NORTH CAROLINA
Environmental Quality

DAQ-03-003.5 Standard Operating Procedure (SOP)

For the Markes-Agilent Gas Chromatography-Mass Spectrometry (GC-MS) Analysis of Volatile Organic Compounds in Ambient Air Collected in 6-Liter Canisters

North Carolina Division of Air Quality (DAQ)

Raleigh Central Office Responsibilities

NORTH CAROLINA
Department of Environmental Quality

1.0 Approval Sign-Off Sheet

I certify that I have read and approve of the contents of the Standard Operating Procedure (SOP) for the Markes-Agilent Gas Chromatography-Mass Spectrometry (GC-MS) Analysis of Volatile Organic Compounds in Ambient Air Collected in 6-Liter Canisters, Raleigh Central Office Responsibilities, with an effective date of June 17, 2022.

Director, Air Quality Division

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Disclaimer:

This document, and any revision hereto, is intended solely as a reference guide to assist the user in the validation of volatile organic compound (VOC) canister sampling data and canister analysis data for the North Carolina Division of Air Quality (DAQ)'s Ambient Monitoring Program.

Acronyms

AQS - Air Quality System (EPA's Air database)
BTEX - Total benzene, toluene, ethylbenzene, and total xylenes
CAR – corrective action report
CC – continuing calibration
COC – Chain of Custody
csv – comma separated value
CV – coefficient of variation
DAQ – North Carolina Division of Air Quality
EPA – United States Environmental Protection Agency
GC-MS – Gas Chromatography-Mass Spectrometry
≥ - greater than or equal to
in Hg – Inches of Mercury
IPA – Instrument Performance Audit
LAB – Laboratory Analysis Branch
LCS – Laboratory control sample
MDL – Method detection limit
min - minutes
mL/min – milliliters per minute
NATTS – National Air Toxics Trends Site
ppb – Part Per Billion
POC – Parameter Occurrence Code
psi – pounds per square inch
PT – Proficiency Testing
QA – Quality assurance
QC – Quality control
RPD – relative percent difference
SOP – Standard Operating Procedure
SSCV – Second source calibration verification
TAD – technical assistance document
UAT – Urban Air Toxics
VOC – Volatile Organic Compound

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2.0 SCOPE AND PURPOSE

This document describes the process of performing the Level 3 validation of VOC canister sampling and analysis data and transforming the VOC analysis data into pipe delimited files.

3.0 OVERVIEW

3.1 VOC Analysis Instrument Reporting Units

The VOC concentration data are reported by the analysis instrument in parts per billion (ppb) and reported to the Air Quality System (AQS) in ppb. This includes concentrations reported for calibration standards, check standards, and performance test samples. The units in AQS are coded with "**008**" and are rounded to the third decimal place.

Note: The standard AQS reports will report the VOC data in parts per billion carbon so the concentrations inputted into AQS may not match the concentrations reported by AQS because of the different units used.

See table 1 below for the AQS codes for site, method, Parameter Occurrence Code (POC), duration, and units. Additionally, this table identifies the site names and the letter code for generating sample numbers. For example, a VOC sample taken from Pitt Ag on 1/4/2022 is identified with sample number: **010422P**.

Table 1: AQS Codes for Site, Method, POC, Frequency, and Unit

Letter Code	Site	Site ID#	Method Code	POC	Duration code	Unit (ppb)
A	AB-Tech-Asheville	37-021-0035	150	1	7	008
B	Eagles Island-Battleship-Wilmington	37-129-0010	150	1	7	008
C	Candor	37-123-0001	150	1	7	008
G	Garinger	37-119-0041	150	2	7	008
M	Millbrook	37-183-0014	150	1	7	008
MD	Millbrook Duplicate	37-183-0014	150	8	7	008
W	Winston Salem-Hattie Ave	37-067-0022	150	2	7	008
P	Pitt-Ag Greenville	37-147-0006	150	1	7	008

3.2 VOC Sampler Reporting Units

The VOC sampler records pressure in inches of mercury (**in Hg**) and pounds per square inch (**psi**). Flow rates are in units of milliliters per minute (**mL/min**).

The VOC sampler data is used to apply null codes or qualifier flags to the VOCs detected in the canister samples that indicate a sample collection issue.

3.3 VOC Analysis Reporting Units

Concentrations are reported to AQS in units of **ppb** and the analysis instrument reports VOC concentrations in units of **ppb**. Unit conversions are NOT performed on the VOC concentrations detected in VOC canisters.

3.4 Rounding Conventions

All VOC concentrations are rounded to 3 decimal places when reported to AQS. The gas chromatography-mass spectrometry (GC-MS) instrument reports VOC concentrations rounded to 3 decimal places in the "Env_Results" reports and to 4 decimal places in the comma separated value (csv) summary files.

Retention times are reported by the GC-MS instrument to 3 decimal places in the "Env_Results" reports and in the csv summary files.

3.5 AQS Pipe Delimited Files

Pipe delimited text files are created through transforming the analysis data reported by the GC-MS instrument and sampling data reported by the sampling equipment. Additionally, sampling and analysis data failing one or more quality control parameters are appropriately null coded or qualifier flagged prior to AQS reporting. Please see **figure 1** and **figure 2** below for an example of a coded pipe delimited file that is ready for reporting to AQS.

Figure 1: Pipe Delimited File Example (Millbrook)

```
RD|I|37|183|0014|42153|1|7|008|150|20210703|00:00||AT|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210709|00:00||AT|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210715|00:00||AT|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210721|00:00|0.173||||7|2|3|4|6||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210727|00:00|0.151||||7|3|2|4|6||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210802|00:00|0.277||||3|2|4|6|||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210808|00:00|0.339||||3|2|4|6|||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210814|00:00||AX|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210820|00:00|0.332||||LB|2|3|4|6||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210826|00:00|0.463||||LB|2|3|4|6||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210901|00:00||AN|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210907|00:00||AX|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210913|00:00|0.028||||7|LB|4|2|3|6|||||||
RD|I|37|183|0014|42153|1|7|008|150|20210919|00:00||AN|||||||||||||
RD|I|37|183|0014|42153|1|7|008|150|20210925|00:00|0.199||||7|2|3|4|6||||||||
```

Figure 2: Pipe Delimited File Example (Millbrook Duplicate)

```
RD|I|37|183|0014|42153|8|7|008|150|20210703|00:00||AT|||||||||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210709|00:00||AT|||||||||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210715|00:00||AT|||||||||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210721|00:00|0.101||||7|2|3|4|6|||||||
RD|I|37|183|0014|42153|8|7|008|150|20210727|00:00|0.236||||3|2|4|6||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210802|00:00|0.363||||3|2|4|6||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210808|00:00|0.787||||3|2|4|6||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210814|00:00||AX|||||||||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210820|00:00|0.295||||LB|2|3|4|6|||||||
RD|I|37|183|0014|42153|8|7|008|150|20210826|00:00|0.511||||LB|2|3|4|6|||||||
RD|I|37|183|0014|42153|8|7|008|150|20210901|00:00|0.611||||2|3|4|6||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210907|00:00|0.027||||7|LB|2|3|4|6|||||||
RD|I|37|183|0014|42153|8|7|008|150|20210913|00:00|0.026||||7|LB|2|3|4|6|||||||
RD|I|37|183|0014|42153|8|7|008|150|20210919|00:00|0.210||||2|3|4|6||||||||
RD|I|37|183|0014|42153|8|7|008|150|20210925|00:00|0.064||||7|2|3|4|6|||||||
```

3.6 VOC Compounds Reported to AQS

The DAQ Laboratory Analysis Branch (LAB) analyzes for the VOC compounds listed in Table 2 below. Additionally, Table 2 lists the compound's AQS parameter code and GC-MS instrument reporting limits.

The compound names that are highlighted in red text are not currently reported to AQS using method 150. Compound names in bold font are the designated National Air Toxics Trends Site (NATTS) Core I and target proficient test (PT) compounds. Compound names neither bold nor red font are additional hazardous air pollutants reported to AQS using method 150.

Table 2: VOC Compound List, AQS parameter code, and reporting limits

	Calibration Range		Qualifier Ions	
Peak Table	**Lower Limit (ppb)**	**Upper Limit (ppb)**	**Primary**	**Secondary**
1,1,1-Trichloroethane 43814	0.2	10.0	97	99, 61
1,1,2,2-Tetrachloroethane 43818	0.2	10.0	83	85
1,1,2-Trichloroethane 43820	0.2	10.0	97	83, 61
1,1-Dichloroethane 43813	0.2	10.0	63	65, 27
1,1-Dichloroethene 43826	0.2	10.0	61	96
1,2,3-Trimethylbenzene 45225	0.2	10.0	105	120
1,2,4-Trichlorobenzene 45810	0.2	10.0	180	182, 184
1,2,4-Trimethylbenzene 45208	0.2	10.0	105	120
1,2-Dibromoethane 43843	0.2	10.0	107	109
1,2-Dichlorobenzene 45805	0.2	10.0	146	148
1,2-Dichloroethane 43815	0.2	10.0	62	64, 27
1,2-Dichloropropane 43829	0.2	10.0	63	41, 62
1,3,5-Trimethyl Benzene 45207	0.2	10.0	105	120

Peak Table	Calibration Range		Qualifier Ions	
	Lower Limit (ppb)	Upper Limit (ppb)	Primary	Secondary
1,3-Butadiene 43218	0.2	10.0	39	54
1,3-dichlorobenzene 45806	0.2	10.0	146	148
1,4-dichlorobenzene 45807	0.2	10.0	146	148, 111
1,4-Dioxane 46201	0.2	10.0	88	58
1-Bromopropane 43853	0.2	10.0	43	41
1-Butanol 43305	0.2	10.0	56	43
2-Pentanone 43562	0.2	10.0	43	86
3-Hexanone 43557	0.2	10.0	43	71
3-Pentanone 43553	0.2	10.0	57	86
Acetaldehyde 43503	0.2	10.0	44	29, 43
Acetone 43551	0.2	10.0	43	42
Acetonitrile 43702	0.2	10.0	41	40
Acrolein 43505	0.2	10.0	56	55
Benzene 45201	0.2	10.0	78	77, 50
Benzyl chloride 45809	0.2	10.0	91	126
Bromodichloromethane 43828	0.2	10.0	83	85
Bromoform 43806	0.2	10.0	173	171, 175
Bromomethane 43819	0.2	10.0	94	96
Carbon disulfide 42153	0.2	10.0	76	44, 78
Carbon Tetrachloride 43804	0.2	10.0	117	119
chlorobenzene 45801	0.2	10.0	112	77, 114
Chloroethane 43812	0.2	10.0	64	66
Chloroform 43803	0.2	10.0	83	85, 47
Chloromethane 43801	0.2	10.0	50	52
Cis-1,3-dichloropropene 43831	0.2	10.0	75	39, 77
Cyclohexane 43248	0.2	10.0	56	41
Cyclopentane 43242	0.2	10.0	42	43
Ethanol 43302	0.2	10.0	45	46
Ethylbenzene 45203	0.2	10.0	91	106
Freon 11 43811	0.2	10.0	101	103
Freon 113 43821	0.2	10.0	151	101
Freon 114 43208	0.2	10.0	85	135
Freon 12 43823	0.2	10.0	51	35
Freon 22 43359	0.2	10.0	85	87
Hexanal 43517	0.2	10.0	56	44

Peak Table	Calibration Range		Qualifier Ions	
	Lower Limit (ppb)	Upper Limit (ppb)	Primary	Secondary
Hexane 43231	0.2	10.0	57	41, 43
Iodomethane 43808	0.2	10.0	142	127
Isobutene 43270	0.2	10.0	41	56
Isoprene 43243	0.2	10.0	67	53
Isopropyl Alcohol 43312	0.2	10.0	45	59
m,p-xylene45109	0.2	10.0	91	106
Methacrolein 43515	0.2	10.0	41	70
Methyl Butyl Ketone 43559	0.2	10.0	58	43
Methyl Ethyl Ketone 43552	0.2	10.0	43	72
Methyl Isobutyl Ketone 43560	0.2	10.0	43	58, 100
Methyl Tert Butyl Ether 43372	0.2	10.0	73	41, 53
Methyl Vinyl Ketone 43558	0.2	10.0	55	70
Methylene Chloride 43802	0.2	10.0	49	84, 86
n-Butanal 43510	0.2	10.0	44	72
n-Pentane 43220	0.2	10.0	43	41
o-Xylene 45204	0.2	10.0	91	106
Propanal 43504	0.2	10.0	58	29, 57
Propylene 43205	0.2	10.0	41	39
Styrene 45220	0.2	10.0	104	78, 103
Tetrachloroethylene 43817	0.2	10.0	166	164, 131
Toluene 45202	0.2	10.0	91	92
Trans-1,2-Dichloroethene 43838	0.2	10.0	61	96
Trans-1,3-dichloropropene 43830	0.2	10.0	75	110
Trichloroethylene 43824	0.2	10.0	130	132, 95
Vinyl Acetate 43447	0.2	10.0	43	86
Vinyl Chloride 43860	0.2	10.0	62	64

4.0 DATA VALIDATION GENERAL OVERVIEW

The Level 3 reviewer transfers the laboratory analysis results from the analysis instrument files to the data validation spreadsheet and transfers null codes and qualifier flags to the data validation spreadsheet. The null code and qualifier flag criteria are tested in the following summary reports: DAQ-03-021 VOC Canister Chain of Custody (COC) Form QC Summary Report, DAQ-03-022 System I VOC Analysis QC Summary Report, DAQ-03-023 System II VOC Analysis QC Summary Report, and DAQ-03-024 Internal Standard Summary Report to the data validation spreadsheet.

The data validation spreadsheet performs a ratio calculation, and the result of the ratio provides an additional layer of confidence that a true ambient air sample was analyzed but only in samples when both VOCs are greater than or equal to (≥) 0.2ppb.

Ratios are calculated between the following pairs of VOCs: (Toluene : Benzene), (m\p-Xylene : o-Xylene), (Benzene : Ethylbenzene), and (n-Pentane : Cyclopentane). In all cases, the **ratios must be ≥ 1** and both **concentrations must be ≥ 0.2ppb**. See figure 3 below showing an example of the imbedded ratio calculation in the data validation spreadsheet and the conditional format that highlights the ratio for being less than 1. In this example the detected concentrations are also less than 0.2ppb so the ratio cannot be used to further apply qualifier flags to the benzene and toluene concentrations detected in the 1/17/22 Asheville, Candor, Pitt Ag, and Eagle Island samples.

Figure 3: Example of Ratio Calculation

Sample Date	Sample Site	Benzene 45201	Toluene 45202	Flag Benzene 45201	Flag Toluene 45202	Toluene vs Benzene
1/17/2022	Asheville	**0.125**	**0.124**			0.9912281
1/17/2022	Candor	**0.123**	**0.114**			0.9205835
1/17/2022	Millbrook	**0.130**	**0.139**			1.0707692
1/17/2022	Millbrook D	**0.127**	**0.131**			1.0267296
1/17/2022	Pitt Ag	**0.118**	**0.117**			0.9915326
1/17/2022	Eagle Island	**0.116**	**0.113**			0.9741824

Lastly, the Level 3 reviewer transforms the VOC results into pipe delimited text files which are formatted according to AQS coding protocols and uploaded by DAQ's database manager to AQS.

4.1 Data Transfer

Data transfer from the instrument summary files to the data validation spreadsheet cannot commence until the Level 1 and Level 2 reviews have been completed. Data transfer occurs on an analysis batch by analysis batch frequency.

4.2 Control Charts

These charts are used to visually identify potential data outliers and to visually identify VOC data points that may require further investigation.

Total benzene, toluene, ethylbenzene, and total xylenes (BTEX) should be detected in sufficient quantities to make comparisons between urban and rural sites and should follow the trend:

urban total BTEX concentration (ppb) ≥ rural total BTEX concentration (ppb). Where the trendlines cross or meet are the areas to focus. If two points exist on the same date from both sites represent the reversal of the typical trend, these two data points require further investigation. An example of urban total BTEX vs rural total BTEX over time is shown in figure 4 below.

Figure 4: Urban vs Rural Total BTEX Control Chart

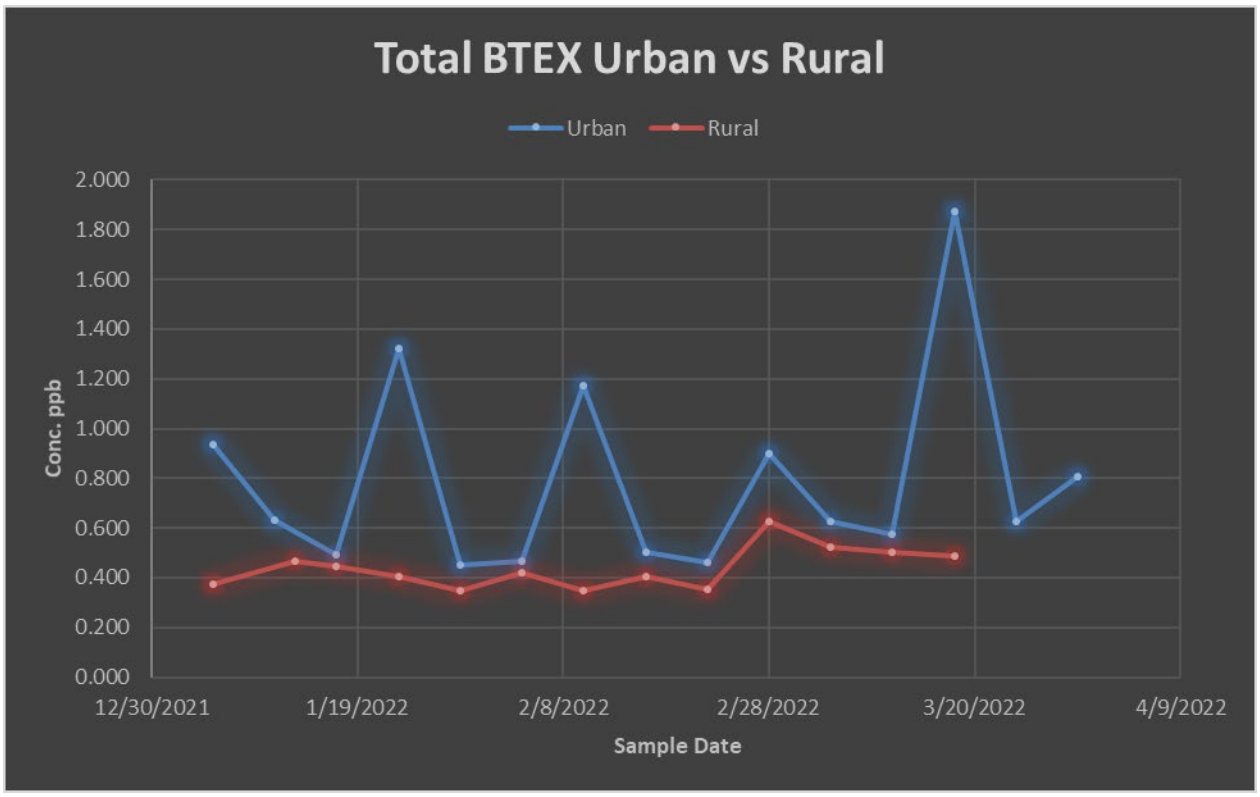
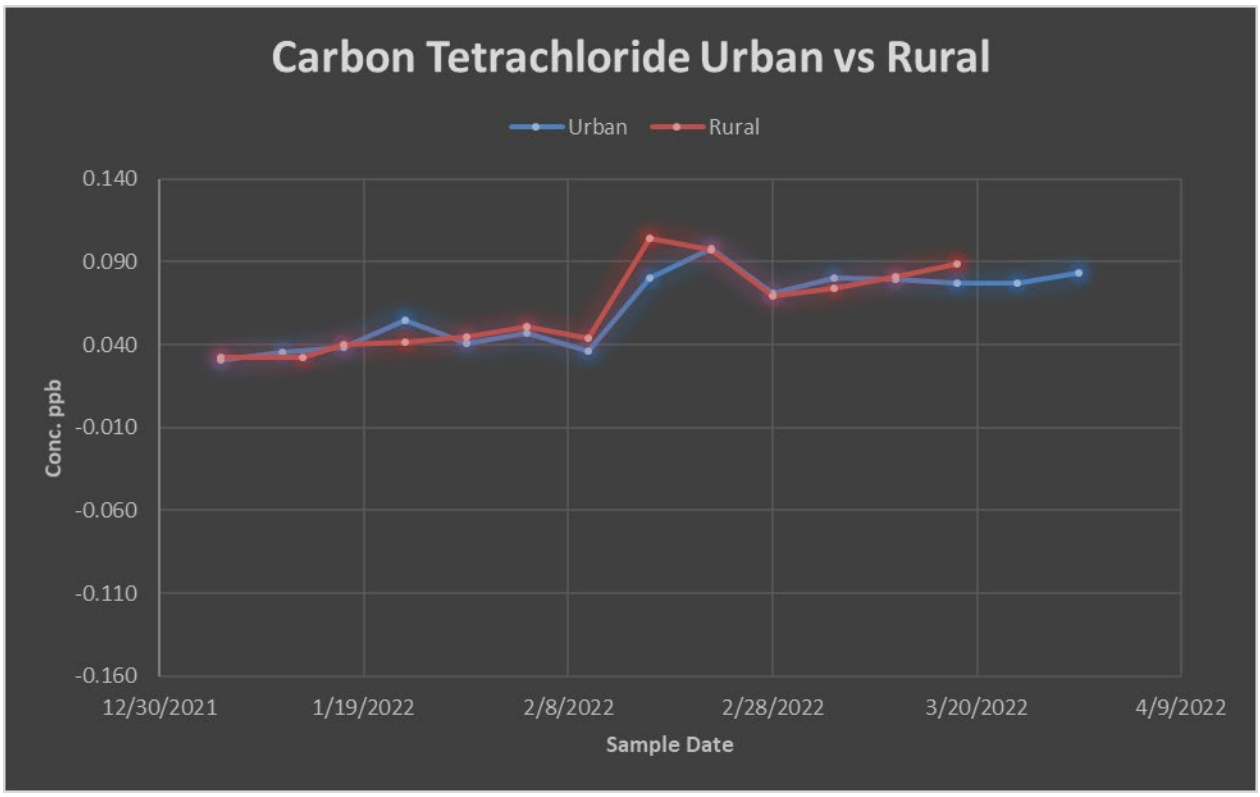


Figure 4 can also be used to identify samples when the total BTEX value is zero or close to zero. When the total BTEX concentration is less than 0.2ppb, further investigation of the chromatogram should be done to ensure target VOCs are properly identified and quantified.

Carbon tetrachloride is also monitored and should be detected and quantified in all field sample canisters that are used to collect ambient air. Carbon tetrachloride is more ubiquitous in the ambient air and is generally the same concentration at urban and rural sites. The typical trend should follow **urban carbon tetrachloride concentration (ppb) should be within ± 0.1ppb of the rural carbon tetrachloride concentration (ppb)**. Instances of a deviation in the trend may indicate that one or both samples have been impacted by a nearby source or provide some doubt that ambient air was collected in the canister. Carbon tetrachloride concentrations should be very similar at both rural and urban sites as shown in figure 5 below.

Figure 5: Urban vs Rural Carbon Tetrachloride Control Chart



4.3 Analysis Instrument Acceptance Criteria

The GC-MS analysis instrument must meet the criteria listed in Table 3 below. If the criteria are not met, the application of a null code or qualifier flag is required before the data point is reported to AQS.

Table 3: VOC Canister Analysis Quality Control (QC) Parameters

Where to look for Null Code or Qualifier Flag	QC parameter	Flag Criteria	AQS Flag	How to apply Flag
DAQ-03-023 analysis summary workbook	Lab blank or method blank	All target carbonyls must be ≤ 0.2 ppb	Quality assurance (QA) qualifier: LB	Apply flag "LB" to all failing VOCs and apply to same VOCs in all field samples analyzed in the same sequence
DAQ-03-023 ICAL summary workbook tab	Calibration curve best fit line type	Best fit line must be Linear or Quadratic	Null: AT	Void failing VOCs with null code AT and apply to same VOCs detected in field samples processed using the calibration curve

Where to look for Null Code or Qualifier Flag	QC parameter	Flag Criteria	AQS Flag	How to apply Flag
DAQ-03-023 ICAL summary workbook tab	Calibration curve regression analysis	R-squared ≥ 0.995	Null: AT	Void failing VOCs with null code AT and apply to same VOCs detected in all field samples processed using the calibration curve
DAQ-03-023 ICAL summary workbook tab	Calibration Curve Accuracy	Relative Error for each level against calibration curve must be ≤ 30% of nominal	Null: AT	Void failing VOCs with null code AT and apply to same VOCs detected in all field samples processed using the calibration curve
DAQ-03-023 Analysis summary workbook tab	Second Source Calibration Verification (SSCV) Accuracy	Relative error for each VOC in the second source must be ≤ 30% of nominal	Null: ST	Void failing VOCs with null code ST and apply to same VOCs detected in all field samples analyzed in the same sequence as the failing SSCV
DAQ-03-023 Analysis Summary workbook tab	Lab Control Sample (LCS) or Continuing Calibration (CC) Accuracy	Relative error for each VOC in the LCS must be ≤ 30% of nominal	QA qualifier: SP	Apply qualifier flag SP to all failing VOCs and apply to same VOCs detected in all field samples analyzed in the same sequence as the failing LCS
DAQ-03-023 Analysis Summary workbook tab	LCS or CC Precision	The relative percent difference (RPD) between the beginning and ending LCS or CC must be ≤ 25%	Null: AX	Void failing VOCs with null code AX and apply to same VOCs detected in all field samples analyzed in the same sequence as the failing LCS precision RPD.
DAQ-03-023 Analysis Summary workbook tab	Replicate Analysis	The RPD between two analyses of the same filed sample must be ≤ 25%	QA qualifier: 4	Apply qualifier flag 4 to all failing VOCs detected in the first injection of the field sample replicate analysis pair
DAQ-03-023 Analysis Summary workbook tab	Primary and Collocated Sample Collection Precision	The RPD between the analyses of the primary and collocated samples must be ≤ 25%	QA qualifier: 3	Apply qualifier flag 3 to all failing VOCs detected in the primary (M) and collocated (MD) samples collected on the same date

Where to look for Null Code or Qualifier Flag	QC parameter	Flag Criteria	AQS Flag	How to apply Flag
DAQ-03-023 Retention Time Summary workbook tab	Measured retention time of the known VOC peak	The VOC peak retention time must be within ± 0.5 minutes (min) of the mean calibration curve retention time	Null: BH	Void the failing VOC with null code BH for the sample failing the retention time criteria
DAQ-03-024	Measured retention time of the internal standard compound	The internal standard retention time must be within ± 0.33 min of the mean calibration curve internal standard retention time	QA qualifier: MX	Apply qualifier flag MX to all VOCs detected in the sample
DAQ-03-024	Measured internal standard response factor	The internal standard response factor must be within ± 40% of the mean calibration curve internal standard response factor	QA qualifier: MX	Apply qualifier flag MX to all VOCs detected in the sample
Pipe delimited AQS file and data validation spreadsheet	Non-detects	The detected concentration = null or “0” or “0.000”	QA qualifier: ND	Applied to all concentrations reported by the analysis instrument that are null concentration values, “0” values, and “0.000” values
Pipe delimited AQS file and data validation spreadsheet	Conc. below the calibration range	The detected concentration < 0.2 ppb (bold font format) in data validation spreadsheet	QA qualifier: 7	Applied to all concentrations reported by the analysis instrument that are < 0.2ppb.
Pipe delimited AQS file and data validation spreadsheet	Conc. above the calibration range	The detected concentration is > 10.0 ppb (bold red font format) in data validation spreadsheet	QA qualifier: EH	Applied to all concentrations reported by the analysis instrument that are > 10.0 ppb

4.4 Sampling Equipment Acceptance Criteria

The sampling equipment used to collect VOCs in ambient air include the sampler and the canister. The sampler is cleaned according to SOP # DAQ-03-001.1. The canisters used to collect field samples are cleaned according to SOP # DAQ-03-005.2.

The following criteria listed in table 4 below must be met or a qualifier flag is applied.

Table 4: VOC Canister and Sampler QC Criteria

Where to look for Null Code or Flag	QC parameter	Flag Criteria	AQS Flag	How to apply Flag
DAQ-03-021 Canister COC summary report and DAQ-03-019 Canister Cleaning Record	Canister Cleaning Batch Certification	All target VOCs ≤ 0.2 ppb.	QA qualifier: CC	Apply flag "CC" to failing VOCs detected in field samples collected using the batch of canisters. To be consider a valid detection in the certification sample the failing VOC must also be ≤ 0.2 ppb in the method blank and within ± 30% of nominal in the calibration verification sample
Sampler Certification Results	Clean Sampler Certification	All VOCs must be ≤ 0.2 ppb.	QA qualifier: SB	Apply flag "SB" to failing VOCs detected in field samples collected using the sampler. To be consider a valid detection in the certification sample the failing VOC must also be ≤ 0.2 ppb in the method blank and within ± 30% of nominal in the calibration verification sample
DAQ-03-021 Canister COC summary report	Canister vacuum at sample start	Canister vacuum must be > 28 in Hg vacuum (-30 in Hg, -29 in Hg, and -28 in Hg all pass this criteria); (-27 in Hg fails this criteria)	QA qualifier: QX	Apply flag "QX" to all VOCs detected in the field sample. Do not apply QX if the VOC was null coded or if another QX qualifier was already applied to the VOC
DAQ-03-021 Canister COC summary report	Sampled Canister Pressure	Sampled canister pressure as measured by the sampler pressure gauge must be between 12-30psig.	QA qualifier: QX	Apply flag "QX" to all VOCs detected in the field sample. Do not apply QX if the VOC was null coded or if another QX qualifier was already applied to the VOC

Where to look for Null Code or Flag	QC parameter	Flag Criteria	AQS Flag	How to apply Flag
DAQ-03-021 Canister COC summary report	Average Flow Rate	The pre and post sampling average flow rate must be between 8-12 mL/min	QA qualifier: W	Apply flag "W" to all VOCs detected in the field sample. Do not apply the flag if the VOC was null coded.
DAQ-03-021 Canister COC summary report	Clean canister holding time	The sample date minus the canister cleaning date must be ≤ 30 days	QA qualifier: QX	Apply flag "QX" to all VOCs detected in the field sample. Do not apply the flag if the VOC was null coded or if another QX qualifier was already applied to the VOC
DAQ-03-021 Canister COC summary report	Sampled canister holding time	The analysis date minus the sample date must be ≤ 30 days	QA qualifier: QX	Apply flag "QX" to all VOCs detected in the field sample. Do not apply the flag if the VOC was null coded or if another QX qualifier was already applied to the VOC

4.5 Sample Date and Duration Criteria

The United States Environmental Protection Agency (EPA) sets the sample schedule every calendar year. The EPA sampling schedules can be found here:

https://www.epa.gov/amtic/sampling-schedule-calendar

VOC canister sampling occurs 1-in-6 days, January through December. Sample duration must be 24 ± 1-hour periods.

Sample dates are recorded on the VOC Canister COC form, document number **DAQ-16-010**. Sample dates are also recorded in the data validation spreadsheet, document number **DAQ-03-011** and in the VOC canister COC summary report, document number **DAQ-03-021**.

4.6 Method Detection Limits and Reporting Limits

Method detection limits (MDLs) are determined minimally once per calendar year, preferably in the 1st calendar quarter. The MDL is statistically determined using the method update rule and guidance in section 4.1 of the NATTS Technical Assistance Document (TAD) revision 3 and according to section 3.4 of SOP # DAQ-03-003.2.

The MDL is calculated using results from a series of seven or eight MDL blank and MDL spike canisters. The greater of the two MDLs is considered the reported MDL and this value is used to further flag reported VOC concentrations. See **Table 2** for a current list of reporting limits.

Current carbonyl MDLs are calculated in an excel workbook stored in the following location:

P:\Toxics\Urban Air Toxics\VOC's Current Year Data\MDL

If the MDL doesn't meet the criteria listed in section 3.4 of SOP # DAQ-03-003.2 and does not meet criteria described in Section 4.1 of NATTS TAD Revision 3, an instrument detection limit must be determined. The instrument detection limit must be determined by analyzing a low-level standard a minimum of seven consecutive times targeting a nominal concentration of ~ 0.200ppb. This low-level standard must be made using the primary source calibration standard mix. Calculate the standard deviation and then multiply the standard deviation by the appropriate student-t value and the result will be the instrument detection limit.

If one or more VOC parameter MDL values fails to meet the criteria in the NATTS TAD Revision 3 or fails to meet criteria in SOP# DAQ-03-003.2 or the MDL could not be completed due to an analysis instrument repair, the MDL value cannot be reported. In these cases, the instrument detection limit is used in-place of the MDL.

5.0 VOC SAMPLING AND ANALYSIS DATA VALIDATION

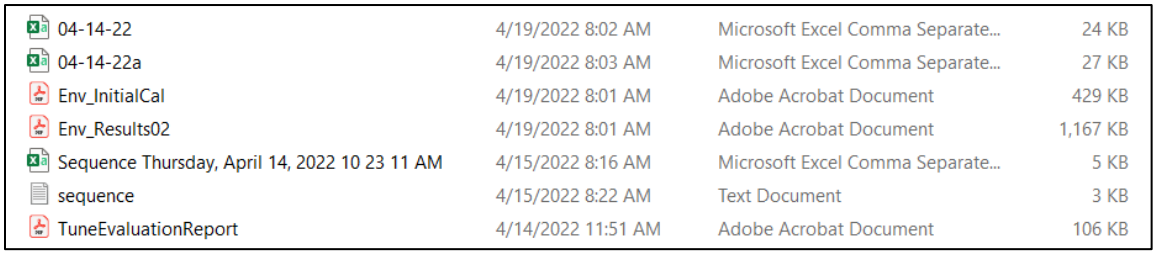
The Level 3 reviewer transfers VOC sampling and analysis data to the data validation spreadsheet and transfers null codes and qualifier flags to the data validation spreadsheet.

5.1 VOC Canister Analysis Data Transfer to The Validation Spreadsheet

Once the Level 1 and Level 2 reviews are complete, the Level 3 reviewer transfers the GC-MS instrument data from the summary files into the data validation spreadsheet.

1. Open the run folder containing the VOC sampling and analysis data that is ready for Level 3 review and validation located here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data.**
2. Open a blank data validation spreadsheet, document number DAQ-03-011 located here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\AQS Test**.
3. Save the data validation spreadsheet with a new name by removing "template" from the file name and adding the current calendar quarter number and two-digit year. For example, if the validation spreadsheet is for samples collected in the 1st quarter of the 2022 year, the file name will be "DAQ-03-011_UAT VOC Canister Data Validation_1Q22" and saved in this location: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\AQS Test\1Q_22.**
4. If a data validation spreadsheet already exists, cancel the creation of the new validation spreadsheet, and open the existing data validation file.
5. Go back to the current run folder and examine the file names to make sure the following files are present and contain sampling and analysis data.
    - DAQ-03-020 VOC Canister Sampling and Analysis Data Review Checklist
    - DAQ-03-021 VOC Canister Form QC Summary Report
    - DAQ-03-022 or DAQ-03-023 System I or System II VOC Analysis QC Summary Report
    - DAQ-03-024 Internal Standard Summary Report
    - Analysis instrument files listed in figure 6 below:

Figure 6: Run Folder Files



04-14-22	4/19/2022 8:02 AM	Microsoft Excel Comma Separate...	24 KB
04-14-22a	4/19/2022 8:03 AM	Microsoft Excel Comma Separate...	27 KB
Env_InitialCal	4/19/2022 8:01 AM	Adobe Acrobat Document	429 KB
Env_Results02	4/19/2022 8:01 AM	Adobe Acrobat Document	1,167 KB
Sequence Thursday, April 14, 2022 10 23 11 AM	4/15/2022 8:16 AM	Microsoft Excel Comma Separate...	5 KB
sequence	4/15/2022 8:22 AM	Text Document	3 KB
TuneEvaluationReport	4/14/2022 11:51 AM	Adobe Acrobat Document	106 KB

6. If any of the files are missing, contact the primary analyst and peer reviewer to produce the missing files.
7. Open the DAQ-03-020 VOC Canister Sampling and Analysis Data Review Checklist to be sure all questions have been answered by the Level 1 and Level 2 reviewer and ensure the form has been signed and dated by both reviewers. Examine the form for completeness and to make sure that all handwritten entries and/or typed entries are legible and easy to understand.
8. Contact the Level 1 and Level 2 reviewer to correct any illegible and/or unreadable entries or comments.
9. Once the data review checklist has been reviewed and is considered complete and readable or has been corrected accordingly by the Level 1 or Level 2 reviewers, open the file “04-14-22”
10. Highlight the top row and turn on “wrap text” formatting.
11. Highlight the peak names from A3 to A76 and select “sort A-Z” a sort warning window should appear.
12. In the “what do you want to do?” select “expand the selection” and click “sort.”
13. This action sorts the data alphabetically by peak name.
14. Copy and paste using the “ctrl” by selecting each individual sample names from the “04-14-22” file and then “paste special values” and “transpose values” into the data validation spreadsheet “sample name” column of the “VOC Data with AQS Flags” workbook tab.
15. Only transfer the sample names for routine field-collected samples. Also, only transfer sample dates that fall within the calendar quarter of data being validated.
16. Run folder names with dates in the months of January, April, July, and October may contain samples from the previous quarter. Pay close attention to the sample date when transferring data points from the instrument summary files to the data validation spreadsheet.
17. Copy and paste the “final conc.” values (row3 to row76) from the “04-14-22” file as “past special value” and “transpose value” into column G. Only transfer the “final conc.” values for routine field-collected samples.
18. Manually enter the run folder name into column E of the data validation spreadsheet for all recently transferred data points.
19. Manually enter the site name into column D of the data validation spreadsheet.
20. Manually enter the sample date into column C of the data validation spreadsheet.
21. Save the data validation spreadsheet.

5.2 Application of Null Codes and Qualifier Flags Due to Analysis and Sampling Issues

After the data has been successfully transferred from the instrument summary files to the data validation spreadsheet "VOC Data with AQS Flags" workbook tab, the application of null codes and qualifier flags in the data validation spreadsheet may begin.

1. Open the System I or System II VOC Analysis QC Summary report for the current run folder and examine the "ICAL summary" workbook tab for red filled cells in columns D-K. A red filled cell in these columns indicates a problem with the instrument calibration curve and requires the application of the "AT" null code to the failing VOC parameter.
2. If a red filled cell exists, find the same VOC parameter in the data validation spreadsheet between rows CD and EZ (cells are shaded gray). For example, **Chloroethane 43812** fails the nominal recovery on the 0.2ppb calibration curve level. Apply the AT flag as shown in Figure 7 below.

Figure 7: AT Null Code Application Example

Sample Date	Sample Site	VOC Run Folder	Sample #	Flag Chloroethane 43812
4/5/2022	Millbrook D	04-14-22 system II	040522MD Millbrook Raleigh duplicate sample	AT
3/30/2022	Huntersville	04-14-22 system II	033022H Huntersville sample	AT
4/5/2022	Candor	04-14-22 system II	040522C Candor sample	AT
4/5/2022	Huntersville	04-14-22 system II	040522H Huntersville sample	AT
4/5/2022	Winston Salem	04-14-22 system II	040522W Winston Salem sample	AT
4/11/2022	Candor	04-14-22 system II	041122C Candor sample	AT
4/11/2022	Winston Salem	04-14-22 system II	041122W Winston Salem sample	AT
4/5/2022	Millbrook	04-14-22 system II	040522M Millbrook Raleigh sample	AT
4/5/2022	Pitt Ag	04-14-22 system II	040522PPitt Ag Greenville sample	AT
4/5/2022	Eagle Island	04-14-22 system II	040522B Eagle Island Wilmington sample	AT
4/11/2022	Pitt Ag	04-14-22 system II	041122P Pitt Ag Greenville sample	AT
4/11/2022	Eagle Island	04-14-22 system II	041122B Eagle Island Wilmington sample	AT

3. Examine the "Analysis Summary" workbook tab columns G and H for red filled cells. A red filled cell indicates the second source verification check for this run folder failed to meet recovery criteria and requires the application of the "ST" null code to all samples in the run folder
4. Find the same VOC parameter in the data validation spreadsheet between rows CD and EZ (cells are shaded gray). For example, in the 04-14-22 system II run, **1,2,4-Trichlorobenzene 45810** failed to recover within acceptable limits and is null coded ST according to Figure 8 below. If an AT null code already exists do not apply the ST null code. Only one null code is reported to AQS.

Figure 8: ST Null Code Application Example

Sample Date	Sample Site	VOC Run Folder	Sample #	Flag 1,2,4-Trichlorobenzene 45810
4/5/2022	Millbrook D	04-14-22 system II	040522MD Millbrook Raleigh duplicate sample	ST
3/30/2022	Huntersville	04-14-22 system II	033022H Huntersville sample	ST
4/5/2022	Candor	04-14-22 system II	040522C Candor sample	ST
4/5/2022	Huntersville	04-14-22 system II	040522H Huntersville sample	ST
4/5/2022	Winston Salem	04-14-22 system II	040522W Winston Salem sample	ST
4/11/2022	Candor	04-14-22 system II	041122C Candor sample	ST
4/11/2022	Winston Salem	04-14-22 system II	041122W Winston Salem sample	ST
4/5/2022	Millbrook	04-14-22 system II	040522M Millbrook Raleigh sample	ST
4/5/2022	Pitt Ag	04-14-22 system II	040522PPitt Ag Greenville sample	ST
4/5/2022	Eagle Island	04-14-22 system II	040522B Eagle Island Wilmington sample	ST
4/11/2022	Pitt Ag	04-14-22 system II	041122P Pitt Ag Greenville sample	ST
4/11/2022	Eagle Island	04-14-22 system II	041122B Eagle Island Wilmington sample	ST

5. Examine the “Analysis Summary” workbook tab column F for any red filled cells. A red filled cell indicates the VOC failed to meet precision RPD criteria and requires the application of the AX null code. Find the VOC parameter in the data validation spreadsheet and apply the AX flag in a similar manner to instructions in #4 above. If an AT or ST null code has already been applied do not apply the AX null code.
6. Examine the “retention time summary” workbook tab for red filled cells in columns K-AO that also contain a number. Red filled cells that contain a number indicate the retention time fails criteria and requires the application of the BH null code. Red filled cells that DO NOT contain a number are considered non-detects and will be coded as ND during the creation of the AQS pipe file. Apply the BH null code in a similar manner to #4 above. Do not apply the BH null code if an AT, ST, or AX null code has already been applied.
7. Examine the “analysis summary” workbook tab and look for red filled cells in columns D and E. A red filled cell indicates the VOC parameter failed the lab control recovery and requires the application of the qualifier flag SP to the VOC parameter in the data validation spreadsheet. If an AT, ST, AX, or BH null code has already been applied do not apply the SP qualifier flag.
8. Examine the “analysis summary workbook tab and look for red filled cells in columns I-L. A red cell indicates the VOC parameter failed to meet blank criteria and requires the application of qualifier flag LB to the VOC parameter in the data validation spreadsheet.
9. If an AT, ST, AX, or BH null code has already been applied do not apply the LB qualifier flag. If a qualifier flag has already been applied, add the LB flag as shown here: “SP,LB”.
10. Examine the “analysis summary” workbook tab and look for red filled cells in column O. A red filled cell indicates a precision failure in the replicate sample analysis and requires the application of the qualifier flag “4” to the VOC parameter in the data validation spreadsheet.

The flag is only applied to the initial injection result of the replicate injection pair as shown in figure 9 below:

Figure 9: "4" Qualifier Flag Application Example

Sample Date	Sample Site	VOC Run Folder	Sample #	Flag 1,2,4-Trimethylbenzene 45208
4/5/2022	Millbrook D	04-14-22 system II	040522MD Millbrook Raleigh duplicate sample	
3/30/2022	Huntersville	04-14-22 system II	033022H Huntersville sample	
4/5/2022	Candor	04-14-22 system II	040522C Candor sample	
4/5/2022	Huntersville	04-14-22 system II	040522H Huntersville sample	
4/5/2022	Winston Salem	04-14-22 system II	040522W Winston Salem sample	
4/11/2022	Candor	04-14-22 system II	041122C Candor sample	
4/11/2022	Winston Salem	04-14-22 system II	041122W Winston Salem sample	
4/5/2022	Millbrook	04-14-22 system II	040522M Millbrook Raleigh sample	4
4/5/2022	Pitt Ag	04-14-22 system II	040522PPitt Ag Greenville sample	
4/5/2022	Eagle Island	04-14-22 system II	040522B Eagle Island Wilmington sample	
4/11/2022	Pitt Ag	04-14-22 system II	041122P Pitt Ag Greenville sample	
4/11/2022	Eagle Island	04-14-22 system II	041122B Eagle Island Wilmington sample	

11. If an AT, ST, AX, or BH null code has already been applied do not apply the "4" qualifier flag. If one or more qualifier flags have already been applied, add the "4" flag as shown here: "SP,LB,4".
12. Examine the "analysis summary" workbook tab for red filled cells in columns R, U, X, or AA. A red filled cell indicates a precision failure in the primary and collocated sample collection and analysis pair and requires the application of the qualifier flag "3" to the VOC parameter in the data validation spreadsheet. The flag is only applied to the VOC parameter in the primary and collocated sample pair.
13. If an AT, ST, AX, or BH null code has already been applied do not apply the "3" qualifier flag. If a qualifier flag has already been applied, add the "3" flag as shown here: "SP,LB,4,3".
14. Open the Internal Standard Summary report for the current run folder, document number DAQ-03-024.
15. Examine the Internal Standard Summary report for red filled cells in rows 17 through 60. A red cell indicates the internal standard retention or internal standard response factor failed to meet criteria and requires the application of the MX qualifier flag to all VOC parameters and sample name in the data validation spreadsheet.
16. If a null code has already been applied, do not apply the MX qualifier flag. If one or more qualifier flags have already been applied, add the MX flag as shown here: "SP,LB,4,3,MX".
17. Open the VOC Canister COC Form QC Summary report, document number DAQ-03-021, that has been generated for the current run folder.

18. Examine the report for red filled cells in columns E, F, G, J, L, P, and Q. A red filled cell indicates a failure with a field sampling parameter and requires the application of the QX qualifier flag. If the same sample contains multiple red filled cells, only apply one QX flag to all VOCs detected in the sample. More than one QX flag cannot be reported.
19. If a null code has already been applied do not apply the QX qualifier. If one or more qualifier flags have already been applied add the QX flag as shown here: "SP,LB,4,3,MX,QX"
20. Examine the report for red filled cells in columns H and K. A red filled cell indicates a failure of the sampler flow rate verification and requires the application of the W qualifier flag to the VOC data validation spreadsheet for all VOCs detected in the sample.
21. If a null code or qualifier flag has been applied do not apply the W qualifier flag. If one or more qualifier flags have already been applied add the W flag as shown here: "SP,LB,4,3,MX,QX,W".
22. Examine the report for compound names listed in column N. Compounds listed in this column failed to meet cleanliness criteria in the clean canister batch certification sample and requires the application of qualifier flag CC to the data validation spreadsheet for the VOCs detected in the sample.
23. If a null code has already been applied, do not apply the CC qualifier flag. If one or more qualifier flags have already been applied, add the CC flag as shown here: "SP,LB,4,3,MX,QX,W,CC".
24. Examine the report for samples that were not analyzed by the laboratory. These samples must be added to the data validation spreadsheet.
25. If a sample name is listed in the COC form summary report that was not analyzed by the laboratory, manually add the sample name, VOC run folder, sample site, and sample date to the data validation spreadsheet as shown in Figure 10 below:

Figure 10: Adding a Sample not Analyzed by the Laboratory

Sample Date	Sample Site	VOC Run Folder	Sample #	Flag 1,1,1-Trichloroethane 43814	Flag 1,1,2,2-Tetrachloroethane 43	Flag 1,1,2-Trichloroethane 43820	Flag 1,1-Dichloroethane 43813	Flag 1,1-Dichloroethene 43826
3/30/2022	Millbrook	04-01-22 system II	033022M Millbrook Raleigh sample					
3/30/2022	Millbrook D	04-01-22 system II	033022MD Millbrook Raleigh duplicate sample					
4/1/2022	LAB cert	04-01-22 system II	Can batch cer # T-1270 can # 29960					
4/1/2022	LAB LCS	04-01-22 system II	1 ppb TO-15 standard can # S6341					
4/1/2022	LAB SSCV	04-01-22 system II	4 ppb TO-15 Secondary Source standard can # S6349					
3/12/2022	Huntersville	04-01-22 system II	031222H	AN	AN	AN	AN	AN

26. The comment section in the COC form summary report should explain why the sample was not analyzed. Apply a null code to all VOCs that best describes the reason for the missed sample. Using the example in figure 10, this sample was missed due to a canister/sampler issue. Apply the AN null code to the data validation spreadsheet for all VOC parameters in the sample.
27. If the comment section of the COC form summary report does not provide an explanation for the unanalyzed sample, locate the canister COC form, and examine this form for operator

comments that would help identify the reason for the missed sample. Contact the laboratory analyst and/or the site operator if the reason for the missed sample cannot be determined.

28. Add a comment to column FB in the data validation spreadsheet with the null codes applied, the reason for the missed sample, and where to find the evidence for the decision to apply the null code. End the comment with the initials of the commenter and date the comment was added to the validation spreadsheet.
29. Using the example in figure 10, the comment for the 031222H missed sample should be “Apply AN to all VOCs due to sampler/canister issues (see COC form in the 04-01-22 system II run folder -sjw 5/5/22)”.
30. Update the control charts in the “urban vs rural” workbook tab.
31. When the typical trend is not observed, and the urban data point and rural data point are collected on the same date, and the VOCs being compared in the control chart are > 0.2ppb, contact the primary analyst to examine the chromatograms for the data points in question. If no adjustments can be made, apply qualifier flag “QX” to the VOC parameter(s) in the data validation spreadsheet for the urban and rural samples analyzed in the current run folder.
32. Additional validation checks are performed automatically in the data validation spreadsheet “VOC Data and AQS Flag” workbook tab.
33. Examine the VOC parameter ratio calculations in columns FN, FO, FP, and FQ and look for yellow filled cells. A yellow filled cell indicates a failure of the VOC parameter ratio and requires the addition of the QX qualifier flag to the VOC parameters tested in the ratio. These ratios include Benzene vs Toluene; m/p-Xylenes vs o-Xylene; Benzene vs Ethylbenzene; and n-Pentane vs Cyclopentane.
34. Before applying the QX qualifier flag to the VOC parameters that fail the ratio criteria, verify both concentration values of the VOC parameters tested in the ratio are > 0.2ppb. If the ratio fails and both concentrations are > 0.2ppb, apply the QX flag to both VOC parameters detected in the sample.
35. If a null code or QX qualifier flag has already been applied do not apply the second QX qualifier flag to the same VOC parameter. If one or more “non-QX” qualifier flags have been applied add the QX flag as shown: “SP,LB,4,3,MX,QX. See figure 11 below for an example of a VOC parameter with several qualifier flags applied to the measurement.

Figure 11: Example of Multiple Qualifier Flags Applied to a VOC Parameter

Sample Date	Sample Site	VOC Run Folder	Sample #	Flag Chloroethane 43812
3/12/2022	Asheville	03-25-22 system II	031222A Asheville sample	LB,MX
3/12/2022	Candor	03-25-22 system II	031222C Candor sample	LB,MX
3/12/2022	Garinger	03-25-22 system II	031222G Garinger Charlotte sample	LB,MX
3/12/2022	Pitt Ag	03-25-22 system II	031222P Pitt Ag Greenville sample	LB,MX
3/12/2022	Eagle Island	03-25-22 system II	031222B Eagle Island Wilmington sample	LB,MX
3/18/2022	Asheville	03-25-22 system II	031822A Asheville sample	LB,MX
3/18/2022	Candor	03-25-22 system II	031822C Candor sample	LB,MX
3/18/2022	Millbrook	03-25-22 system II	031822M Millbrook Raleigh sample	LB,3,MX
3/18/2022	Millbrook D	03-25-22 system II	031822MD Millbrook Raleigh duplicate sample	LB,3,MX
3/18/2022	Pitt Ag	03-25-22 system II	031822P Pitt Ag Greenville sample	LB,4,MX
3/18/2022	Eagle Island	03-25-22 system II	031822B Eagle Island Wilmington sample	LB,MX

36. After the application of null codes and qualifier flags due to sampling and analysis issues is complete the “Overall Precision (CV)” workbook tab in the data validation spreadsheet can be updated with primary and collocated sample collection and analysis precision data.
37. The primary and collocated precision data is added to rows 2-45. Insert additional rows as needed for the number of primary and collocated sample pairs.
38. Examine the embedded formulas and conditional formats in every third row starting with row 4 and ending with row 46.
39. Examine the embedded formulas and conditional formats in rows 49-86. These formulas pool the calculated RPDs and perform a coefficient of variation (CV) calculation to evaluate overall precision of the VOC canister sampling and analysis method. This method only works for VOC parameters that are detected in the primary and collocated canister samples.

6.0 INDEPENDENT ACCURACY AUDITS

Independent accuracy audits consist of proficiency testing (PT) and instrument performance audits (IPA). These assessments are also used to evaluate and control bias in the Urban Air Toxics (UAT) VOC canister sampling and analysis methods.

6.1 VOC Laboratory Analysis Proficiency Testing (PT)

Proficiency testing (PT) samples are used to assess bias in the UAT VOC canister analysis method. The DAQ LAB participates in the NATTS PT program. The DAQ LAB receives PT samples from an EPA approved contractor, analyzes the spiked canister, and submits the results to the EPA contractor.

At a minimum, two times per calendar year (actual frequency depends on availability of EPA contractor supplies) the DAQ LAB ships out a certified clean field sample canister under vacuum to the approved EPA contractor. The EPA contractor spikes the canister with a known concentration of VOCs that is only known to the EPA and the EPA contractor. The DAQ does not know the nominal VOC concentration or whether the VOC was spiked in the PT canister. The PT canister is analyzed alongside other routine field samples.

The results of the PT canister are reported to the EPA contractor by the Level 3 reviewer and a report is generated by the EPA contractor comparing the reported results against designated NATTS laboratory average results, the referee laboratory average results, and the nominal VOC concentration spiked in the PT canister.

6.2 Instrument Performance Audit (IPA)

The Xontech 911 VOC canister sampler flows are verified before and after each sample event by the site operator using a NIST traceable flow meter. The sampler flow must be maintained between 8-12mL/min. The site operators are instructed to adjust the variable flow rate controller when flow rate verifications fall outside the acceptable limit and may also adjust the flow rate if the measured flow is approaching the limit but has not yet exceeded the limit.

In addition to these pre and post sampling flow rate verifications, the Level 3 reviewer performs an instrument performance audit once per calendar year and every 365 days thereafter using a NIST traceable flow meter that is different from the flow meter used to perform routine pre and post sampling flow rate verifications.

The result of the IPA is logged in the site logbook. The IPA should be performed on a sample setup date while the site operator is present so the level 3 reviewer and site operator flow rate results can be compared.

7.0 PRECISION CHECK DATA

VOC precision is measured in two ways. The first precision measurement is derived from replicate analysis of a field-collected sample during an analysis sequence (also known as duplicate injection analysis). This precision evaluation only tests VOCs that are detected in the field sample and may not evaluate all VOC parameters. To counter this gap in precision evaluation, the laboratory control sample is also analyzed twice during an analysis sequence so that all VOCs in the standard mix can be evaluated for precision.

The second precision measurement is derived from the primary and collocated sample pairs collected in the UAT VOC canister network (also known as overall method precision). The Millbrook VOC canister site is the designated primary and collocated precision site. The VOC concentrations detected in the primary sample are reported to AQS using POC **1** and the VOC concentrations detected in the collocated sample are reported to AQS as POC **8**.

Please see figure 12 below for the precision equation. For all VOC parameters, the CV must be ≤ 15%.

Figure 12: Formula for Calculating CV

$$CV = \sqrt{\frac{\sum_{i}^{n}\left[\frac{(p-r)}{0.5*(p+r)}\right]^2}{2n}}$$

p = primary value
r = replicate, repeated or collocated value
n = number of pairs and n must be ≥ 2

When a single pair of precision data are being evaluated, the RPD is calculated using part of the formula bracketed in figure 11. This equation is simply defined as the **difference/average** and the RPD must be ± 25% for individual pairs of precision data. The equation is as follows: ***RPD = [(p-r)/(0.5*(p+r))]*100***

Within 90 days of the conclusion of the calendar quarter, the Level 3 reviewer prepares and sends a UAT VOC Sampling and Analysis Precision (CV) Report representing the CV for each VOC parameter for the calendar quarter to DAQ management and staff directly involved in UAT operations. This report includes CVs calculated from the primary and collocated sample collection pair RPDs and provides a visual pass-fail status using green and red conditional formats to highlight VOC parameters that meet precision criteria and VOC parameters that do not meet precision criteria.

8.0 AIR QUALITY SYSTEM TRANSACTION FILE GENERATION

The level 3 reviewer generates pipe delimited transaction files which are used to upload UAT VOC canister data to EPA's AQS.

8.1 Raw Data Transaction File Generation

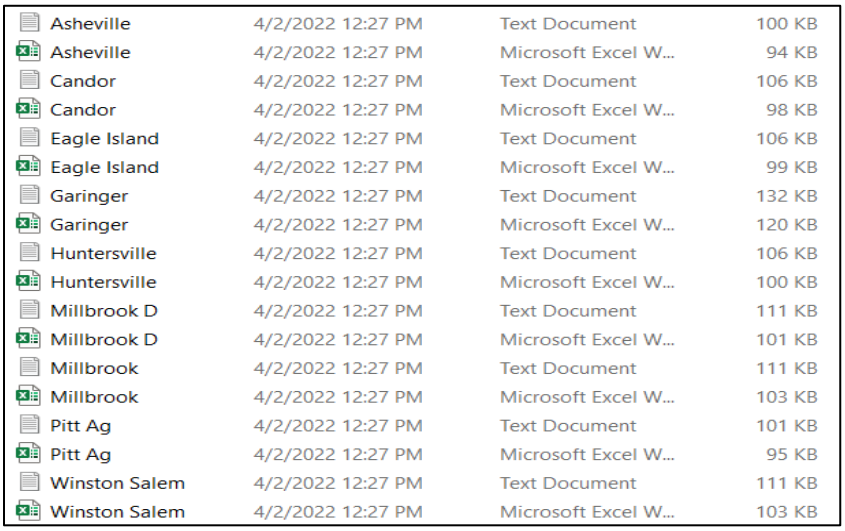
The data validation spreadsheet is a macro-enabled workbook and has an embedded macro that automatically transforms the data in the "VOC Data and AQS Flag" workbook tab into individual Excel files and tab delimited text files. The excel files are then reviewed for accuracy and edited as needed. The edited files are saved in a manner that does not overwrite the original files generated by the macro. Once the files have been generated, reviewed, and edited, the Level 3 reviewer contacts the data base manager to inform him or her that UAT VOC canister data is ready for AQS upload. These transaction files are uploaded to AQS within 90 days from the end of the calendar quarter.

1. Open the data validation spreadsheet for the quarter of VOC data that is ready for AQS upload.
2. Examine the "VOC Data and AQS Flags" workbook tab and verify the sample dates of the field samples match the EPA national sampling schedule for 1-in-6 day sampling.
3. Each VOC site must report something to AQS for each EPA national sampling schedule sample date. Missed samples must be reported but with a null code that explains the reason for the missed sample. Samples that are taken on dates outside the 1-in-6 EPA national sampling schedule are still reported to AQS, but an error warning will be sent by AQS at the completion of the file loading process. The error does not prevent any data from being accepted by AQS

and only warns the user of the reported sample date not matching the EPA national sampling schedule.

4. Examine the sample site names and make sure that no spaces are present before or after the value entered in the sample site cell. Highlight the sample site column and toggle the left align and right align to check the presence of spaces. Remove the spaces if found. Spaces will cause the macro to create multiple files for the same site which is ok and can be corrected later if some spaces are missed during this process.
5. Once all sample dates for the quarter are present and spaces in the sample site names are removed, the data is ready for transformation into AQS ready files.
6. Click the "view" tab and select "macros" and then "view macros."
7. The macro named "KevinOursFunction3" should be highlighted.
8. Select "edit" to open visual basic and allow the user to manually set the path for the macro to save the generated target files.
9. Set the path in the macro to a location on your computer's C: drive. An example file path for 1st quarter 2022 data: **C:\AQS DATA\VOC\2022\Q1**. The file folder in the path may have to be created before the macro is ran to make sure the path is found. If the macro cannot find the path it will fail to generate the AQS ready files.
10. After the path is set, save the data validation spreadsheet.
11. Click "view" and then select "macro" and select "view macros."
12. Highlight the KevinOursFuntion3 macro and click "run."
13. The macro may take several minutes up to 30 minutes to complete depending on how much data is being transformed.
14. When the macro completes the following files shown in figure 13 below will be generated in the path applied to the macro.

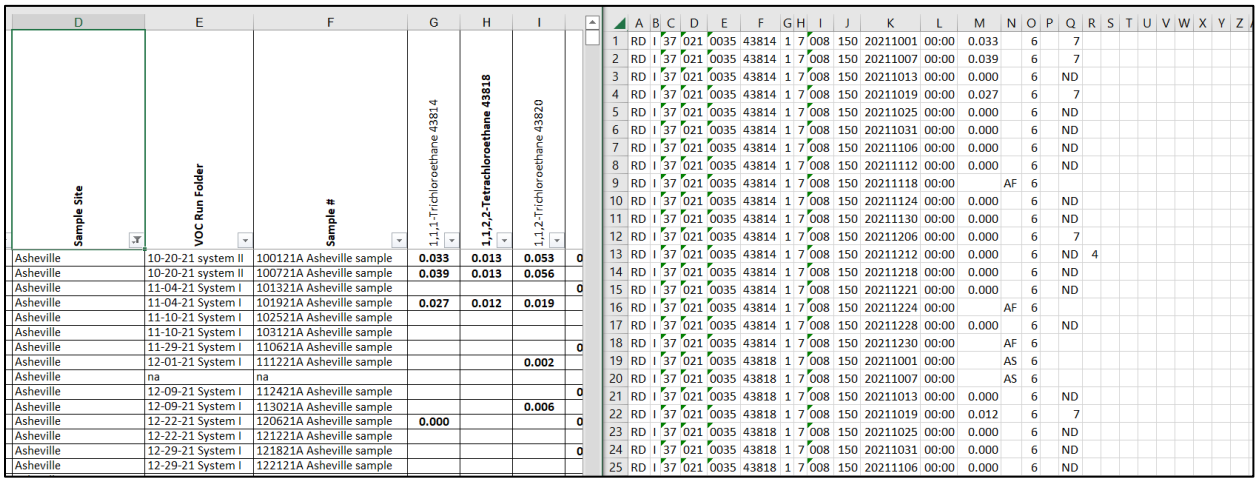
Figure 13: Macro Output File List



Asheville	4/2/2022 12:27 PM	Text Document	100 KB
Asheville	4/2/2022 12:27 PM	Microsoft Excel W...	94 KB
Candor	4/2/2022 12:27 PM	Text Document	106 KB
Candor	4/2/2022 12:27 PM	Microsoft Excel W...	98 KB
Eagle Island	4/2/2022 12:27 PM	Text Document	106 KB
Eagle Island	4/2/2022 12:27 PM	Microsoft Excel W...	99 KB
Garinger	4/2/2022 12:27 PM	Text Document	132 KB
Garinger	4/2/2022 12:27 PM	Microsoft Excel W...	120 KB
Huntersville	4/2/2022 12:27 PM	Text Document	106 KB
Huntersville	4/2/2022 12:27 PM	Microsoft Excel W...	100 KB
Millbrook D	4/2/2022 12:27 PM	Text Document	111 KB
Millbrook D	4/2/2022 12:27 PM	Microsoft Excel W...	101 KB
Millbrook	4/2/2022 12:27 PM	Text Document	111 KB
Millbrook	4/2/2022 12:27 PM	Microsoft Excel W...	103 KB
Pitt Ag	4/2/2022 12:27 PM	Text Document	101 KB
Pitt Ag	4/2/2022 12:27 PM	Microsoft Excel W...	95 KB
Winston Salem	4/2/2022 12:27 PM	Text Document	111 KB
Winston Salem	4/2/2022 12:27 PM	Microsoft Excel W...	103 KB

15. Open each excel file and examine the file for accuracy by comparing the parameter codes, sample dates, concentration values, and null codes and qualifier flags in the output files against the data validation spreadsheet.
16. Open the “Asheville” excel file and in the data validation spreadsheet filter the site names in the “VOC Data and AQS flag” for all Asheville samples.
17. Spot check the concentrations, sample dates, parameter codes, and flags in both files for accuracy. See figure 14 below for an example of this comparison:

Figure 14: Macro Output File vs Validation Spreadsheet Comparison



Sample Site	VOC Run Folder	Sample #	1,1,1-Trichloroethane 43814	1,1,2,2-Tetrachloroethane 43818	1,1,2-Trichloroethane 43820	
Asheville	10-20-21 system II	100121A Asheville sample	**0.033**	**0.013**	**0.053**	0
Asheville	10-20-21 system II	100721A Asheville sample	**0.039**	**0.013**	**0.056**	
Asheville	11-04-21 System I	101321A Asheville sample				0
Asheville	11-04-21 System I	101921A Asheville sample	**0.027**	**0.012**	**0.019**	
Asheville	11-10-21 System I	102521A Asheville sample				
Asheville	11-10-21 System I	103121A Asheville sample				
Asheville	11-29-21 System I	110621A Asheville sample				0
Asheville	12-01-21 System I	111221A Asheville sample			**0.002**	
Asheville	na	na				
Asheville	12-09-21 System I	112421A Asheville sample				0
Asheville	12-09-21 System I	113021A Asheville sample			**0.006**	
Asheville	12-22-21 System I	120621A Asheville sample	**0.000**			0
Asheville	12-22-21 System I	121221A Asheville sample				
Asheville	12-29-21 System I	121821A Asheville sample				0
Asheville	12-29-21 System I	122121A Asheville sample				

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z
1	RD	I	37	021	0035	43814	1	7	008	150	20211001	00:00	0.033		6		7									
2	RD	I	37	021	0035	43814	1	7	008	150	20211007	00:00	0.039		6		7									
3	RD	I	37	021	0035	43814	1	7	008	150	20211013	00:00	0.000		6		ND									
4	RD	I	37	021	0035	43814	1	7	008	150	20211019	00:00	0.027		6		7									
5	RD	I	37	021	0035	43814	1	7	008	150	20211025	00:00	0.000		6		ND									
6	RD	I	37	021	0035	43814	1	7	008	150	20211031	00:00	0.000		6		ND									
7	RD	I	37	021	0035	43814	1	7	008	150	20211106	00:00	0.000		6		ND									
8	RD	I	37	021	0035	43814	1	7	008	150	20211112	00:00	0.000		6		ND									
9	RD	I	37	021	0035	43814	1	7	008	150	20211118	00:00		AF	6											
10	RD	I	37	021	0035	43814	1	7	008	150	20211124	00:00	0.000		6		ND									
11	RD	I	37	021	0035	43814	1	7	008	150	20211130	00:00	0.000		6		ND									
12	RD	I	37	021	0035	43814	1	7	008	150	20211206	00:00	0.000		6		7									
13	RD	I	37	021	0035	43814	1	7	008	150	20211212	00:00	0.000		6		ND	4								
14	RD	I	37	021	0035	43814	1	7	008	150	20211218	00:00	0.000		6		ND									
15	RD	I	37	021	0035	43814	1	7	008	150	20211221	00:00	0.000		6		ND									
16	RD	I	37	021	0035	43814	1	7	008	150	20211224	00:00		AF	6											
17	RD	I	37	021	0035	43814	1	7	008	150	20211228	00:00	0.000		6		ND									
18	RD	I	37	021	0035	43814	1	7	008	150	20211230	00:00		AF	6											
19	RD	I	37	021	0035	43818	1	7	008	150	20211001	00:00		AS	6											
20	RD	I	37	021	0035	43818	1	7	008	150	20211007	00:00		AS	6											
21	RD	I	37	021	0035	43818	1	7	008	150	20211013	00:00	0.000		6		ND									
22	RD	I	37	021	0035	43818	1	7	008	150	20211019	00:00	0.012		6		7									
23	RD	I	37	021	0035	43818	1	7	008	150	20211025	00:00	0.000		6		ND									
24	RD	I	37	021	0035	43818	1	7	008	150	20211031	00:00	0.000		6		ND									
25	RD	I	37	021	0035	43818	1	7	008	150	20211106	00:00	0.000		6		ND									

18. Repeat the spot check for all output files. If changes are made to these output files, save the edited file using a new name by adding a version number to the end of the file name. After the spot check, combine the unedited and/or edited macro-output files reported by DAQ into one file and name this file “UAT VOCs_1Q22 All DAQ Sites” and save this file in the same location as the path set in the macro.
19. Examine the combined file. Highlight column O and then right click the mouse and select “clear contents”. Do not delete the column or any column from A to AC because deleting a column causes the pipe file to be formatted improperly. Qualifier flags may not get uploaded to AQS if columns are deleted.
20. To correct instances of improperly qualified concentration values = 0.000 select all the data and perform a custom sort first by column M, then by column Q, then by column R, then by column S, and by column T. A warning window pops up select ok.
21. For all concentrations = 0.000 a “ND” flag must also be reported in column Q in the same row. If the ND flag is missing add it to column Q and remove all other qualifier flags reported in the row. ND flags cannot be reported to AQS with other qualifier flags in the same row. Also, the ND flag must have a conc. = 0.000.
22. For all concentrations between 0.001 and 0.200 a 7-qualifier flag should be reported in column Q. 0.2ppb should not be reported with a 7-qualifer so pay close attention to the concentrations that = 0.199ppb, 0.200ppb, and 0.201ppb to be sure the 7 flag is applied correctly.

23. For all concentrations > 0.000 multiple qualifier flags can be reported so if multiple qualifier flags are reported the concentration value must be > 0.000.
24. For all concentrations > 10.0ppb a qualifier "EH" should be reported in column Q. Add EH to column Q as needed.
25. After adjusting the flags perform the custom sort again to verify that all ND flags are reported when conc. values = 0.000 and that no other qualifier flags are reported in the same row as a ND flag. Also verify 7 flags are reported to conc. values between 0.001 and 0.200. Also verify EH flags are reported to conc. values > 10.0.
26. Check the null concentrations in column M and make sure that a null code is also reported in the same row but in column N. Make sure that rows containing null codes do not contain qualifier flags in the same row. Null codes and qualifier flags cannot be reported for the same row.
27. Remove all the custom sort options except for one and perform a custom sort by column F only.
28. Remove rows with the following parameter codes: 43302, 43305, 43312, 43503, 43504, 43510, 43517, and 43551.
29. Custom sort the file by column D, E, G, F, and K. Save the excel file and then save the excel file as a "tab delimited" text file.
30. Close the excel file and open the tab delimited text file.
31. Highlight the space between RD and I and select copy.
32. Select "edit" and "replace" to open the edit and replace window.
33. In the "find what" field, paste the recently copied space and make sure only space is entered in this field. Multiple spaces in this field will cause the pipe file to be formatted improperly and will cause some qualifier flags to not be reported to AQS.
34. In the "replace with" field add the pipe character.
35. Select "replace all."
36. Next in the "find what" field enter "end."
37. In the "replace with" field, leave it blank and make sure nothing is entered even spaces.
38. Select "replace all."
39. Scroll to the bottom of the file and remove excess rows and pipes that do not contain data.
40. Save the pipe delimited file using the following naming convention, using 1st quarter 2022 as the example: "RDTX1Q22_VOCsAlldaqSites."
41. Repeat steps 19-40 with the AQS output files reported to AQS by Mecklenburg county. These files are Garinger and Huntersville. When naming the pipe files remove "AlldaqSites" and add "Garinger" then perform the same naming for the Huntersville site file.
42. Create a folder on the p-drive to store all the files generated during the macro process and any edited files. Create a folder in this location: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\AQS Test\1Q_22**. Using 1st quarter 2022 data as the example. Adding these files to the p-drive ensure the files characterizing the data transformations are properly backed up.
43. Add the RDTX pipe files to the manual upload folder on the p-drive and contact the database manager with the file names and location of the file names that are ready for AQS upload.
44. The database manager uploads the files and will provide a LOAD report from AQS if any errors or warnings are found by the AQS upload process.

9.0 ROUTINE TROUBLESHOOTING AND CORRECTIVE ACTIONS

Routine troubleshooting and corrective actions are described in the following sub sections.

9.1 Data Validation Issues

9.1.1 Level 1 and Level 2 Data Reviews

During data validation, the Level 3 reviewer may discover issues from the Level 1 and Level 2 data reviews. The Level 3 reviewer contacts the Level 1 and Level 2 reviewer for issue correction. Reports or checklists that may be edited/changed/altered during this process must be saved in a manner, so the original report or checklist is preserved along with the corrected report or checklist. This is typically achieved by adding a "version #" or "update #" to the end of the original file name.

9.1.2 Correcting Data Uploaded to AQS

If AQS errors are discovered, correction must be made by the Level 3 reviewer. The documentation of the data correction is recorded in the "AQS Audit Report and Data Update Form 9.0." Contact the PPB chemist responsible for this form and request a blank copy. Fill in all fields of the form that are applicable to the correction. See Appendix 11.2 for an example of this form and some of the fields.

The best practice is to find the raw, output file generated by the data validation macro that contains the error and resave the file with "update1" added to the file name. Make the corrections to this file and re-format the file to AQS pipe delimited format. Save the file with "update 1" added to the end of the file name and place the updated file in the manual upload folder on the p-drive and contact the database manager to upload the corrected file to AQS. Request the database manager to send the LOAD report if one is generated.

9.2 NATTS PT Issues

VOC compounds reported between two consecutive failing PT sample assessment dates must be qualified "QX,4" in AQS. This action may require data already uploaded to AQS to be updated with the "QX,4" qualifier flags. Follow guidance in section 9.1.2 to update the AQS data. These qualifier flags must remain on the data until a passing PT result is achieved.

Immediately following the NATTS PT failure, the Level 3 review prepares an "in-house" PT sample. The Level 3 reviewer spikes a certified clean canister with a known amount of primary calibration standard mix.

The "in-house" PT sample is handed directly to the primary VOC analyst to be analyzed in the next sequence. The level 3 reviewer generates and sends an "in-house PT sample results report" to the DAQ management and staff directly involved in the VOC sampling and analysis method. This report summarizes the results of the "in-house" PT and provides and "pass/fail" indicator for each VOC parameter spiked in the canister. If the VOC parameter spiked in the in-house PT sample passes, application of the "QX,4" flag is not required as described in this sub section.

9.3 Instrument Performance Audit Issues

In the event of a failing IPA, the Level 3 reviewer locates a second NIST flow standard and performs the IPA again. If the IPA passes, the original flow meter used by the Level 3 reviewer may need to be

serviced. If the IPA continues to fail, the sampler must be removed from the field for service and recertification before being re-deployed to the field. The failing sampler can be replaced by a sampler that's been recently certified as clean. Perform an IPA on the newly installed sampler.

When a failing IPA is confirmed, the Level 3 reviewer must flag all VOCs reported back to the last passing IPA with "QX,W" to indicate the samples were collected using a sampler that recently failed an IPA. This may require updates to data already reported to AQS so follow instructions in section 9.1.2 for updating data already reported to AQS.

9.4 Primary and Collocated Sample Collection Pair RPD Issues

When three consecutive VOC parameters detected in sufficient amounts (>0.200 ppb) from primary and collocated sample collection pairs fail the RPD acceptable limits (± 25%); a corrective action report (CAR) must be generated to determine the root cause of the systematic failure of the precision RPD. The CAR is generated according to **SOP# DAQ-15-002**.

10.0 REVISION HISTORY

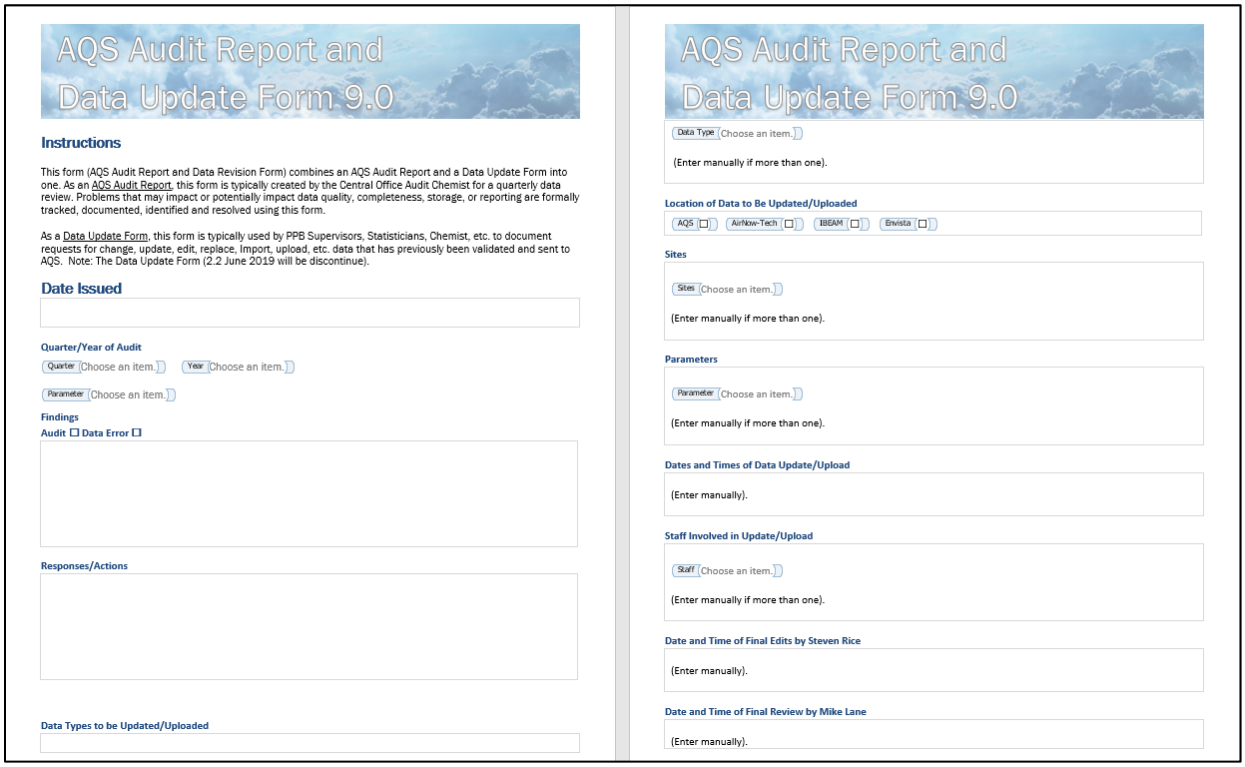
1. Revision 0, SJW 5/13/2022

11.0 APPENDICES

11.1 UAT VOC Precision Summary Report

UAT VOC Sampling and Analyis Precision (CV)		
Peak Table (Bold = NATTS Core I and Target PT compounds)	**4Q21 CV**	**Comment**
1,1,1-Trichloroethane 43814	#DIV/0!	could not be calculated insufficient number of valid pairs
1,1,2,2-Tetrachloroethane 43818	#DIV/0!	could not be calculated insufficient number of valid pairs
1,1,2-Trichloroethane 43820	5.89%	
1,1-Dichloroethane 43813	#DIV/0!	could not be calculated insufficient number of valid pairs
1,1-Dichloroethene 43826	#DIV/0!	could not be calculated insufficient number of valid pairs
1,2,3-Trimethylbenzene 45225	1.67%	
1,2,4-Trichlorobenzene 45810	#DIV/0!	could not be calculated insufficient number of valid pairs
1,2,4-Trimethylbenzene 45208	9.29%	
1,2-Dibromoethane 43843	#DIV/0!	could not be calculated insufficient number of valid pairs
1,2-Dichlorobenzene 45805	#DIV/0!	could not be calculated insufficient number of valid pairs
1,2-Dichloroethane 43815	9.73%	
1,2-Dichloropropane 43829	3.86%	
1,3,5-Trimethyl Benzene 45207	1.66%	
1,3-Butadiene 43218	7.23%	
1,3-dichlorobenzene 45806	#DIV/0!	could not be calculated insufficient number of valid pairs
1,4-dichlorobenzene 45807	7.31%	
1,4-Dioxane 46201	#DIV/0!	could not be calculated insufficient number of valid pairs
1-Bromopropane 43853	5.43%	
1-Butanol 43305	#DIV/0!	could not be calculated insufficient number of valid pairs
2-Pentanone 43562	3.62%	
3-Hexanone 43557	10.74%	
3-Pentanone 43553	8.45%	
Acetaldehyde 43503	#DIV/0!	could not be calculated insufficient number of valid pairs
Acetone 43551	3.36%	
Acetonitrile 43702	9.26%	
Acrolein 43505	1.15%	
Benzene 45201	3.52%	
Benzyl chloride 45809	#DIV/0!	could not be calculated insufficient number of valid pairs
Bromodichloromethane 43828	#DIV/0!	could not be calculated insufficient number of valid pairs
Bromoform 43806	#DIV/0!	could not be calculated insufficient number of valid pairs
Bromomethane 43819	#DIV/0!	could not be calculated insufficient number of valid pairs
Carbon disulfide 42153	#DIV/0!	could not be calculated insufficient number of valid pairs
Carbon Tetrachloride 43804	6.73%	
chlorobenzene 45801	#DIV/0!	could not be calculated insufficient number of valid pairs
Chloroethane 43812	#DIV/0!	could not be calculated insufficient number of valid pairs
Chloroform 43803	3.16%	
Chloromethane 43801	6.15%	
Cis-1,3-dichloropropene 43831	#DIV/0!	could not be calculated insufficient number of valid pairs
Cyclohexane 43248	3.35%	
Cyclopentane 43242	8.95%	
Ethanol 43302	#DIV/0!	could not be calculated insufficient number of valid pairs
Ethylbenzene 45203	3.44%	
Freon 11 43811	2.61%	
Freon 113 43821	9.91%	
Freon 114 43208	#DIV/0!	could not be calculated insufficient number of valid pairs
Freon 12 43823	4.04%	
Freon 22 43359	1.64%	
Hexanal 43517	#DIV/0!	could not be calculated insufficient number of valid pairs
Hexane 43231	3.24%	
Iodomethane 43808	#DIV/0!	could not be calculated insufficient number of valid pairs
Isobutene 43270	2.58%	
Isoprene 43243	14.30%	
Isopropyl Alcohol 43312	5.50%	
m,p-xylene45109	2.35%	
Methacrolein 43515	14.47%	
Methyl Butyl Ketone 43559	3.18%	
Methyl Ethyl Ketone 43552	9.84%	
Methyl Isobutyl Ketone 43560	#DIV/0!	could not be calculated insufficient number of valid pairs
Methyl Tert Butyl Ether 43372	#DIV/0!	could not be calculated insufficient number of valid pairs
Methyl Vinyl Ketone 43558	11.78%	
Methylene Chloride 43802	6.78%	
n-Butanal 43510	2.55%	
n-Pentane 43220	5.39%	
o-Xylene 45204	4.02%	
Propanal 43504	10.22%	
Propylene 43205	3.03%	
Styrene 45220	2.95%	
Tetrachloroethylene 43817	9.86%	
Toluene 45202	2.87%	
Trans-1,2-Dichloroethene 43838	5.85%	
Trans-1,3-dichloropropene 43830	9.96%	
Trichloroethylene 43824	#DIV/0!	could not be calculated insufficient number of valid pairs
Vinyl Acetate 43447	2.38%	
Vinyl Chloride 43860	#DIV/0!	could not be calculated insufficient number of valid pairs

11.2 AQS Audit and Data Update Form



AQS Audit Report and Data Update Form 9.0

Instructions

This form (AQS Audit Report and Data Revision Form) combines an AQS Audit Report and a Data Update Form into one. As an AQS Audit Report, this form is typically created by the Central Office Audit Chemist for a quarterly data review. Problems that may impact or potentially impact data quality, completeness, storage, or reporting are formally tracked, documented, identified and resolved using this form.

As a Data Update Form, this form is typically used by PPB Supervisors, Statisticians, Chemist, etc. to document requests for change, update, edit, replace, Import, upload, etc. data that has previously been validated and sent to AQS. Note: The Data Update Form (2.2 June 2019 will be discontinue).

Date Issued

Quarter/Year of Audit

Quarter Choose an item. Year Choose an item.

Parameter Choose an item.

Findings

Audit ☐ Data Error ☐

Responses/Actions

Data Types to be Updated/Uploaded

AQS Audit Report and Data Update Form 9.0

Data Type Choose an item.

(Enter manually if more than one).

Location of Data to Be Updated/Uploaded

AQS ☐ AirNow-Tech ☐ IBEAM ☐ Envista ☐

Sites

Sites Choose an item.

(Enter manually if more than one).

Parameters

Parameter Choose an item.

(Enter manually if more than one).

Dates and Times of Data Update/Upload

(Enter manually).

Staff Involved in Update/Upload

Staff Choose an item.

(Enter manually if more than one).

Date and Time of Final Edits by Steven Rice

(Enter manually).

Date and Time of Final Review by Mike Lane

(Enter manually).

12.0 REFERENCES

1. EPA Compendium Method TO-15, second addition, January 1999: https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-15r.pdf

2. National Air Toxics Trends Station, Technical Assistance Document, Revision 3, October 2016: https://www3.epa.gov/ttn/amtic/files/ambient/airtox/NATTS%20TAD%20Revision%203_FINAL%20October%202016.pdf

3. Technical Assistance Document for Sampling and Analysis of Ozone Precursors for PAMS, Revision 2, April 2019: https://www.epa.gov/sites/default/files/2019-11/documents/pams_technical_assistance_document_revision_2_april_2019.pdf