## APPENDIX F

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, OLM04.3, Exhibit D Pesticides/Aroclors.

## SECTION I: PRESERVATION \& HOLDING TIME CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-I-B for preservation and holding time data validation criteria.

## SECTION II: GC/ECD INSTRUMENT PERFORMANCE CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-II-B for GC/ECD Instrument Performance Check data validation criteria and the following method GC/ECD instrument performance check QC criteria:

## Resolution Check Mixture

A Resolution Check Mixture containing the following analytes must be analyzed at the beginning of the initial calibration sequence. The resolution between two adjacent peaks in the Resolution Check mixture must be greater than or equal to $60.0 \%$.

Analyte Concentration ( $\mathrm{ng} / \mathrm{mL}$ )

| gamma-Chlordane | 10.0 |
| :--- | :--- |
| Endosulfan I | 10.0 |
| 4,4 '-DDE | 20.0 |
| Dieldrin | 20.0 |
| Endosulfan sulfate | 20.0 |
| Endrin ketone | 20.0 |
| Methoxychlor | 100.0 |
| Tetrachloro-m-xylene (surrogate) | 20.0 |
| Decachlorobiphenyl (surrogate) | 20.0 |

Resolution Check - The \% Resolution is calculated using the following equation:

$$
\% \text { Resolution }=\frac{V}{H} \times 100
$$

Where,
$\mathrm{V}=$ Depth of valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.
$\mathrm{H}=$ Height of the shorter peak.

## Example

IF:
Height of Peak $\mathrm{A}=2560$
Height of Peak B $=1435$

The resolution (depth of the valley) between the two peaks must be at least $60.0 \%$ of the shorter peak or Peak B in this situation $(0.6 \times 1435=861)$. If the resolution between Peak A and Peak B is less than 861 , the data will need to be qualified as indicated in Tables Pest/PCB-II-1 and Pest/PCB-II-2.

## Performance Evaluation Mixture

A Performance Evaluation Mixture (PEM) containing the analytes listed below must be analyzed at the beginning of the initial calibration sequence, immediately after the Resolution Check mixture and at the end of the initial calibration sequence. It must also be analyzed once during every 24 hours of the analytical sequence as part of the calibration verification. The resolution between two adjacent peaks in the PEM must be greater than or equal to $90.0 \%$. The percent breakdown for both DDT and Endrin in each PEM must be less than or equal to $20.0 \%$ for both GC columns. The combined percent breakdown for DDT and Endrin in each PEM must be less than or equal to $30.0 \%$ for both GC columns.

Analyte Concentration (ng/mL)

| gamma-BHC | 10.0 |
| :--- | :--- |
| alpha-BHC | 10.0 |
| $4,4^{\prime}-$ DDT | 100.0 |
| beta-BHC | 10.0 |
| Endrin | 50.0 |
| Methoxychlor | 250.0 |
| Tetrachloro-m-xylene (surrogate) | 20.0 |
| Decachlorobiphenyl (surrogate) | 20.0 |

Percent Difference (\% D) - The \% D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM analysis of the initial calibration sequence on each GC column must be less than or equal to $\pm 25.0 \%$. The $\% \mathrm{D}$ is calculated using the following equation:

$$
\text { \% Difference }=\frac{C_{\text {calc }}-C_{\text {nom }}}{C_{\text {nom }}} \times 100
$$

Where,

$$
\begin{aligned}
& \mathrm{C}_{\text {calc }}=\text { Calculated concentration of each analyte from the analyses of the calibration standards. } \\
& \mathrm{C}_{\text {nom }}=\text { Nominal concentration of each analyte. }
\end{aligned}
$$

```
            4,4'-DDT/ENDRIN BREAKDOWN CALCULATIONS
            % Breakdown DDT = { Amount found (ng)(DDD + DDE)
% Breakdown Endrin = Amount found(ng) (Endrin Aldehyde + Endrin Ketone)
Combined % Breakdown = % Breakdown DDT + % Breakdown Endrin
```


## SECTION III: INITIAL CALIBRATION CRITERIA

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

Each GC/ECD system must be initially calibrated using the following sequence:

## INITIAL CALIBRATION SEQUENCE

1. Resolution Check
2. Performance Evaluation Mixture
3. Aroclor 1016/1260
4. Aroclor 1221
5. Aroclor 1232
6. Aroclor 1242
7. Aroclor 1248
8. Aroclor 1254
9. Toxaphene
10. Low Point Standard A
11. Low Point Standard B
12. Midpoint Standard A
13. Midpoint Standard B
14. High Point Standard A
15. High Point Standard B
16. Instrument Blank
17. Performance Evaluation Mixture

Table App.F.III-1 - ANALYTES CONTAINED IN INDIVIDUAL STANDARD MIXTURE A AND
INDIVIDUAL STANDARD MIXTURE B

IND STD MIXTURE A
alpha-BHC

## LOW POINT CONCENTRATION (ng/mL)

5.0

Heptachlor
5.0
gamma-BHC 5.0
Endosulfan I 5.05.0

| Dieldrin | 10.0 |
| :--- | :--- |
| Endrin | 10.0 |
| 4,4'-DDD | 10.0 |
| 4,4'-DDT | 10.0 |
| Methoxychlor | 50.0 |
| Tetrachloro-m-xylene (surrogate) | 5.0 |
| Decachlorobiphenyl (surrogate) | 10.0 |
|  |  |
| IND STD MIXTURE B | LOW POINT CONCENTRATION (ng/mL) |
|  |  |
| beta-BHC | 5.0 |
| delta-BHC | 5.0 |
| Aldrin | 5.0 |
| Heptachlor epoxide | 5.0 |
| alpha-Chlordane | 5.0 |
| gamma-Chlordane | 5.0 |
| 4,4'-DDE | 10.0 |
| Endosulfan sulfate | 10.0 |
| Endrin aldehyde | 10.0 |
| Endrin ketone | 10.0 |
| Endosulfan II | 10.0 |
| Tetrachloro-m-xylene (surrogate) | 5.0 |
| Decachlorobiphenyl (surrogate) | 10.0 |

## Table App.F.III-2 - MULTICOMPONENT ANALYTE LOW POINT CONCENTRATIONS

## MULTICOMPONENT ANALYTES

LOW POINT CONCENTRATIONS (ng/mL)

| Aroclor 1016 | 100 |
| :--- | :--- |
| Aroclor 1221 | 200 |
| Aroclor 1232 | 100 |
| Aroclor 1242 | 100 |
| Aroclor 1248 | 100 |
| Aroclor 1254 | 100 |
| Aroclor 1260 | 100 |
| Toxaphene | 500 |

Multicomponent standards including the Aroclors and Toxaphene must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture.

## RETENTION TIME WINDOW CALCULATION

In the initial calibration (low, mid, and high point) the absolute retention times (RTs) are measured for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte. The mean RTs for single component standards and surrogates are calculated as the average of the three values and the RTs for multicomponent analytes are based on one value.

A retention time window for each single component pesticide and surrogate and for three to five major peaks of each multicomponent analyte are calculated using the values in Table App.F.III-3.

The following equation is used when calculating a mean absolute retention time for each single component pesticide and surrogate:

$$
\overline{R T}=\frac{\sum_{i=1}^{n}}{n} R T_{i}
$$

Where,
RT $=$ Mean absolute retention time of analyte.
$\mathrm{RT}_{\mathrm{i}}=$ Absolute retention time of analyte.
$\mathrm{n} \quad=\quad$ Number of measurements (3).

## Example

The retention time window is calculated by first taking the mean of the retention times from the low, mid, and high concentration of the individual standards in the initial calibration. For example the retention times for Endrin are:

Low - 9.86 Mid - 9.85 High - 9.86

Mean $=9.86$

Since we know from Table App. F. III-3 that the retention time window for Endrin is $\pm 0.07$, we add and subtract 0.07 to and from the Mean to calculate the retention time window for Endrin from the initial calibration.

RT Window for Endrin = 9.79-9.93

Table App.F.III-3 - RETENTION TIME WINDOWS FOR ANALYTES AND SURROGATES

| Analyte | Retention Time Window in Minutes |
| :--- | :---: |
| alpha-BHC | $\pm 0.05$ |
| beta-BHC | $\pm 0.05$ |
| gamma-BHC | $\pm 0.05$ |
| delta-BHC | $\pm 0.05$ |
| Heptachlor | $\pm 0.05$ |
| Aldrin | $\pm 0.05$ |
| alpha-Chlordane | $\pm 0.07$ |
| gamma-Chlordane | $\pm 0.07$ |
| Heptachlor Epoxide | $\pm 0.07$ |


| Analyte | Retention Time Window in Minutes |
| :--- | :---: |
| Dieldrin | $\pm 0.07$ |
| Endrin | $\pm 0.07$ |
| Endrin Aldehyde | $\pm 0.07$ |
| Endrin Ketone | $\pm 0.07$ |
| DDD | $\pm 0.07$ |
| DDE | $\pm 0.07$ |
| DDT | $\pm 0.07$ |
| Endosulfan I | $\pm 0.07$ |
| Endosulfan II | $\pm 0.07$ |
| Endosulfan Sulfate | $\pm 0.07$ |
| Methoxychlor | $\pm 0.07$ |
| Aroclors | $\pm 0.07$ |
| Toxaphene | $\pm 0.07$ |
| Tetrachloro-m-xylene (surrogate) | $\pm 0.05$ |
| Decachlorobiphenyl (surrogate) | $\pm 0.10$ |

## CALIBRATION FACTOR CALCULATION

In the initial calibration, peak areas or peak heights are measured to determine the analyte Calibration Factor (CF). The Calibration Factor for each single component pesticide and surrogate and for each peak in the selected set of 3 to 5 major peaks for each multicomponent analyte is calculated using equation III-1. The mean CF for each single component pesticide and surrogate is calculated using equation III-2. Note: The single component pesticide mean CF is only used in calculating the \% RSD and not for quantifying sample results.

Equation III-1 is as follows:

$$
C F=\frac{\text { Peak Area (height) of the standard }}{\text { Mass injected (ng) }}
$$

Equation III-2 is as follows:

$$
\overline{C F}={\frac{\sum_{i=1}^{n}}{n} C F_{i}}_{n}
$$

Where,
$\overline{\mathrm{CF}}=$ Mean calibration factor.
$\mathrm{CF}_{\mathrm{i}}=$ Calibration factor.
$\mathrm{n} \quad=\quad$ Total number of values (3).
\% RSD CALCULATION
Initial calibration data is used to assess the linearity of each GC/ECD system used for sample analysis. The linearity of the GC is assessed by calculating a \% RSD of the calibration factors for each single component pesticide and surrogate.

Equation III-3 is as follows:

$$
\% R S D=\frac{S D_{C F}}{\overline{C F}} \times 100
$$

Where,

$$
S D_{C F}=\sqrt{\sum_{i=1}^{n} \frac{\left(C F_{i}-\overline{C F}\right)^{2}}{(n-1)}}
$$

$$
\begin{aligned}
\overline{\mathrm{CF}} & =\text { Mean calibration factor. } \\
\mathrm{CF} & =\text { Each individual Calibration Factor used to calculate the mean. } \\
\mathrm{n} & =\text { Total number of values (3). } \\
\% \mathrm{RSD} & =\text { Percent Relative Standard Deviation. } \\
\mathrm{SD}_{\mathrm{CF}} & =\text { Standard Deviation of the calibration factors. }
\end{aligned}
$$

## SECTION IV: CALIBRATION VERIFICATION CRITERIA

Refer to the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IV-B for calibration verification data validation criteria and the following method calibration verification QC criteria:

The analyses of the instrument blanks, Performance Evaluation Mixtures (PEM), and the midpoint concentration of Individual Standard Mixtures A and B constitute the calibration verification. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

A valid analysis sequence is given below:

1. Sequence

| Time | Injection \# | Material Injected |
| :---: | :---: | :---: |
|  | 1-15 | First 15 steps of the Initial Calibration |
| 0 hr . | 16 | Instrument Blank* at the end of the Initial Calibration |
|  | 17 | PEM at the end of the Initial Calibration |
|  | 18 | First Sample |
|  | o |  |
|  | o | Subsequent Samples |
|  | o |  |
| 12 hr . | o | Last Sample |
|  | 1st injection past | Instrument Blank* |
|  | 12:00 hr. |  |
|  | 2 nd and 3rd injections | Individual Standard |
|  | past 12:00 hr. | Mixtures A and B |
|  | o | Sample |
|  | o |  |
|  | o | Subsequent Samples |
|  | o |  |
| Another 12 hr . | o | Last Sample |
|  | 1st injection past $12: 00 \mathrm{hr}$. | Instrument Blank* |
|  | 2nd injection past 12:00 hr. | Performance Evaluation Mixture |
|  | o | Sample |
|  | o |  |
|  | o | Subsequent Samples |
|  | o |  |
| Another 12 hr . | o | Last Sample |
|  | 1st injection past 12.00 hr | Instrument Blank* |
|  | 2 nd and 3rd injections | Individual Standard |
|  | past 12:00 hr . | Mixtures A and B |
|  | o | Sample |
|  | o |  |
|  | o | Subsequent Samples |

*The instrument blank contains only the surrogate analytes: Tetrachloro-m-xylene and Decachlorobiphenyl.
PERCENT DIFFERENCE (\% D) - The \% D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and Individual Mixture runs of the calibration verification on each GC column must be less than or equal to $\pm 25.0 \%$. The $\% \mathrm{D}$ is calculated using the following equation:

$$
\frac{\circ}{\circ} D=\frac{C_{\text {nom }}-C_{c a l c}}{C_{\text {nom }}} \times 100
$$

Where,
$\mathrm{C}_{\text {calc }}=$ Calculated concentration of each analyte from the analyses of the standards.
$C_{\text {nom }}=$ Nominal concentration of each analyte.

## Example

The nominal amount of gamma-BHC added to the PEM is $10 \mathrm{ng} / \mathrm{mL}$. The calculated amount of gamma-BHC is found to be $12 \mathrm{ng} / \mathrm{mL}$. Using the above equation, the percent difference is calculated as follows:

$$
\% \mathrm{D}=\frac{12-10}{10} \times 100=20 \%
$$

## Example

The nominal amount of $4,4^{\prime}$-DDT added to the INDA is $10 \mathrm{ng} / \mathrm{mL}$. The calculated amount of $4,4^{\prime}$-DDT is found to be $9.0 \mathrm{ng} / \mathrm{mL}$. Using the above equation, the percent difference is calculated as follows:

$$
\% \mathrm{D}=\frac{9.0-10}{10} \times 100=-10 \%
$$

## Example

The nominal amount of Aldrin added to the INDB is $5.0 \mathrm{ng} / \mathrm{mL}$. The calculated amount of Aldrin is found to be 7.0 $\mathrm{ng} / \mathrm{mL}$. Using the above equation, the percent difference is calculated as follows:

$$
\% \mathrm{D}=\frac{7.0-5.0}{5.0} \times 100=40 \%
$$

## SECTION V: BLANK CRITERIA

## Method Required Blanks

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-V-B for blank data calibration criteria and the following method QC criteria:

1. 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 entire set of samples. Surrogate analytes must recover between $30-150 \%$. A method blank must be extracted and analyzed once per each SDG, or each matrix within an SDG, or each extraction procedure within an SDG, or whenever samples are extracted, whichever is most frequent.
2. Sulfur Cleanup The sulfur cleanup blank is a modified form of the method blank. It is a volume of clean solvent spiked with the surrogates and carried through the sulfur cleanup and analysis procedures. This blank is used to determine the levels of contamination associated with the separate sulfur cleanup steps. Surrogate analytes must recover between $30-150 \%$. The sulfur cleanup blank is prepared separately when only part of a set of samples
extracted together requires sulfur removal. If all of the samples associated with a given method blank require sulfur cleanup, then the method blank must be subjected to sulfur cleanup and no separate sulfur cleanup blank is required.
3. Instrument Blank - The instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrument analysis itself, such as the carry-over of analytes from standards or highly contaminated samples into other analyses. An instrument blank must be analyzed after a sample whose concentration exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the system is considered contaminated.

A GPC blank, a type of instrument blank, is analyzed after the initial calibration of the GPC. It is not spiked with surrogate analytes.

## SECTION VI: SURROGATE ANALYTE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VI-B for the surrogate analyte data validation criteria and the following method surrogate analyte QC criteria:

Table App.F.VI-1 - SURROGATE RETENTION TIME WINDOWS

| Surrogate | Retention Time Window in Minutes |
| :---: | :---: |
| Tetrachloro-m-xylene | $\pm 0.05$ |
| Decachlorobiphenyl | $\pm 0.10$ |

The surrogate \% recovery is calculated using the following equation:

$$
\text { Surrogate Percent Recovery }=\frac{Q_{d}}{Q_{a}} \times 100 \%
$$

$Q_{d}=$ Quantity of surrogate determined by analysis.
$\mathrm{Q}_{\mathrm{a}}=$ Quantity of surrogate added to sample/blank.

Table App.F.VI-2 - SURROGATE SPIKE RECOVERIES

| Surrogate | Percent Recovery* <br> (Soil/Sediment and Water) |
| :---: | :---: |
| Tetrachloro-m-xylene | $30-150$ |
| Decachlorobiphenyl | $30-150$ |

[^0]
## SECTION VII: PESTICIDE CLEANUP CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST-VII-B for pesticide cleanup data validation criteria and the following method pesticide cleanup QC criteria:

## GPC

The Initial GPC Calibration consists of analyzing the GPC Calibration and GPC Calibration Verification solutions 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:

| corn oil | perylene |
| :--- | :--- |
| bis-(2-ethylhexyl)phthalate | sulfur |
| methoxychlor |  |

2. The GPC blank consists of methylene chloride.

Table App.F.VII-1 - GPC CALIBRATION ACCEPTANCE CRITERIA

| Peak | Corn Oil and phthalate peaks must exhibit $>85 \%$ resolution. <br> Resolution <br> Phthalate and methoxychlor peaks must exhibit $>85 \%$ resolution. <br> Methoxychlor and perylene peaks must exhibit $>85 \%$ resolution. <br> Perylene and sulfur peaks must not be saturated and must exhibit <br> $>90 \%$ baseline resolution. |
| :--- | :--- |
| Peak Shape | Peaks must be observed and symmetrical for all analytes in the <br> calibration solution. |
| Retention <br> Time | The retention times must not vary more than $\pm 5.0 \%$ between <br> calibrations. |
| Blanks | A GPC blank must be analyzed after each initial GPC calibration and <br> target analytes cannot be present at greater than the quantitation limit <br> for any target analyte. |

The Continuing GPC Calibration consists of analyzing the following two solutions in sequence every 7 days.

1. The Pesticide GPC Calibration Verification Solution contains the following 6 pesticide analytes (same as matrix spiking solution) in methylene chloride:

| gamma-BHC | dieldrin |
| :--- | :--- |
| heptachlor | endrin |
| aldrin | $4,4^{\prime}$-DDT |

2. The PCB GPC Calibration Verification Solution contains $2 \mathrm{ug} / \mathrm{mL}$ each of Aroclors 1016 and 1260 in methylene chloride.

## Table App.F.VII-2 - GPC CALIBRATION VERIFICATION CRITERIA

| \% Recovery of Single <br> Component Pesticides | $80-110 \%$ |
| :--- | :--- |
| Aroclor Patterns | Elution patterns must be the same as those from the <br> Aroclor 1016 and Aroclor 1260 standards in the <br> initial calibration sequence. |

## Florisil Cartridge

The Florisil Cartridge Performance Check consists of testing every lot of Florisil prior to use or every 6 months, whichever is more frequent, by analyzing the following two solutions in a mixture.

1. The Florisil Cartridge Check Solution contains 2,4,5-trichlorophenol in acetone, at a concentration of $0.1 \mathrm{ug} / \mathrm{mL}$.
2. The Pesticide Spiking Solution contains the following Standard Mixture A analytes at the midpoint concentration:

| alpha-BHC | $4,4 '^{\prime}-$ DDD |
| :--- | :--- |
| heptachlor | $4,4^{\prime}-$ DDT |
| gamma-BHC | methoxychlor |
| endosulfan I | tetrachloro-m-xylene |
| dieldrin | decachlorobiphenyl |
| endrin |  |

Table App.F.VII-3 - FLORISIL CARTRIDGE PERFORMANCE CHECK CRITERIA

| INDA Analyte \% Recovery | $80 \%-120 \%$ |
| :---: | :---: |
| $2,4,5-T C P$ Recovery | $2,4,5$-Trichlorophenol must recover at $<5.0 \%$ |
| Other Target Analytes | No interfering peaks with the target analytes |

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

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-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 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.

The following advisory matrix spike analyte recoveries and RPDs are listed below:

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

| Analyte | Method QC Criteria |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
|  | Water |  | Soil/Sediment |  |
|  | \% Recovery* | RPD** | \% Recovery | RPD |
| gamma-BHC (Lindane) | $56-123$ | 15 | $46-127$ | 50 |
| Heptachlor | $40-131$ | 20 | $35-130$ | 31 |
| Aldrin | $40-120$ | 22 | $34-132$ | 43 |
| Dieldrin | $52-126$ | 18 | $31-134$ | 38 |
| Endrin | $56-121$ | 21 | $42-139$ | 45 |
| 4,4 '-DDT | $38-127$ | 27 | $23-134$ | 50 |

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

$$
\text { Matrix Spike Recovery }=\frac{S S R-S R}{S A} \times 100
$$

Where,
SSR $=$ Spike Sample Result.
SR = Sample Result.
$\mathrm{SA}=$ Spike Added.
**The MS/MSD relative percent difference (RPD) is calculated using the following equation:

$$
\text { Relative Percent Difference }=\frac{|M S R-M S D R|}{1 / 2(M S R+M S D R)} \times 100
$$

Where,
MSR = Matrix Spike Recovery.
MSDR $=$ Matrix Spike Duplicate Recovery.
Note: The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always positive.

## SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IX-B for field duplicate data validation criteria.

## SECTION X: SENSITIVITY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-X-B for sensitivity check data validation criteria.

## SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XI-B for accuracy check data validation criteria.

## SECTION XII: TARGET ANALYTE IDENTIFICATION CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XII-B for target analyte identification data validation criteria.

## SECTION XIII: ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIII-B for analyte quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Pesticide/PCB analytes must be quantified using the external standard method. The midpoint calibration factor from the most recent compliant initial calibration must be used to quantify sample single component pesticides on both columns and the lower sample concentration must be reported. For multicomponent analytes, the calibration factors for three to five major peaks from the most recent compliant initial calibration are each used to quantify the multicomponent analyte in the sample. The three to five concentrations are then averaged and an analyte mean concentration for the sample is calculated for each column. The lower mean concentration is reported. Pesticides/PCBs must be reported to the CRQLs listed below:

## Table App.F.XIII-1 - TARGET ANALYTE LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

|  | Quantitation Limits <br> Water <br> $\mathrm{ug} / \mathrm{L}$ |  |  |
| :--- | :--- | :---: | :--- |
| Pesticides/Aroclors | Soil <br> $\mathrm{ug} / \mathrm{kg}$ | On Column <br> $(\mathrm{pg})$ |  |
| alpha-BHC | 0.050 | 1.7 | 5 |
| beta-BHC | 0.050 | 1.7 | 5 |
| delta-BHC | 0.050 | 1.7 | 5 |
| gamma-BHC | 0.050 | 1.7 | 5 |
| Heptachlor | 0.050 | 1.7 | 5 |
| Aldrin | 0.050 | 1.7 | 5 |
| Heptachlor epoxide | 0.050 | 1.7 | 5 |
| Endosulfan I | 0.050 | 1.7 | 5 |
| Dieldrin | 0.10 | 3.3 | 10 |
| 4,4'-DDE | 0.10 | 3.3 | 10 |


| Endrin | 0.10 | 3.3 | 10 |
| :--- | :--- | :--- | :--- |
| Endosulfan II | 0.10 | 3.3 | 10 |
| $4,4^{\prime}-$ DDD | 0.10 | 3.3 | 10 |
| Endosulfan sulfate | 0.10 | 3.3 | 10 |
| 4,4'-DDT | 0.10 | 3.3 | 10 |
| Methoxychlor | 0.50 | 17 | 50 |
| Endrin ketone | 0.10 | 3.3 | 10 |
| Endrin aldehyde | 0.10 | 3.3 | 10 |
| alpha-Chlordane | 0.050 | 1.7 | 5 |
| gamma-Chlordane | 0.050 | 1.7 | 5 |
| Toxaphene | 5.0 | 170 | 500 |
| Aroclor-1016 | 1.0 | 33 | 100 |
| Aroclor-1221 | 2.0 | 67 | 200 |
| Aroclor-1232 | 1.0 | 33 | 100 |
| Aroclor-1242 | 1.0 | 33 | 100 |
| Aroclor-1248 | 1.0 | 33 | 100 |
| Aroclor-1254 | 1.0 | 33 | 100 |
| Aroclor-1260 | 1.0 | 33 | 100 |

SAMPLE CONCENTRATION - Concentrations of single component pesticides and surrogates are calculated for both GC columns using the calibration factor from the initial calibration for the midpoint concentration of the external calibration standard in the following equations:

Sample concentrations for waters:

$$
u g / L=\frac{\left(A_{x}\right)(D F)\left(V_{t}\right)(G P C)}{(C F)\left(V_{o}\right)\left(V_{i}\right)}
$$

Sample concentration for soils:

$$
u g / K g=\frac{\left(A_{x}\right)(D F)\left(V_{t}\right)(G P C)}{(C F)\left(W_{s}\right)(D)\left(V_{i}\right)}
$$

Where,

$\mathrm{D}=\frac{100-\% \text { Moisture }}{100}$
GPC $=$ GPC factor (If no GPC is performed, GPC $=1$; if GPC is performed, then GPC $=2.0$ ).

## Adjusted CRQL $=$ Non-adjusted CRQL x Sample Dilution Factor

Sample concentrations for waters:

$$
\underset{C R Q L}{\text { Adjusted }}=\underset{C R Q L}{\text { Contract }} \times \frac{\left(V_{x}\right)\left(V_{t}\right)\left(V_{y}\right)(D F)}{\left(V_{o}\right)\left(V_{c}\right)\left(V_{i}\right)}
$$

Where,
$V_{t}, D F, V_{o}$, and $V_{i}$ are as given in the sample concentration equation above.
$\mathrm{V}_{\mathrm{x}}=$ Contract sample volume $(1000 \mathrm{~mL})$.
$\mathrm{V}_{\mathrm{y}}=$ Contract injection volume ( 1 uL or 2 uL$)$.
$\mathrm{V}_{\mathrm{c}} \quad=$ Contract concentration extract volume $(10,000 \mathrm{uL}$ if GPC was not performed and $5,000 \mathrm{uL}$ if GPC was performed).

Sample concentrations for soil/sediments:

$$
\underset{C R Q L}{\text { Adjusted }}=\underset{C R Q L}{\text { Contract }} \times \frac{\left(W_{x}\right)\left(V_{t}\right)\left(V_{y}\right)(D F)}{\left(W_{s}\right)\left(V_{c}\right)\left(V_{i}\right)(D)}
$$

Where,
$V_{t}, D F, V_{o}$, and $V_{i}$ are as given in the sample concentration equation above.
$\mathrm{W}_{\mathrm{x}} \quad=\quad$ Contract sample weight (30 g).
$\mathrm{V}_{\mathrm{y}}=$ Contract injection volume ( 1 uL or 2 uL$)$.
$\mathrm{V}_{\mathrm{c}}=$ Contract concentrated extract volume (GPC is required: 5000 uL ).

For example, the adjusted CRQL for a water sample with a 1.0 U non-diluted CRQL and a 1 to 100 dilution ( 100 dilution factor) would be 100 U , according to the following equation:

$$
100 \mathrm{U}=1.0 \mathrm{U} \times 100
$$

Adjusting for dry weight, the CRQL would be calculated as:

$$
\text { Dry Wt. CRQL }=\frac{\frac{\text { Non-adjusted Weight }}{100-\frac{\%}{\text { Moisture }}}}{100}
$$

For example, the dry weight CRQL for a soil sample with a 33 U non-adjusted CRQL and $10 \%$ moisture would be 37 U , according to the following equation:

$$
37 U=\frac{33 U}{\frac{100-10}{100}}
$$

The lower of the two concentrations calculated for each single component pesticide is reported on the tabulated Report Form I. For multicomponent analytes the lower mean concentration is reported. The percent difference between the two values is reported on Form X and calculated using the following equation:

PERCENT DIFFERENCE CALCULATION

$$
\% D=\frac{C_{h}-C_{1}}{C_{1}} \times 100
$$

Where,
$\mathrm{C}_{\mathrm{h}}=$ The higher of the two concentrations for the target analyte in question.
$\mathrm{C}_{1}=$ The lower of the two concentrations for the target analyte in question.

## SECTION XIV: SYSTEM PERFORMANCE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XIV-B for system performance data validation criteria.

## SECTION XV: OVERALL ASSESSMENT CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XV-B for overall assessment data validation criteria.


[^0]:    * Advisory for sample analysis. Mandatory for method blanks/sulfur cleanup blanks

