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DAQ-03-003.2 Standard Operating Procedure (SOP)
For the Markes-Agilent GC-MS Analysis of Volatile Organic Compounds
in Ambient Air Collected in 6-Liter Canisters
North Carolina Division of Air Quality (DAQ)
Operator Responsibilities



1.0 Approval Sign-Off Sheet

I certify that I have read and approve of the contents of DAQ-03-003.2 Standard Operating Procedure For the Markes-Agilent GC-MS Analysis of Volatile Organic Compounds in Ambient Air Collected 6-Liter Canisters, Operator Responsibilities, with an effective date of May 25, 2022.

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Disclaimer:

This document, and any revision hereto, is intended solely as a reference guide to assist operators in the successful operation of VOC analysis equipment and VOC sampling media related to the North Carolina Division of Air Quality's (DAQ) Ambient Monitoring Program. Mention of products, services, or materials does not constitute endorsement or requirement for use by DAQ.

SOP Acronym Glossary

AQS - Air Quality System

BFB – Bromofluorobenzene

COC – Chain of Custody

DAQ - North Carolina Division of Air Quality

°C – Degrees Celsius

EPA – United States Environmental Protection Agency

GC – Gas Chromatograph

inHg – inches of mercury

MDL – Method detection limit

MS – Mass Spectrometer

NATTS – National Air Toxics Trends Station

NIST – National Institute of Standards and Technology

ppb – part per billion

QA – Quality assurance

QC – Quality control

psig – Pounds Per Square Inch Gauge

RRF – Relative Response Factor

RT – Retention Time

SOP - Standard operating procedure

UAT – Urban Air Toxics

UHP – Ultra High Purity

USB – Universal Serial Bus

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2.0 SCOPE AND PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the steps followed by the Laboratory Analysis Branch (LAB) chemist to successfully analyze volatile organic compounds (VOCs) in ambient air samples collected in 6-Liter canisters.

Summary of Method:

Ambient air is collected in 6-Liter silica lined canisters using a Xontech 911 sampler. The sampling procedure for air samples is detailed in the Xontech 911 sampler SOP. All the operational procedures and sampling conditions for each sample are documented in the field using the chain of custody (COC) sampling information form. The COC form is generated at the LAB after a batch of canisters has been cleaned and certified. The form accompanies each canister to the field sample site. The field sampling information is recorded on the form and sent back to the VOC laboratory. The sample is then analyzed according to this document.

The ambient air samples are introduced into the analytical system from a 6-Liter silica lined canister with the aid of a mass flow controller (MFC) and a vacuum system. A digital readout attached to the MFC provides a visual indication of the proper sample flow during sampling. Automated sampling of up to 16 canisters can be accomplished using the system's multi-position stream selector valve.

The VOCs in the ambient air sample are concentrated for injection onto the gas chromatograph (GC) column by means of a Markes CIA Advantage and Markes Unity pre-concentrator using a micro-scale purge and trap method, and MARKES Kori Xr sample water management system.

The sample flows through the Kori Xr water management system and is slowly flushed with helium to deliver the VOC's to a second Tenax[®] trap which has been cooled to -30 °C, leaving most of the water behind. The second trap allows the CO₂ in the sample to pass through it and out to the pump. The VOCs are then thermally desorbed at 275 degrees Celsius (°C). The focuser is then rapidly heated to inject the sample, and the GC is started.

The sample mixture is separated into individual components by their interaction with the capillary column's stationary phase, using temperature-programmed gas chromatography. A single quadrupole mass spectrometer (MS) ionizes the eluting compounds, which are then detected, identified, and quantified. Identification of a compound in a sample is based upon both the retention time and the mass spectral matching. The response of the primary quantitation ion is used to quantify the peak of interest. Quantitation is accomplished via a six-point calibration curve. A standard of known concentration for TO-15 target compounds is prepared and introduced to the analytical system in concentrations of 0.2, 0.5, 1.0, 2.0, 5.0, and 10.0 ppb for the required points of the calibration curve. Unknown samples are analyzed in relation to this curve. Each sample is also analyzed with a known volume of an internal standard mix.

3.0 INSTRUMENT CHECKS

The following instrument checks are performed on the GC-MS instrument and support equipment.

3.1 List of Equipment and Materials

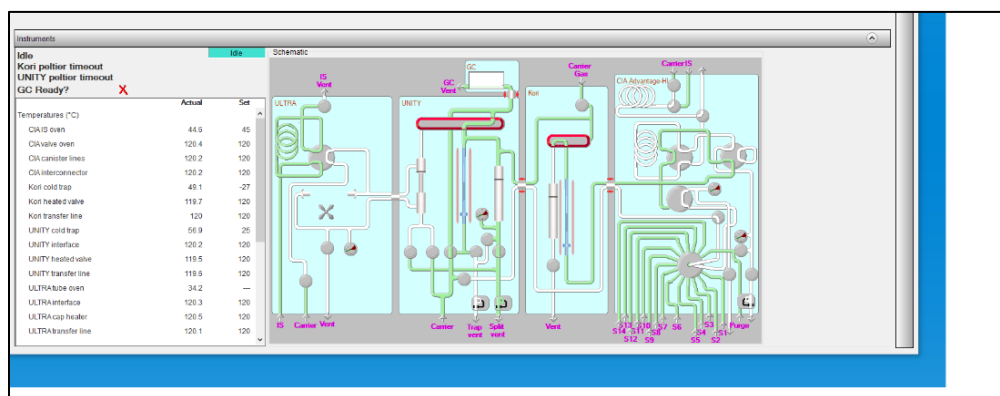
- GC-MS Instrument modules
- Computer and software
- 6 Liter (L) Silica Lined Canisters
- Wrenches, tools
- VOC COC forms
- Instrument logbook
- National Institute of Standards and Technology (NIST) traceable pressure gauge
- VOC Standard, Zero-Air, and Ultra High Purity (UHP) Nitrogen cylinders
- Entech Dilution System

3.2 Markes Equipment Checks

The Markes software controls the Kori Xr water management system; the CIA Advantage autosampler tree; and the Ultra Xr thermal desorber.

1. Check the pressure of the UHP nitrogen cylinder. The pressure should be ≥ 400 pounds per square inch gauge (psig).
2. Install a new cylinder of nitrogen if the pressure is below the limit.
3. Turn on each unit of the Markes Unity, i.e., Kori Xr, CIA Advantage, and the Ultra Xr.
4. Click on the **M TD MIC 2.0** to activate the **MARKES software control** window. The Markes Instrument Control window will open. **Workflow** will appear with **Method editor**, **Sequence**, **Sequence History**.
5. **Tools** window with **Instrument configuration**, **Setting**, **About**.
6. **Instruments** window will indicate **running or idle**.
7. See display on the Markes instrument control editor window to ensure that the MARKES Unity Xr is connected and ready. **Figure 1** below shows the Markes instrument control editor.

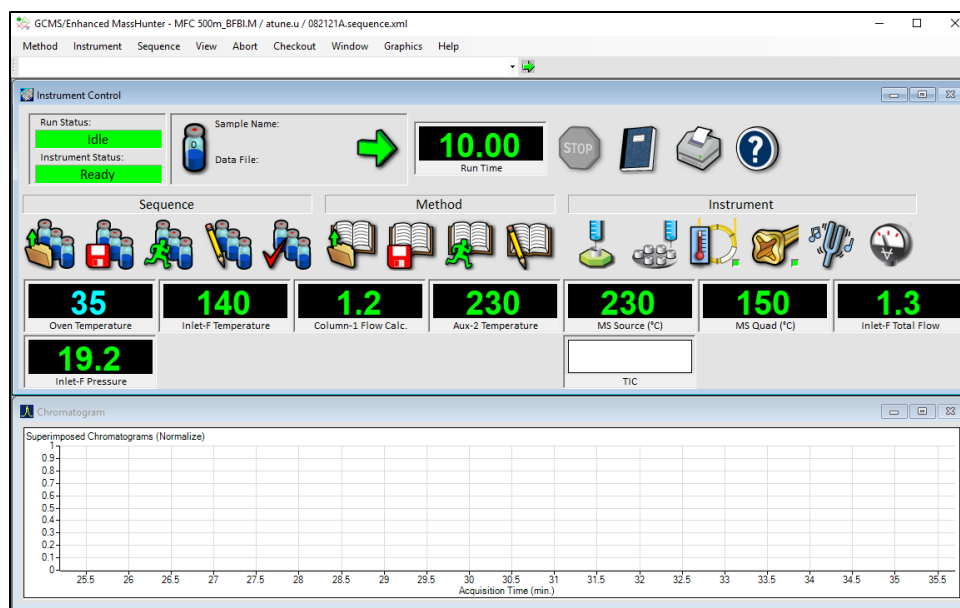
Figure 1: Markes instrument control editor



3.3 Agilent GC-MS Checks

1. Check the carrier gas (helium) cylinder; the gauge pressure should be ≥ 400 psig.
2. Check the internal standard gas cylinder; make sure the tank contains ≥ 200 psig and the line pressure is set to 40 psig.
3. Load the method file which will contain the temperature program, carrier gas flow, and column type for the method.
4. See **figure 2** below showing the Gas Chromatograph – Mass Spectrometer (GC-MS) instrument software control panel.

Figure 2: GC-MS Instrument Software Control Panel



3.3.1 GC-MS Tuning

To ensure the Mass Spectrometer (MS) detector is properly tuned, 4-Bromofluorobenzene (BFB) is injected into the detector. The internal standard cylinder used by the lab contains the BFB tuning compound which means BFB is added with every injection. The results of the first injection of the method blank are used for tune evaluation. The following criteria must be met before the instrument can be calibrated and tuning criteria must be met prior to sample analysis.

Table 1: GC-MS Tuning Criteria

Mass	Ion Abundance Criteria
50	15.0% to 40.0% of m/z 95
75	30% to 60% of mass 95
95	Base Peak, 100% Relative Abundance

Table 1: GC-MS Tuning Criteria

Mass	Ion Abundance Criteria
96	5% to 9% of mass 95
173	≤2% of mass 174
174	50% to 100% of mass 95
175	5% to 9% of mass 174
176	95% to 101% of mass 174
177	5% to 9% of mass 176

3.3.2 GC-MS Internal Standard

An aliquot of an internal standard mix of Hexane-d14, 1,4-Difluorobenzene, Chlorobenzene-d5, and 4-Bromofluorobenzene is added to all GC-MS analysis injections. The internal standard is connected to the Markes system which also controls how much internal standard is added to the injection and when the internal standard is added to the injection. The results of the internal standard compound relative response factors (RRFs) measured with every injection are tracked to evaluate matrix effects of field-collected samples.

3.3.2 GC-MS Cleanliness

The second injection of the method blank is used to assess overall background contamination contribution from the GC-MS, internal standards, and sample canisters. The method blank is made using a certified clean canister that is filled with humidified zero-air employed from the dilution system. The dilution system is used to generate GC-MS calibration standards and GC-MS calibration verification standards.

The method blank is analyzed twice at the beginning of each analytical sequence.

3.3.3 GC-MS Calibration

A six-point instrument calibration (ICAL) curve is generated at the following nominal levels: **0.2ppb**, **0.5ppb**, **1.0ppb**, **2.0ppb**, **5.0ppb**, and **10.0ppb**. The best fit line applied to the calibration curve level results must be **linear** or **quadratic** and each calibration level must meet accuracy requirements.

3.3.4 GC-MS Calibration Verification and Continuing Calibration

The GC-MS calibration verification is performed by analyzing a canister filled with a known amount of target VOCs made from the second source cylinder. This sample is analyzed immediately after calibration standard levels are injected and in the same analysis sequence. This sample is also analyzed at least once in analysis sequences that contain field collected samples. This quality control (QC) sample targets a nominal concentration in the mid-range of the calibration curve and measures overall calibration curve accuracy.

Additionally, GC-MS calibration verification is performed by analyzing a canister filled with a known amount of target VOCs made from the primary source cylinder. This sample is also called the laboratory control sample. This QC sample is analyzed at the beginning and end of each analysis sequence that contains field collected samples. This QC sample targets a nominal concentration in the lower-third range of the calibration curve and measures overall calibration curve accuracy and overall GC-MS precision for all target VOCs.

3.3.5 VOC Compound List

The VOC compounds analyzed by the laboratory are listed in **Table 2**. Bold font compound names indicate Core I and target proficiency testing (PT) compounds for the National Air Toxics Trends Station (NATTS) network.

Compound names in italic font and underlined are not reported to the Environmental Protection Agency's (EPA's) air quality system (AQS) using the VOC analysis method. Acetone, 1-butanol, isopropyl alcohol, ethanol, acetaldehyde, hexanal, n-butanol, and propanal are not reported to AQS due to persistent issues exhibited by the GC-MS instrument for these compounds.

Acetaldehyde, hexanal, n-butanol, and propanal are collected, analyzed, and reported using AQS method 202 (Carbonyls by TO-11A).

All VOCs in the calibration standard mix and the calibration verification standard mix are listed in **Table 2**. Additionally, the primary and secondary qualifier ions are listed for reference.

Table 2: VOC Compound List

Peak Table	Calibration Range		Qualifier Ions	
	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
1,3-Butadiene 43218	0.2	10.0	39	54
1,3-dichlorobenzene 45806	0.2	10.0	146	148

Peak Table	Calibration Range		Qualifier Ions	
	Lower Limit (ppb)	Upper Limit (ppb)	Primary	Secondary
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
<u>1-Butanol 43305</u>	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
<u>Acetaldehyde 43503</u>	0.2	10.0	44	29, 43
<u>Acetone 43551</u>	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
<u>Ethanol 43302</u>	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
<u>Hexanal 43517</u>	0.2	10.0	56	44
Hexane 43231	0.2	10.0	57	41, 43
Iodomethane 43808	0.2	10.0	142	127

Peak Table	Calibration Range		Qualifier Ions	
	Lower Limit (ppb)	Upper Limit (ppb)	Primary	Secondary
Isobutene 43270	0.2	10.0	41	56
Isoprene 43243	0.2	10.0	67	53
<i>Isopropyl Alcohol 43312</i>	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
<i>n-Butanal 43510</i>	0.2	10.0	44	72
n-Pentane 43220	0.2	10.0	43	41
o-Xylene 45204	0.2	10.0	91	106
<i>Propanal 43504</i>	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

Table 2 Note: Bold font peak names = NATTS Core I and PT compounds. Italic and underlined peak names = compounds not reported to AQS using this method (150) or are not reported to AQS due to excessive analysis QC issues.

3.4 Method Sensitivity

Sensitivity is determined using the method detection limit (MDL) determination guidance described in Section 4.1.3.1 of the NATTS Technical Assistance Document Revision 3.

The MDL is determined from the analysis of at least seven separate MDL blank canisters and seven separate MDL spike canisters. The statistical method used to calculate the MDL are described in more details in SOP# DAQ-03-003.5. The following criteria must be met for the MDL to be considered valid:

- The canisters designated MDL spikes and MDL blanks must be pulled from at least 3 different canister cleaning batches.
- The cleaning batches cannot occur on consecutive days of the week.

- The analysis of the MDL samples must take place in at least three different analysis batches.
- The analysis batches cannot occur on consecutive days of the week.
- At least seven separate MDL blank canister results and seven separate MDL spike canister results must be included in the MDL computations.
- MDLs must be performed once per calendar year and every 365 days thereafter or whenever instrument adjustments or maintenance affects detector sensitivity.

3.5 Canister Cleanliness

All canisters used for the collection of pressurized ambient air and creation of QC standards are cleaned according to SOP# DAQ-03-005.2.

3.6 Sampler Cleanliness

Samplers used for canister sampling are cleaned according to SOP# DAQ-03-001.1.

4.0 SITE CHECKS

This section is reserved. Site checks do not apply to analysis instruments housed in a secure laboratory facility.

5.0 DETAILED PROCEDURES

The following sections describe detailed procedures for shipping unsampled canisters, receiving sampled canisters, installation of canisters onto instrument auto-sampler, sampled canister analysis sequence development, instrument calibration curve sequence development, and data processing

5.1 Unsampled Canister Shipping

Canisters must be certified clean before shipping to the field. Additionally, these canisters must be under vacuum (≥ 28 inHg vacuum) prior to field deployment and verified using a NIST traceable certified pressure gauge.

1. Obtain a blank air sampling canister COC form for each canister being shipped. The main shipping boxes are heavy duty metal boxes that hold two canisters. At a minimum, ship two canisters to the field site. If a single canister is being shipped, use the cardboard shipping box designed for a single canister shipment.
2. Assign a sampling site and record the sample site on the air sampling canister COC form
3. Assign a sample # using the EPA national sampling schedule date and the site name abbreviation. For example, if the canister is being shipped to **Candor** for the **1/29/2022** EPA national sample date, the **sample#** is **012922C**. Reference **Table 3** below describing the site name and abbreviation.

Table 3 Site Names and Abbreviations

Site Name	Abbreviation
Asheville	A
Candor	C
Winston Salem	W
Millbrook	M
Millbrook Collocated	MD
Eagle Island	B
Pitt Ag	P
Garinger	G
Huntersville	H

4. Record the canister# on the COC form and the cleaning batch number.
5. Attach a NIST certified pressure gauge to the canister. The pressure gauge units should be in "inHg vacuum".
6. Open the canister valve and allow a few seconds to elapse to stabilize the pressure gauge reading. The absolute value of the displayed gauge pressure reading should be ≥ 28 inHg vacuum. Do not send the canister to the field if the canister pressure is < 28 inHg vacuum. If the canister fails this pressure criteria, obtain another canister and repeat steps 1-6.
7. If the canister pressure passes criteria, record the pressure gauge reading on the COC form.
8. Close the canister valve and remove the pressure gauge.
9. Install a quick connect on the canister inlet if quick connects are in use at the field site or cap the canister using the attached canister inlet cover
10. Obtain a canister valve cover and a COC seal.
11. Place the valve cover over the canister and place the COC seal in the cover and snap the COC seal closed.
12. Record the COC seal number on the COC form and record the initials of the person performing the canister shipping and the date and time the canister valve was sealed on the COC form.
13. Tear off the bottom copy of the COC form for storage in the laboratory.
14. Label the outside of the shipping box with the assigned sample number using a permanent marker and tape.
15. Obtain one shipping label and one return shipping label for each shipping box being sent to the field.
16. Place the canister, assigned COC form, and return shipping label in the shipping box.
17. Seal the metal shipping boxes using the attached clips and straps. Seal the cardboard shipping box using packing tape.
18. Coordinate a shipping pickup with the preferred shipping carrier.
19. Update the shipping dates on the current year's canister sample tracking chart for all sites and sample dates shipped to the field. If needed, reference the sites and sample dates on the gold copies of the COC forms when updating the VOC canister sample tracking chart.

5.2 Sampled Canister Receiving

Samples are delivered to the laboratory in metal shipping boxes or cardboard shipping boxes via Federal Express and/or United Parcel Service. The metal shipping boxes can transport two canisters and the cardboard shipping boxes transport one canister.

1. Open the shipping box and remove the canister(s) and COC form(s).
2. One COC form should be present for each canister in the shipping box. If the shipment is missing a COC form, contact the site operator that shipped the canisters to the laboratory and attempt to recover the COC form.
3. Examine the COC form to make sure the **Pre-Sample (Lab)** and the **Sample Setup and Collection (Field)** sections are completely filled out. Also ensure the hand-written entries are legible. Contact the site operator for assistance when correcting illegible and/or missing entries.
4. Verify the canister manufacturer number stamped on the canister matches the canister number recorded on the COC form and the canister number recorded on the canister tag. Place a check mark next to the canister number field if they match and physically circle the canister number if they don't match and place a comment on the COC form such as "canister # issue."
5. Verify the sample # recorded on the COC form matches the site name and sample date recorded on the COC form and verify the sample# on the COC form matches the sample# on the canister tag.
6. If the sample date does not match the sample # or if the COC form and canister tag do not match, check the COC form for site operator comments. If the deviation can be explained, correct the sample # on the COC form and canister tag to match the sample date on the COC form. If no comments are present, contact the site operator to clarify the mismatched sample# and sample date issue.
7. Verify the canister valve COC seal is intact, undamaged, and the COC number on the form matches the number on the canister. Note deviations in the COC form comments section.
8. If no deviations are observed, place a check mark in the **Canister COC seal intact** and **Same # as sealed** boxes on the COC form.
9. Enter the initials of the person checking-in the sample and the date and time the sample was received in the **Sample Received by** field on the COC form.
10. Place the COC sealed canister and COC form on the GC-MS analysis laboratory staging table.
11. Update the received dates on the canister sample tracking chart for all samples received.

5.3 Canister Install on CIA Advantage Autosampler

5.3.1 Field Sample Canisters

1. Break the VOC canister COC seal and record the initials of the person breaking the COC seal and the date and time the COC is broken in the **COC Broken by Initial, Date & Time** field on the COC form.
2. Measure the canister pressure using the NIST traceable certified pressure gauge in the units of psig and record this pressure in the **Canister Pressure psig (Lab)** field on the COC form.

3. Install canister on the Markes autosampler tree and record the port number, sample #, and canister number in the instrument logbook.
4. Repeat steps 1-3 for all samples being analyzed.

5.3.2 Calibration Standard Canisters and Quality Control Canisters

Calibration standard canisters and QC canisters do not have a COC form. The dilution system logbook contains the written record of when the calibration standard canisters and QC canisters were made; what concentration of VOCs were added to the calibration standard canister and QC canister; and the type of standard mix and diluent gas added to the canister. Additional details on making calibration canisters and QC canisters are described in SOP # DAQ-03-006.2.

5.4 Analytical Sequences

Analysis sequences are created using the Markes instrument control software and the GC-MS instrument control software. There are three types of sequences: calibration curve sequences, field sample sequences, and if needed, certification sample sequences. Regardless of the sequence type, all sequences must contain the following QC samples:

- **Method blank** (routine field sample canister filled with zero-air from the dilution system and filled using the dilution system).
- **Lab Control Sample or Continuing Calibration** (canister filled with primary calibration standard mix targeting a nominal concentration in the lower third of the calibration curve range, typically 1.0ppb).
- Calibration curve sequences and field sample sequences require the additional analysis of the **Second Source** standard (canister filled with secondary source standard mix targeting a nominal concentration in the upper-third of the calibration curve range, typically 4.0ppb)

5.4.1 Calibration Curve Sequences

Calibration sequences must contain, at a minimum, the following injections in chronological order by injection date/time:

1. **method blank** (BFB tune criteria only)
2. **method blank** (instrument and sampling media background)
3. **calibration curve standards** (0.2ppb, 0.5ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb)
4. **lab control sample or continuing calibration** (calibration curve accuracy)
5. **second source** (calibration curve accuracy)

5.4.2 Field Sample Sequences

Field sample sequences must contain, at a minimum, the following injections in chronological order by injection date/time (#6 is not required if 10 or less field samples are being analyzed in the field sample sequence):

1. **method blank** (BFB tune criteria only)
2. **method blank** (instrument and sampling media background)
3. **lab control sample or continuing calibration** (accuracy)

4. **field samples** not to exceed 10 consecutive injections and must include at least one **replicate injection** of a field sample (analysis precision)
5. **second source sample** (calibration verification)
6. **field samples** (no more than 10 consecutive injections)
7. **method blank** (instrument and sampling media background)
8. **lab control sample or continuing calibration** (same as #3 and used for accuracy and precision)

5.4.3 Certification Sample Sequences

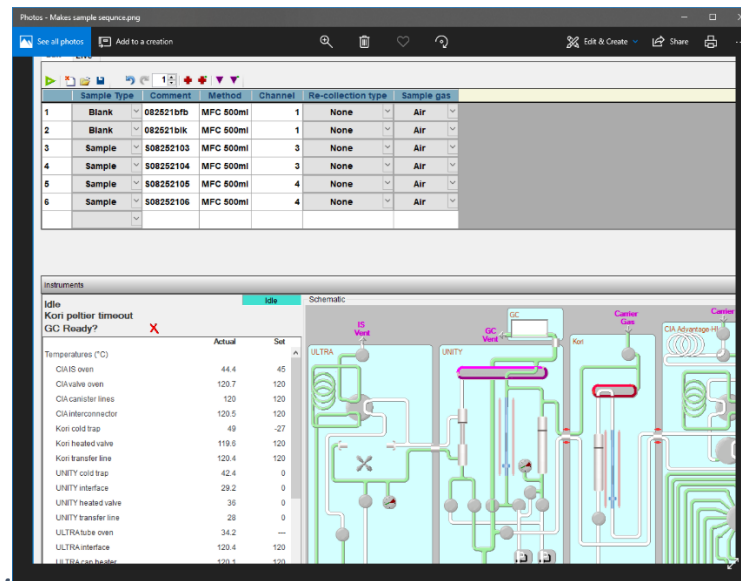
Certification samples include canister cleaning batch certification canisters and clean sampler certification canisters. The results of these two certification samples are used to assess canister cleanliness and sampler cleanliness. It is encouraged to analyze certification samples alongside routine field sample canisters but due to the immediate need for certification sample results, certification samples can be analyzed by themselves or with other certification samples if an insufficient number of field samples are ready for analysis. When certification samples are analyzed by themselves or with other certification samples, the following injections must be included (chronological order by injection date/time):

1. **method blank** (BFB tune criteria only)
2. **method blank** (instrument and sampling media background)
3. **lab control sample** (accuracy)
4. **batch cert** or **sampler cert** canisters (not to exceed 10 consecutive)
5. **lab control sample** (accuracy)

5.4.4 Analysis Sequence Creation

1. Click the **MTD MIC 2.0** icon which activates the **Markes instrument control** window to open.
2. Select the **Sequence** icon to allow the operator to enter information into the sequence window which includes the sample list which should correlate with the sample list on the Sequence list on the Instrument Control on the GCMS sample list. See figure 3 below for the Markes sequence editor screen.

Figure 3: Markes Sequence Editor Screen



3. In the **Sample Types** select either sample, blank, unknown, standard or quality control.
4. Enter the sample # in the **Comment** field.
5. Click **method** and select the method named "MFC 500ml".
6. The **Channel** refers to the Absorbent tubes in the Ultra Xr, or the port on the CIA Advantage on the canister auto-sampler tree. Enter the port number into the Markes sequence.
7. For the **Re-collection type** select "none" to force Markes to sample from the canister tree.
8. For the **Sample gas** select "Air".
9. Next click the "GCMS icon to open the **GCMS\Enhanced MassHunter** software. See **figure 4** below for an example of the Masshunter sequence editor window.

Figure 4: Masshunter Sequence Editor Window

Name	Vial	Method Path	Method File	Data Path	Data File	Type	Level
1 Cont. ac blank sample	1	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221fb	Blank	
2 Zero ac blank sample	1	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221bk	Blank	
3 1 ppb TO-15 standard	2	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215c	CC	1.0
4 1 ppb TO-15 standard	2	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215c	CC	1.0
5 080221A Asheville sa...	3	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221A	Sample	
6 080221B Eagle Islan...	4	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221B	Sample	
7 080221A Asheville sa...	5	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221A	Sample	
8 080221P Pitt Ag Gr...	6	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221P	Sample	
9 081121G Gangee C...	7	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081121G	Sample	
10 080821H Huntersvill...	8	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080821H	Sample	
11 080821B Eagle Islan...	9	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080821B	Sample	
12 081121C Carder sa...	10	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081121C	Sample	
13 081421G Gangee C...	11	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081421G	Sample	
14 081421G Gangee C...	11	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081421G	Sample	
15 081421H Huntersvill...	12	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081421H	Sample	
16 081421M Millbrook R...	13	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081421M	Sample	
17 081421D Millbrook R...	14	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	081421D	Sample	
18 080221P Pitt Ag Gr...	3	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221P	Sample	
19 0.2 ppb TO-15 stand...	4	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215-2	Sample	
20 0.2 ppb TO-15 stand...	4	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215-2	Sample	
21 Zero ac blank sample	1	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	080221bk	Blank	
22 1 ppb TO-15 standard	2	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215d	CC	1.0
23 1 ppb TO-15 standard	2	D:\Ma...methods	MFC 500m_BFB.M	D:\MassHunter\GCMS1\data\080221A	0802215d	CC	1.0

10. Click Sequence and select "edit" sequence.
11. Select save as and save the sequence with today's run date and add an "A" at the end of the file name indicating the first sequence analyzed for the current day. If a second sequence is made and started on the same day, save the sequence with "A1" on the end of the file name.
12. The sequence table opens and can now be edited.
13. The NAME column must include sample# and site name.
14. The VIAL should match the vial number in the logbook and the Markes sequence.
15. The METHOD PATH must be D:\MassHunter\GCMS\1\methods.
16. The METHOD FILE must be MFC 500m_BFB.
17. The DATA PATH must be selected by clicking the box to the right of the path name and selecting the sequence name that was just created and saved.
18. The DATA FILE is manually entered for field sample injection and should match the sample# in the NAME field.
19. The DATA FILE is manually entered for QC injections (blanks, ICAL, and check standards). The data file name will be MMDDYY with "bfb" on the end for tuning samples and "S4" on the end for the 4ppb check standard. The duplicate injection data file name has a "-" added to the end. ICAL standard data file names have "cal1", "cal2", "cal3", "cal4", "cal5", and "cal6" added to the name. Samples receive the sample # and site abbreviation.
20. The TYPE should be blank for the bfb injection and method blank injections; CC for all check standards; and sample for all field collected samples. ICAL standard type should be cal.
21. Compare the GC sequence to the Markes sequence.
22. If no errors are found save the GC sequence again.
23. Select the run sequence icon.
24. The start sequence window will open. Select "run sequence" using the GC software.
25. Next start the Markes sequence by clicking the green arrow on the Markes Instrument Control Sequence editor window.

5.5 GC-MS Data Processing

These are the steps for processing the analysis data following the completion of the analysis sequence.

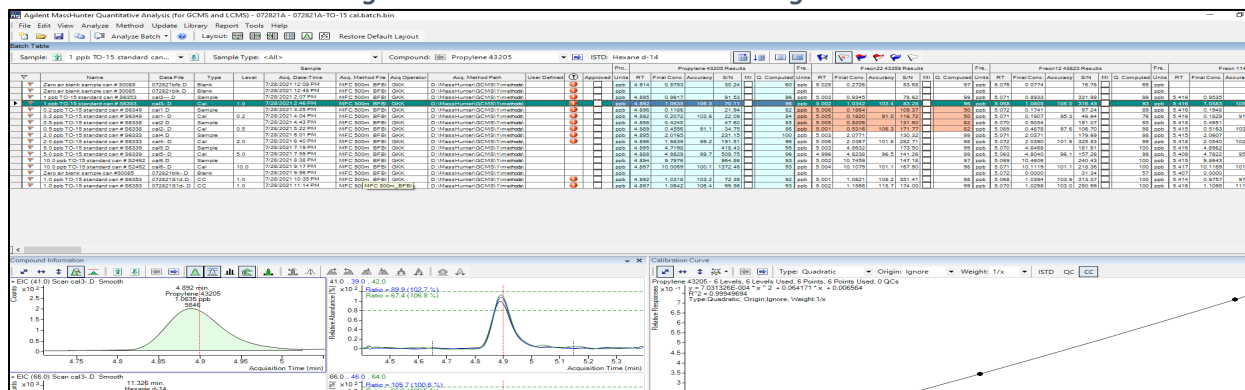
5.5.1 Processing a New Calibration Curve

1. Open the MS Quantitation icon on the desktop. The Agilent MassHunter Quantitative Analysis (for GCMS and LCMS) window opens.
2. Click file and select new batch.
3. Select the sequence folder name that was recently saved and analyzed that also contains the calibration standard injection results. The sequence folder is in the MMDDYYA format.
4. Click method and then select new method from acquired scan with library search.
5. Use the 1ppb curve level results to perform the library search and verify retention times of the standard mix.
6. Save the calibration curve with a new name such as MMDDYY TO-15cal. This file name will be used to apply existing calibration curve results to future analysis sequences.

5.5.2 Processing Samples Using an Existing Calibration Curve

1. Open the MS Quantitation icon on the desktop. The Agilent MassHunter Quantitative Analysis (for GCMS and LCMS) window opens.
2. Click File and select new batch.
3. Select the sequence folder name that was recently saved and analyzed.
4. Type in a file name MMDDYYA. The MMDDYY must match the analysis date the sequence was analyzed. Click create to start a new data processing batch.
5. The Add Sample window opens; click ok.
6. Go to method and select open.
7. Open method from existing file and select the most recent/current calibration file name MMDDYY TO15cal and select open.
8. Select Analyze batch and a screen will pop up as shown in Figure 5.

Figure 5: MassHunter Data Processing Window

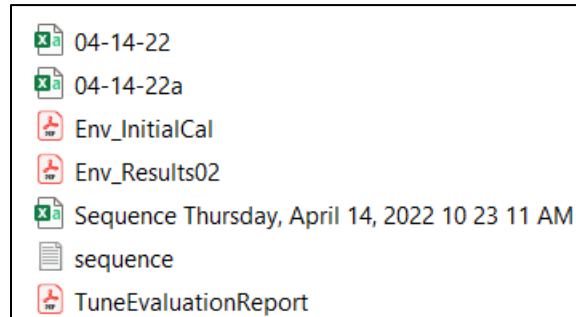


9. Examine the processing batch for flags and outliers. Perform manual integration of each injection and peak if necessary. Typically, the instrument software does a good job properly identifying and integrating target VOC peaks.

5.6 Generating Run Folder Files for Calibration Curve and Field Sample Sequences

The files listed in figure 6 are generated using the GC-MS instrument control software and Markes instrument control software. All files in figure 6 are generated for each hardcopy version of the current run folder and the electronic version of the current run folder except for the two files “04-14-22” and “04-14-22a”.

Figure 6: Run Folder Files



These two files are not formatted to print correctly and therefore not included in the hardcopy version of the current run folder.

1. After processing the GC-MS data, obtain one empty file folder and label it with the current run folder name such as “04-14-22 system II”. Also, create an electronic copy of the run folder by creating a new folder on the p-drive here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data** and naming newly created file folder as “04-14-22 system II”.
2. Using the GC-MS instrument computer, select report from the MassHunter pulldown menu.
3. Go to export table to generate the csv files (04-14-22 and 04-14-22a).
4. Choose compound table and flat table for the two csv files.
5. Select generate on the top line. Save the csv files and transfer them to the electronic copy of the current run folder.
6. Go to report method and Choose **C:\user\DAQ\Document\sample_results Tic.m** to generate the “Env_Results02” report for all injections in the sequence.
7. Go to report method and choose **C:\user\DAQ\Document\ICAL** to generate the “Env_InitialCal” report.
8. Select process to generate the reports. Print the Env_Results and Env_InitialCal reports and store them in the hardcopy version of the current run folder and save an electronic copy of these reports in the electronic version of the current run folder.
9. To generate the Markes sequence file **Sequence Thursday, April 14, 2022 10 23 11 AM**, open the Markes control software and on the sequence history select export and choose a drive. Save the file in the current run folder on the p-drive and print the file.
10. To generate the **Sequence** .txt report go to the sequence pulldown menu on the MassHunter software and select view sequence log.
11. Open the sequence based on the mm/dd/yy of analysis. Save the file in the current run folder on the p-drive and print the sequence.

12. The **Tune Evaluation Report** is generated by selecting the tune evaluation shortcut on the GC-MS computer.
13. Select file pulldown menu and open to find the data file designated the BFB tune and select data.
14. Evaluate the BFB, select report, save the report in the current run folder on the p-drive, and print the report.
15. Gather the VOC Canister COC forms that were analyzed in the sequence and gather VOC Canister COC forms (recently received) that indicate a failed field-sample collection and will not be analyzed by the laboratory.
16. Scan these forms into a single pdf file or multiple pdf files and store these files in the **04-14-22 system II** run folder. If a single scan is created save the file as "VOC Canister COC forms_04-14-22 system II." For example, if five individual scans of the VOC canister COC forms are created, add text "1 of 5"; "2 of 5"; "3 of 5" ; "4 of 5" ; and "5 of 5" at end of the file name.

5.7 Generating Files for Certification Samples and Certification Sample Sequences

It is strongly recommended to analyze certification samples such as canister cleaning batch certification samples and clean sampler certification samples alongside routine field samples or calibration curve samples. Due to the immediate need for valid certification sample results, the certification samples may be analyzed by themselves or with other certification samples.

1. If a certification sample is analyzed in the same sequence as routine field samples or analyzed in the same sequence as the calibration curve samples or analyzed in a dedicated certification sample sequence with other certification samples, the "Env_Results02" report is generated and printed for each certification sample injection. Additionally, the "Env_Results02" report is generated for the method blank sample injection and lab control sample injections. Lastly, print the tune evaluation report.
2. All these files are reviewed by the analyst using an indelible ink pen, highlighter, or both.
3. Examine the tune evaluation report to ensure all criteria indicate a "pass" status. If the tune fails, the results obtained from an "out of tune mass spectrometer" are considered invalid and the samples should be re-analyzed after a passing tune is achieved.
4. If the tune passes, examine the printed method blank results report and circle or highlight concentration values > 0.2ppb and circle or highlight the compound name.
5. Examine the lab control sample results and circle or highlight concentration values not between 0.700 ppb and 1.30 ppb.
6. Examine the certification sample results for any VOC concentration > 0.2ppb.
7. If the detected VOC in the certification sample is > 0.2ppb and passes the method blank criteria and passes the lab control accuracy criteria, circle or highlight the concentration value and compound name in the certification sample results report.
8. If the detected VOC in the certification sample is > 0.2ppb and the same VOC is detected in the method blank sample with a concentration value > 0.2 ppb or the same VOC detected in the certification sample is not between 0.700 ppb and 1.300 ppb in the lab control sample, the detected VOC in the certification sample is considered invalid and does not count toward a failing status.

9. Do not circle or highlight VOC concentration or compound names detected in the certification sample that also fail method blank criteria or fail lab control accuracy criteria.
10. If the detected VOC in the certification sample is > 0.2ppb and passes method blank criteria and passes lab control accuracy criteria, the detection in the certification sample is considered valid and counts toward a failing status for batch of canisters being cleaned or the sampler being cleaned. Circle or highlight the VOC concentration and compound name in the certification sample results report.
11. Certification results that fail for one or more VOC compounds may still be used for field operations but the results for the failing VOC in the certification sample will be flagged if the same VOC is detected in the field sample collected using the batch of canisters or field samples collected using the sampler.

5.8 Quality Control Parameters and Summary Reports

VOC canister sampling data and VOC canister analysis data should meet quality control parameters listed in Table 4 below. To document the testing of these quality control parameters, the following summary reports are generated with each run folder:

- DAQ-03-020 VOC Canister COC Form QC Summary Report
- DAQ-03-022 System I VOC Canister Analysis QC Summary Report
- DAQ-03-023 System II VOC Canister Analysis QC Summary Report
- DAQ-03-024 Internal Standard Summary Report

Table 4: VOC Canister Sampling and Analysis Quality Control Parameters

Quality Control Parameter Description	AQS Flag or Code	Type	Criteria
VOC Canister Sampling Quality Control Parameters			
Pre-sampling canister vacuum/pressure	QX	QUALIFIER	Applied to all VOC detected in field-collected canisters with a pre-sampling vacuum reading < 28 inHg vacuum.
Post-sampling canister pressure	QX	QUALIFIER	Applied to all VOCs detected in field-collected canisters where the post sampling canister pressure is not between 12-30 psig.
Pre and post sampling flow rate verification	W	QUALIFIER	Applied to all VOCs detected in field-collected canisters where the pre or post flow average is not between 8-12 mL/min.
Clean canister Hold Time	QX	QUALIFIER	Applied to all VOCs detected in field-collected canisters with holding times > 30 days.
Lab verified canister pressure and Markes pre-sampling canister pressure	QX	QUALIFIER	Applied to all VOCs detected in field-collected canisters where the lab verified canister pressure or Markes pre-sampling canister pressure is ≤ 0.2 psig.

Quality Control Parameter Description	AQS Flag or Code	Type	Criteria
Sampled Canister Holding Time	QX	QUALIFIER	Applied to all VOCs detected in field-collected canisters with holding times > 30 days.
VOC Canister Analysis Quality Control Parameters			
Clean Canister Batch Certification	CC	QUALIFIER	Applied to VOCs detected in the certification sample > 0.2ppb. To be considered a valid detection in the certification sample, the failing VOC must pass method blank and accuracy criteria. Also apply the flag to same VOCs detected in canisters from the out-of-control cleaning batch.
Clean Sampler Certification (zero bias)	SB	QUALIFIER	Applied to VOCs detected in the certification sample > 0.2ppb. To be considered a valid detection in the certification sample, the failing VOC must pass method blank and accuracy criteria. Also apply the flag to same VOCs detected in canisters filled using the out-of-control sampler.
GC-MS Calibration Curve Type (linear or quadratic)	AT	NULL	Applied to VOCs with a calibration curve best fit line that is not linear or quadratic. Also apply the flag to same VOCs detected in field-sample canisters processed using the out-of-control calibration curve.
GC-MS Calibration Curve R ²	AT	NULL	Applied to VOCs with R ² < 0.995. Also apply the flag to same VOCs detected in field-sample canisters processed using the out-of-control calibration curve.
GC-MS Calibration Curve Level Accuracy	AT	NULL	Applied to VOCs outside ± 30% of nominal. Also apply the flag to same VOCs detected in field-sample canisters processed using the out-of-control calibration curve.
GC-MS Calibration Verification Accuracy (second source)	ST	NULL	Applied to VOCs outside ± 30% of nominal. Also apply the flag to same VOCs detected in field-sample canisters analyzed in the same analysis batch as the out-of-control second source standard accuracy.
Method Blank (Zero-air blank)	LB	QUALIFIER	Applied to VOCs detected in the method blank sample > 0.2ppb. Also apply the flag to the same VOCs detected in field-sample canisters that are analyzed in the same analysis batch as the out-of-control method blank.

Quality Control Parameter Description	AQS Flag or Code	Type	Criteria
GC-MS Laboratory Control Sample (continuing calibration) Accuracy	SP	QUALIFIER	Applied to VOCs outside $\pm 30\%$ of nominal. Also apply the flag to the same VOCs detected in field-sample canister that are analyzed in the same analysis batch as the out-of-control lab control sample accuracy.
GC-MS Laboratory Control Sample (continuing calibration) Precision	AX	NULL	Applied to VOCs when the RPD between the initial lab control sample VOC concentration and ending lab control sample VOC concentration exceeds $\pm 25\%$. Also apply flag to same VOCs detected in field-sample canisters that are analyzed in the same analysis batch as the out-of-control lab control sample RPD.
GC-MS Replicate Analysis (duplicate injection analysis) of a Field Sample	4	QUALIFIER	Applied to VOCs when the RPD between the initial field-sample VOC concentration and replicate field-sample VOC concentration exceeds $\pm 25\%$.
Primary/Collocated Sample Collection and Analysis Precision	3	QUALIFIER	Applied to all VOCs detected in the primary and collocated samples when the RPD between the primary and collocated results exceeds $\pm 25\%$.
Below Reportable Limit	7	QUALIFIER	Applied to all detected VOCs in field samples <0.2ppb
Above Reportable Limit	EH	QUALIFIER	Applied to all detected VOCs in field samples >10ppb
Internal Standard Response Factor	MX	QUALIFIER	The internal standard response factor must be within $\pm 40\%$ of the mean ICAL internal standard response factor. If the internal standard response factor is outside this limit, the detected VOCs in the sample will be qualified MX prior to reporting.
Internal Standard Retention Time Window	MX	QUALIFIER	Each internal standard retention time must be within ± 0.33 min of its mean calibration curve retention time. If an internal standard retention is outside this limit, the detected VOCs in the sample will be qualified MX prior to reporting.
Target VOC Retention Time Window	BH	NULL	Applied to VOCs detected in the sample when the VOC retention time is not between ± 0.5 min of its mean calibration curve retention time.

Quality Control Parameter Description	AQS Flag or Code	Type	Criteria
Target VOC Detection	ND	QUALIFIER	Applied to target VOCs when the GC-MS reports concentration values = 0 ppb or blank/null value.

5.8.1 VOC Analysis QC Summary Report

This report is generated using the processed GC-MS analysis data stored in the current run folder. The analysis QC summary reports are unique to the analytical system being used. The VOC Analysis QC Summary reports are assigned to a specific GC-MS system.

1. Open a blank "System II VOC Analysis QC Summary Report_Template" located here:
P:\Toxics\Urban Air Toxics\VOC's Current Year Data\Data Review Templates.
2. Select save as and rename the file by removing template and entering the current run folder name. For example, if the report is for the **04-14-22 system II** run folder, the summary report is named "System II VOC Analysis QC Summary Report_04-14-22 system II."
3. Create a sub folder named "data review" in the electronic version of the **04-14-22 system II** run folder and store the recently created summary report in the "data review" sub folder.
4. In the summary report there are four workbook tabs: ICAL Summary, Analysis Summary, Retention Time Summary, and QC Criteria. Data entry is required for the ICAL Summary, Analysis Summary and Retention Time Summary workbook tabs.
5. Add the current run folder name "04-14-22 system II" to the title of the Analysis Summary workbook tab.
6. Add the calibration curve run folder name to the title of the ICAL Summary workbook tab. In this example, the original calibration curve accuracy results are in the **03-09-22 system II new curve** run folder.
7. Open the **03-09-22 system II new curve** run folder and open the .csv format excel file named **03-09-22**.
8. Highlight cells A3 to A76 and select "sort A-Z" and a sort warning window will be displayed, select expand selection and click sort. This will sort all the data in the file in alphabetical order by target VOC peak name.
9. Open the file named "Env_InitialCal" located in the **04-14-22 system II** run folder.
10. Click "export pdf" then select "spreadsheet" and select "Microsoft excel workbook" then click "export" and a "save as" file window opens.
11. Select the **04-14-22 system II** run folder and save the exported file as "Env_InitialCal_04-14-22 system II."
12. The adobe export engine will process the file and open the excel version of the file.
13. In the exported file, highlight rows 92 to 165.
14. In the menu bar, deactivate the "merge & center" formatting for all the highlighted rows.
15. Highlight all data from cells A92 to L165 and select "sort A-Z."

16. Copy and paste the peak names from cells A92 to A165 as “paste special values” into the ICAL Summary workbook tab cells C4 to C77.
17. Copy and paste the “curve fit” column data from the exported excel file as “paste special values” into cells D4 to D77 of the ICAL Summary workbook tab.
18. Copy and paste the R-squared value from the exported excel file as “paste special values” into cells E4 to E77 of the ICAL Summary workbook tab.
19. Open the .csv format excel file named **03-09-22**. Highlight row 1 and select “wrap text.”
20. Locate the “0.2 ppb TO-15...” “final conc.” values, copy and “paste special values” into cells F4 to F77 of the ICAL Summary workbook tab. Repeat this process for the 0.5ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.ppb calibration curve levels.
21. Examine the response factors for null or blank values in the “Env_InitialCal_04-14-22 system II.”
22. If a null or blank response factor value is found, this means the point was removed from the calibration curve and results of the accuracy test do not apply. Enter the number of “null or blank” response factors for each target VOC into cells L4 to L77. For example, if two null or blank response factors are discovered for Propylene, enter “2” into cell L69 of the ICAL Summary workbook tab. If no blank or null response factors are discovered, enter “0” into cells L4 to L77 of the ICAL Summary workbook tab.
23. Locate the first “second source” injection sample name and copy and “paste special values” into cell M3 of the ICAL Summary workbook tab.
24. For the same injection, locate the “final conc.” values, copy and “paste special values” into cells M4 to M77 of the ICAL Summary workbook tab. Repeat steps 21 and 22 if there is a second injection of the secondary source standard, place these values into cells N3 and N4 to N77 of the ICAL Summary workbook tab.
25. Locate the first laboratory control sample injection and copy and “paste special values” the value in row 1 into cell O3 of the ICAL Summary workbook tab.
26. For the same injection, locate the “final conc.” values, copy and “paste special values” into cells O4 to O77 of the ICAL Summary workbook tab. Repeat steps 23 and 24 if there is a second injection of the laboratory control sample, place these values into cell P3 and P4 to P77 of the ICAL Summary workbook tab.
27. Locate the second method blank injection and copy and “paste special values” the value in row 1 into cell Q3 of the ICAL Summary workbook tab.
28. For the same injection locate the “final conc. values, copy and “paste special values” into cells Q4 to Q77 of the ICAL Summary workbook tab. The first method blank injection is used for BFB tuning criteria only. Repeat steps 25 and 26 if there is a third injection of the method blank, place these values into cell R3 and R4 to R77 of the ICAL Summary workbook tab.
29. Open the **04-14-22 system II** run folder and open the .csv format excel file named **04-14-22**.
30. Highlight cells A3 to A76 and select “sort A-Z”; a sort warning will be displayed; select expand selection and click sort. This will sort all data in the file in alphabetical order by target VOC peak name.
31. Copy and “paste special” the peak names from the .csv format excel file named **04-14-22** as values into cells C4 to C77 of the Analysis Summary workbook tab.

32. In the .csv format excel file named **04-14-22**, highlight row 1 and select “wrap text” to better read the sample names.
33. Locate the initial and final laboratory control sample injection in the .csv format excel file named **04-14-22** and paste the sample name and final conc. values in cells D3 and D4 to D77 (initial LCS) and cells E3 and E4 to E77 (final LCS) of the Analysis Summary workbook tab.
34. Locate the second source injection in the .csv format excel file named **04-14-22** and paste the sample name and final conc. values into cells G3 and G4 to G77. If a second, second source injection is present add these values to cells H3 and H4 to H77 of the Analysis Summary workbook tab.
35. Locate the second injection of the method blank in the .csv format excel file named **04-14-22** and paste the sample name and final conc values into cells I3 and I4 to I77. If a third, fourth or fifth method blank injection is found in the .csv format excel file named **04-14-22**, paste the sample names and final conc. values into cells J3 and J4 to J77, K3 and K4 to K77, and L3 and L4 to L77 of the Analysis Summary workbook tab.
36. Locate the replicate analysis or duplicate injection analysis pair of a field collected sample in the .csv format excel file named **04-14-22**. Paste the first injection results into cells M3 and M4 to M77 and paste the second injection results into cells N3 and N4 to N77 of the Analysis Summary workbook tab.
37. Locate the M and MD primary/collocated sample collection analysis pairs that contain the same sample dates in the .csv format excel file named **04-14-22**. For example, paste the “040521M” injection results into cells P3 and P4 to P77 of the Analysis Summary workbook tab and paste the “040521MD” injection results into cells Q3 and Q4 to Q77 of the Analysis Summary workbook tab. Repeat if a second, third or fourth pair of M and MD primary/collocated sample collection analysis pair are found in the .csv format excel file named **04-14-22** and paste values into the next group of M/MD pairs of the Analysis Summary workbook tab.
38. Fill out the Retention Time Summary workbook tab using the .csv format excel files, already sorted A-Z and named **03-09-22** and **04-14-22**.
39. The sample names and retention times from the calibration curve level injections in the .csv format file named **03-09-22** go into cells B1 to G1 and cells B3 to G76 of the Retention Time Summary workbook tab.
40. The sample names and retention times from the .csv format excel file named **04-14-22** go into cells K1 to AN1 and K3 to AN76 of the Retention Time Summary workbook tab.
41. Delete unused gray colored columns in the Retention Time Summary workbook tab.

5.8.2 Internal Standard Summary Report

This report evaluates the internal standard response factors and retention times in the current analysis batch against the mean calibration curve internal standard response factors and retention times. This report is generated using a macro that is built into the summary report.

1. Open a blank “Internal Standard Summary Report_Template” located here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\Data Review Templates**. Select save as and rename the file by removing template and entering the current run folder name. For example, if the

report is for the **04-14-22 system II** run folder, the report is named "Internal Standard Summary Report_04-14-22 system II" and stored in the "data review" subfolder in the electronic version of the **04-14-22 system II** run folder.

2. Open the "Env_Result02" .pdf file for the current run folder. Select "export pdf" then select "spreadsheet" and select "Microsoft excel workbook" then click "export" and a "save as" file window opens.
3. Select the current run folder and sub folder and save the file as "Env_Results02_04-14-22 system II".
4. The adobe export engine will process the file and open an excel workbook using the data within the pdf file. Close the excel workbook file and the "Env_Results02" pdf file.
5. Open the original calibration curve run folder (in this example the original calibration curve run folder is named: "03-09-22 system II new curve". Open this run folder and then open the pdf file named "Env_Results02". Select "export pdf" then select "spreadsheet" and select "Microsoft excel workbook" then click "export" and a "save as" file window opens.
6. Select the current run folder and sub folder and save the file as "Env_Results02_03-09-22 system II new curve".
7. The adobe export engine will process the file and open an excel workbook using the data within the pdf file. Close the excel workbook file and the "Env_Results02" pdf file.
8. Re-visit the recently saved summary report and enter the current run folder name into cell A2.
9. Select the "view" tab and then select down arrow below "macros" to view the current macro within the summary report.
10. The "Kevin_Ours_ISC_Report_Function" macro name should appear; highlight this macro name, and select "edit". Microsoft visual basic screen should appear.
11. Look for the green font colored comment "Manually set the path to the source files".
12. Using the **04-14-22 system II** run folder example set the path to source files "P:\Toxics\Urban Air Toxics\VOC's Current Year Data\04-14-22 system II\Data Review\"
13. Immediately below the path is the source file identification. Set Source1File = "Env_Results02_03-09-22 system II.xlsx" (recently exported file created in step #5 above).
14. Set Source2file = "Env_Results02_04-14-22 system II.xlsx" (recently exported file created in step #2 above).
15. Click the save icon and close the visual basic window and click save again in the excel format internal standard summary report.
16. Select the "view" tab and then select down arrow below "macro" to view the current macro and make sure the "Kevin_Ours_ISC_Report_Function" macro is highlighted. Click "run".
17. The macro should take several seconds to a minute to run, and the end result will be a filled out internal standard summary report that flags internal standard response factors and retention times that fail acceptance limits. *Please note: the macro search function places the results of the first method blank injection (also known as the BFB tune check injection) at the end of the sample name list in the internal standard summary report.*
18. Save the summary report.

5.8.3 VOC Canister COC Form QC Summary Report

This report is used to summarize the VOC canister field sampling data and the canister cleaning data. Fill out this report for each run folder. To fill out this report the following documents and records are needed: VOC canister COC forms, canister cleaning records, and the current run folders' "MMDDYYa" .csv format report.

1. Open a blank "VOC Canister COC Form QC Summary Report_Template" located here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\Data Review Templates**. Select save as and rename the file by removing template and entering the current run folder name. For example, if the report is for the **04-14-22 system II** run folder, the report is named "VOC Canister COC Form QC Summary Report_04-14-22 system II" and stored in the "data review" subfolder in the electronic version of the **04-14-22 system II** run folder.
2. Each gray colored header in the report requests a specific data point from the VOC canister COC forms, canister cleaning records, and analysis records stored in the current run folder. Enter the requested information into the report for all valid and invalid field-collected canisters. *Please note: some fields will be blank for invalid-sample canisters.*
3. Once the data has been entered, remove/delete unused rows, and enter the current run folder name into cell A2 in the summary report.
4. Save the summary report in the current run folder.

6.0 DATA REVIEW

This section describes the steps required to perform self-review (level 1) and peer review (level 2) on the VOC canister sampling data and VOC canister analysis data.

6.1 Self Review (Level 1)

Self-review is performed by the VOC analyst that setup the GC-MS to analyze field samples or calibration curve samples; processed the GC-MS analysis batch, created the run folder files, and created the QC summary reports.

1. Open a blank review form, document number DAQ-03-020, located here: **P:\Toxics\Urban Air Toxics\VOC's Current Year Data\Data Review Templates**.
2. This form can be filled out electronically or printed as a blank form and filled out by hand using an indelible ink pen. If filling out this form electronically, save this file as "DAQ-03-020_04-14-22 system II (using the same run folder example as in 5.8.3 above) and store this file in the **04-14-22 system II** run folder.
3. Enter the run folder name and begin to answer all questions on the data review form.
4. Once complete, initial and date the bottom of the form.
5. If filling out the data review form electronically, save the form in the current folder. If filling out a blank form by hand using an indelible ink pen, scan the completed form and save the scan as "DAQ-03-020_04-14-22 system II" and store in the **04-14-22 system II** run folder on the p-drive.
6. Store the handwritten form (if filled in by hand) in the run folder file that is used to store the "Env_Results" reports and "Env_InitialCal" reports generated for the current run folder.

7. Notify the Level 2 reviewer that VOC canister sampling and analysis data are ready for review.

6.2 Peer Review (Level 2)

Peer-review is performed by a trained laboratory person that was not directly involved with analyzing the samples, processing the GC-MS analysis batch, creating the run folder files, creating the QC summary reports, and performing the Level 1 review.

1. Open the **04-14-22 system II** run folder and open the form named "DAQ-03-020_04-14-22 system II".
2. Verify all Level 1 questions have been answered.
3. If unanswered questions are present and the same questions also contain blank comments sections, contact the self-reviewer to rectify the issue. Also contact the self-reviewer to clarify illegible entries/comments.
4. If all questions are answered by the self-reviewer and the entries are legible and comments clearly explain unanswered questions; the peer-review can move-forward, and this form can be filled out by hand using indelible ink or filled out electronically using Microsoft Word.
5. If filling out the data review form by hand using an indelible ink pen, print the form and store the completed form in the hardcopy of the run folder. If filling out the form electronically, save the form as "DAQ-03-020_04-14-22 system II-Level2" and store it in the **04-14-22 system II** run folder.
6. Begin answering all questions on the data review form and placing answers in the Level 2 columns on the form.
7. Once complete, sign and date the bottom of the form as the Level 2 reviewer.
8. If filling out the data review form electronically, save the form in the current run folder. If filling out the form by hand using an indelible ink pen, scan the completed form and save the scan as "DAQ-03-020_04-14-22 system II-Level2" and store in the **04-14-22 system II** run folder on the p-drive.
8. Store the original handwritten copy of the review checklist in the run folder file that is used to store the "Env_Results" reports and "Env_InitialCal" reports generated for the current run folder.
9. Notify the Level 3 data reviewer that VOC canister sampling and analysis data are ready for review.

7.0 VOC FILE MANAGEMENT

7.1 GC-MS Instrument Files

The GC-MS instrument files such as sequences, results files in pdf and excel formats, tune reports and calibration reports are stored on the GC-MS instrument computer in appropriately named file folders using a naming system established by the primary analyst.

Additionally, these files are stored electronically on Universal Serial Bus (USB) storage devices and internal network drives and some files are stored as hard copies inside the hardcopy of the run folder

file. See section 5.5 for more details on GC-MS instrument file naming conventions and storage locations.

7.2 VOC Canister COC Forms

The hardcopies of the VOC Canister COC forms are stored in a secure file cabinet in the laboratory. Scans of the VOC canister COC forms that are analyzed in a specific run folder will be stored in the run folder saved to internal network drives. For example, scans of the VOC canister COC forms analyzed in the 04-14-22 system II run folder will be stored in the 04-14-22 system II run folder saved on internal network drives. See section 5.6 for more details on VOC Canister COC form naming conventions and storage locations.

7.3 QC Summary Reports and Data Review Checklists

The QC summary reports: VOC Canister Analysis QC Summary Report, VOC Canister COC Form QC Summary, Internal Standard Summary, and DAQ-03-020 (data review checklist) are stored in the current run folder on internal network drives. For example, these reports and data review checklists generated for the 04-14-22 system II run will be stored in the 04-14-22 system II run folder saved on internal network drives. See sections 5.7, 6.1, and 6.2 for more details on file naming conventions and storage locations. Occasionally, the data review checklist may be filled out by hand using an indelible ink pen. In these cases, the original handwritten copies are stored in the hardcopy version of the current run folder.

8.0 QUALITY ASSURANCE FILES AND DATA HANDLING

8.1 VOC Standard Mix and Internal Standard Mix Certificates of Analysis Storage

The VOC standard cylinders and VOC internal standard cylinders purchased by the laboratory must be traceable to NIST and contain a certificate of analysis generated by the cylinder manufacturer that confirms the measured concentrations of target VOC and/or concentrations of internal standard compounds in the cylinder. The certificates of analysis are stored in a designated file folder controlled by the VOC analyst. Scans of the certificates of analysis may also be stored on the p-drive here:

P:\Toxics\Urban Air Toxics\VOC's Current Year Data\VOC Standard CofA using a naming convention such as this for an internal standard connected to GC-MS system I: "System1_Internal Standard CofA_1-18-22to1-18-23".

8.2 GC-MS Instrument Logbook

This logbook is stored in the VOC laboratory next to the GC-MS system. This logbook contains a handwritten log of sample canisters, calibration canisters, certification canisters, and QC canisters analyzed by the laboratory and the dates of analysis. Also included are brief summaries of GC-MS instrument maintenance activities.

8.3 Quarterly GC-MS Instrument Computer Backups

At the end of each calendar quarter, the primary analyst backs up the GC-MS instrument data and methods using a specially formatted 4-terabyte, external hard drive. Plug in the external hard drive and click the toolkit app on the GC-MS computer and the backup process will start automatically.

9.0 TROUBLESHOOTING AND CORRECTIVE ACTIONS

This section describes some basic troubleshooting and corrective actions for GC-MS instruments and support equipment modules including errors and issues found during the data review process. All GC-MS and support module maintenance activities should be documented in the instrument logbook.

9.1 GC-MS Instrument

Agilent certified technicians are best equipped to handle most of the mechanical issues and software issues that develop over time during instrument operation. The GC-MS is under an annual service agreement with Agilent service technicians and the primary analyst and GC-MS operators are encouraged to call the instrument manufacturer for technical support and troubleshooting problems.

The Agilent service technicians perform preventative maintenance according to Agilent's protocols which includes rough pump oil changing, transfer line O-ring replacement, column changing, ion source cleaning, and general performance checks to ensure the system is operating as designed.

Agilent technicians perform preventative maintenance once per year and every 365 days thereafter. Due to the consistent operation of the instrument and support modules, the primary analyst may have to perform some of the following maintenance activities in the days between the preventative maintenance performed by the Agilent service technicians and may include the following:

- Changing the oil of the rough pump once per year and every 365 days thereafter. As the instrument and rough pump age, more frequent oil changes may be required.
- Disassembling and cleaning the ion trap as necessary when tuning checks fail acceptance criteria.
- Scheduling regular preventative maintenance of the GC-MS and support modules through contacting Agilent service technicians (1-800-227-9770).

9.2 GC-MS Support Modules (Markes Instruments)

The support modules include the Unity Xr thermal desorber, Kori Xr water management, and CIA Advantage autosampler tree. Regularly check the support modules for leaks and check the transfer lines between the GC-MS and support modules for leaks. Regularly replace the two O-rings that seal the transfer line on both sides when leaks are discovered. Replace the desorber trap once per year and every 365 days thereafter or whenever a leak is detected, and it's determined the cause of the leak is a broken and worn trap.

See **Agilent User Manual** loaded on the GC-MS instrument computer and operating software for any other instrument maintenance issues not specified in Section 9.0. Follow the instructions on the videos for filament replacement, and source cleaning for the Mass Spectrometer.

9.3 Data Review Errors

Errors may be found within the instrument files, summary reports, and data review checklists during data review procedures.

If an error or typo is found in an excel file or word document file and will be corrected electronically, go to the error and highlight the error. Select the "strikethrough" font type. This will show the error in "strikethrough" text font. Place your cursor in the space immediately after the "strikethrough" text and turn off "strikethrough" text format function. Enter the "(initials and date)" of person making correction and then add the correction immediately after the last parenthesis of the "(initial and date)". If the excel file or word document is equipped with a "comments" section or "comments" field, the person making the change can add their initials and date of the correction in this field.

Errors found on records that cannot be changed electronically should be changed manually by crossing out the error using an indelible ink pen using a single line to cross out the error and writing the correction. Add the initials and date of the person making the change.

10.0 REVISION HISTORY

1. Revision 0, Steven Walters, Chemist II, 05/03/2022.

11.0 REFERENCES

1. Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 3, Prepared for: U.S. Environmental Protection Agency Office of Air Quality Planning and Standards (C304-06) Research Triangle Park, NC 27711.
https://www3.epa.gov/ttnamti1/files/ambient/airtox/NATTS%20TAD%20Revision%203_FINAL%20October%202016.pdf
2. Compendium Method TO-15 Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography Mass Spectrometry (GC-MS), Center of Environmental Research Information Office of Research and Development U.S Environmental Protection Agency Cincinnati, OH 45268, January 1999.
<https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-15r.pdf>

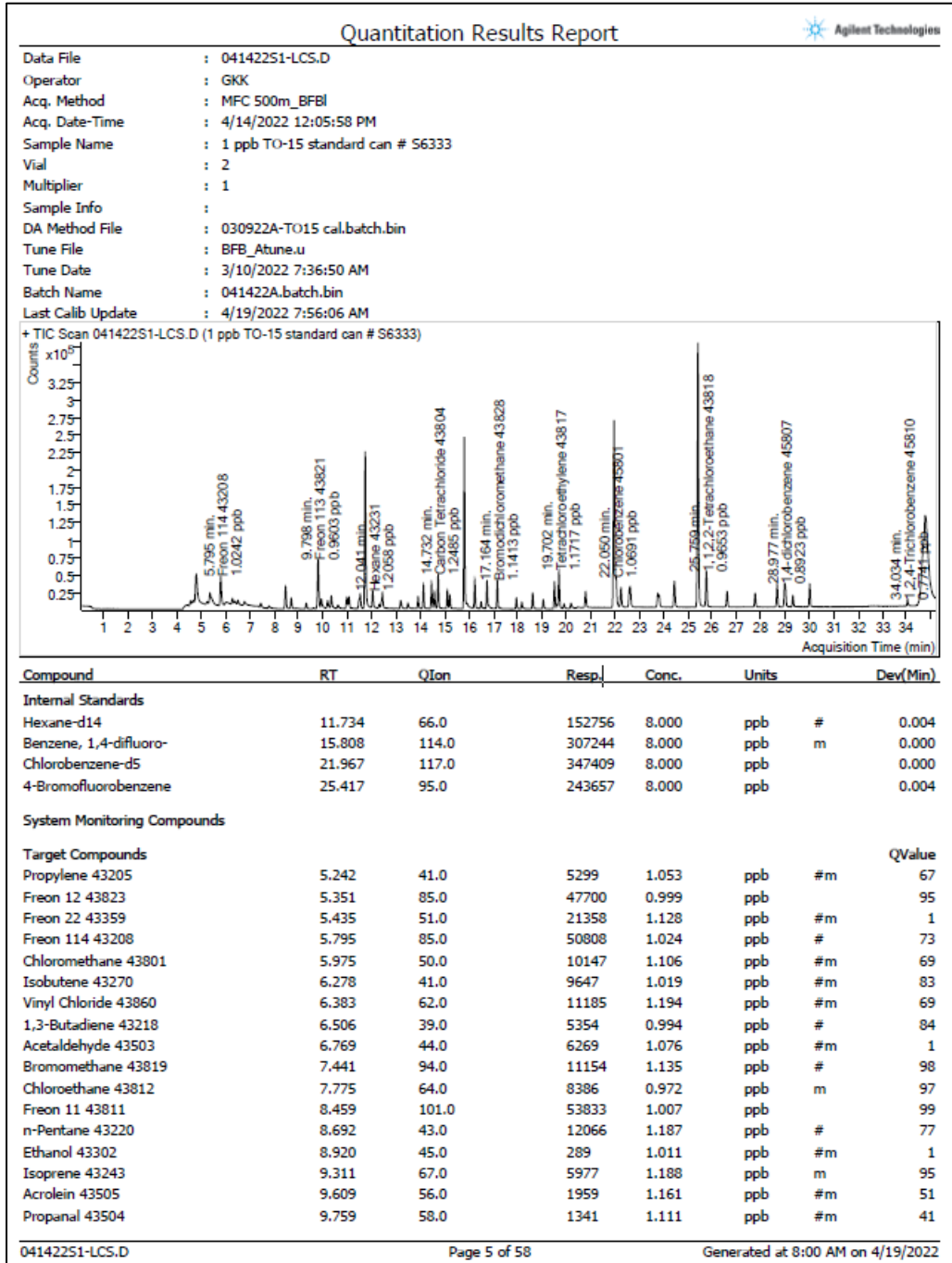
12.0 APPENDICES

12.1 VOC Canister Chain of Custody Form

NC DAQ Laboratory Analysis Branch Air Sampling Canister Chain of Custody							
NC Division of Air Quality Laboratory 4403 Reedy Creek Rd Raleigh, NC 27607 (919) 733-9777				Sampling Site Sample #			
Pre-Sample (Lab)							
Canister #	Cleaning Batch #	Canister Vacuum Inches Hg (Lab)	Canister COC #	Canister COC Sealed by Initials, Date, & Time (Lab)			
Sample Setup and Collection (Field)							
Sampler ID#: _____ <input type="checkbox"/> Exp. Date Checked		Flow meter ID#: _____ <input type="checkbox"/> Exp. Date Checked		<input type="checkbox"/> Sampler COC seal intact <input type="checkbox"/> Same # as sealed		<input type="checkbox"/> Canister COC seal intact <input type="checkbox"/> Same # as sealed	
Sampler COC Broken by Initials, Date, & Time	Canister COC Broken by Initials, Date, & Time	Canister Vacuum Inches Hg (Field)	Leak Check (psig) (≤ 0.1 psi over 10 min)				
			Start	End	Diff.	Pass/Fail	
<input type="checkbox"/> Timer Clock checked against NIST <input type="checkbox"/> Timer Adjusted (Details noted in comments below)							
Pre-Sampling Avg Flow Check: _____ mL/min <small>Acceptable Range: 8 mL/min to 12 mL/min (max)</small>			#1:	#2:	#3:	#4:	
Sampling Date & Day	Timer Start Time	Timer End Time	Canister Pressure (psig)	Sampler COC #	Canister and Sampler COCs Sealed by Initials, Date, & Time		
Post-Sampling Avg Flow Check: _____ mL/min <small>Acceptable: 8 mL/min to 12 mL/min (max)</small>			#1:	#2:	#3:	#4:	
Adjusted Sampling Avg Flow Check: _____ mL/min <small>Acceptable: 8 mL/min to 12 mL/min (max) (NA = No Adjustment)</small>			#1:	#2:	#3:	#4:	
Comments:							
Post Sampling (Lab)							
Sample Received by Initials, Date, & Time	<input type="checkbox"/> Canister COC seal intact <input type="checkbox"/> Same # as sealed		COC Broken by Initials, Date, & Time	Canister Pressure psig (Lab)			
Comments:							
Intra-Laboratory Canister Transfers							
VOC Lab Relinquished by: _____				Date/Time: _____			
PAMS Lab Received by: _____				Date/Time: _____			

12.2 GC-MS Results Report Example

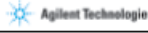
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Quantitation Results Report							Agilent Technologies
Freon 113 43821	9.798	101.0	43067	0.960	ppb	#	86
1,1-Dichloroethene 43826	9.798	61.0	15254	1.013	ppb		99
Acetone 43551	9.930	43.0	29607	1.020	ppb	#m	90
Iodomethane 43808	10.189	142.0	17869	0.965	ppb	#	75
Isopropyl Alcohol 43312	10.193	45.0	4623	1.186	ppb	#m	86
Carbon disulfide 42153	10.343	76.0	50675	1.096	ppb		96
Acetonitrile 43702	10.619	41.0	9808	1.148	ppb	#m	90
Methylene Chloride 43802	10.975	49.0	15766	1.059	ppb	#m	70
Cyclopentane 43242	11.067	42.0	11684	1.107	ppb	#	84
Methyl Tert Butyl Ether 43372	11.479	73.0	14384	1.029	ppb	#	81
Trans-1,2-Dichloroethene 43838	11.532	61.0	17207	1.140	ppb		95
Hexane 43231	12.041	57.0	12965	1.206	ppb	#m	90
Methacrolein 43515	12.309	41.0	4128	1.148	ppb	#m	58
Vinyl Acetate 43447	12.397	43.0	13833	1.222	ppb	#m	92
1,1-Dichloroethane 43813	12.445	63.0	25562	1.200	ppb	#m	89
Methyl Vinyl Ketone 43558	13.191	55.0	4163	1.184	ppb	#m	1
n-Butanal 43510	13.191	44.0	2413	0.812	ppb	#m	1
Methyl Ethyl Ketone 43552	13.490	43.0	13332	1.145	ppb	#m	1
1-Bromopropane 43853	13.911	43.0	14309	1.191	ppb	#m	80
Chloroform 43803	14.131	83.0	40687	1.201	ppb		96
1,1,1-Trichloroethane 43814	14.460	97.0	36078	1.266	ppb	#	89
Cyclohexane 43248	14.579	56.0	9232	1.205	ppb	#m	82
Carbon Tetrachloride 43804	14.732	117.0	47293	1.249	ppb		100
Benzene 45201	15.114	78.0	36836	1.227	ppb		100
1,2-Dichloroethane 43815	15.215	62.0	23031	1.299	ppb	#	87
1-Butanol 43305	15.931	56.0	990	0.980	ppb	#m	1
Trichloroethylene 43824	16.238	130.0	22495	1.066	ppb		96
2-Pentanone 43562	16.501	43.0	15378	1.280	ppb		98
3-Pentanone 43553	16.738	57.0	13692	1.201	ppb	#	82
1,2-Dichloropropane 43829	16.743	63.0	14602	1.186	ppb	m	93
1,4-Dioxane 46201	16.817	88.0	2790	0.973	ppb	#	54
Bromodichloromethane 43828	17.164	83.0	40483	1.141	ppb	#	96
Cis-1,3-dichloropropene 43831	17.950	75.0	14540	1.239	ppb		98
Methyl Isobutyl Ketone 43560	18.174	58.0	3832	1.487	ppb	#	84
Toluene 45202	18.617	91.0	30469	1.070	ppb	#	97
Trans-1,3-dichloropropene 43830	19.056	75.0	13111	1.271	ppb		98
1,1,2-Trichloroethane 43820	19.513	97.0	22234	1.140	ppb		97
3-Hexanone 43557	19.622	57.0	3807	1.231	ppb	#m	83
Tetrachloroethylene 43817	19.702	166.0	32840	1.172	ppb		99
Methyl Butyl Ketone 43559	19.908	58.0	2682	1.053	ppb	#m	52
Hexanal 43517	20.198	56.0	2699	1.243	ppb	#	58
1,2-Dibromoethane 43843	20.799	107.0	28196	1.095	ppb		98
Chlorobenzene 45801	22.050	112.0	45696	1.069	ppb		93
Ethylbenzene 45203	22.252	91.0	40326	0.931	ppb		99
m,p-xylene45109	22.625	91.0	55301	0.944	ppb	#	94
o-Xylene 45204	23.762	91.0	24961	1.030	ppb		97
Styrene 45220	23.828	104.0	18485	0.916	ppb	#	94
Bromoform 43806	24.451	173.0	42014	1.017	ppb		99
1,1,2,2-Tetrachloroethane 43818	25.759	83.0	50839	0.965	ppb	#	1
1,2,4-Trimethylbenzene 45208	26.615	105.0	24698	1.030	ppb		98
1,2,3-Trimethylbenzene 45225	27.770	105.0	22477	1.039	ppb		96
1,3-dichlorobenzene 45806	28.670	146.0	30521	0.919	ppb	#	100
1,4-dichlorobenzene 45807	28.977	146.0	28185	0.892	ppb		98
1,3,5-Trimethyl Benzene 45207	29.021	105.0	23294	0.903	ppb		95
Benzyl chloride 45809	29.319	91.0	23057	0.946	ppb		97
1,2-Dichlorobenzene 45805	30.017	146.0	28800	0.903	ppb		99

Page 3 of 3:

Quantitation Results Report						
1,2,4-Trichlorobenzene 45810	34.034	180.0	6555	0.774	ppb	99
(#) = Qualifier Out of Range; (m) = Manual Integration; (+) = Area Summed; (*) = Surrogate Percent Recovery Out of Range; (d): Zeroed Peak						
04142251-LCS.D	Page 7 of 58				Generated at 8:00 AM on 4/19/2022	

12.6 DAQ-03-022 System I VOC Analysis QC Summary Report (Retention Time Summary Workbook)

DAQ-03-022 Retention Time Summary		*Calibration Curve Run Folder Name*		
Net File	File Name	Run Date	Run Time	Run Date
11-11-2021-0001	11-11-2021-0001	11/11/21	12:00:00	11/11/21
11-11-2021-0002	11-11-2021-0002	11/11/21	12:05:00	11/11/21
11-11-2021-0003	11-11-2021-0003	11/11/21	12:10:00	11/11/21
11-11-2021-0004	11-11-2021-0004	11/11/21	12:15:00	11/11/21
11-11-2021-0005	11-11-2021-0005	11/11/21	12:20:00	11/11/21
11-11-2021-0006	11-11-2021-0006	11/11/21	12:25:00	11/11/21
11-11-2021-0007	11-11-2021-0007	11/11/21	12:30:00	11/11/21
11-11-2021-0008	11-11-2021-0008	11/11/21	12:35:00	11/11/21
11-11-2021-0009	11-11-2021-0009	11/11/21	12:40:00	11/11/21
11-11-2021-0010	11-11-2021-0010	11/11/21	12:45:00	11/11/21
11-11-2021-0011	11-11-2021-0011	11/11/21	12:50:00	11/11/21
11-11-2021-0012	11-11-2021-0012	11/11/21	12:55:00	11/11/21
11-11-2021-0013	11-11-2021-0013	11/11/21	13:00:00	11/11/21
11-11-2021-0014	11-11-2021-0014	11/11/21	13:05:00	11/11/21
11-11-2021-0015	11-11-2021-0015	11/11/21	13:10:00	11/11/21
11-11-2021-0016	11-11-2021-0016	11/11/21	13:15:00	11/11/21
11-11-2021-0017	11-11-2021-0017	11/11/21	13:20:00	11/11/21
11-11-2021-0018	11-11-2021-0018	11/11/21	13:25:00	11/11/21
11-11-2021-0019	11-11-2021-0019	11/11/21	13:30:00	11/11/21
11-11-2021-0020	11-11-2021-0020	11/11/21	13:35:00	11/11/21
11-11-2021-0021	11-11-2021-0021	11/11/21	13:40:00	11/11/21
11-11-2021-0022	11-11-2021-0022	11/11/21	13:45:00	11/11/21
11-11-2021-0023	11-11-2021-0023	11/11/21	13:50:00	11/11/21
11-11-2021-0024	11-11-2021-0024	11/11/21	13:55:00	11/11/21
11-11-2021-0025	11-11-2021-0025	11/11/21	14:00:00	11/11/21
11-11-2021-0026	11-11-2021-0026	11/11/21	14:05:00	11/11/21
11-11-2021-0027	11-11-2021-0027	11/11/21	14:10:00	11/11/21
11-11-2021-0028	11-11-2021-0028	11/11/21	14:15:00	11/11/21
11-11-2021-0029	11-11-2021-0029	11/11/21	14:20:00	11/11/21
11-11-2021-0030	11-11-2021-0030	11/11/21	14:25:00	11/11/21
11-11-2021-0031	11-11-2021-0031	11/11/21	14:30:00	11/11/21
11-11-2021-0032	11-11-2021-0032	11/11/21	14:35:00	11/11/21
11-11-2021-0033	11-11-2021-0033	11/11/21	14:40:00	11/11/21
11-11-2021-0034	11-11-2021-0034	11/11/21	14:45:00	11/11/21
11-11-2021-0035	11-11-2021-0035	11/11/21	14:50:00	11/11/21
11-11-2021-0036	11-11-2021-0036	11/11/21	14:55:00	11/11/21
11-11-2021-0037	11-11-2021-0037	11/11/21	15:00:00	11/11/21
11-11-2021-0038	11-11-2021-0038	11/11/21	15:05:00	11/11/21
11-11-2021-0039	11-11-2021-0039	11/11/21	15:10:00	11/11/21
11-11-2021-0040	11-11-2021-0040	11/11/21	15:15:00	11/11/21
11-11-2021-0041	11-11-2021-0041	11/11/21	15:20:00	11/11/21
11-11-2021-0042	11-11-2021-0042	11/11/21	15:25:00	11/11/21
11-11-2021-0043	11-11-2021-0043	11/11/21	15:30:00	11/11/21
11-11-2021-0044	11-11-2021-0044	11/11/21	15:35:00	11/11/21
11-11-2021-0045	11-11-2021-0045	11/11/21	15:40:00	11/11/21
11-11-2021-0046	11-11-2021-0046	11/11/21	15:45:00	11/11/21
11-11-2021-0047	11-11-2021-0047	11/11/21	15:50:00	11/11/21
11-11-2021-0048	11-11-2021-0048	11/11/21	15:55:00	11/11/21
11-11-2021-0049	11-11-2021-0049	11/11/21	16:00:00	11/11/21
11-11-2021-0050	11-11-2021-0050	11/11/21	16:05:00	11/11/21
11-11-2021-0051	11-11-2021-0051	11/11/21	16:10:00	11/11/21
11-11-2021-0052	11-11-2021-0052	11/11/21	16:15:00	11/11/21
11-11-2021-0053	11-11-2021-0053	11/11/21	16:20:00	11/11/21
11-11-2021-0054	11-11-2021-0054	11/11/21	16:25:00	11/11/21
11-11-2021-0055	11-11-2021-0055	11/11/21	16:30:00	11/11/21
11-11-2021-0056	11-11-2021-0056	11/11/21	16:35:00	11/11/21
11-11-2021-0057	11-11-2021-0057	11/11/21	16:40:00	11/11/21
11-11-2021-0058	11-11-2021-0058	11/11/21	16:45:00	11/11/21
11-11-2021-0059	11-11-2021-0059	11/11/21	16:50:00	11/11/21
11-11-2021-0060	11-11-2021-0060	11/11/21	16:55:00	11/11/21
11-11-2021-0061	11-11-2021-0061	11/11/21	17:00:00	11/11/21
11-11-2021-0062	11-11-2021-0062	11/11/21	17:05:00	11/11/21
11-11-2021-0063	11-11-2021-0063	11/11/21	17:10:00	11/11/21
11-11-2021-0064	11-11-2021-0064	11/11/21	17:15:00	11/11/21
11-11-2021-0065	11-11-2021-0065	11/11/21	17:20:00	11/11/21
11-11-2021-0066	11-11-2021-0066	11/11/21	17:25:00	11/11/21
11-11-2021-0067	11-11-2021-0067	11/11/21	17:30:00	11/11/21
11-11-2021-0068	11-11-2021-0068	11/11/21	17:35:00	11/11/21
11-11-2021-0069	11-11-2021-0069	11/11/21	17:40:00	11/11/21
11-11-2021-0070	11-11-2021-0070	11/11/21	17:45:00	11/11/21
11-11-2021-0071	11-11-2021-0071	11/11/21	17:50:00	11/11/21
11-11-2021-0072	11-11-2021-0072	11/11/21	17:55:00	11/11/21
11-11-2021-0073	11-11-2021-0073	11/11/21	18:00:00	11/11/21
11-11-2021-0074	11-11-2021-0074	11/11/21	18:05:00	11/11/21
11-11-2021-0075	11-11-2021-0075	11/11/21	18:10:00	11/11/21
11-11-2021-0076	11-11-2021-0076	11/11/21	18:15:00	11/11/21
11-11-2021-0077	11-11-2021-0077	11/11/21	18:20:00	11/11/21
11-11-2021-0078	11-11-2021-0078	11/11/21	18:25:00	11/11/21
11-11-2021-0079	11-11-2021-0079	11/11/21	18:30:00	11/11/21
11-11-2021-0080	11-11-2021-0080	11/11/21	18:35:00	11/11/21
11-11-2021-0081	11-11-2021-0081	11/11/21	18:40:00	11/11/21
11-11-2021-0082	11-11-2021-0082	11/11/21	18:45:00	11/11/21
11-11-2021-0083	11-11-2021-0083	11/11/21	18:50:00	11/11/21
11-11-2021-0084	11-11-2021-0084	11/11/21	18:55:00	11/11/21
11-11-2021-0085	11-11-2021-0085	11/11/21	19:00:00	11/11/21
11-11-2021-0086	11-11-2021-0086	11/11/21	19:05:00	11/11/21
11-11-2021-0087	11-11-2021-0087	11/11/21	19:10:00	11/11/21
11-11-2021-0088	11-11-2021-0088	11/11/21	19:15:00	11/11/21
11-11-2021-0089	11-11-2021-0089	11/11/21	19:20:00	11/11/21
11-11-2021-0090	11-11-2021-0090	11/11/21	19:25:00	11/11/21
11-11-2021-0091	11-11-2021-0091	11/11/21	19:30:00	11/11/21
11-11-2021-0092	11-11-2021-0092	11/11/21	19:35:00	11/11/21
11-11-2021-0093	11-11-2021-0093	11/11/21	19:40:00	11/11/21
11-11-2021-0094	11-11-2021-0094	11/11/21	19:45:00	11/11/21
11-11-2021-0095	11-11-2021-0095	11/11/21	19:50:00	11/11/21
11-11-2021-0096	11-11-2021-0096	11/11/21	19:55:00	11/11/21
11-11-2021-0097	11-11-2021-0097	11/11/21	20:00:00	11/11/21
11-11-2021-0098	11-11-2021-0098	11/11/21	20:05:00	11/11/21
11-11-2021-0099	11-11-2021-0099	11/11/21	20:10:00	11/11/21
11-11-2021-0100	11-11-2021-0100	11/11/21	20:15:00	11/11/21

12.8 DAQ-03-020 Data Review Checklist

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VOC Canister Data Review Checklist (Level 1 and Level 2)

Run Folder Name: _____

Verify all files are present in the hard copy and/or electronic copy versions of the run folder name stored on p-drive (asterisk "*" indicates files that should be present in the electronic copy of the run folder stored on network drives and will not be present in the hard copy version of the run folder):

Name of Document	Level 1 Review		Level 2 Review		Comments
	YES	NO	YES	NO	
*Two GC-MS summary files in .csv format that contain the same date in the file names and this date is identical to the date in the run folder name.					
One or multiple Markes sequence files in excel format that also contains a month, day, and year in the file name that is identical to the date in the run folder name.					
File named "sequence" in .txt format.					
PDF report named "Env_InitialCal."					
PDF report named "Env_Results02."					
PDF file named "TuneEvaluationReport."					
*Form "DAQ-03-021" for current run folder.					
*Form "DAQ-03-022" or "DAQ-03-023" (not both) for current run folder.					
*Form "DAQ-03-024" for the current run folder. If no, provide comment.					
Excel file "Env_InitialCal_ current run folder name."					
Excel file "Env_Results02_ current run folder name."					
Excel file "Env_Results02_calibration run folder name."					
Scans of the VOC Canister COC forms in a single file or multiple files.					

Answer the following questions regarding information recorded in the Run Folder Files:

Questions	Level 1 Review		Level 2 Review		Comments
	YES	NO	YES	NO	
Open the Tune Evaluation Report, do all test parameters pass criteria? If no, list the failing tune check parameter in comments.					
Does the Tune Evaluation Report "Acq on:" date match the date in the current run folder name and was the tune check performed using the first					

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Questions	Level 1 Review		Level 2 Review		Comments
	YES	NO	YES	NO	
Injection of the method blank? If no, list the "Acq on:" date and injection # in the comments.					
Open the "sequence" .txt format file. Is each VOC canister COC form sample # found? If no, list sample# in comments. Also list sample# in comments that are listed in the "sequence" .txt file but do not have a VOC canister COC form.					
Open the Markes sequence file(s) in .csv format. Is each VOC canister COC form sample # found? If no, list sample# in comments. Also list sample# in comments that are listed in the sequence file but do not have a VOC canister COC form.					
In the Marke's sequence, are all pre-sample canister pressures > 0.2psi? If no, list sample# in comments.					
Open both GC-MS summary files (.csv format). Is each VOC canister COC form sample # found in both files? If no, list sample# and summary file name in comments. Also list sample# in comments that are listed in the summary files but do not have a VOC canister COC form.					
Do the injection names match in both summary files? If no, list mis-matched injection names in the comments.					
Does the trap fire time in the Markes Sequence match the Acq date-times in the GC-MS summary file in .csv format? If no, list mis-matched sample names in the comments.					
Does the trap fire time in the Markes Sequence match the Acq date and time in the GC-MS "Env_Results02" pdf file? If no, list mis-matched sample names in the comments.					
Does the vial numbers and comment values in the Markes Sequence match the vial numbers and sample names in the "Env_Results02" pdf report? If no, list the mismatches in the comments					
Open the "Env_Results02" pdf report and the "Env_Results_current run folder name" excel report (pdf file extracted as excel workbook). Is each VOC canister COC form sample# found in both files? If no, list sample# and file name in comments					
In the VOC canister analysis QC summary report, verify the ICAL summary workbook tab contains the current calibration curve results					
In the VOC canister analysis QC summary report, verify the Analysis summary workbook tab contains the current run folder analysis results					

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Questions	Level 1 Review		Level 2 Review		Comments
	YES	NO	YES	NO	
Level 2 Reviewer Only: Do the conditional formats embedded in the ICAL Summary workbook tab in the VOC canister analysis QC summary report correctly flag concentration values and RPD values that are outside control limits?					
Level 2 Reviewer only: Do the formulas embedded in the Analysis Summary workbook tab in the VOC canister analysis QC summary report correctly calculate the RPD values?					
Is the data recorded in the Retention Time Summary workbook tab match the data in the calibration curve summary file (.csv format) and the current run folder summary file (.csv format)?					
Level 2 Reviewer only: Are the calculation in the retention time summary workbook tab determining the correct average and upper and lower control limits?					
Do the data recorded in the Internal Standard Summary report match the data in the "Env_Results_calibration curve run folder name" and the "Env_Results_current run folder name" (both "files" are extracted from pdf "Env_Results02" reports)? If no, list mis-matched sample# and file name in the comment					
Level 2 Reviewer only: Are the calculations in the Internal Standard Summary Report correctly determining the correct average and upper and lower control limits?					
Is the data recorded in the VOC canister COC form QC summary report match the recorded data in the canister COC forms?					
Does the canister cleaning data recorded in the VOC canister COC form QC summary report match the canister cleaning records?					
Level 2 Reviewer only: Do the condition formats and embedded calculations in the VOC canister COC form QC summary report correctly identify out-of-control data and correctly calculate clean canister holding times and sampled canister holding times?					

Level 1 Reviewer: _____ Date of Level 1 Review: _____

Level 2 Reviewer: _____ Date of Level 2 Review: _____