

## APPENDICES

The following appendices were developed to assist the validator in volatile and semivolatile data validation. The appendices include method-specific Quality Control requirements for the following QC parameters: preservation and technical holding times, GC/MS instrument performance check (tuning), initial and continuing calibrations, blanks, surrogate compounds, internal standards, matrix spike/matrix spike duplicates, field duplicates, sensitivity check, PE samples/accuracy check, target compound identification, compound quantitation and reported quantitation limits, tentatively identified compounds, semivolatile cleanup, system performance, and overall evaluation of data.

Appendices are included for the following methods:

- Appendix A: CLP SOW OLM03.2/Volatile Organic Analysis
- Appendix B: CLP SOW OLM03.2/Semivolatile Organic Analysis
- Appendix C: CLP SOW OLC02.1/Low Concentration Volatile Organic Analysis
- Appendix D: CLP SOW OLC02.1/Low Concentration Semivolatile Organic Analysis

Additional appendices for other methods may be added as required.

Appendix E: VOA/SV Functional Guidelines Action Tables

## Appendix A

CLP/SOW OLM03.2/Volatile Organic Analysis  
Method QC criteria, Equations, and Definitions

## APPENDIX A

The following method QC criteria, equations, and definitions apply to data generated according to the USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM03.2, Exhibit D Volatiles.

**SECTION I: PRESERVATION & TECHNICAL HOLDING TIME CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-I-B for preservation and technical holding time data validation criteria.

**SECTION II: GC/MS INSTRUMENT PERFORMANCE CHECK (TUNING) CRITERIA**

Refer to the following method GC/MS instrument performance (tuning) QC criteria for data validation:

The analysis of the instrument performance (tuning) check solution (50 ng BFB on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, bromofluorobenzene (BFB), for volatile analysis must meet the ion abundance criteria given below:

<u>m/z</u>	<u>ION ABUNDANCE CRITERIA</u>
50	8.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95
95	Base Peak, 100% Relative Abundance
96	5.0 - 9.0% of m/z 95 (see note)
173	Less than 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120.0% that of m/z 95.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Part of the BFB peak must not be background subtracted.

**SECTION III: INITIAL CALIBRATION CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

The initial calibration standards must be analyzed upon contract award, whenever corrective action is taken which may change or affect the initial calibration criteria or if the continuing calibration acceptance criteria have not been met. Initial calibrations must be analyzed after the analysis of a compliant instrument performance check.

The initial calibration standards must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

All initial calibration standards must be analyzed at the following concentration levels: 10, 20, 50, 100, 200 ug/L (unheated for aqueous and medium level soils/heated for low level soils).

**Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**

**RELATIVE RESPONSE FACTOR (RRF)** - A measure of the relative mass spectral response of an analyte compared to its' internal standard. The RRF is calculated using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where,

- A<sub>x</sub> = Area of primary quantitation ion response (EICP) for the compound to be measured
- A<sub>is</sub> = Area of primary quantitation ion response (EICP) for the internal standard
- C<sub>is</sub> = Concentration of the internal standard
- C<sub>x</sub> = Concentration of the compound to be measured

**AVERAGE (MEAN) RELATIVE RESPONSE FACTOR (RRF)** - The average or mean RRF is determined by the analysis of five different standard concentrations and is used in calculating compound concentrations in samples. The RRF is calculated using the following equation:

$$\overline{RRF} = \sum_{i=1}^n \frac{RRF_i}{n}$$

Where,

RRF<sub>i</sub> = The individual RRFs for various concentration levels  
 n = The number of RRFs

**PERCENT RELATIVE STANDARD DEVIATION (%RSD)** - The % RSD for each compound is a measure of the linearity of the calibration curve. The % RSD is calculated using the following equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

Where,

$$\text{Standard Deviation} = \sqrt{\sum_{i=1}^n \frac{(x_i - \bar{x})^2}{(n-1)}}$$

$\bar{x}$  = Mean  
 n = total number of values  
 x<sub>i</sub> = each individual value used to calculate the mean

#### SECTION IV: CONTINUING CALIBRATION CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IV-B for continuing calibration data validation criteria and the following method continuing calibration QC criteria:

The continuing calibration standard must be analyzed once every 12 hours, following the analysis of a compliant instrument performance check and initial calibration, and prior to the analysis of field samples, QC samples and blanks.

The continuing calibration standard must include the target compounds listed in the Target Compound List (TCL) in Section XIII of this Appendix, as well as the internal standards and the system monitoring compounds.

Continuing calibration standards must be analyzed at a concentration level of 50 ug/L (unheated for aqueous and medium level soils/heated for low level soils).

**Note: The CLP SOW OLM03.2 minimum response factor method acceptance criterion differs from the Region I Functional Guidelines initial and continuing calibration minimum response factor validation criterion. If data quality objectives allow for greater variability of data, then an expanded minimum response factor validation criterion should be documented in the EPA-approved site-specific QAPjP or amendment to the QAPjP. If response factors less than 0.05 are allowed, then the validator should ensure that there is sufficient QC data to support the use of low response factors in sample calculations.**

**PERCENT DIFFERENCE (%D)** - The % D is used to compare the initial calibration RRF with the continuing calibration RRF50. The % Difference indicates both the direction and the magnitude of the comparison, i.e., the % Difference may be either negative, positive or zero.

$$\% \text{ Difference} = \frac{RRF_c - \overline{RRF}_i}{\overline{RRF}_i} \times 100$$

Where,

$\overline{RRF}_i$  = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

$RRF_c$  = Relative response factor from continuing calibration standard

## SECTION V: BLANK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-V-B for blank validation criteria and the following method QC criteria:

### Method Required Blanks

1. Method Blank - A 5.0 mL aliquot of reagent water or purified solid matrix approximate in weight or volume to the samples that is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. The method blank must be analyzed at least once during every 12 hour time period on each GC/MS system used for volatile analysis.
2. Storage Blank - Consists of two 40 mL VOA vials filled with reagent water prepared by the laboratory when the first samples in an SDG are received. The vials are stored, under the same conditions, with the field samples. After all of the samples in the SDG are analyzed, a 5.0 mL aliquot of the storage blank is analyzed to determine whether contamination was introduced during storage of the samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. A minimum of one storage blank must be analyzed per SDG after all samples for that SDG have been analyzed.
3. Instrument Blank - A 5.0 mL aliquot of reagent water that is carried through the entire analytical procedure and is analyzed following highly contaminated samples containing target compounds that exceed the initial calibration range. The instrument blanks are used to determine if contamination is introduced by a previous sample and the level associated with the analytical instrument. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. An instrument blank must be analyzed after a sample that exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the

system is considered contaminated.

## SECTION VI: SURROGATE COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VI-B for surrogate compound data validation criteria and the following method surrogate compound QC criteria:

The proper surrogate compounds must be quantified using correctly assigned internal standards and the correct primary quantitation ions.

10 uL of a 25 ug/mL solution of surrogate compounds Toluene-d<sub>8</sub>, 4-Bromofluorobenzene, 1,2-Dichloroethane-d<sub>4</sub> are added to 5 mL/5 g of sample, standard, QC sample, or blank for a final concentration of 50 ug/L or 50 ug/kg. 10 uL of a 25 ug/mL solution of internal standards Chlorobenzene-d<sub>5</sub> and Bromochloromethane are added to 5 mL/5 g of sample for a final concentration of 50 ug/L or 50 ug/kg. 10 uL of a 25 ug/mL solution of surrogate compounds Toluene-d<sub>8</sub>, 4-Bromofluorobenzene, 1,2-Dichloroethane-d<sub>4</sub> are added to 5 mLs of reagent water containing an aliquot of the methanol medium level soil/sediment extract.

Table App.A.VI-1 - CHARACTERISTIC IONS FOR SURROGATE COMPOUNDS

Surrogate	Characteristic Ions		Internal Standard
	Primary Quantitation Ion	Secondary Ion(s)	
Toluene-d <sub>8</sub>	98	70, 100	Chlorobenzene-d <sub>5</sub>
4-Bromofluorobenzene	95	174, 176	Chlorobenzene-d <sub>5</sub>
1,2-Dichloroethane-d <sub>4</sub>	65	102	Bromochloromethane

The surrogate % recovery is calculated using the following equation:

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Q<sub>d</sub> = Quantity of surrogate determined by analysis

Q<sub>a</sub> = Quantity of surrogate added to sample/blank

Table App.A.VI-2 - SURROGATE RECOVERY LIMITS

Surrogate	Method QC Criteria	
	Percent Recovery (Water)	Percent Recovery (Soil/Sediment)
Toluene-d <sub>8</sub>	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d <sub>4</sub>	76-114	70-121

If one or more sample surrogate recovery does not meet the acceptance criteria, the sample must be reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Reanalysis is not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate surrogate recoveries failed to meet the acceptance criteria. If the sample was reanalyzed and the surrogate recovery(ies) was acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the reanalysis also recovers the surrogate(s) outside of the acceptance limits, then both analyses should have been submitted.

## SECTION VII: INTERNAL STANDARDS CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VII-B for internal standard data validation criteria and the following method internal standard QC criteria:

The correct internal standard must be used for sample compound quantitation and the correct internal standard primary quantitation ion must be used for quantitation.

10 uL of a 25 ug/mL solution of internal standards Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d<sub>5</sub> are added to 5 mL/5 g of sample, standard, QC sample, or blank for a final concentration of 50 ug/L or 50 ug/kg.

Table App.A.VII-1 - VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

IS	IS	IS
<u>Bromochloromethane</u>	<u>1,4) Difluorobenzene</u>	<u>Chlorobenzene) d5</u>
Chloromethane	1,1,1) Trichloroethane	2) Hexanone
Bromomethane	Carbon Tetrachloride	4) Methyl) 2) Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2) Dichloropropane	1,1,2,2) Tetrachloroethane
Methylene Chloride	trans) 1,3) Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1) Dichloroethene	1,1,2) Trichloroethane	Styrene
1,1) Dichloroethane	Benzene	Xylene (total)
1,2) Dichloroethene(tot.)	cis) 1,3) Dichloropropene	4-Bromofluorobenzene (surr)
Chloroform	Bromoform	Toluene-d8 (surr)
1,2) Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d4 (surr)		

Table App.A.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR VOLATILE COMPOUNDS

Internal Standard	Characteristic Ions	
	Primary Quantitation Ion	Secondary Ion(s)
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d5	117	82, 119

Internal standard area counts for each of the internal standards must be within the inclusive range of -50.0% and +100.0% of the response of internal standards in the associated daily continuing calibration standard.

The retention time of the internal standard must not vary by more than  $\pm 30$  seconds from the retention time of the associated daily continuing calibration standard.

If one or more internal standard area count and/or retention time does not meet the acceptance criteria, then the sample must be reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Reanalysis is not required if the sample is a QC sample and both the matrix spike and matrix spike duplicate failed to meet the internal standard acceptance criteria. If the sample was reanalyzed and the internal standard area counts and/or retention times were acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the reanalysis also recovers the internal standard outside of the area count and/or retention time acceptance criteria, then both analyses should have been submitted.

#### **SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

The MS/MSD spike compounds listed below are spiked at 10 uL in a 25 ug/mL solution into the 5 mL or 5 g sample for a final concentration of 50 ug/L or 50 ug/kg.

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for each SDG, or each matrix within an SDG or each group of samples of a similar concentration level (soils only) whichever is most frequent. The following advisory matrix spike compound recoveries and RPDs are listed below:

#### **Table App.A.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS**

Compound	Method QC Criteria			
	Water		Soil/Sediment	
	% Recovery*	RPD**	% Recovery	RPD
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

\*The MS/MSD % recovery is calculated using the following equation:

$$\text{Matrix Spike Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

Where,

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

\*\*The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$\text{Relative Percent Difference} = \frac{|MSR - MSDR|}{1/2 (MSR + MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

Note: The vertical bars in the formula indicate the absolute value of the difference, hence RPD is always positive.

#### SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-IX-B for field duplicate data validation criteria.

#### SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-X-B for sensitivity check data validation criteria.

**SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XI-B for accuracy check data validation criteria.

**SECTION XII: TARGET COMPOUND IDENTIFICATION CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XII-B for target compound identification data validation criteria.

**SECTION XIII: COMPOUND QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIII-B for compound quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Volatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix A, Section VII. The daily RRF50 must be used for sample quantitation. The sample target compounds must be quantified using the following primary quantitation ions and must be reported to the CRQLs listed below.

Table App.A.XIII-1 - TARGET COMPOUND LIST (TCL), PRIMARY QUANTITATION AND SECONDARY IONS, AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR OLM03.1 SOW VOLATILE ORGANIC COMPOUNDS

Volatiles	CAS Number	Quantitation Limits				Characteristic Ions	
		Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)	Primary	Secondary
Chloromethane	74) 87) 3	10	10	1200	(50)	50	52
Bromomethane	74) 83) 9	10	10	1200	(50)	94	96
Vinyl Chloride	75) 01) 4	10	10	1200	(50)	62	64
Chloroethane	75) 00) 3	10	10	1200	(50)	64	66
Methylene Chloride	75) 09) 2	10	10	1200	(50)	84	49, 51, 86
Acetone	67) 64) 1	10	10	1200	(50)	43	58
Carbon Disulfide	75) 15) 0	10	10	1200	(50)	76	78
1,1) Dichloroethene	75) 35) 4	10	10	1200	(50)	96	61, 98
1,1) Dichloroethane	75) 34) 3	10	10	1200	(50)	63	65, 83, 85, 98, 100
1,2) Dichloroethene (total)	540) 59) 0	10	10	1200	(50)	96	61, 98
Chloroform	67) 66) 3	10	10	1200	(50)	83	85
1,2) Dichloroethane	107) 06) 2	10	10	1200	(50)	62	64, 100, 98
2) Butanone	78) 93) 3	10	10	1200	(50)	43*	57
1,1,1) Trichloroethane	71) 55) 6	10	10	1200	(50)	97	99, 117, 119
Carbon Tetrachloride	56) 23) 5	10	10	1200	(50)	117	119, 121
Bromodichloromethane	75) 27) 4	10	10	1200	(50)	83	85
1,2) Dichloropropane	78) 87) 5	10	10	1200	(50)	63	65, 114
cis) 1,3) Dichloropropene	10061) 01) 5	10	10	1200	(50)	75	77

Appendix A

OLM03.2/VOA

\* - m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Table App.A.XIII-1 - TARGET COMPOUND LIST (TCL), PRIMARY QUANTITATION AND SECONDARY IONS, AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR OLM03.1 SOW VOLATILE ORGANIC COMPOUNDS (CONT.)

Quantitation Limits								
Volatiles	CAS Number	Water ug/L	Low			On Column (ng)	Characteristic Primary	Ions Secondary
			Soil ug/Kg	Med. Soil ug/Kg	High Soil ug/Kg			
Trichloroethene	79) 01) 6	10	10	1200	(50)	130	95,97,132	
Dibromochloromethane	124) 48) 1	10	10	1200	(50)	129	208,206	
1,1,2) Trichloroethane	79) 00) 5	10	10	1200	(50)	97	83,85,99,132,134	
Benzene	71) 43) 2	10	10	1200	(50)	78	--	
trans) 1,3) Dichloropropene	10061) 02) 6	10	10	1200	(50)	75	77	
Bromoform	75) 25) 2	10	10	1200	(50)	173	171,175,250,252,254,256	
4) Methyl) 2) pentanone	108) 10) 1	10	10	1200	(50)	43	58,100	
2) Hexanone	591) 78) 6	10	10	1200	(50)	43	58,57,100	
Tetrachloroethene	127) 18) 4	10	10	1200	(50)	164	129,131,166	
Toluene	108) 88) 3	10	10	1200	(50)	91	92	
1,1,2,2) Tetrachloroethane	79) 34) 5	10	10	1200	(50)	83	85,131,133,166	
Chlorobenzene	108) 90) 7	10	10	1200	(50)	112	114	
Ethylbenzene	100) 41) 4	10	10	1200	(50)	106	91	
Styrene	100) 42) 5	10	10	1200	(50)	104	78,103	
Xylenes (Total)	1330) 20) 7	10	10	1200	(50)	106	91	

**SAMPLE CONCENTRATION** - The amount of analyte present in a sample is calculated using the RRF50 of the continuing calibration standard in the following equations:

Sample concentration for water:

$$\text{ug/L} = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Sample concentration for low level soil/sediment:

$$\text{ug/Kg (Dry weight basis)}' \frac{(A_x)(IS)}{(A_{is})(RRF)(W_s)(D)}$$

Sample concentration for medium level soil/sediment:

$$\text{ug/Kg (Dry weight basis)}' \frac{(A_x)(IS)(V_t)(1000)(Df)}{(A_{is})(RRF)(V_a)(W_z)(D)}$$

Where,

- $A_x$  = Area of the primary quantitation ion response (EICP) for the compound to be measured  
 $A_{is}$  = Area of the primary quantitation ion response (EICP) for the specific internal standard  
 IS = Amount of internal standard added in nanograms (ng)  
 RRF = Relative Response Factor from the (Ambient temperature purge for water and medium level soil/sediment and heated purge for low level soil/sediment) calibration standard  
 $V_o$  = Volume of water purged in milliliters (mL)  
 Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e.,  $V_o$  above) to the number of mL of the original water sample used for purging. If no dilution is performed, Df=1.  
 The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as follows:  

$$\frac{\text{uL most conc. extract used to make dilution} + \text{uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$
 $W_s$  = Weight of the sample added to the purge tube in grams (g)  
 $D = \frac{100 - \% \text{ Moisture}}{100}$   
 $V_t$  = Total volume of the methanol extract in milliliters (mL)  
 $V_a$  = Volume of aliquot of the methanol extract in microliters (uL) added to reagent water for purging  
 $W_z$  = Weight of soil/sediment extracted in grams (g)

## CRQL CALCULATIONS

Water:

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(V_x)}{(V_o)} \times (Df)$$

Where,

$V_o$  and  $Df$  are defined in the sample concentration equation above

$V_x$  = Contract sample volume (5 mL)

Soil/Sediment-Low:

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)}{(W_s)(D)}$$

Where,

$W_s$  and  $D$  are defined in the sample concentration equation above

$W_x$  = Contract sample weight (5 g)

Soil/Sediment-Med:

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(W_x)(V_t)(V_y)(Df)}{(W_s)(V_c)(V_a)(D)}$$

Where,

$V_t$ ,  $Df$ ,  $W_s$ ,  $V_a$ , and  $D$  are defined in the sample concentration equation above

$W_x$  = Contract sample weight (4 g)

$V_y$  = Contract soil aliquot volume from soil methanol extract (100 uL)

$V_c$  = Contract soil methanol extract volume (10,000 uL)

## SECTION XIV: TENTATIVELY IDENTIFIED COMPOUND CRITERIA

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XIV-B for tentatively identified compound (TIC) data validation criteria and the following method

TIC QC criteria:

The validator is required to report up to 30 TICs in the Data Validation Memorandum.

**TENTATIVELY IDENTIFIED COMPOUND CONCENTRATION** - The estimated concentration for non-target compounds tentatively identified shall be determined by the internal standard method using the following equations:

Sample concentration for water:

$$\text{ug/L} = \frac{(A_x)(IS)(Df)}{(A_{is})(RRF)(V_o)}$$

Sample concentration for low level soil/sediment:

$$\text{ug/Kg} = \frac{(A_x)(IS)}{(A_{is})(RRF)(W_s)(D)}$$

Sample concentration for medium level soil/sediment:

$$\text{ug/Kg} = \frac{(A_x)(IS)(V_t)(1000)(Df)}{(A_{is})(RRF)(V_a)(W_z)(D)}$$

Where,

- $A_x$  = Area of the primary quantitation ion response (EICP) for the non-target compound to be measured
- $A_{is}$  = Area of the primary quantitation ion response (EICP) for the specific internal standard
- IS = Amount of internal standard added in nanograms (ng)
- RRF = Relative Response Factor assumed to be 1
- $V_o$  = Volume of water purged in milliliters (mL)
- Df = Dilution Factor - The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e.,  $V_o$  above) to the number of mL of the original water sample used for purging. If no dilution is performed, Df=1. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as follows:  

$$\frac{\text{uL most conc. extract used to make dilution} + \text{uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$
- $W_s$  = Weight of sample added to the purge tube in grams (g)
- D =  $\frac{100 - \% \text{ Moisture}}{100}$
- $V_t$  = Total volume of the methanol extract in milliliters (mL)
- $V_a$  = Volume of aliquot of the methanol extract in microliters (uL) added to reagent water for purging
- $W_z$  = Weight of soil/sediment extracted in grams (g)

#### SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

Not applicable to volatile analysis.

**SECTION XVI: SYSTEM PERFORMANCE CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVI-B for system performance data validation criteria.

**SECTION XVII: OVERALL ASSESSMENT CRITERIA**

Refer to Region 1, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, Section VOA/SV-XVII-B for overall assessment data validation criteria.