

**Hazardous Waste Support Section**  
**SOP NO. HW-31, Revision 6**  
**Analysis of Volatile Organic Compounds in Air Contained in Canisters by**  
**Method TO-15**



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## **NOTICE**

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment with provided justification supersedes any guidance listed in this document.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

<http://www.epa.gov/region2/qa/documents.htm>

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## ACRONYMS

<b>%D</b>	Percent Difference
<b>%RSD</b>	Percent Relative Standard Deviation
<b>ASB</b>	Analytical Services Branch
<b>BFB</b>	Bromofluorobenzene
<b>CCV</b>	Continuing Calibration Verification
<b>CF</b>	Calibration Factor
<b>CRQL</b>	Contract Required Quantitation Limit
<b>DAR</b>	Data Assessment Report
<b>DAT</b>	Data Assessment Tool
<b>DQA</b>	Data Quality Assessment
<b>DQO</b>	Data Quality Objective
<b>EDD</b>	Electronic Data Deliverable
<b>ESAT</b>	Environmental Services Assistance Team
<b>GC</b>	Gas Chromatograph
<b>GC/MS</b>	Gas Chromatograph/Mass Spectrometer
<b>HWSS</b>	Hazardous Waste Support Section
<b>LCS</b>	Laboratory Control Sample
<b>MA</b>	Modified analysis
<b>OSRTI</b>	Office of Superfund Remediation and Technology Innovation
<b>PO</b>	Project Officer (responsible for the Regional Contract Laboratory)
<b>QA</b>	Quality Assurance
<b>QAC</b>	Quality Assurance Coordinator
<b>QAPP</b>	Quality Assurance Project Plan
<b>QC</b>	Quality Control
<b>RAS</b>	Routine Analytical Services
<b>RIC</b>	Reconstructed Ion Chromatogram
<b>RPD</b>	Relative Percent Difference
<b>RRF</b>	Relative Response Factor
<b>RRF</b>	Mean Relative Response Factor
<b>RRT</b>	Relative Retention Time
<b>RSCC</b>	Regional Sample Control Center Coordinator
<b>RSD</b>	Relative Standard Deviation
<b>RT</b>	Retention Time
<b>SAP</b>	Sampling and Analysis Plan
<b>SDG</b>	Sample Delivery Group
<b>SIM</b>	Selected Ion Monitoring
<b>SMO</b>	Sample Management Office
<b>SOP</b>	Standard Operating Procedure
<b>SOW</b>	Statement of Work
<b>TCL</b>	Target Compound List
<b>TIC</b>	Tentatively Identified Compound
<b>TOPO</b>	Task Order Project Officer
<b>TR/COC</b>	Traffic Report/Chain of Custody Record
<b>USEPA</b>	United States Environmental Protection Agency

## INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data from the analysis of Volatile Organic Compounds in air samples taken in canisters and analyzed by method TO-15. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process. The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the method and/or addenda to it (e.g. modified analysis request), and (b) the usability /validity and extent of bias of any data not meeting the specific technical and QC criteria established in the method. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability/ feasibility for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

## DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

<b>U</b>	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
<b>J</b>	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
<b>J+</b>	The result is an estimated quantity, but the result may be biased high.
<b>J-</b>	The result is an estimated quantity, but the result may be biased low.
<b>NJ</b>	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
<b>UJ</b>	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
<b>R</b>	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.

## DATA PACKAGE INSPECTION

Validation should include inspection of the data package to identify any missing and/or incorrect information or need for reanalysis. The laboratory may submit a reconciliation package for any missing items or to correct data.

If there are any concerns regarding the data package, Regional Laboratory Contract Project Officer (PO) should be contacted. Initial Review of data package should include any need for reanalysis on priority basis because of holding time and preservation reasons.

## HWSS DATA VALIDATION PROCESS

The data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

- |                                  |             |
|----------------------------------|-------------|
| A. case number                   | i.e., 12345 |
| B. SDG name                      | i.e., BXY12 |
| C. level of validation performed | i.e., VM    |

Examples:     **12345\_BXY12\_VM.xls**  
                  **12345\_BXY12\_VM.pdf**

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

<https://epaqp.rtp.epa.gov/hwssclpdeliverables>

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report, when applicable (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).



## **PRELIMINARY REVIEW**

This document is for the review of analytical data generated through the 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), and any supplements, additions and future editorial revisions of this method as appropriate. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Case, Sample Delivery Group (SDG) at hand. The exact number of samples, their assigned numbers, their matrix, their location, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP supersede this Standard Operating Procedure. The reviewer should contact the appropriate Regional Program Project Officer (PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final data usability.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
  - a. Sample locations
  - b. Sample matrix;
  - c. Field blanks and trip blanks;
  - d. Field duplicates;
  - e. QC audit samples;
  - f. Shipping dates;
  - g. Laboratories involved.
  - h. Initial/final canister pressure
  - i. Initial/final canister temperature

The TR/COC documentation includes sample descriptions, date(s) of sampling, starting canister pressure, temperature and time, and ending canister pressure, temperature and time. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrix, canister pressure, insufficient sample volume for analysis or reanalysis, samples received in abused containers/clogged flow controllers with high negative, zero or positive pressure and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any e-mail or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory and the Project Manager, USEPA Region 2. If the laboratory SDG narrative is inadequate and fails to mention important issues, validator should note it in the report and bring it to the attention of the Project Officer.

## **Sample Integrity and Preservation**

### **1. Presampling Criteria:**

#### **Canister Suitability:**

Canister used for the sampling of the ambient air must be demonstrated clean, and leak free prior to sample collection. This cleanliness is demonstrated by analysis of an individual canister or analysis of a representative canister, if only batch cleaning was required. Leak proof testing is performed on individual canisters. Canisters are used in conjunction with gauges, valves and flow controllers. Therefore, canister should be demonstrated clean and leak free inclusive of these components as appropriate.

#### **a. Leak Proof Test :**

Canisters are tested by their ability to hold vacuum/pressure within +/- 2 psi for a period of 24 hours preceding sampling. Any nonconformance issues must be reported to the laboratory, regional PO and sampler immediately and their explanation considered. Actions for use of canisters with failing leak test criteria are indicated in the Table 1 below.

**Table 1. Canister Leak test Actions for TO-15 Analysis\***

Matrix	Difference in initial and 24 hour pressure (psi) Criteria	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
Air	$\leq 5$	No qualification	
Air	$> 5$	J	UJ or R

\*Excessive time period (> 3months) elapsed between leak test and actual use should be considered in evaluation of canister integrity.

#### **b. Cleanliness:**

Integrity of the canister used for sampling of air for analysis should be maintained at all times including the time of shipment to the field, sampling, shipment back to the laboratory and time of analysis. Analytical results of canister cleaning verification must be taken into account in the validation of sample results. Canister contamination actions are stated in Table 2 below.

**Table 2. Canister Contamination Actions for TO-15 Analyses**

Contamination Type/level	Canister Cleaning Result	Sample Result	Action for Samples
Clean Canister analysis	Detects	Analytes found in clean canister analysis are non-detects	No qualification required
	<CRQL	< CRQL	Report CRQL value with a U
		$\geq$ CRQL and < 2x the CRQL	Report concentration of sample with a U
		$\geq$ 2x the CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		$\geq$ CRQL and $\leq$ clean canister value	Report clean canister value with a U
		$\geq$ CRQL and > clean canister value	No qualification required
	= CRQL	$\leq$ CRQL	Report CRQL value with a U
		> CRQL	No qualification required

## 2. Post Sampling Criteria

### Holding Times and Sample Integrity:

Specially prepared SUMMA canisters are designed to minimize sample changes or loss for majority of the analytes. Method TO-15 states, “Fortunately, under conditions of normal usage for sampling ambient air, most VOCs may be recovered from canisters near their original concentrations after storage times of up to thirty days”. This assumes that sample integrity is maintained by ensuring the system is closed tight and canister pressure from the time of sampling to the time of analysis is maintained within a difference allowable due to temperature change.

Qualify sample results using technical holding time information as stated in Table 3:

- If there is no evidence that the samples were properly preserved (pressure inside the canister maintained within +/- 5 psi from sampling to check in the laboratory or analysis) and samples were analyzed within technical holding time [30 days from sample collection], qualify detects as estimated (J) and non-detects as “UJ”.
- If there is no evidence that the samples were properly preserved (pressure inside the canister maintained within +/- 5 psi from sampling to check in the laboratory or analysis) and the samples were analyzed outside of the

- technical holding time [30 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).
- c. If the samples were properly preserved (pressure inside the canister maintained within +/- 5 psi from sampling to analysis), and the samples were analyzed within the technical holding time [30 days from sample collection], no qualification of the data is necessary.
  - d. If the samples were properly preserved (pressure inside the canister maintained within +/- 5 psi from sampling to analysis), but were analyzed outside of the technical holding time [30 days from sample collection], qualify detects as estimated (J) and non-detects as "UJ".
2. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.

**Table 3. Holding Time Actions for TO-15 Volatile Analyses**

Matrix	Preserved (Pressure difference between sampling and analysis ≤ 5psi)	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Air	Yes	< 30 days	No qualification	
	Yes	>30 days	J	UJ
Air	No	< 30 days	J	UJ
	No	>30 days	J	R

### 3. QC for canister cleaning verification:

It is expected that for canister cleaning analysis laboratory will use identical method and QC criteria as for the analysis of samples contained in the canister. Any QC defects and omissions in clean canister GCMS analysis should be evaluated by the validator and any deficiencies noted and rectified as necessary in collaboration with PO. These findings and defects should be noted in the data assessment narrative and reported to the PO. Professional judgment should be used in this evaluation to qualify the data. Gross multiple exceedances in the QC of canister cleaning analysis can be used to invalidate canister cleaning verification and reject data with professional judgment and justification.

## **Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance** **Check**

### **Action:**

**NOTES:** This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable.

**NOTES:** No data should be qualified based on BFB or DFTTP failure. Instances of this should be noted in the narrative.

All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

1. If samples are analyzed without a preceding valid instrument performance check, qualify all data in those samples as unusable (R).
2. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.
3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data and notify the Project Officer (PO).
4. If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. When applying professional judgment to this topic, the most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance. This issue is more critical for Tentatively Identified Compounds (TICs) than for target analytes.
5. Note, in the Data Review Narrative, decisions to use analytical data associated with BFB instrument performance check failures (not meeting contract requirements).
6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in the Compendium method TO-15 entitled "Determination Of Volatile Organic Compounds(VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry(GC/MS)", section 10.4, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation.
7. Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

## **METHOD DETECTION LIMITS (MDL)**

Data validator should be familiar with the MDL requirements in the QAPP and the method used. MDLs should be lower than reporting limits and satisfy data quality requirements for samples as well as and clean canister analysis. 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) states that to qualify under Compendium Method TO-15 the method detection limit should be  $\leq 0.5$  ppbv (section 11.1.1). The method also states that “any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used.” Table 4 of the compendium method TO-15 lists method detection limits for certain analytes. Much lower detection limits are generally achievable. Document, “Supplement to Compendium Method TO-15-Reduction of Method Detection Limits to Meet Vapor Intrusion Monitoring Needs” suggests that requirements for monitoring at  $10^{-6}$  risk levels are possible with TO-15 method. In addition, reporting limits should be  $\leq$  reporting limits (RL). Any observations in this regard must be reported to the PO, noted in the data assessment report and considered during validation.

## Initial Calibration

Instrument calibration compliance requirements are established to ensure that the instrument is capable of generating acceptable data for qualitative as well as quantitative use. Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing linear calibration curve and provides Mean Relative Response Factors (RRFs) suitable for quantitation.

**Table 4. TO 15 Volatile Compounds List\***

Compound	CAS Number	Synonyms
Acetone	67-64-1	Dimethyl ketone; Dimethylformaldehyde; 2-Propanone
Allyl chloride	107-05-1	3-Chloropropene; 3-Chloroprene
Benzene	71-43-2	Benzol; Benzine
Benzyl chloride	100-44-7	Chloromethylbenzene; alpha-Chlorotoluene
Bromodichloromethane	75-27-4	Monobromodichloromethane; Methane-bromodichloro
Bromoethene	593-60-2	Vinyl bromide; Monobromoethene
Bromoform	75-25-2	Tribromoethane
Bromomethane	74-83-9	Methyl bromide; Monobromomethane
1,3-Butadiene	106-99-0	Biethylene; Erythrene; Pyrrolyene
<b>Carbon disulfide</b>	<b>75-15-0</b>	<b>Carbon bisulfide; Carbon sulfide</b>
Carbon tetrachloride	56-23-5	Carbon tet; Tetrachloromethane
Chlorobenzene	108-90-7	Monochlorobenzene; Chlorobenzol; Benzene chloride
<b>Chloroethane</b>	<b>75-00-3</b>	<b>Ethyl chloride; Chlorene; Chloryl</b>
Chloroethene	75-01-4	Vinyl chloride; Ethylene monochloride
Chloroform	67-66-3	Trichloromethane; Methyltrichloride; Methane trichloride
<b>Chloromethane</b>	<b>74-87-3</b>	<b>R40; Methyl chloride; Monochloromethane</b>
Cyclohexane	110-82-7	Hexamethylene; Hexahydrobenzene; Hexanaphthene
Dibromochloromethane	124-48-1	Chlorodibromomethane
<b>1,2-Dibromoethane</b>	<b>106-93-4</b>	<b>EDB; Ethylene dibromide</b>
1,2-Dichlorobenzene	95-50-1	ODB; Chloroben
1,3-Dichlorobenzene	541-73-1	meta-Dichlorobenzene; m-Phenylenedichloride
1,4-Dichlorobenzene	106-46-7	para-Dichlorobenzene; Parazene; Santochlor
1,1-Dichloroethane	75-34-3	Ethylidene chloride; Ethylidene dichloride
1,2-Dichloroethane	107-06-2	Ethylene dichloride; Glycol dichloride; 1,2-DCA
1,1-Dichloroethene	75-35-4	1,1-DCE; Vinylidene chloride
cis-1,2-Dichloroethylene	156-59-2	cis-1,2-DCE; cis-Acetylene dichloride
trans-1,2-Dichloroethylene	156-60-5	trans-1,2-DCE; trans-Acetylene dichloride
<b>1,2-Dichloropropane</b>	<b>78-87-5</b>	<b>Propylene dichloride; Propylene chloride</b>
cis-1,3-Dichloropropene	10061-01-5	1-Propene, 1,3-dichloro-, (z)-; cis-1,3-Dichloro-1-Propene
trans-1,3-Dichloropropene	10061-02-6	trans-1,3-Dichloro-1-Propene; trans-1,3-Dichloropropylene
<b>1,4-Dioxane</b>	<b>123-91-1</b>	<b>Diethylene dioxide; Diethylene ether</b>
Ethyl acetate	141-78-6	Acetic acid ethyl ester; Acetic ether
Ethylbenzene	100-41-4	Ethylbenzol; Phenylethane
4-Ethyltoluene	622-96-8	1-Ethyl-4-methyl benzene; p-Methylethylbenzene
Freon 11 (CCl3F)	75-69-4	Trichlorofluoromethane; Fluorotrichloromethane; Fluorocarbon 11

Freon 12 (CCl <sub>2</sub> F <sub>2</sub> )	75-71-8	Dichlorodifluoromethane; Fluorocarbon 12
Freon 113 (C <sub>2</sub> Cl <sub>3</sub> F <sub>3</sub> )	76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane; Fluorocarbon 113; 1,1,2-Trichlorotrifluoroethane
Freon 114 (C <sub>2</sub> Cl <sub>2</sub> F <sub>4</sub> )	76-14-2	1,2-Dichlorotetrafluoroethane; Halocarbon 114; 1,2-Dichloro-1,1,2,2-tetrafluoroethane
Heptane	142-82-5	Dipropylmethane; Heptyl hydride
Hexachlorobutadiene	87-68-3	1,3-Hexachlorobutadiene; Perchlorobutadiene
Hexane	110-54-3	n-Hexane; Hexyl hydride
2-Hexanone	591-78-6	Methyl butyl ketone; Butyl methyl ketone; Hexan-2-one
Isopropyl alcohol	67-63-0	2-Propanol; Isopropanol
<b><i>Methylene chloride</i></b>	<b><i>75-09-2</i></b>	<b><i>Dichloromethane; Methylene dichloride</i></b>
<b><i>Methyl ethyl ketone</i></b>	<b><i>78-93-3</i></b>	<b><i>MEK; 2-Butanone; Ethyl methyl ketone</i></b>
Methyl isobutyl ketone	108-10-1	MIBK; 2-Pentanone; Hexone; Isopropylacetone
Methyl tert-butyl ether	1634-04-4	MTBE; 2-Methoxy-2-methylpropane; tert-Butyl methyl ether
Propylene	115-07-1	Propene; Methylenehydride
Styrene	100-42-5	Vinylbenzene; Phenylethylene
1,1,2,2-Tetrachloroethane	79-34-5	Tetrachloroethane; Acetylene tetrachloride; Bonoform
Tetrachloroethene	127-18-4	PCE; PERC; Perchloroethylene; Ethylene tetrachloride; Carbon bichloride; Carbon dichloride
Tetrahydrofuran	109-99-9	Diethylene oxide; Butylene oxide
Toluene	108-88-3	Toluol; Methylbenzene
1,2,4-Trichlorobenzene	120-82-1	1,2,4-Trichlorobenzol
1,1,1-Trichloroethane	71-55-6	Methyl chloroform; Trichloroethane
1,1,2-Trichloroethane	79-00-5	beta-Trichloroethane; Ethane trichloride; Vinyl trichloride
Trichloroethene	79-01-6	TCE; Acetylene trichloride; Ethinyl trichloride
1,2,4-Trimethylbenzene	95-63-6	Pseudocumene; Pseudocumol
1,3,5-Trimethylbenzene	108-67-8	Mesitylene; Trimethylbenzol
2,2,4-Trimethylpentane	540-84-1	Iso-octane; Isobutyltrimethylmethane
Vinyl acetate	108-05-4	Acetic acid ethenyl ether; Ethenyl acetate
p-Xylene	106-42-3	p-Methyltoluene; 1,4-dimethylbenzene
m-Xylene	108-38-3	m-Methyltoluene; 1,3-dimethylbenzene
o-Xylene	95-47-6	o-Methyltoluene; 1,2-Dimethylbenzene

\*Laboratories use different sets and subsets of analytes on as needed basis.

#### NOTES:

Compounds in bold italicized letters may have poor GCMS response. These poor response compounds are evaluated using more relaxed relative response factor criteria as stated below.

#### Action:

Qualify all volatile target compounds, using the following criteria (see Table 5):

- If any volatile target compound has an RRF value less than the minimum criterion of 0.01 for poor response compounds and 0.05 for all other compounds listed in the table 4 above, use professional judgment for detects, based on mass spectral identification to qualify the data as estimated (J).



- b. If any volatile target compound has an RRF value less than the minimum criterion of 0.01 for poor response compounds and 0.05 for other compounds listed in Table 4, qualify non-detected compounds as unusable (R).
  - c. If any of the poor response target compounds listed in Table 4 has %RSD greater than 40.0%, qualify detects as estimated (J), and non-detected compounds using professional judgment.
  - d. For all other volatile target compounds, if %RSD is greater than 30.0%, qualify detects as estimated (J), and non-detected compounds using professional judgment.
- 2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:
  - a. If any volatile target compound has a %RSD greater than the maximum criterion of 30.0%, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
    - i. Qualify detects for that compound(s) as estimated (J).
    - ii. Qualify non-detected volatile target compounds using professional judgment.
  - b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
    - i. Qualify detects outside of the linear portion of the curve as estimated (J).
    - ii. No qualifiers are required for detects in the linear portion of the curve.
    - iii. No qualifiers are required for volatile target compounds that were not detected.
  - c. If the low-point of the curve is outside of the linearity criteria:
    - i. Qualify low-level detects in the area of non-linearity as estimated (J).
    - ii. No qualifiers are required for detects in the linear portion of the curve.
    - iii. For non-detected volatile compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
- 3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
- 4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
- 5. Note, for Project Officer (PO) action, if calibration criteria are grossly exceeded.

**Table 5. Initial Calibration Actions for TO-15 Analyses**

Criteria for TO-15 Analysis	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (poor response volatile target compounds, Table 4) RRF < 0.050 (all other volatile target compounds)	J (based on mass spectral identification)	R
RRF > 0.010 (poor response volatile target compounds, Table 4) RRF > 0.050 (all other volatile target compounds)	No qualification	
% RSD > 40.0 or < -40.0 (poor response volatile target compounds, Table 4) % RSD > 30.0 or < -30.0 (all other Volatile target compounds)	No qualification	
% RSD < 40.0 and > -40.0 (poor response volatile target compounds, Table 4) % RSD < 30.0 and > -30.0 (all other volatile target compounds)	J	Use professional judgment

### **Continuing Calibration Verification (CCV)**

**Action:**

1. If a CCV/daily calibration was not run at the appropriate frequency ( $\leq 20$  field samples or 24 hours), qualify data using professional judgment.
2. Qualify all volatile target compounds using the following criteria:
  - a. For a CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.01), use professional judgment for detects, based on mass spectral identification, to qualify the data as estimated (J).
  - b. For a CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.010), qualify non-detected compounds as unusable (R).
  - c. For a CCV, if the Percent Difference value for poor performance volatile target compound is outside the  $\pm 40.0\%$  criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - d. For a CCV, if the Percent Difference value for any other volatile target compound is outside the  $\pm 30.0\%$  criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - e. If the volatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.
3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary

information. If the information is not available, the reviewer must use professional judgment to assess the data.

4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
5. Note, for Laboratory Project Officer (PO) action, if calibration criteria are grossly exceeded.

**Table 6. Continuing Calibration Verification (CCV) Actions for TO-15 Analyses**

Criteria for CCV	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (poor response volatile target compounds, Table 4) RRF < 0.050 (all other volatile target compounds)	J (based on mass spectral identification)	R
RRF > 0.010 (poor response volatile target compounds, Table 4) RRF > 0.050 (all other volatile target compounds)	No qualification	
%D > 40.0 or < -40.0 (poor response volatile target compounds, Table 4) %D > 30.0 or < -30.0 (all other Volatile target compounds)	J	UJ
%D < 40.0 and > -40.0 (poor response volatile target compounds, Table 4) %D < 30.0 and > -30.0 (all other volatile target compounds)	No qualification	

If the % D for daily calibration exceeds -90, use professional judgment to see if non-detects need to be qualified as unusable "R"

## **Blanks**

### **Action:**

If trip blanks are present, the data reviewer should evaluate this data to ensure that it can be used for qualification of samples.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a volatile compound is found in a method blank or trip blank, but not found in the sample, no qualification of the data is necessary.
2. If the method or trip blanks contain a volatile Target Compound List (TCL) compound(s) at a concentration less than the CRQL and:
  - a. The sample concentration is less than the CRQL, report the CRQL value with a "U".
  - b. The sample concentration is greater than or equal to the CRQL, and less than 2x the CRQL, report the concentration of the compound in the sample and qualify with a "U".
  - c. The sample concentration is greater than or equal to 2x the CRQL, no qualification of the data is necessary.
3. If the method, or trip blanks contain a volatile TCL compound(s) at a concentration greater than the CRQL and:
  - a. The sample concentration is less than the CRQL, report the CRQL value with a "U".
  - b. The sample concentration is greater than or equal to the CRQL, and less than the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a "U".
  - c. The sample concentration is greater than or equal to the CRQL and greater than the blank concentration, no qualification is required.
4. If the method, storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration equal to the CRQL and:
  - a. The sample concentration is less than or equal to the CRQL, report the CRQL value with a "U".
  - b. The sample concentration is greater than the CRQL, no qualification is required.
5. If gross contamination exists (i.e., blank contamination > 2x the CRQL) in the method, or trip blanks, raise the CRQL to the level of the blank contamination and report the associated sample data below this level as CRQL-U.
6. If contaminants are found in the trip blank, the following is recommended:
  - a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
    - i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

- ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area or during sample transport. Consider all associated samples for possible cross-contamination.
7. Tentatively Identified Compounds (TICs) should only be considered if requested. For TICs, if the concentration in the sample is less than two times the concentration in the most contaminated associated blank (TIC concentration < 2xblank concentration), qualify the sample data as unusable (R).
8. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s) (i.e., exceeding the calibration range), evaluate the sample analysis results immediately after the high concentration sample for carryover. The system is considered uncontaminated if the target analyte is below the CRQL. Use professional judgment to determine if instrument cross-contamination has affected any positive compound identification(s). Note, for PO action, if instrument cross-contamination is suggested and suspected of having an effect on the sample results.

**NOTE:** There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

**Table 7. Blank Actions for TO-15 Analyses**

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument***	Detects	Not detected	No qualification required
	< CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL* and < 2x the CRQL**	Report concentration of sample with a “U”
		≥ 2x the CRQL**	No qualification required
	> CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL* and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL* and > blank concentration	No qualification required
	= CRQL*	≤ CRQL*	Report CRQL value with a U
		> CRQL*	No qualification required
	Gross contamination **	Detects	Report blank value for sample concentration with a U

\* 2x the CRQL for methylene chloride, 2-butanone and acetone.

\*\* 4x the CRQL for methylene chloride, 2-butanone, and acetone.

\*\*\* Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

Field or Trip blank when available should be assessed for possible contaminants in the canister used for trip blank. This canister and its analytical results are specific to the trip blank sample **only**. If contaminants are present in the canister used for trip blank, its suitability for use as trip blank can be assessed using the following criteria.

**Table 8. Field/Trip Blank suitability based on Canister contamination**

<b>Clean canister Result</b>	<b>Field/Trip Blank Result</b>	<b>Action for Field/Trip Blank</b>
Detects	Not detected	No qualification, no action for samples is required
Detects	< clean canister result or $\geq$ clean canister result but < 2X the clean canister result	Report as non-detect “U”, invalid as trip blank, no action for samples is required.
	$\geq$ 2x the clean canister result	No qualification, valid trip blank for sample actions.

## **Laboratory Control Sample/Laboratory Control Sample Duplicate**

Data for Laboratory control sample (LCS) and Laboratory Control Sample Duplicate (LCSD) is generated to ensure accuracy and reproducibility of the method and the laboratory. LCS and LCSD samples are analyzed using concentration in the middle of the calibration range and under the same conditions as samples to be analyzed. LCS/LCSD analysis should be performed once per 24 hour analytical sequence and concurrently with the samples in a given SDG. Actions for LCS/LCSD criteria are summarized below.

### **Action:**

**Table 9. LCS/LCSD Actions for TO-15 Analyses**

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
Percent recovery Criteria		
%R > Upper Acceptance Limit (>130%)	J	No qualification
%R in the acceptable range, 70-130%	No qualification	
%R < Lower Acceptance Limit (< 70 %)	J	UJ
%R < 50%	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	
Relative Percent Difference Criteria		
% RPD ≤ 25%	No qualification	
% RPD > 25 %	J	UJ

## **Internal Standards**

### **Action:**

1. If an internal standard area count for a sample or blank is greater than 140.0% of the area for the associated standard (CCV or mid-point standard from initial calibration) (see Table 10):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
  - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 60.0% of the area for the associated standard (CCV or mid-point standard from initial calibration):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
  - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 60.0%, and less than or equal to 140% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. If an internal standard RT varies by more than 20.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 20.0 seconds, no qualification of the data is necessary.
6. Note, for Laboratory Project Officer (PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.



**Table 10. Internal Standard Actions for TO-15 Analyses**

Criteria	Action	
	Detected Associated Compounds*	Non-detected Associated Compounds*
Area counts > 140% of CCV or mid-point standard from initial calibration)	J-	No qualification
Area counts < 60% of CCV or mid-point standard from initial calibration)	J+	R
Area counts $\geq$ 60% but $\leq$ 140% of CCV or mid-point standard from initial calibration)	No qualification	
RT difference > 20.0 seconds between samples CCV or mid-point standard from initial calibration)	R*	
RT difference < 20.0 seconds between samples and CCV or mid-point standard from initial calibration)	No qualification	

\* Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.

**Standards Data**

**Action:**

If any calibration standards data are missing, contact the Laboratory Project Officer to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

### **Target Compound Identification**

**Action:**

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Laboratory Project Officer (PO) action, the necessity for numerous or significant changes.

### **Tentatively Identified Compounds (TICs)**

#### **Action:**

**NOTE:** Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations.
2. General actions related to the review of TIC results are as follows:
  - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to “unknown” or another appropriate identification, and qualify the result as estimated (J).
  - b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as “either compound X or compound Y”. If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1, 3, 5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).
5. Target compounds from other fractions and suspected laboratory contaminants should be marked as “non-reportable”.
6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.

**Compounds Quantitation and Reported Contract Required Quantitation Limits (CRQLs)**

**Action:**

1. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample. Replace concentrations that exceed the calibration range in the original analysis by crossing out the “E” and its corresponding value on the original Form I and substituting the data from the diluted sample. This result value consolidation is also reflected in the EDDs and documented in the data assessment report.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. Note, for Laboratory Project Officer (PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

### **Field Duplicates**

**Action:**

**NOTE:** Criteria provided in the QAPP should be applied. In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Note large RPDs (> 50%) in the narrative. Use professional judgment to qualify data when RPD is > 50%.

### **System Performance**

**Action:**

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Note, for Laboratory Project Officer (PO) action, any degradation of system performance which significantly affected the data.

**Regional Quality Assurance (QA) and Quality Control (QC)**

**Action:**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Note, for Laboratory Project Officer (PO) action, unacceptable results for PE samples.



### **Overall Assessment of Data**

**Action:**

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for Laboratory Project Officer (PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

## APPENDIX A: GLOSSARY

**Analyte** -- The element of interest, ion, or parameter an analysis seeks to determine.

**Analytical Sample** -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), and daily calibration. Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); duplicate samples; serial dilution samples; Laboratory Control Samples (LCSs).

**Associated Samples** -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

**Blank** -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

**Calibration** -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

**Calibration Curve** -- A plot of instrument response versus concentration of standards.

**Calibration Standards** -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

**Case** -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

**Contract Compliance Screening (CCS)** -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract.

**Continuing Calibration Verification (CCV)** -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

**Laboratory Project Officer (PO)** -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a laboratory.

**Contract Required Quantitation Limit (CRQL)** -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

**Duplicate** -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

**Field Blank** -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

**Field Duplicate** -- A duplicate sample generated in the field, not in the laboratory.

**Holding Time** -- The maximum amount of time samples may be held before they are processed.

**Contractual** -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

**Technical** -- The maximum amount of time that samples may be held from the collection date until analysis.

**Initial Calibration** -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

**CCV or daily calibration**—Prior to sample analysis but after tuning criteria have been met, the initial calibration of each GC/MS must be routinely checked by analyzing a daily calibration standard to ensure initial calibration still holds and the instrument continues to remain under control. Typically this standard is the mid-level calibration standard that contains all the target compounds.

**Internal Standard** -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

**Matrix** -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrix is air.

**Method Detection Limit (MDL)** -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

**Narrative (SDG Narrative)** -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

**Office of Solid Waste and Emergency Response (OSWER)** -- The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

**Percent Difference (%D)** -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

**Performance Evaluation (PE) Sample** -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

**Method Blank** -- An analytical control that contains humid air and internal standards, which is analyzed under the same conditions as standards and sample.

**Relative Percent Difference (RPD)** -- As used in this document and the Statement of Work to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

**Relative Standard Deviation (RSD)** -- As used in this document and the Statement of Work, the mean divided by the standard deviation, expressed as a percentage.

**Sample** -- A single, discrete portion of material to be analyzed, which is contained in a canister and identified by a unique Sample Number.

**Sample Delivery Group (SDG)** -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
- b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

**Statement of Work (SOW)** -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

## APPENDIX B: ORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 2  
DESA/HWSB/HWSS  
2890, Woodbridge Avenue, Edison, NJ 08837

### EXECUTIVE NARRATIVE

Case No. :  
Site:  
Number of Samples:  
Analysis:

SDG No.:  
Laboratory:  
Sampling dates:

QAPP  
HWSS #:  
Contractor Document #:

#### **SUMMARY:**

**Critical:** Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

**Major:** A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated.

**Minor:** The level of uncertainty is acceptable. No significant bias in the data was observed.

#### Critical Findings:

#### Major Findings:

#### Minor Findings:

COMMENT:

Reviewer Name(s):

Approver's Signature:

Date:

Name:

Affiliation: USEPA/R2/HWSB/HWSS

**APPENDIX C: ELECTRONIC DATA DELIVERABLE TEMPLATE**

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		

# REQUEST FOR SOP CHANGE

Requestor Name:	Narendra Kumar	Date of Initiation:	July 20, 2023															
Dept.:	LSASD-HWSB-HWSS	SOP #: HW-31	Revision #: 6	Date: Sept. 2016														
SOP Title:	<b>Analysis of Volatile Organic Compounds in Air Contained in Canisters by Method TO-I5</b>																	
Please Check One		MINOR REVISION   .....		MAJOR REVISION   X														
<b>CHANGE(S)</b> (Use attachment if necessary):																		
<p><b><u>CHANGE FROM:</u></b></p> <p>1)</p> <table border="1" style="margin-left: auto; margin-right: auto; border-collapse: collapse;"> <thead> <tr> <th rowspan="2" style="text-align: center;">Matrix</th> <th rowspan="2" style="text-align: center;">Difference in initial and 24 hour pressure (psi) Criteria</th> <th colspan="2" style="text-align: center;">Action</th> </tr> <tr> <th style="text-align: center;">Detected Associated Compounds</th> <th style="text-align: center;">Non-Detected Associated Compounds</th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">Air</td> <td style="text-align: center;"><math>\leq 5</math></td> <td colspan="2" style="text-align: center;">No qualification</td> </tr> <tr> <td style="text-align: center;">Air</td> <td style="text-align: center;"><math>&gt; 5</math></td> <td style="text-align: center;">J</td> <td style="text-align: center;">UJ or R</td> </tr> </tbody> </table> <p>2)</p> <p><b><u>Field Duplicates</u></b>  <b>Action:</b>  <b>NOTE:</b> Criteria provided in the QAPP should be applied. In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.</p> <p>Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Note large RPDs (<math>&gt; 50\%</math>) in the narrative. Use professional judgment to qualify data when RPD is <math>&gt; 50\%</math>.</p> <p>3)</p> <p><b><u>Target Compound Identification</u></b>  <b>Action:</b></p> <p>1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were</p>					Matrix	Difference in initial and 24 hour pressure (psi) Criteria	Action		Detected Associated Compounds	Non-Detected Associated Compounds	Air	$\leq 5$	No qualification		Air	$> 5$	J	UJ or R
Matrix	Difference in initial and 24 hour pressure (psi) Criteria	Action																
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## REQUEST FOR SOP CHANGE

made, qualify all such data as unusable (R).

2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.

3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Laboratory Project Officer (PO) action, the necessity for numerous or significant changes.

### **CHANGE TO:**

1)

Matrix	Difference in initial and 24-hour pressure (psi) Criteria	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
Air	$\leq 2$	No qualification	
Air	$> 2$	J	UJ or R

2)

### **Field Duplicates**

#### **Action:**

- Identify which samples within the data package are field duplicates.
- Calculate the relative percent difference (RPD) of the results of these samples for each target analyte if both are detects.
- Note/highlight large RPDs ( $>$  QAPP criteria) in the narrative.
- Criteria and guidance provided in the QAPP should then be applied.
- In the absence of QAPP guidance for validating data for field duplicates, if both the results are detects,  $\geq$  CRQL and RPD is  $> 50\%$  note the difference in the Field Duplicates section of the narrative.

3)

### **Target Compound Identification**

#### **Action:**

- The Relative Retention Time (RRT) for a positively identified target analyte should be within  $\pm 0.06$  RRT units of the RRT for the same analyte in the associated opening CCV or mid-point standard from the associated ICAL.
- All ions present in the calibration standard mass spectrum should be present in the sample spectrum.

If mass spectrum does not match or the RRT is outside the specified RRT windows, qualify detects as unusable (R), or report the result at QL and qualify as non-detect (U).



## REQUEST FOR SOP CHANGE

<b>REASON(S) FOR CHANGE(S):</b> To clarify and add details for Data validation actions.		
<b>APPROVAL</b>	<b>NAME:</b>	<b>Signature/Date</b>
<b>EPA Branch Chief / Section Chief/Team Leader</b>	<b>Donna Ringel</b>	<i>Donna Ringel</i> 07/20/2023
<b>EPA TOCOR</b>	<b>Narendra Kumar</b>	<i>Narendra Kumar</i> 07/20/2023
<b>REQUESTOR</b>	<b>Narendra Kumar</b>	<i>Narendra Kumar</i> 07/20/2023
<b>Effective Date</b>	July 25, 2023	

# REQUEST FOR SOP CHANGE

Requestor Name:	Narendra Kumar	Date of Initiation:	April 9, 2024																											
Dept.:	LSASD-HWSB-HWSS	SOP #: HW-31	Revision #: 6      Date: Sept. 2016																											
SOP Title:	Analysis of Volatile Organic Compounds in Air Contained in Canisters by Method TO-15																													
Please Check One	MINOR REVISION    X	MAJOR REVISION    .....																												
<b>CHANGE(S)</b> (Use attachment if necessary):																														
<p><b>CHANGE FROM:</b></p> <p><b>b. Cleanliness:</b>  Integrity of the canister used for sampling of air for analysis should be maintained at all times including the time of shipment to the field, sampling, shipment back to the laboratory and time of analysis. Analytical results of canister cleaning verification must be taken into account in the validation of sample results. Canister contamination actions are stated in Table 2 below.</p> <p style="text-align: center;"><b>Table 2. Canister Contamination Actions for TO-15 Analyses</b></p> <table border="1" style="width: 100%; border-collapse: collapse; margin-top: 10px;"> <thead> <tr> <th style="width: 20%;">Contamination Type/level</th> <th style="width: 15%;">Canister Cleaning Result</th> <th style="width: 20%;">Sample Result</th> <th style="width: 45%;">Action for Samples</th> </tr> </thead> <tbody> <tr> <td rowspan="8" style="text-align: center; vertical-align: middle;">Clean Canister analysis</td> <td style="text-align: center;">Detects</td> <td>Analytes found in clean canister analysis are non-detects</td> <td>No qualification required</td> </tr> <tr> <td rowspan="3" style="text-align: center;">&lt;CRQL</td> <td style="text-align: center;">&lt; CRQL</td> <td>Report CRQL value with a U</td> </tr> <tr> <td style="text-align: center;">≥ CRQL and &lt; 2x the CRQL</td> <td>Report concentration of sample with a U</td> </tr> <tr> <td style="text-align: center;">≥ 2x the CRQL</td> <td>No qualification required</td> </tr> <tr> <td rowspan="3" style="text-align: center;">&gt; CRQL</td> <td style="text-align: center;">&lt; CRQL</td> <td>Report CRQL value with a U</td> </tr> <tr> <td style="text-align: center;">≥ CRQL and ≤ clean canister value</td> <td>Report clean canister value with a U</td> </tr> <tr> <td style="text-align: center;">≥ CRQL and &gt; clean canister value</td> <td>No qualification required</td> </tr> <tr> <td rowspan="2" style="text-align: center;">= CRQL</td> <td style="text-align: center;">≤ CRQL</td> <td>Report CRQL value with a U</td> </tr> <tr> <td style="text-align: center;">&gt; CRQL</td> <td>No qualification required</td> </tr> </tbody> </table>				Contamination Type/level	Canister Cleaning Result	Sample Result	Action for Samples	Clean Canister analysis	Detects	Analytes found in clean canister analysis are non-detects	No qualification required	<CRQL	< CRQL	Report CRQL value with a U	≥ CRQL and < 2x the CRQL	Report concentration of sample with a U	≥ 2x the CRQL	No qualification required	> CRQL	< CRQL	Report CRQL value with a U	≥ CRQL and ≤ clean canister value	Report clean canister value with a U	≥ CRQL and > clean canister value	No qualification required	= CRQL	≤ CRQL	Report CRQL value with a U	> CRQL	No qualification required
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## REQUEST FOR SOP CHANGE

### CHANGE TO:

#### **b. Cleanliness:**

Integrity of the canister used for sampling of air for analysis should be maintained at all times including the time of shipment to the field, sampling, shipment back to the laboratory and time of analysis. Analytical results of canister cleaning verification must be taken into account in the validation of sample results. Canisters may be requested individually cleaned or batch cleaned only. Missing, incomplete or inappropriate canister cleaning information should be addressed during data validation. Batch cleaning in and of itself should not be construed as a defect, if so requested. Project and SOW requirements should be considered in this evaluation along with professional judgment to qualify the data. Canister contamination actions are stated in Table 2 below.

**Table 2. Canister Contamination Actions for TO-15 Analyses**

Contamination Type/Level	Canister Cleaning Result	Sample Result	Action for Samples
Canister cleaning analysis	Missing, incomplete or inappropriate*	Detects	J
		Non-detects	No qualification
	Detects	Analytes found in clean canister analysis are non-detects	No qualification required
	< QL	< QL	Report QL value with a "U"
		$\geq$ QL and < 2x the QL	Report concentration of sample with a "U"
		$\geq$ 2x the QL	No qualification required
	$\geq$ QL	< QL	Report QL value with a "U"
		$\geq$ QL and $\leq$ clean canister value	Report clean canister value with a "U"
		$\geq$ QL and > clean canister value	No qualification required

\* Batch cleaning alone, if so requested, should not be considered incomplete or inappropriate.

## REQUEST FOR SOP CHANGE

**REASON(S) FOR CHANGE(S):**

To clarify actions for batch canister cleaning actions when individual canister cleaning was requested.

APPROVAL	NAME:	Signature/Date
EPA Branch Chief / Section Chief/Team Leader	Donna Ringel	<i>Donna Ringel</i> 04/10/2024
EPA TOCOR	Narendra Kumar	<i>Narendra Kumar</i> 04/09/2024
REQUESTOR	Narendra Kumar	
Effective Date	Date of signature	