Appendix B

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

APPENDIX B

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 Semivolatiles**.

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 DFTPP on column) must be performed at the beginning of each **12-hour** period during which samples or standards are analyzed. The tuning check, decafluorotriphenylphosphine (DFTPP), for semivolatile analysis must meet the ion abundance criteria given below:

m/z	ION ABUNDANCE CRITERIA
51	30.0 - 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base Peak, 100% Relative Abundance (see note)
199	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 - 110.0% of m/z 198
443	15.0 - 24.0% of m/z 442

Note: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110% that of m/z 198.

The mass spectrum of DFTPP 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 DFTPP. Part of the DFTPP 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 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.

2 uL of the initial calibration standard must be injected and all initial calibration compounds and system monitoring compounds must be analyzed at the following concentration levels; 10, 20, 40, 60, 80 ng/uL. However, the following eight compounds only require a four point calibration and are injected at concentration levels of 50, 80, 120, and 160 ng/uL: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol since detection at less than 50 ng per injection is difficult.

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_x = Area of primary quantitation ion response (EICP) for the compound to be measured
A_{is} = Area of primary quantitation ion response (EICP) for the internal standard
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

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

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

Where,

RRF_i = 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:

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

Where,

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

$\bar{x}$ = Mean
n = total number of values
x_i = 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 calibrations must be analyzed at a concentration level of 50 ng/2 uL except for the following eight compounds which are analyzed at a concentration level of 100 ng/2 uL: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, and pentachlorophenol since detection at less than 50 ng per injection is difficult.

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 $\overline{RRF}$ 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.

$$\% \ Difference = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \ x \ 100$$

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 data validation criteria and the following method QC criteria:

Method Required Blank

Method Blank - A volume of reagent water or purified solid matrix approximate in weight or volume to the samples which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the samples. All blanks are spiked with internal standards and surrogate compounds and blank analysis must meet internal standard and surrogate compound criteria. Method blank extraction and analysis must be performed once per each SDG, or each 20 samples in an SDG, or whenever samples are extracted by the same procedure, whichever is most frequent, and analyzed on each GC/MS system used to analyze associated samples.

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.

Surrogate compounds Nitrobenzene-d_5, 2-Fluorobiphenyl, Terphenyl-d_{14}, and 1,2-Dichlorobenzene-d_4 (advisory) are added to all samples, standards, QC samples, and blanks at a concentration of 100 ug/mL and surrogate compounds Phenol-d_5, 2-Fluorophenol, 2,4,6-Tribromophenol, and 2-Chlorophenol-d_4 (advisory) are added to all samples, standards, QC samples, and blanks at a concentration of 150 ug/mL.

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

Surrogate	Characteristic Ions		Internal Standard
	Primary Quantitation Ion	Secondary Ion(s)	
Nitrobenzene-d_5	82	128, 54	Naphthalene-d_8
2-Fluorobiphenyl	172	171	Acenaphthene-d_{10}
Terphenyl-d_{14}	244	122, 212	Chrysene-d_{12}
Phenol-d_5	99	42, 71	1,4-Dichlorobenzene-d_4
2-Fluorophenol	112	64	1,4-Dichlorobenzene-d_4
2,4,6-Tribromophenol	330	332, 141	Phenanthrene-d_{10}
2-Chlorophenol-d_4 (Recovery limits advisory)	132	68, 134	1,4-Dichlorobenzene-d_4
1,2-Dichlorobenzene-d_4 (Recovery limits advisory)	152	115, 150	1,4-Dichlorobenzene-d_4

The % surrogate recovery is calculated using the following equation:

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

Q_d = Quantity of surrogate determined by analysis

Q_a = Quantity of surrogate added to sample/blank

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

Surrogate	Method QC Criteria	
	Percent Recovery (Water)	Percent Recovery (Soil/Sediment)
Nitrobenzene-d_5 (B/N)	35-114	23-120
2-Fluorobiphenyl (B/N)	43-116	30-115
Terphenyl-d_{14} (B/N)	33-141	18-137
Phenol-d_5 (Acid)	10-110	24-113
2-Fluorophenol (Acid)	21-110	25-121
2,4,6-Tribromophenol (Acid)	10-123	19-122
2-Chlorophenol-d_4 (Acid)	33-110 (advisory)	20-130 (advisory)
1,2-Dichlorobenzene-d_4 (B/N)	16-110 (advisory)	20-130 (advisory)

If two or more acid or base neutral surrogate compounds fail to meet their recovery acceptance criteria, the laboratory should check calculations, sample preparation logs, the surrogate compound spiking solutions, and the instrument operation. If sample surrogate recoveries do not meet the acceptance criteria, as a result of the above mentioned problems or other unknown problems, the sample should be re-extracted and reanalyzed to determine if the sample matrix is interfering with the surrogate recoveries. Re-extraction and reanalysis are 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. Reanalysis is required if the failed surrogate recoveries are the result of instrument malfunction. If the sample was re-extracted and reanalyzed and the surrogate recoveries were acceptable in the reanalysis, then only the reanalysis should have been submitted. However, if the re-extracted/reanalyzed sample also recovers the surrogates 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 and the correct internal standard primary quantitation ion must be used for sample compound quantitation.

The internal standard compounds listed below are added into all samples, standards, QC samples, and blanks at a concentration of 40 ng/2 uL.

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

IS 1,4–Dichlorobenzene–d4	IS Naphthalene–d8	IS Acenaphthene–d10	IS Phenanthrene–d10	IS Chrysene–d12	IS Perylene–d12
Phenol	Nitrobenzene	Hexachlorocyclo–pentadiene	4,6–Dinitro–2–methylphenol	Pyrene	Di–n–octyl-phthalate
bis(2–Chloroethyl) ether	Isophorone	2,4,6–Trichloro–phenol	N–nitroso-di–phenylamine	Butylbenzyl-phthalate	Benzo(b)fluor–anthene
2–Chlorophenol	2–Nitrophenol	2,4,5–Trichloro–phenol	4–Bromophenyl-phenolether	3,3'–Dichloro–benzidine	Benzo(k)fluor–anthene
1,3–Dichlorobenzene	2,4–Dimethyl–phenol	2–Chloronaphthalene	Hexachloro–benzene	Benzo(a)–anthracene	Benzo(a)pyrene
1,4–Dichlorobenzene	bis(2–Chloro–ethoxy)methane	2–Nitroaniline	Pentachloro–phenol	bis(2–ethyl-hexyl)phthalate	Indeno(1,2,3–cd)-pyrene
1,2–Dichlorobenzene	2,4–Dichloro–phenol	Dimethylphthalate	Phenanthrene	Chrysene	Dibenz(a,h)-anthracene
2–Methylphenol	1,2,4–Trichloro–benzene	Acenaphthylene	Carbazole	Terphenyl–d14 (surr)	Benzo(g,h,i)-perylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3–Nitroaniline	Anthracene		
4–Methylphenol	4–Chloroaniline	Acenaphthene	Di–n–butyl-phthalate		
N–Nitroso–Di–n–propylamine	Hexachloro–butadiene	2,4–Dinitrophenol	Fluoranthene		
Hexachloroethane	4–Chloro–3–methylphenol	4–Nitrophenol	2,4,6-Tribromo-phenol (surr)		
2–Fluorophenol (surr)	2–Methylnaph-thalene	Dibenzofuran			
Phenol–d5 (surr)	Nitrobenzene–d5 (surr)	2,4–Dinitrotoluene			
2-Chlorophenol-d4 (surr)		2,6–Dinitrotoluene			
1,2-Dichlorobenzene-d4 (surr)		Diethylphthalate			
		4–Chlorophenyl phenylether			
		Fluorene			
		4–Nitroaniline			
		2–Fluorobiphenyl (surr)			

(surr) = surrogate compound

Table App.B.VII-2 - CHARACTERISTIC IONS FOR INTERNAL STANDARDS FOR SEMIVOLATILE COMPOUNDS

Internal Standard	Characteristic Ions	
	Primary Quantitation Ion	Secondary Ion
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

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 ± 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:

A matrix spike and matrix spike duplicate must be extracted and analyzed 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. The following advisory matrix spike compound recoveries and RPDs are listed below:

MS/MSD base neutral compounds 1,4-dichlorobenzene, N-nitroso-di-n-propylamine, 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, and pyrene are spiked at a concentration of 100 ug/mL and MS/MSD acid compounds phenol, 2-chlorophenol, 4-chloro-3-methylphenol, 4-nitrophenol, and pentachlorophenol are spiked at a concentration of 150 ug/mL.

Table App.B.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	Method QC Criteria			
	Water		Soil/Sediment	
	% Recovery*	RPD**	% Recovery	RPD
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

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

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

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

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

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

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 the 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:

Semivolatile target compounds must be quantitated using the internal standard method with the internal standards assigned in Appendix B, 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.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLMO3.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS

		Quantitation Limits					
		Low Water	Med Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
Phenol	108-95-2	10	330	10000	(20)	94	65,66
bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)	93	63,95
2-Chlorophenol	95-57-8	10	330	10000	(20)	128	64,130
1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)	146	148,113
1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)	146	148,113
1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)	146	148,113
2-Methylphenol	95-48-7	10	330	10000	(20)	108	107
2,2'-oxybis (1-Chloropropane)#	108-60-1	10	330	10000	(20)	45	77,79
4-Methylphenol	106-44-5	10	330	10000	(20)	108	107
N-Nitroso-di-n-propylamine	621-64-7	10	330	10000	(20)	70	42,101,130
Hexachloroethane	67-72-1	10	330	10000	(20)	117	201,199
Nitrobenzene	98-95-3	10	330	10000	(20)	77	123,65
Isophorone	78-59-1	10	330	10000	(20)	82	95,138
2-Nitrophenol	88-75-5	10	330	10000	(20)	139	65,109
2,4-Dimethylphenol	105-67-9	10	330	10000	(20)	107	121,122
bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)	93	95,123
2,4-Dichlorophenol	120-83-2	10	330	10000	(20)	162	164,98
1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)	180	182,145
Naphthalene	91-20-3	10	330	10000	(20)	128	129,127
4-Chloroaniline	106-47-8	10	330	10000	(20)	127	129
Hexachlorobutadiene	87-68-3	10	330	10000	(20)	225	223,227

Previously known by the name bis(2-Chloroisopropyl) ether

Table App.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLMO3.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits					
		Low Water	Med Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
4—Chloro—3—methylphenol	59—50—7	10	330	10000	(20)	107	144,142
2—Methylnaphthalene	91—57—6	10	330	10000	(20)	142	141
Hexachlorocyclopentadiene	77—47—4	10	330	10000	(20)	237	235,272
2,4,6—Trichlorophenol	88—06—2	10	330	10000	(20)	196	198,200
2,4,5—Trichlorophenol	95—95—4	25	830	25000	(50)	196	198,200
2—Chloronaphthalene	91—58—7	10	330	10000	(20)	162	164,127
2—Nitroaniline	88—74—4	25	830	25000	(50)	65	92,138
Dimethylphthalate	131—11—3	10	330	10000	(20)	163	194,164
Acenaphthylene	208—96—8	10	330	10000	(20)	152	151,153
2,6—Dinitrotoluene	606—20—2	10	330	10000	(20)	165	89, 121
3—Nitroaniline	99—09—2	25	830	25000	(50)	138	108,92
Acenaphthene	83—32—9	10	330	10000	(20)	153	152,154
2,4—Dinitrophenol	51—28—5	25	830	25000	(50)	184	63,154
4—Nitrophenol	100—02—7	25	830	25000	(50)	109	139,65
Dibenzofuran	132—64—9	10	330	10000	(20)	168	139
2,4—Dinitrotoluene	121—14—2	10	330	10000	(20)	165	63,182
Diethylphthalate	84—66—2	10	330	10000	(20)	149	177,150
4—Chlorophenyl—phenylether	7005—72—3	10	330	10000	(20)	204	206,141
Fluorene	86—73—7	10	330	10000	(20)	166	165,167
4—Nitroaniline	100—01—6	25	830	25000	(50)	138	92,108
4,6—Dinitro—2—methylphenol	534—52—1	25	830	25000	(50)	198	182,77
N—nitroso-di-phenylamine	86—30—6	10	330	10000	(20)	169	168,167
4—Bromophenyl—phenylether	101—55—3	10	330	10000	(20)	248	250,141
Hexachlorobenzene	118—74—1	10	330	10000	(20)	284	142,149
Pentachlorophenol	87—86—5	25	830	25000	(50)	266	264,268
Phenanthrene	85—01—8	10	330	10000	(20)	178	179,176
Anthracene	120—12—7	10	330	10000	(20)	178	179,176
Carbazole	86-74-8	10	330	10000	(20)	167	166,139
Di—n—butylphthalate	84—74—2	10	330	10000	(20)	149	150,104
Fluoranthene	206—44—0	10	330	10000	(20)	202	101,100
Pyrene	129—00—0	10	330	10000	(20)	202	101,100
Butylbenzylphthalate	85—68—7	10	330	10000	(20)	149	91, 206

Table App.B.XIII-1 - TARGET COMPOUND LIST (TCL), CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs), PRIMARY QUANTITATION IONS, AND SECONDARY IONS FOR OLMO3.1 SOW SEMIVOLATILE ORGANIC COMPOUNDS (CONT.)

		Quantitation Limits					
		Low Water	Med Soil	Soil	Column	Characteristic Ions	
Semivolatiles	CAS Number	ug/L	ug/Kg	ug/Kg	(ng)	Primary	Secondary
3,3'—Dichlorobenzidine	91—94—1	10	330	10000	(20)	252	254,126
Benzo(a)anthracene	56—55—3	10	330	10000	(20)	228	229,226
Chrysene	218—01—9	10	330	10000	(20)	228	226,229
bis(2—Ethylhexyl)phthalate	117—81—7	10	330	10000	(20)	149	167,279
Di—n—octylphthalate	117—84—0	10	330	10000	(20)	149	--
Benzo(b)fluoranthene	205—99—2	10	330	10000	(20)	252	253,125
Benzo(k)fluoranthene	207—08—9	10	330	10000	(20)	252	253,125
Benzo(a)pyrene	50—32—8	10	330	10000	(20)	252	253,125
Indeno(1,2,3—cd)pyrene	193—39—5	10	330	10000	(20)	276	138,227
Dibenz(a,h)anthracene	53—70—3	10	330	10000	(20)	278	139,279
Benzo(g,h,i)perylene	191—24—2	10	330	10000	(20)	276	138,277

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

Sample concentration for water:

$$ug/L = \frac{(A_x)\,(IS)\,(V_t)\,(Df)\,(GPC)}{(A_{is})\,(RRF)\,(V_i)\,(V_o)}$$

Sample concentration for low and medium level soil/sediment:

$$ug/Kg\ (Dry\ weight\ basis) = \frac{(A_x)\,(IS)\,(V_t)\,(Df)\,(GPC)}{(A_{is})\,(RRF)\,(V_i)\,(W_s)\,(D)}$$

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 most recent continuing calibration standard
Df = Dilution Factor - The dilution factor for analysis of water and soil/sediment samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.
W_s = Weight of sample extracted in grams (g)
D = $\frac{100 - \%\ \text{Moisture}}{100}$
V_t = Volume of the concentrated extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)
GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0)

CRQL CALCULATIONS

Water:

$$Adjusted\ CRQL = Contract\ CRQL\ x \frac{(V_x)\,(V_t)\,(V_y)\,(Df)}{(V_o)\,(V_c)\,(V_i)}$$

Where,

V_t, V_o, V_i and Df are defined in the sample concentration equation above
V_x = Contract sample volume (1000 mL)
V_y = Contract injection volume (2 uL)
V_c = Contract concentrated extract volume (1000 uL if GPC is not performed, 500 uL if GPC was performed).

CRQL CALCULATIONS

Soil/Sediment:

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

Where,

V_t, Df, W_s, V_i and D are defined in the sample concentration equation above
W_x = Contract sample weight (30 g for low level and 1 g for medium level soil/sediment samples).
V_y = Contract injection volume (2 uL)
V_c = Contract concentrated extract volume (1000 uL if GPC is not performed, 500 uL if GPC was performed).

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:

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

Sample concentration for low and medium level soil/sediment:

$$ug/Kg = \frac{(A_x)\ (IS)\ (V_t)\ (Df)\ (GPC)}{(A_{is})\ (RRF)\ (V_i)\ (W_s)\ (D)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the non-target compound to be measured
A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
IS = Amount of internal standard added in nanograms (ng)
RRF = Relative Response Factor is assumed to be 1
Df = Dilution Factor - The dilution factor for analysis of water and soil/sediment samples for semivolatiles by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.
W_s = Weight of sample extracted in grams (g)
D = $\frac{100 - \%\ Moisture}{100}$
V_t = Volume of the concentrated extract in microliters (uL)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (uL)
GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0)

SECTION XV: SEMIVOLATILE CLEANUP CRITERIA

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

GPC

Initial GPC calibration consists of analyzing the GPC calibration solution to establish the correct "Collect" and "Dump" time periods and a GPC blank to ensure that the system is free of contaminants.

1. The GPC Calibration Solution contains the following analytes in methylene chloride at the specified concentrations:

   corn oil - 25.0 mg/mL
   bis-(2-ethylhexyl)phthalate - 0.5 mg/mL
   methoxychlor - 0.2 mg/mL
   perylene - 0.02 mg/mL
   sulfur (optional) - 0.08 mg/mL

2. The GPC blank consists of methylene chloride.

The GPC must be recalibrated every 7 days with the GPC Calibration Solution followed by a methylene chloride GPC blank.

Table App.B.XVI-1 - INITIAL AND CONTINUING GPC CALIBRATION CRITERIA

Peak Resolution	Corn Oil and phthalate peaks must exhibit > 85% resolution. Phthalate and methoxychlor peaks must exhibit > 85% resolution. Methoxychlor and perylene peaks must exhibit > 85% resolution. Perylene and sulfur peaks (if sulfur was added) must not be saturated and must exhibit > 90% baseline resolution.
Peak Shape	Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
Retention Time	The retention times must not vary more than ± 5.0% between calibrations.
Blanks	A GPC blank must be analyzed after each initial GPC calibration. Target analytes cannot be present at greater than the CRQL for any target analyte except phthalate esters, which must be < 5x CRQL.

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.