VI. SURROGATE ANALYTES

A. OBJECTIVE

Sample matrix effects and laboratory performance on individual samples are assessed by spiking the samples with surrogate analytes prior to extraction and analysis and determining their recoveries. Evaluation of surrogate recoveries is not necessarily straightforward. Interfering matrix effects, including high concentrations of target and/or non-target analytes, are frequently outside the control of the laboratory and may present relatively unique problems. Therefore, the evaluation and review of the surrogate analyte results are frequently subjective, demanding extensive analytical experience and professional judgment. Accordingly, this section consists primarily of guidance with several optional approaches suggested.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. The correct method-required surrogate analytes must be added to all samples, QC samples and blanks at the proper concentrations.

2. Recoveries for mandatory and advisory surrogate analytes in samples, QC samples and blanks must be within the QC acceptance criteria specified in the method.

3. The retention times for surrogates in samples, QC samples and blanks must be within the calculated retention time windows.

4. If surrogate analyte recoveries are outside the method QC acceptance criteria, then the pesticide/PCB sample must be reanalyzed in accordance with method requirements. If the recoveries are still outside the criteria, then the samples must be reextracted and reanalyzed.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from surrogate analyte anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
*1. Verify that the correct analytes were used as surrogate analytes and were added at the required concentration and frequency to all samples, QC samples, and blanks.	1. a. If surrogate analytes were not added to all samples, QC samples, and blanks, were added at the wrong concentration (for example a sample was "double" spiked) or an incorrect analyte was used, then the validator should use professional judgment to qualify or reject sample data. b. If surrogate analytes were diluted out of a sample, then the validator should use professional judgment to qualify or reject sample data. Greater than five-fold dilutions result in surrogate recovery data that may be analytically unusable.
2. Review Form II PEST to verify that no mandatory or advisory surrogate analyte recovery is outside the method QC acceptance criteria for pesticide/PCB field, QC, and blank samples. a. Determine whether or not a surrogate analyte was reported with a recovery above the upper QC acceptance limit on any GC column.	2. a. If a surrogate analyte in the pesticide/PCB sample has a recovery greater than the upper QC acceptance limit on any GC column, then the validator should: i. Use professional judgment to qualify positive detects in the affected sample based on the magnitude of the recovery and whether or not the upper limit was exceeded on more than one column. ii. Accept non-detects in the affected sample.

C. EVALUATION	D. ACTION
2. b. Determine whether or not a surrogate analyte was reported with a recovery below the lower QC acceptance limit on any GC column. If low surrogate recoveries are observed, then the validator should investigate whether the low recoveries were a result of sample dilution.	2. b. If a surrogate analyte in the pesticide/PCB sample has a recovery greater than or equal to 10% but less than the lower QC acceptance limit on any GC column, then the validator should: i. Use professional judgment to qualify positive detects in the affected sample based on the magnitude of the recovery and whether or not the lower limit was exceeded on more than one column. ii. Estimate (UJ) the sample quantitation limit for non-detects in the affected sample.
c. Determine if surrogate analytes were reported with extremely low recoveries, less than 10% on any GC column.	c. If a surrogate analyte in the pesticide/PCB sample recovers at less than 10% on any column, then the validator should: i. Estimate (J) positive detects in the affected sample. ii. Reject (R) non-detects as unusable in the affected sample. iii. If extremely low surrogate recoveries (less than 10%) were reported for the majority of samples in the sample delivery group, then the validator should use professional judgment to reject the entire pesticide/PCB fraction as unusable.

C. EVALUATION	D. ACTION
2. d. Determine if blank surrogate analyte recovery results meet method QC acceptance criteria.	2. d. In the special case of a blank analysis with a surrogate analyte recovery outside the method QC acceptance criteria, the validator must give special consideration to the validity of the associated sample data. The basic concern is whether or not the blank results represent an isolated problem with the blank, or whether there is a fundamental problem with the analytical process. For example, if most of the samples including other types of blanks in the batch demonstrate acceptable surrogate analyte recoveries, then the validator may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows the use of some of the affected data, analytical problems should be noted in the Data Validation Memorandum. All samples that were extracted with or analyzed after an out of control blank should be noted in the Data Validation Memorandum. Also, note in the Data Validation Memorandum if there are potential contractual problems associated with the failure to reextract and/or reanalyze blanks with surrogate analyte recoveries that were outside the method QC criteria.
3. a. Verify from Form VIII PEST that the absolute retention times for surrogates in the samples, QC samples and blanks are within the established retention time windows.	3. a. Retention time windows are essential to the qualitative identification of target analytes. Non-target analytes may appear as interferences in the retention time windows. The validator should be on guard for this possibility and look for interference trends throughout the entire case. If the surrogate analytes are not within the established retention time windows, then the validator should carefully evaluate the associated sample, QC sample, and blank results and raw data. This will necessitate a Tier III review.
* b. If reported retention times of the surrogate analytes are not within the established retention time windows, check the raw data for accurate identification of GC peaks. Non-recovery of surrogates may be due to shifts in retention time or matrix interference.	b. If the retention time of a surrogate analyte in the samples, QC samples, or blanks is outside of the calculated retention time windows, then the validator must use professional judgment to qualify the sample data. Refer to Section II. GC/ECD Instrument Performance Check, D.2 for guidance.

C. EVALUATION	D. ACTION
*3. c. Ten percent of the surrogate analyte raw retention time data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent should be checked. If errors are found in the additional ten percent, then the retention times of all peaks in the data package should be checked to evaluate whether or not results were reported accurately.	3. c. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. For Pesticide/PCB samples, verify that if surrogate analytes are outside the method QC acceptance criteria, then the required reextraction/reanalysis was performed to confirm that the non-compliance was due to sample matrix effects rather than poor laboratory performance.	4. If a laboratory fails to reextract and reanalyze a sample which is out of specification, then the sample data should be qualified or rejected according to the guidelines above. The validator should note this method deviation/contractual deficiency in the Data Validation Memorandum.
*5. a. Check raw data (e.g., chromatograms and quantitation reports) to verify that surrogate recoveries were reported accurately on the Surrogate Recovery Forms (Form II PEST-1 and Form II PEST-2).	5. a. If there are any transcription errors, then the validator should have the laboratory resubmit all corrected raw data and forms.
* b. Ten percent of the surrogate analyte recovery data should be checked for calculation and/or transcription errors. If errors are detected in this ten percent, then an additional ten percent of the data should be checked. If errors are found in the additional ten percent, then all surrogate analyte recovery calculations and transcriptions in the data package should be checked.	b. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.1, C.3.b, C.3.c, C.5.a, C.5.b

Table Pest/PCB-VI-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SURROGATE ANALYTE RECOVERIES

	Surrogate Analyte Recovery			
Sample Results	**One or more surrogates %Rec < 10%**	**One or more surrogates 10% ≤ %Rec < LL**	**All surrogates LL ≤ %Rec ≤ UL**	**One or more surrogates %Rec > UL**
Detects	J	Professional Judgment	A	Professional Judgment
Non-detects	R	UJ	A	A

LL - Lower Limit of method QC acceptance criteria
UL - Upper Limit of method QC acceptance criteria

Note: The surrogate recoveries in the method blank and the instrument blank must be within criteria for the analytical sequence to be valid.

E. EXAMPLES

Example #1: (Both pesticide surrogate recoveries < 10% on both columns)

Soil sample SA521, analyzed by CLP SOW OLM04.3, had TCX and DCB recoveries below 10% on both columns. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. SA521	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	5	7	30 - 150
Column 2	8	6	30 - 150

The validator estimates (J) positive detects and rejects (R) non-detects in sample SA521 on the Data Summary Table. The validator notes that low recoveries may be due to losses that occurred during the clean-up/extraction processes or chromatography problems and notes this in the Data Validation Memorandum.

E. EXAMPLES

Example #2: (One pesticide surrogate analyte recovery high on one column)

Soil sample MY207, analyzed by CLP SOW OLM04.3, had DCB recovered within advisory QC acceptance criteria on Column #1 and outside the upper limit of acceptance criteria on Column #2. TCX met advisory QC acceptance criteria on both columns. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. MY207	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	80	110	30 - 150
Column 2	125	155*	30 - 150

Non-detects are accepted. The validator uses professional judgment to determine that the high DCB surrogate recovery on Column #2 does not warrant qualification of the positive detects given that the criteria was only slightly exceeded on one column. The validator notes this in the Data Validation Memorandum.

Example #3: (Both pesticide surrogate analyte recoveries low on one column)

Soil sample NA351, analyzed by CLP SOW OLM04.3, had TCX and DCB recovered below the lower limit of QC acceptance criteria on Column #1 only (but greater than 10%). The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. NA351	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	15	12	30 - 150
Column 2	65	60	30 - 150

The validator uses professional judgment to determine that the TCX and DCB surrogate recoveries on Column #1 warrants qualification of the data. The validator notes that Column #1 data are suspect due to low recoveries and uses Column #2 to quantitate sample results. The validator estimates (J) positive detects and estimates (UJ) non-detects on the Data Summary Table. The validator documents this in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (One pesticide surrogate analyte recovery high on both columns)

Soil sample ZY409, analyzed by CLP SOW OLM04.3, had DCB recovered above the upper limit of QC acceptance criteria on both Column #1 and #2. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. ZY409	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	100	200	30 - 150
Column 2	95	180	30 - 150

Aroclor 1254 and 1260 were detected in sample ZY409. The validator reviews the sample and standard chromatograms. The validator uses professional judgment to surmise that DCB recoveries were enhanced by coelution with unidentified contamination from the sample and disregards the high DCB surrogate recoveries. The validator also notes that the multicomponent peaks chosen for quantitation did not interfere with the DCB peak, and therefore, determines that the Aroclor quantitation is accurate. The validator accepts positive Aroclor detects in sample ZY409 based upon the compliant TCX surrogate recoveries. The validator notes this in the Data Validation Memorandum.

Example #5: (Both pesticide surrogate analytes low on both columns)

Aqueous sample QA129, analyzed by CLP SOW OLM04.3, had TCX and DCB recovered below the lower limit of QC acceptance criteria on both Column #1 and #2, but above 10%. The following table lists the surrogate % recoveries and the QC acceptance criteria:

Sample No. QA129	TCX % Recovery	DCB % Recovery	QC Acceptance Criteria
Column 1	27	23	30 - 150
Column 2	25	28	30 - 150

The validator uses professional judgment to determine that the low TCX and DCB surrogate recoveries on both Column #1 and #2 warrants qualification of positive detects. The validator estimates (J) the positive detects and estimates (UJ) the non-detects in sample QA129. The validator notes this in the Data Validation Memorandum.

VII. PESTICIDE/PCB CLEANUP

A. OBJECTIVE

Pesticide/PCB cleanup procedures are utilized to remove matrix interferences from sample extracts prior to analysis. If not removed from the sample extracts, matrix interferences can inhibit accurate analyte identification and quantitation resulting in highly suspect data. Pesticide/PCB cleanup procedures are evaluated by spiking the cleanup columns or cartridges with target analytes and assessing the recovery of these analytes through the cleanup procedure.

Several types of pesticide cleanup procedures exist, including but not limited to:

1. **Gel Permeation Chromatography (GPC)** - removes high molecular weight contaminants

   GPC is a size exclusion procedure that utilizes organic solvents and hydrophobic gels to separate of macromolecules. The packing gel is porous and is characterized by the exclusion range (range of uniformity) of that pore size. The exclusion range must be greater than those of the molecules to be separated.

   General applications of GPC as a cleanup procedure include the removal of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids and dispersed high molecular-weight analytes from the sample extract.

   Under CLP SOW OLM04.3, the GPC column is packed with bead-like packing and connected to a UV detector. After the GPC is calibrated and a blank analyzed, sample extracts are loaded into sample loops and an automated sequence is started. The target analytes are eluted with methylene chloride and collected during the pre-determined retention times. The high molecular weight interferences, those outside the exclusion range, elute earlier than the pesticide/PCB analytes during the “dump” phase, while the smaller interferents such as sulfur elute with a later volume of solvent during the “wash” phase.

2. **Florisil Cartridge Cleanup** - reduces matrix interferences

   Florisil is a magnesium silicate with basic properties that is used in column chromatography to reduce matrix interferences caused by polar analytes in pesticide/PCB sample extracts.

   Florisil is used in the cleanup of pesticide residues and other chlorinated hydrocarbons, the separation of nitrogen analytes from hydrocarbons, and the separation of aromatic analytes from aliphatic-aromatic mixtures. Florisil is also used in separating steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates from pesticide analytes.

   A Florisil cleanup of pesticide/PCB extracts in hexane may be performed by transferring the extract to the top of a Florisil column and then eluting the column with a hexane/acetone mixture. The interferences are retained on the Florisil and the pesticide/PCB fraction is collected, concentrated and analyzed. Refer to CLP SOW OLM04.3 for method specific requirements.

   In some methods the Florisil cleanup is performed using multiple elutions of the cleanup column with hexane/ether mixtures of increasing polarity. The various eluant fractions, each containing different pesticide/PCB analytes, are then concentrated and analyzed either separately or as a combined extract. Refer to the EPA SW-846 method 3620B, December 1996 (or most recent revision), or EPA water method 608 for method-specific requirements.

3. **Sulfur Cleanup** - removes sulfur

   Sulfur cleanup eliminates elemental sulfur. Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analysis of the later eluting pesticides. Three techniques available to remove sulfur are: the Mercury Technique, the Copper Technique and the Tetrabutylammonium (TBA) -Sulfite Reagent Technique. Refer to the CLP SOW OLMO4.2 for mercury and copper clean-up method-specific requirements. Refer to the EPA SW-846 method 3660B, December 1996 (or most recent revision) for copper and TBA-sulfite clean-up method-specific requirements.

4. **Sulfuric Acid/Permanganate Cleanup - suitable only for PCB analysis** - removes most organic chemicals

   Sulfuric Acid/Permanganate cleanup destroys most organic chemicals including the pesticides Aldrin, Dieldrin, Endrin, Endosulfan (I and II), and Endosulfan sulfate. This method is suitable for the rigorous cleanup of sample extracts prior to analysis for PCBs. This method is used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs. Refer to the EPA SW-846 method 3665A, December 1996 (or most recent revision) for method-specific requirements.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP - Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. **Gel Permeation Chromatography**

   a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

   b. The GPC system must be calibrated initially in accordance with the method prior to the analysis of field samples, QC samples, or blanks to ensure acceptable solid phase activation, peak shape, and resolution of target analytes and interferents.

      i. GPC system must be calibrated and verified on a continuing basis at the frequency specified in the method.

      ii. The method-required GPC calibration and calibration verification solutions must contain target analytes and interferents at the method-required concentrations.

      iii. The calibration verification solution must be analyzed according to the analytical method. Target analyte recoveries must meet method QC acceptance criteria.

      iv. Aroclor patterns between standards that have undergone GPC and those that have not must be similar.

      v. Peak shapes must be symmetrical and resolution must meet method QC criteria.

vi. Retention time shifts between GPC calibrations must not exceed ±5% for bis(2-ethylhexyl)phthalate and perylene.

c. i. A GPC instrument blank must be analyzed after each GPC calibration and prior to sample analysis.

ii. Target analytes must not be present at greater than or equal to the quantitation limit for any target analyte in the GPC instrument blank.

2. **Florisil Cartridge Cleanup**

a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

b. Each lot number of cleanup cartridges must be checked in accordance with the method prior to use to ensure acceptable solid phase activation and acceptable recovery of target analytes.

c. i. The cartridge performance check must be conducted at the frequency specified in the method.

ii. The cartridge performance check must be analyzed on a GC/EC meeting the initial calibration and calibration verification technical acceptance criteria.

iii. Percent recoveries for Florisil Cartridge Performance Check solutions, which contain analytes of interest and surrogate analytes must meet method QC acceptance criteria.

d. i. All QC samples associated with the sample extracts that are cleaned up using this method must also be processed through this cleanup method. QC samples must meet method QC acceptance criteria after Florisil cartridge cleanup.

3. **Sulfur Cleanup**

a. Pesticide/PCB sample extracts, QC sample extracts, and method blank extracts must undergo all cleanup procedures required by the method.

b. Sulfur removal is used for sample extracts containing sulfur that may interfere with the analysis of target analytes.

c. i. A sulfur blank is prepared separately when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then the method blank must also be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

ii. The sulfur cleanup blank is a modified form of the method blank, and, other than the frequency stated above, must meet all method QC criteria specified for the method blank.

4. **Sulfuric Acid/Permanganate Cleanup - suitable only for PCB analysis**

a. Sample extracts, QC sample extracts, and method blank extracts for PCB analysis must undergo all cleanup procedures required by the method.

b. Sulfuric Acid/Permanganate cleanup is used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

c. i. Blanks and replicate analysis samples must be subjected to the same cleanup procedures as the samples associated with them.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from pesticide cleanup anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Gel Permeation Chromatography (GPC)	**1. Gel Permeation Chromatography (GPC)**
a. Verify from Form I PEST and Form IX PEST-2 that GPC cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If GPC was not performed according to the analytical method on all method-required extracts, then the raw data should be reviewed for the presence of high molecular weight contaminants and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if GPC was required by the method.

C. EVALUATION	D. ACTION
*1. b. GPC Calibration	1. b. GPC Calibration
* i. Verify that the GPC system was calibrated initially in accordance with the method requirements and that peak shape and resolution criteria were met.	i. If the GPC system was not calibrated initially in accordance with the method (prior to the analysis of field samples, QC samples or blanks) or fails to meet peak shape and/or resolution criteria or the initial calibration data are not available for review, then the validator should evaluate the last calibration verification analyzed just prior to sample analysis.
* ii. Review the raw GPC calibration data to verify that peaks are symmetrical and resolution meets method QC acceptance criteria for target analytes and interferents in the GPC calibration solution.	ii. If the GPC calibration method QC acceptance criteria do not meet peak shape and analyte resolution, then the raw sample data should be examined for the presence of high molecular-weight interferences or the loss of late eluting target analytes and professional judgment should be used to qualify or reject sample data. The validator should discuss the impact of unacceptable peak shape and resolution on the sample data in terms of high or low bias and/or the possibility of false negatives and note this in the Data Validation Memorandum.
* iii. Check the raw GPC calibration data to verify that retention times for bis(2-ethylhexyl)phthalate and perylene in the GPC calibration solution did not vary more than ± 5% between calibrations	iii. Retention time shifts indicate instrument performance problems that require laboratory corrective actions. If retention time shifts are excessive, the GPC cleanup procedure may be the cause of analyte losses and false negatives, and the validator should evaluate the sample data carefully and document all deficiencies in the Data Validation Memorandum.
* iv. Check the collect and dump cycle times in the GPC calibration chromatogram and compare it with the samples collect and dump cycle times. Verify that retention times have not shifted between the calibration and the sample runs.	iv. All samples collect and dump cycle times should be consistent with the calibration. If retention times have shifted, the dump and collection times determined by the calibration standard no longer will be appropriate. Professional judgement should be used to evaluate the data and qualify the data appropriately.

C. EVALUATION	D. ACTION
*1. c. GPC Blank	1. c. GPC Blank
* i. Verify that a GPC instrument blank was analyzed after each GPC calibration and prior to sample analysis.	i. If a GPC instrument blank was not analyzed at the correct frequency and in the proper sequence, then the validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
* ii. Verify that there are no target analytes present at greater than or equal to the quantitation limit in the GPC instrument blank.	ii. If any target analytes are detected in the GPC instrument blank at greater than or equal to the quantitation limit, then the quality of the GPC operation is suspect. The validator must use professional judgment in conjunction with the blank guidance provided in Section V to qualify or reject sample data.
d. GPC Calibration Verification	d. GPC Calibration Verification
* i. Confirm from the raw data that the GPC calibration verification was performed at the method-required frequency.	i. If GPC calibration verifications have not been performed at the method-required frequency, then the quality of the GPC operation may be suspect and the validator should use professional judgment to qualify or reject sample data.
* ii. Verify that a GPC calibration verification solution was analyzed in accordance with the method and that the correct target analytes, interferents, and concentrations were used.	ii. If a GPC calibration verification solution was not analyzed in accordance with the method or the correct analytes and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
1. d. iii. Verify from Form IX PEST-2 that GPC calibration verification solution analyses meet method QC acceptance criteria for target analyte recoveries.	1. d. iii. If GPC calibration verification method QC acceptance criteria are not met, then the GPC calibration verification solution results should be used to qualify sample data for specific analytes included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution analytes, taking into consideration the analyte's chemical class. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) all positive detects in all samples associated with that GPC calibration verification to indicate potential high bias. - Accept all quantitation limits for non-detects in all samples associated with that GPC calibration verification. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification to indicate potential low bias.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration verification to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that GPC calibration verification to indicate potential low bias. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If a GPC calibration verification analyte recovery is less than 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that GPC calibration verification to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that GPC calibration verification to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
1. d. iii. Continued from above.	1. d. iii. Continued from above. • If more than half of the GPC calibration verification analyte recoveries are less than 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that GPC calibration verification to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that GPC calibration verification to indicate that the data are unusable due to the possibility of false negatives. Professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the GPC calibration verification analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the GPC column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum. • If more than half of the GPC calibration verification analyte recoveries are outside the method QC acceptance limits in one GPC calibration verification, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that GPC calibration verification.

C. EVALUATION	D. ACTION
1. d. iv. Verify that Aroclor patterns in the GPC calibration verification analysis are similar to the corresponding Aroclor standard patterns of the Initial Calibration sequence.	1. d. iv. If Aroclor patterns of GPC calibration verification are not similar to the corresponding Aroclor patterns of the Initial Calibration sequence, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.
* e. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	e. If the laboratory made any calculation and/or transcription errors, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
f. Review surrogate, MS/MSD, and PES data to evaluate the operational effectiveness of the GPC cleanup.	f. If any analyte or analyte class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided in Sections VI, VIII and XI of Part III-Pest/PCB.

C. EVALUATION	D. ACTION
2. Florisil Cartridge Cleanup	**2. Florisil Cartridge Cleanup**
a. Verify from Form IX PEST-1 that a Florisil cartridge cleanup was performed according to the analytical method on all method-required sample extracts, QC sample extracts, and method blank extracts.	a. If Florisil cartridge cleanup was not performed according to the analytical method on all method-required extracts, then the data should be reviewed for the presence of interferents and professional judgment should be used to qualify or reject sample data. The validator should request sample cleanup and reanalysis if Florisil cartridge cleanup was required by the method.
b. i. Verify from Form IX PEST-1 that each Florisil cartridge lot used to cleanup samples was checked at least once prior to use, and at the proper frequency, in accordance with method requirements. Cartridges should be checked at least once for every 300 cartridges of a particular lot (EPA SW-846 method 3620B) or every 6 months of use for a particular lot (CLP SOW OLM04.3).	b. i. If each Florisil cartridge lot was not checked or was not checked at the proper frequency, then the solid phase may not be properly activated potentially resulting in unacceptable target analyte recoveries, the presence of interferents and possibly the loss of target analytes (false negatives). The validator should review the Florisil Cartridge Check recovery data associated with each batch of Florisil cartridge cleanups to ascertain if any target analytes should be qualified or rejected using the guidance provided in b.iii and c.
ii. Verify from Form IX PEST-1 that a Florisil Cartridge Performance Check solution was prepared and analyzed in accordance with the method and that the correct target and surrogate analytes, interferents, and concentrations were used.	ii. If a Florisil Cartridge Performance Check solution was not prepared and analyzed in accordance with the method or the correct analytes and/or concentrations were not used, then the data quality may be adversely affected. In these circumstances, the validator should use professional judgment to qualify or reject sample data.

C. EVALUATION	D. ACTION
2. b. iii. Check the reported data from the Florisil Cartridge Performance Check solution analyses on Form IX PEST-1 to verify that the target analyte recoveries meet method QC acceptance criteria.	2. b. iii. If Florisil Cartridge Check method QC acceptance criteria are not met, then the Florisil Cartridge Performance Check solution results should be used to qualify sample data for specific analytes included in the check solution. Professional judgment should be used to qualify or reject sample data for non-check solution analytes. The validator should discuss the impact of unacceptable recoveries on the sample data in terms of high or low bias and note this in the Data Validation Memorandum. • If a Florisil Cartridge Performance Check solution analyte recovery is greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are greater than the upper limit of the method QC acceptance criteria, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that Florisil Cartridge Performance Check solution.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If a Florisil Cartridge Performance Check solution analyte recovery is less than the lower limit but greater than 10% of the method QC acceptance criteria, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are less than the lower limit of the method QC acceptance criteria but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If a Florisil Cartridge Performance Check solution analyte recovery is less than the 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any sample associated with that Florisil Cartridge Performance Check solution to indicate that the data are unusable due to the possibility of false negatives. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are less 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that Florisil Cartridge Performance Check solution to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. b. iii. Continued from above.	2. b. iii. Continued from above. • If more than half of the Florisil Cartridge Performance Check solution analyte recoveries are outside the method QC acceptance limits in one Florisil Cartridge Check, where some recoveries are low and some recoveries are high, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes, or the entire fraction for samples associated with that Florisil Cartridge Check. • If the 2,4,5-trichlorophenol recovery was ≥ 5% in the analysis of the Florisil Cartridge Performance Check solution, then the Florisil is not adequately removing non-target analytes and the sample data must be evaluated for potential interferences. Professional judgment should be used to qualify or reject sample data. This may necessitate a Tier III validation. In all cases, professional judgement should be used to evaluate positive detects for analytes which had acceptable recoveries in the Florisil Cartridge Performance Check solution analyses. These analytes may be acceptable after taking into consideration the chemical class of the analytes and their elution order on the Florisil column. The validator should also document and justify all technical decisions made based on professional judgement in the Data Validation Memorandum.
* iv. Verify that surrogate analyte recoveries in the Florisil Cartridge Performance Check solution meet method QC acceptance criteria.	iv. If surrogate analyte recoveries and/or retention times in the Florisil Performance Check solution do not meet method QC acceptance criteria, then the validator should use professional judgement to assess the sample data and qualify the sample data in accordance with Section VI, Surrogate Analytes.

C. EVALUATION	D. ACTION
2. c. Verify from Form IX PEST-1 that all QC samples and method blanks associated with the sample extracts that were Florisil cleaned were also Florisil cleaned. All QC samples and method blanks must meet method-specified criteria after Florisil cleanup.	2. c. If Florisil cartridge cleanup was not performed for associated QC samples and/or method blanks, then the data should be reviewed for potential impacts and professional judgment should be used to qualify or reject sample data. If the QC samples and method blanks do not meet QC criteria after Florisil cleanup, then the validator should refer to the appropriate section of Part III-Pest/PCB, and use professional judgment to qualify sample data.
* d. Compare the raw data, if available, to the reported results and verify that no calculation and/or transcription errors have occurred. If result forms are not available, then the validator must review the cleanup logs to confirm that method required cleanups were performed.	d. If the laboratory made any calculation and/or transcription errors, then the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
e. Review MS/MSD, surrogate, and PES data to evaluate the operational effectiveness of the Florisil Cartridge cleanup.	e. If any analyte or analyte class has zero recovery indicating the possibility of false negatives and/or recovers low indicating a potential low bias, then the validator should discuss the possible false negatives and/or potential low bias in the Data Validation Memorandum and qualify and/or reject sample results according to the guidance provided in Sections VI, VIII and XI of Part III-Pest/PCB.

C. EVALUATION	D. ACTION
3. Sulfur Cleanup	**3. Sulfur Cleanup**
a. i. Review the Form I Pest to ascertain if sulfur cleanup was performed on any sample extracts, and associated QC samples and method blanks.	a. i. If a Tier II validation is being performed, then the validator should note that sulfur cleanup was performed and that reducing conditions may exist at the sample site location.
* ii. Check the field sample GC chromatograms to determine whether or not there is a flat baseline. A rising baseline may indicate the presence of sulfur. Confirm that all pesticide/PCB peaks are adequately resolved and are symmetrical.	ii. If a method-required sulfur cleanup was not performed on sample extracts that contain sulfur or adequate sulfur removal was not achieved, then the validator should carefully assess the impact on the sample data. If only minor sulfur interference is observed, then the validator should use professional judgment to estimate (J) positive detects for analyte(s) that coelute with sulfur and reject (R) non-detects. If the sulfur peak obscures a limited, discrete portion of the chromatogram, then the validator should use professional judgment to reject (R) the positive detects and non-detects for analytes coeluting with sulfur in that portion of the chromatogram and accept the unaffected sample results. If the sulfur contamination is gross and the majority of the chromatogram is obscured, then the validator should use professional judgment to reject (R) the entire pesticide/PCB analysis for that sample. The validator should request sample reanalysis that includes sulfur removal.
* iii. Confirm from the raw data, laboratory bench sheets, or SDG Narrative, that a method-required cleanup technique was used to remove any sulfur present in the samples	iii. If a method-required sulfur cleanup technique was not used for sulfur removal, then the validator should request sample cleanup and reanalysis and document all technical decisions in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
3. b. i. Verify from Form IV PEST that a sulfur cleanup blank was prepared and analyzed along with samples, or that the associated method blank was also sulfur cleaned.	3. b. i. If a sulfur cleanup blank was not prepared and/or analyzed with the samples, or the associated method blank was not also sulfur cleaned, then the validator should use professional judgment to qualify sample data.
ii. Verify that the sulfur cleanup blank met all method QC acceptance criteria specified for the method blank (refer to Section V, Blanks).	ii. If the sulfur cleanup blank does not meet QC criteria after sulfur cleanup, then the validator should refer to Section V, Blanks, and use professional judgment to qualify sample data.
* iii. Verify from the raw data that there are no target analytes greater than the quantitation limit present in the sulfur cleanup blank.	iii. If any target analytes are detected in the sulfur cleanup blank greater than or equal to the quantitation limit, then the sulfur cleanup may be a source of contamination. The validator must use professional judgment in conjunction with guidance provided in Section V, Blanks to qualify sample data.
* iv. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred.	iv. If discrepancies between the raw and reported data are found, the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
4. Sulfuric Acid/Permanganate Cleanup - suitable for PCB analysis only	**4. Sulfuric Acid/Permanganate Cleanup**
* a. i. Review the raw data, laboratory bench sheets, or SDG Narrative, to ascertain if sulfuric acid/permanganate cleanup was performed on all method-required sample extracts, QC sample extracts, and method blank extracts	a. i. If a method-required sulfuric acid/permanganate cleanup technique was not used, then the validator should request sample cleanup and reanalysis and document all technical decisions in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
*4. a. ii. Check the field sample GC chromatograms to determine whether or not there are interferences causing elevated baselines or overly complex chromatograms. Confirm that all PCB peaks are adequately resolved and are symmetrical.	4. a. ii. If a method-required sulfuric acid/permanganate cleanup was not performed on sample extracts that contain interferences, or adequate interference removal was not achieved, then the validator should carefully assess the impact on the sample data. The validator should use professional judgment to accept, qualify, or reject the data.
* b. i. Verify from the raw data that the associated QC samples and method blanks was also sulfuric acid/ permanganate cleaned.	b. i. If the associated QC samples and/or method blank was not also sulfuric acid/permanganate cleaned, then the validator should assess the potential impacts on the sample data and use professional judgment to qualify the data.
ii. Verify that the associated QC samples and method blanks met all method-specified QC acceptance criteria after sulfuric acid/permanganate cleanup (refer to the appropriate sections of Part III-Pest/PCB).	ii. If the QC samples and method blanks does not meet QC criteria after sulfuric acid/permanganate cleanup, then the validator should refer to the appropriate sections of Part III-Pest/PCB, and use professional judgment to qualify sample data.
* iii. Compare the raw data to the reported results, if available, and verify that no calculation and/or transcription errors have occurred.	iii. If discrepancies between the raw and reported data are found, the validator should have the laboratory recalculate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.b, C.1.c.i, C.1.c.ii, C.1.c.iv, C.1.c.v, C.1.c.vi, C.1.d.i, C.1.d.ii, C.1.e, C.2.b.iv, C.2.d, C.3.a.ii, C.3.a.iii, C.3.b.iii, C.3.b.iv, C.4.a.i, C.4.a.ii, C.4.b.i, C.4.b.iii.

Table Pest/PCB-VII-1:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON GPC CALIBRATION QUALITY CONTROL

	Criteria	Action
Peak Resolution	As per method QC acceptance criteria.	Professional Judgment
Peak Shape	Peak shapes must be symmetrical.	Professional Judgment
Aroclor Pattern	After GPC is performed, Aroclor 1016 and 1260 standard patterns must be similar to Aroclor patterns in the Initial Calibration sequence.	Professional Judgment
Retention Time	Retention time shifts between GPC calibrations for bis(2-ethylhexyl)phthalate and perylene must not exceed ± 5%.	Professional Judgment
GPC Instrument Blank	Target analytes must be less than the quantitation limit.	Refer to Section V for Blank Actions

Table Pest/PCB-VII-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON GPC CALIBRATION VERIFICATION QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A

Note: Professional judgment should be used in applying the guidance above to qualify or reject sample data.

Table Pest/PCB-VII-3:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON FLORISIL CARTRIDGE CLEANUP QUALITY CONTROL

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < Lower Limit	Lower Limit ≤ %Rec ≤ Upper Limit	%Rec > Upper Limit
Detects	J	J	A	J
Non-detects	R	UJ	A	A
2,4,5-TCP Recovery Criterion	If 2,4,5-Trichlorophenol recovers at ≥ 5%, then the Florisil is not working properly and the data must be evaluated for potential interferences.			

Note: Professional judgment should be used to qualify the data when a combination of low recoveries and high recoveries are obtained.

Table Pest/PCB-VII-4:

QUALIFICATION OF PESTICIDE/PCB ANALYTES BASED ON SULFUR CLEANUP QUALITY CONTROL

Sample Results	Degree of Sulfur Interference		
	Minor	Limited to discrete part of the sample chromatogram	Major
Detects	Estimate (J) positive detects for the affected analytes.	Accept positive detects that are not impacted by sulfur interference. Reject (R) positive detects for those analytes coeluting with the sulfur peak.	Reject (R) all detects for the affected sample and request sample reanalysis that includes sulfur cleanup.
Non-detects	Use professional judgement to evaluate the non-detects.	Accept non-detects that are not impacted by sulfur interference. Reject (R) non-detects for those analytes coeluting with the sulfur peak.	Reject (R) all non-detects for the affected sample and request sample reanalysis that includes sulfur cleanup.

Note: Professional judgment should be used in applying the above guidance to qualify or reject sample data.

E. EXAMPLES

Example #1: (Florisil % Rec > 120% for one analyte)

The validator examines Form IX PEST-1 to verify that the percent recoveries from the Florisil Cartridge Check Solution analysis meet QC acceptance criteria (80-120%). The validator notes that dieldrin was recovered at 150%. The validator uses professional judgment to estimate (J) the positive dieldrin detects associated with this Florisil batch and accept (A) the quantitation limits for dieldrin non-detects on the Data Summary Table. The validator notes in the Data Validation Memorandum that a high bias exists for dieldrin and that positive detects for dieldrin may actually be lower than the reported results.

Example #2: (Florisil % Rec < 80% for six analytes)

The validator examines Form IX PEST-1 to verify that the percent recoveries from the Florisil Cartridge Check Solution analysis meet QC acceptance criteria (80-120%). The validator notes that alpha-BHC, heptachlor, endosulfan I, endrin, 4,4'-DDT, and methoxychlor showed the following recoveries: 75%, 65%, 32%, 70%, 41%, and 9%, respectively. The validator concludes that the Florisil batch used for sample cleanup has resulted in a low bias for pesticide and PCB results. Therefore, the validator uses professional judgment to qualify all sample data associated with this Florisil batch. The validator estimates (J) the positive pesticide/PCB detects and estimates (UJ) all the quantitation limits for non-detects with the exception of the quantitation limits for methoxychlor which are rejected (R). The validator reports the qualified data on the Data Summary Table and discusses the low bias in the Data Validation Memorandum.

Example #3: (GPC % Rec < 80% for one analyte)

The validator examines Form IX PEST-2 to verify that the percent recoveries from the GPC Calibration Verification meet QC acceptance criteria (80-110%). The validator notes that endrin was recovered at 60%. The validator also reviews the GPC calibration data for peak shape, resolution, and retention time shift to verify that the proper collection and dump cycles were utilized to ensure that all interferences were removed without loss of target analytes. The validator concludes that the GPC was calibrated correctly. The validator uses professional judgment to estimate (J) the positive endrin detects and estimate (UJ) the quantitation limits for endrin non-detects for all samples associated with the non-compliant GPC Calibration Verification. The validator reports the qualified data on the Data Summary Table and discusses the reasons for sample qualification and the low bias in the Data Validation Memorandum.

VIII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. OBJECTIVE

Data for matrix spike/matrix spike duplicates (MS/MSDs) are generated at the time of sample preparation and analysis to determine laboratory precision and method bias for specific sample matrices. MS/MSD data can be used to determine long-term interlaboratory precision and bias of an analytical method for various matrices and are used in setting quality control acceptance criteria for spiking analytes. MS/MSD data should be used in conjunction with other QC data, such as field duplicate data and surrogate analyte recoveries, to determine if a sample or an entire sample group should be qualified.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications, or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP. If no deviations or modifications to the method QC acceptance criteria have been defined, then the QC acceptance criteria in the method would be applied in the data validation.

1. In accordance with the SAP, QAPP, and/or method, a field sample of each matrix is spiked in duplicate with known concentrations of specific target analytes to generate an MS/MSD pair. Concurrently, the laboratory analyzes an unspiked aliquot and the MS/MSD pair of the field sample.

2. a. Field samples (not trip, equipment, or bottle blanks and not PE samples) must be spiked to assess matrix effects.

   b. Field samples chosen for MS/MSD analysis should not contain high levels of MS/MSD spiking analytes prior to spiking. Preferably, field samples chosen for MS/MSD analysis should contain low levels of the spiking analytes.

3. Recovery of the spiked analytes must be within the QC acceptance criteria specified in the QAPP/SAP or method.

4. Relative percent differences (RPDs) between MS and MSD recoveries must be within the QC acceptance criteria specified in the QAPP/SAP or method.

5. The percent relative standard deviation (%RSD) between positively detected non-spiked analytes in the unspiked sample, MS, and MSD must be less than or equal to 50%.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from matrix spike/matrix spike duplicate anomalies should be noted in the Data Validation Memorandum. Contractual non-compliance issues concerning the MS/MSD requirements must be discussed in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Verify that the correct analytes were added at the required concentrations; that MS/MSD samples were analyzed at the proper frequency; and that MS/MSD results are provided for each sample matrix.	1. If the laboratory did not use the required analytes at the concentration and frequency specified in the method for each sample matrix, then the validator must use professional judgment and the results from the other QC parameters, such as surrogate analyte recoveries and field duplicate precision, to determine the proper qualifications for the sample results.
2. a. Verify that a field sample was chosen for the MS/MSD.	2. a. If an equipment or bottle blank, or PE sample was spiked with the MS solution for the MS/MSD, then the validator should note this information in the Data Validation Memorandum and discuss the impact on assessing laboratory precision, method bias, sample matrix effects and ultimately data usability.
b. Determine if an inappropriate sample containing high levels of the spiking analytes was chosen for the MS/MSD pair.	b. If the MS/MSD analytes were present in the field sample at high concentrations (e.g., 4x spike concentration) before spiking, then the validator must use professional judgment in assessing matrix spike recoveries and RPDs.
c. Ascertain if the MS/MSD analyses required dilutions.	c. If no MS/MSD data can be reported because of sample dilution, then the validator should note this problem in the Data Validation Memorandum and discuss its impact on assessing data usability in the case where laboratory precision and method bias information are absent.

C. EVALUATION	D. ACTION
3. Verify that all spike recoveries are within the QC acceptance criteria specified in the QAPP/SAP or method.	3. a. If any spiked analyte recovery result is greater than the upper limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Accept the non-detect for that affected analyte in the unspiked sample. b. If any recovery result is greater than or equal to 10%, but less than the lower limit of the method QC acceptance criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Estimate (UJ) the non-detect for that affected analyte in the unspiked sample. c. If any recovery result is less than 10%, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Reject (R) the non-detect for that affected analyte in the unspiked sample. d. If the majority of spike analyte recoveries are outside the method QC acceptance criteria, then the validator may use professional judgment to estimate (J) or reject (R) <u>all</u> positive detects and estimate (UJ) or reject (R) <u>all</u> non-detects in the unspiked sample. Consideration should also be given to qualifying all the results of a particular matrix. See section C.8 for additional guidance.

C. EVALUATION	D. ACTION
4. Verify that the RPDs between the MS and MSD meet the QC acceptance criteria specified in the QAPP/SAP or method.	4. If an RPD result is outside the method QC acceptance criteria, then the validator should: a. Assess whether or not the appropriate RPD acceptance criteria were applied for the situation at hand. b. Estimate (J) the positive detect for that affected analyte in the unspiked sample. c. Estimate (UJ) the non-detect for that affected analyte in the unspiked sample. d. If the majority of the matrix spike RPDs are outside method QC acceptance criteria, then the validator should use professional judgment to estimate (J) all positive detects and estimate (UJ) or reject (R) all non-detects in the unspiked sample. Consideration should also be given to the possibility of qualifying all the results of a particular matrix. Refer to section C. 8 and 9 for additional guidance.
5. a. Calculate the % RSD for the non-spiked target positive detects in the unspiked sample, the MS, and the MSD.	5. a. If a non-detected result or a detect less than the quantitation limit is reported for an analyte in one of the samples in the MS, MSD, or unspiked sample set, then the validator should use the sample quantitation limit value for that analyte to calculate the %RSD. If a non-detected result or a detect less than the quantitation limit is reported for an analyte in two of the samples in the MS, MSD, or unspiked sample set, then the validator should not calculate the %RSD but should use professional judgment to qualify sample data.

C. EVALUATION	D. ACTION
5. b. The unspiked sample, MS, and MSD may be considered a triplicate in determining the overall precision of the analytical method. Therefore, evaluate the %RSD data for positive detects in the triplicate set.	5. b. If any %RSD is greater than the method-specific criteria, then the validator should: i. Estimate (J) the positive detect for that affected analyte in the unspiked sample. ii. Use professional judgment to estimate (UJ) or accept the non-detect for that affected analyte in the unspiked sample. If overall laboratory precision for the unspiked field sample, MS, and MSD is poor, then the validator may use professional judgment to qualify <u>all</u> positive detects and non-detects in the unspiked sample. The Data Validation Memorandum should include a discussion of the potential impact of laboratory precision on representativeness and usability of the data in meeting the project DQOs.
*6. Check and recalculate the analytical concentrations and percent recovery for at least one spiked analyte per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	6. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
*7. Check and recalculate the RPD for at least one spiked analyte per MS/MSD fraction. Verify that the recalculated value agrees within ± 10% of the reported value.	7. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
8. Evaluate the appropriateness of qualifying the entire data set based on MS/MSD laboratory precision and method/matrix bias results.	8. Generally, no action is taken based on the MS/MSD data alone to qualify all samples of a particular matrix. The qualification is limited to the unspiked sample associated with the MS/MSD. However, professional judgment may be used to qualify sample results across a matrix (i.e., all associated groundwater samples or a homogeneous soil matrix).
9. Evaluate MS/MSD precision data to confirm the laboratory's ability to generate precise data in conjunction with surrogate analyte recoveries and field duplicate precision data to assess overall precision.	9. If precision data for the laboratory MS/MSD pair, surrogate analyte recoveries, and the field duplicate pair indicate a heterogenous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify <u>all</u> affected analytes and/or <u>all</u> field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting the project DQOs should be discussed. Refer to Section IX for additional guidance.

* **Note: The following subsections are applicable only to a Tier III data validation:**

C.6, C.7

Table Pest/PCB-VIII-1:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MATRIX SPIKE RECOVERIES AND RPDS **

Sample Results	Recovery < 10%	10% ≤ Recovery < Lower QC Limit	Lower QC Limit ≤ Recovery ≤ Upper QC Limit	Recovery > Upper QC Limit	RPD > QC Limit
Detects	J	J	A	J	J
Non-detects	R	UJ	A	A	UJ

** Note that qualification and rejection generally are limited to the spiking analytes, however, the validator may use professional judgment to qualify or reject all positive detects or non-detects in the unspiked sample, or even all results of a particular matrix, if the majority of spike analyte recoveries and/or RPDs are outside the method QC acceptance criteria.

Table Pest/PCB-VIII-2:

QUALIFICATION OF ORGANIC ANALYTES IN THE UNSPIKED FIELD SAMPLE BASED ON MS, MSD, AND UNSPIKED SAMPLE %RSD

Sample Results	%RSD ≤ 50%*	%RSD > 50%*	Two out of three sample results reported as non-detects
Detects	A	J	Professional Judgment
Non-detects	A	Professional Judgment	Professional Judgment

* If a non-detect is reported for an analyte in only one of the samples in the MS, MSD, or unspiked sample set, then the validator should use the sample quantitation limit value for that analyte to calculate the %RSD.

E. EXAMPLES

Example #1: (High MS/MSD RPD for one analyte)

Soil QC samples SAA99MS and SAA99MSD, analyzed under CLP SOW OLM04.3, have unacceptable RPD results for aldrin. Aldrin was detected in the unspiked sample, SAA99.

Sample No.	Analyte	MS/MSD % Recovery	MS/MSD % Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA99MS SAA99MSD	Aldrin	60/116	34 - 132	64*	43

* outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator then concludes that the lack of laboratory precision in this sample is due to poor laboratory technique. The validator estimates (J) the positive detect for aldrin in the unspiked sample, SAA99, on the Data Summary Table. The validator discusses the lack of laboratory precision for one analyte, aldrin, in the Data Validation Memorandum and notes that laboratory precision for other pesticide matrix spike analytes was acceptable.

Example #2: (Low MS/MSD recoveries for one analyte)

Soil QC samples SAA22MS and SAA22MSD, analyzed under CLP SOW OLM04.3, have one unacceptable recovery result but an acceptable RPD result for heptachlor. Heptachlor was not detected in the unspiked sample, SAA22.

Sample No.	Analyte	MS/MSD Recovery	MS/MSD Recovery Criteria	MS/MSD RPD	MS/MSD RPD Criteria
SAA22MS SAA22MSD	Heptachlor	30*/40	35 - 130	29	31

*outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator concludes that the sample matrix causes a reproducible negative bias for heptachlor in soil samples SAA22MS and SAA22MSD. The validator estimates (UJ) the non-detect for heptachlor in the unspiked sample, SAA22, on the Data Summary Table. The validator discusses the low matrix spike recovery in the Data Validation Memorandum and notes that recoveries for the other pesticide matrix spike analytes were acceptable.

Example #3: (High %RSD; High RPD, poor laboratory precision)

Soil samples SAA55, SAA55MS, and SAA55MSD, analyzed under CLP SOW OLM04.3, had high RSDs for 4,4'-DDD and 4,4'-DDE. The validator assesses the matrix spike results and notes that the pesticides had acceptable recoveries, however, three of the pesticides, gamma-BHC (67%), heptachlor (55%), and aldrin (72%) had high RPDs.

Sample No.	Analyte	MS Conc. Dry Weight (ug/kg)	MSD Conc. Dry Weight (ug/kg)	Unspiked Sample Conc. Dry Weight (ug/kg)	% RSD	% RSD Criteria
SAA55	4,4'-DDD	22	7	40	72*	50
SAA55	4,4'-DDE	5	13	3.3U	73*	50

* outside QC limit

The validator evaluates the field duplicate pair and determines that the RPDs for all positive detects are less than 50%, indicating acceptable overall precision for this sampling event. The validator uses professional judgement to estimate (J, UJ) just the three MS analytes (gamma-BHC, heptachlor, and aldrin) with high % RPD in only the unspiked sample SAA55.

The unspiked sample chromatogram is also examined and no interfering peaks are noted. The validator considers that although the DDT MS recovery was acceptable, the poor laboratory precision was a result of inlet degradation effects that interfere with the efficient reproducible analysis of 4,4'-DDD and 4,4'-DDE. The validator uses professional judgment to estimate (J) the positive 4,4'-DDD detect and (UJ) the 4,4'-DDE non-detect on the Data Summary Table. The validator notes the sample qualifications in the Data Validation Memorandum.

E. EXAMPLES

Example #4: (Low MS/MSD recoveries for multiple analytes)

Soil QC samples SAA09MS and SAA09MSD, analyzed under CLP SOW OLM04.3, have low spike analyte recoveries for four of the six analytes in the matrix spike and matrix spike duplicate (less than the specified QC acceptance criteria but greater than 10%). The validator notes that the recoveries for both pesticide surrogates are acceptable but slightly low in the MS and MSD unspiked samples.

Sample No.	Analyte	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Recovery	RPD
SAA09 MS/MSD	Heptachlor	21*	28*	29	35-130	31
	Aldrin	29*	31*	6.7	34-132	43
	Dieldrin	27*	21*	25	31-134	38
	Endrin	30*	34*	13	42-139	45
	DCB (surrogate)	40	45	NA	30-150	NA
	TCX (surrogate)	38	35	NA	30-150	NA

* outside QC limit

Upon review of the MS/MSD results and surrogate recoveries, the validator notes that the sample matrix causes a reproducible negative bias for pesticide analytes in the MS/MSD samples. The validator reviews the unspiked sample surrogate analyte recoveries and notes that they are also low but within QC acceptance criteria (at the low end of the QC acceptance range). The validator then reviews the surrogate analyte recoveries for all samples with this matrix associated with the sample delivery group to ascertain if surrogate recoveries are also low in the remaining samples.

Several samples, including the field duplicates, show low surrogate recoveries that were greater than 10%. The validator estimates (J) all positive detects in the unspiked sample and estimates (UJ) all non-detects in the unspiked sample. The validator uses professional judgment to estimate (J) the positive detects and estimate (UJ) the non-detects in all other samples associated with this sample delivery group in which surrogates recovered low. The validator reports qualified data in the Data Summary Table and discusses the low bias in the Data Validation Memorandum.

E. EXAMPLES

Example #5: (High MS/MSD RPDs for multiple analytes)

Aqueous QC samples SAA01MS and SAA01MSD, analyzed under CLP SOW OLM04.3, have high RPD values for five out of the six analytes in the matrix spike/matrix spike duplicate pair. The matrix spike and matrix spike duplicate analyte recoveries were all within QC acceptance criteria. The surrogate recoveries were acceptable for both the MS and the MSD. The validator notes the lack of precision in the unspiked analytes.

Sample No.	Analyte	MS % Recovery	MSD % Recovery	RPD	QC Acceptance Criteria	
					% Recovery	RPD
SAA01	gamma-BHC (Lindane)	58	86	39*	56-123	15
	Heptachlor	45	85	62*	40-131	20
	Aldrin	70	118	51*	40-120	22
	Endrin	75	120	46*	56-121	21
	4,4'-DDT	50	100	66*	38-127	27

*outside QC limits

Sample No.	Analyte	MS Conc. (ug/L)	MSD Conc. (ug/L)	Unspiked Sample Conc. (ug/L)	% RSD	% RSD Criteria
SAA01	4,4'-DDE	10	65	90	74*	50
	delta-BHC	45	25	10U	93*	50

*outside QC limits

Upon review of the MS/MSD results, surrogate recoveries, and the % RSDs, the validator notes the laboratory imprecision and suspects that problems occurred during extraction and/or analysis of the MS/MSD sample and/or unspiked sample. The validator then reviews the field duplicate data and surrogate recoveries for the remaining samples and QC samples in the sample delivery group to assess precision and bias data.

Surrogate recoveries in all other samples were acceptable. The field duplicate RPD data were also acceptable. Therefore, the validator determines that poor precision was limited to the MS/MSD pair. The validator used professional judgment to estimate (J) all positive detects and estimate (UJ) all non-detects in the unspiked sample SAA01 on the Data Summary Table. The validator notes this problem in the Data Validation Memorandum.

IX. FIELD DUPLICATES

A. OBJECTIVE

Field duplicates measure the cumulative effects of both field and laboratory precision and hence provide an indication of overall precision. Therefore, field duplicates may have greater variability than laboratory duplicates which measure only laboratory precision. It is also expected that non-aqueous matrices will have a greater variance than aqueous matrices due to the heterogeneity of most non-aqueous samples (such as soil/sediment samples).

B. CRITERIA

1. The frequency of field duplicate analysis must support the site-specific Data Quality Objectives (DQOs) and be documented in the EPA approved QAPP or SAP.

2. a. The Relative Percent Difference (RPD) for all analytes detected at concentrations greater than the sample quantitation limit in aqueous matrices must be less than or equal to 30 percent.

   b. The RPD for all analytes detected at concentrations greater than the sample quantitation limit in non-aqueous matrices must be less than or equal to 50 percent.

3. In situations where the RPD criteria are not specified in the QAPP, it is recommended to use the criteria found in 2.a. and 2.b. above.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
	All potential impacts on the sample data resulting from field duplicate anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. a. Identify which samples are field duplicates from the Chain-of-Custody form and/or the Traffic Report.	1. a. If field duplicates are not listed on the Chain-of-Custody form or the Traffic Report, then the validator should contact the sampler to determine if field duplicates were collected. If the forms were completed incorrectly or if field duplicates were not collected, then the validator should document this on the Data Validation Worksheet and in the Data Validation Memorandum.
b. Verify that the appropriate number of field duplicates per matrix sampled were collected and analyzed to support the project DQOs.	b. If field duplicates were not collected at the required frequency to support the project DQOs, then the validator should note the absence of field precision data in the Data Validation Memorandum and discuss how the lack of field precision data might potentially increase uncertainty surrounding site decisions.

C. EVALUATION	D. ACTION
2. Calculate the RPD for all analytes detected at concentrations greater than or equal to the sample quantitation limit in the field duplicate sets. Record the RPDs on the appropriate worksheet.	2. a. If any analyte is detected at a concentration greater than or equal to twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should estimate (J) the positive detects for that analyte in both samples. If any analyte is detected at a concentration greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both aqueous field duplicate samples and has an RPD greater than 30%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that analyte in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs. b. If any analyte is detected at a concentration greater than or equal to twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should estimate (J) the positive detects for that analyte in both samples. If any analyte is detected at a concentration greater than or equal to the sample quantitation limit but less than twice the sample quantitation limit in both non-aqueous field duplicate samples and has an RPD greater than 50%, then the validator should use professional judgment to accept, qualify, or reject the positive detects for that analyte in the field duplicate samples taking into consideration the increased variability of data near the sample quantitation limit and the site-specific DQOs.

C. EVALUATION	D. ACTION
2. Continued from above.	2. c. If any analyte in a field duplicate pair has one positive detect that is greater than or equal to twice the sample quantitation limit and a duplicate positive detect that is less than twice the sample quantitation limit, and the RPD exceeds field duplicate precision criteria for that matrix, then the validator should use professional judgment to qualify the positive detects for that analyte in the field duplicate samples. d. If any analyte in a field duplicate pair has one non-detect and a duplicate positive detect that is greater than or equal to twice the sample quantitation limit, then the validator should estimate (J) the positive detect and (UJ) the non-detect for that analyte in the field duplicate samples. (RPDs should not be evaluated for those duplicate pairs.) e. If any analyte in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is at or above the sample quantitation limit but less than twice the sample quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that analyte in the field duplicate samples taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs. (RPDs should not be evaluated for those duplicate pairs.) f. If any analyte in a field duplicate pair has one non-detect or a reported value below the sample quantitation limit and a duplicate positive detect that is less than the quantitation limit, then the validator should use professional judgment to qualify the positive detects and non-detects for that analyte in the field duplicate samples taking into consideration the increased variability of data at the sample quantitation limit and the project DQOs. (RPDs should not be evaluated for those duplicate pairs.)

C. EVALUATION	D. ACTION
*3. Check and recalculate the analytical concentrations for at least one positive detect and one sample quantitation limit (for a diluted sample or soil sample) for each fraction, in every field duplicate sample, in accordance with Section Pest/PCB-XIII, C.	3. If calculation and/or transcription errors are detected, then the validator should follow the procedures outlined in Section Pest/PCB XIII, D.
4. Evaluate the appropriateness of qualifying the entire data set based on field duplicate results.	4. If field duplicate data indicate poor field precision and general sample heterogeneity and/or possible sampling error, then professional judgment may be used to qualify data for <u>all</u> samples of the same matrix.
5. Evaluate field duplicate precision data to assess overall precision and to verify the field sampler's ability to collect representative duplicate samples. MS/MSD precision data should be evaluated to verify the laboratory's ability to generate precise data. Surrogate recovery data can also be evaluated to identify laboratory precision issues and overall matrix precision issues.	5. If precision data for the field duplicate pair, surrogate analyte recoveries, and the laboratory MS/MSD pair indicate a heterogeneous matrix at the site or potential sampling error, then the validator may use professional judgment to qualify <u>all</u> affected analytes and/or <u>all</u> affected field sample results. This problem should be noted in the Data Validation Memorandum and the potential impact on the representativeness and usability of the data in meeting project DQOs should be discussed. Refer to Section VIII for additional guidance.

*** Note: The following subsections are applicable only to a Tier III validation:**

C.3

Table Pest/PCB-IX-1:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 1: POSITIVE DETECTS IN BOTH FIELD DUPLICATES

Relative Percent Difference	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%	Aqueous > 30% Non-Aqueous > 50%
Sample Results	**Both duplicate sample concs. ≥ 2 X QL**	**QL ≤ both duplicate samples concs. < 2 X QL**	**One sample conc. ≥ 2 X QL QL ≤ One sample conc. < 2 X QL**
Detects	J	Professional Judgment	Professional Judgment

* QL = Sample Quantitation Limit
N/A = Not Applicable

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

Table Pest/PCB-IX-2:

QUALIFICATION OF ORGANIC ANALYTES IN FIELD DUPLICATES
SITUATION 2: POSITIVE DETECT IN ONLY ONE FIELD DUPLICATE**

Aqueous and Non-Aqueous		
Sample Results	**One Sample Conc. = ND (or values reported as less than the QL) QL ≤ One Sample Conc. < 2 X QL**	**One sample conc. = ND (or values reported as less than the QL) One sample conc. ≥ 2 X QL**
Detects	Professional Judgment	J
Non-detects	Professional Judgment	UJ

* QL = Sample Quantitation Limit

** RPD should not be evaluated for these duplicate pairs

Note: Qualification refers to field duplicate sample results only. Professional judgment may be used to apply field duplicate actions to all samples of the same matrix.

E. EXAMPLES

Example #1: (Both field duplicate sample concentrations ≥ 2X QL; %RPD > 50%; Acceptable laboratory precision)

Soil samples SAA11 and SAA12 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 89% and 85% solids, respectively. Sample SAA11 has a detected concentration of Aroclor-1254 of 100 ug/kg. Sample SAA12 has a detected concentration of Aroclor-1254 of 250 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 86%. The validator notes that both results are greater than twice the sample Quantitation Limit (QL). The QL for Aroclor-1254 in sample SAA11 is 37 ug/kg and the QL for Aroclor-1254 in SAA12 is 39 ug/kg. The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. As a result, the validator estimates (J) the positive Aroclor-1254 detects in the field duplicate samples only, on the Data Summary Table, and notes the qualification and justification in the Data Validation Memorandum. The validator also notes that poor field precision may be due to a heterogenous matrix or a result of sampling error.

Analyte	SAA11		SAA12		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
Aroclor 1254	100	37	250	39	86

Example #2: (QL ≤ both field duplicate sample concentrations < 2X QL; RPD > 50%; Acceptable laboratory precision)

Soil samples SAA21 and SAA22 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 51% and 50% solids, respectively. Sample SAA21 has a detected concentration of alpha-Chlordane of 3.8 ug/kg. Sample SAA22 has a detected concentration of alpha-Chlordane of 6.5 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 52%. The sample QL for alpha-Chlordane in sample SAA21 is 3.3 ug/kg based on 51% solids sample and the sample QL for alpha-Chlordane in sample SAA22 is 3.4 ug/kg based on 50% solids. The validator reviews the MS/MSD results and determines that laboratory precision is acceptable. The validator notes that there were no blank actions applicable for alpha-Chlordane to the samples arising from blank contamination. The validator notes that both field duplicate results are between the sample QL and twice the sample QL. As a result the validator uses professional judgment to accept the alpha-Chlordane results in the field duplicate samples taking into consideration the increased variability of data near the quantitation limit. The validator notes in the Data Validation Memorandum that field duplicate precision was acceptable.

Analyte	SAA21		SAA22		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
alpha-chlordane	3.8	3.3	6.5	3.4	52

E. EXAMPLES

Example #3: (One sample concentration = ND; One sample concentration ≥ 2X QL; Acceptable laboratory precision)

Aqueous samples SAA31 and SAA32 are field duplicates, analyzed under CLP SOW OLM04.3. Sample SAA31 has a detected concentration of endrin ketone of 8.0 ug/L. Endrin ketone was not detected in sample SAA32. The validator notes that the positive endrin ketone detect in sample SAA31 is greater than twice the sample QL of 0.1 ug/L. The validator reviews the MS/MSD data and determines that laboratory precision was acceptable. The validator reviews the preceding blank and sample runs for potential contribution of endrin ketone to the sample, and determines that there was no apparent endrin ketone contamination. The validator estimates (J) the positive endrin ketone detect in sample SAA31 and estimates (UJ) the quantitation limit of the endrin ketone non-detect in sample SAA32 on the Data Summary Table based on poor field precision. The validator notes the qualification in the Data Validation Memorandum and also suggests that poor field precision may be due to sampling error.

Analyte	SAA31		SAA32		RPD
	Sample Conc. (ug/L)	Sample QL (ug/L)	Sample Conc. (ug/L)	Sample QL (ug/L)	
endrin ketone	8.0	0.1	ND	0.1	NA

Example #4: (One sample concentration = ND; One sample concentration < 2X QL; Acceptable laboratory precision)

Soil samples SAA41 and SAA42 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 90% and 85% solids, respectively. Sample SAA41 has a detected concentration of Aroclor-1260 of 65 ug/kg. Aroclor-1260 was not detected in sample SAA42. The validator notes that the positive Aroclor-1260 detect is between the sample QL and twice the sample QL. The sample QL for Aroclor-1260 in sample SAA41 is 37 ug/kg and the sample QL for Aroclor-1260 in sample is SAA42 is 39 ug/kg. The validator reviews the MS/MSD results and determines that RPD criteria were met for the pesticides indicating acceptable laboratory precision. The validator verifies the identification of the multi-component analyte in the samples and determines that the results were correct as reported. The validator reviews the preceding blank and sample runs for potential contribution of Aroclor-1260 to the sample, and determines that there was no apparent Aroclor-1260 contamination. As a result, the validator uses professional judgment to accept the positive Aroclor-1260 detect in SAA41 and to accept the Aroclor-1260 non-detect in sample SAA42, taking into consideration the increased variability of data near the quantitation limit. The validator reports the results on the Data Summary Table and notes this in the Data Validation Memorandum.

Analyte	SAA41		SAA42		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
Aroclor 1260	65	37	ND	39	NA

E. EXAMPLES

Example #5: (Both duplicate sample concentrations ≥ 2X QL; Poor field and laboratory precision)

Soil samples SAA34 and SAA35 are field duplicates, analyzed under CLP SOW OLM04.3, and they contain 90% and 95% solids, respectively. Sample SAA34 has a detected concentration of aldrin of 10 ug/kg. Sample SAA35 has a detected concentration of aldrin of 40 ug/kg. The validator calculates the Relative Percent Difference (RPD) and determines that the RPD equals 120%. The validator notes that both results are greater than twice the sample QL. The sample QL for aldrin in sample SAA34 is 1.9 ug/kg and the sample QL for aldrin in sample SAA35 is 1.8 ug/kg. The validator reviews the MS/MSD data for samples SAA34MS/MSD and determines that the RPD for aldrin equals 60% which is outside the criteria. The validator is unable to determine the source of the imprecision since both the lab and field precision were poor; therefore, the validator uses professional judgment to estimate (J) the aldrin positive detects in all samples associated with the sample delivery group and estimate (UJ) the quantitation limits for aldrin non-detects in all samples associated with the sample delivery group. The validator reports the qualified data on the Data Summary Table and justifies the qualification in the Data Validation Memorandum. The validator notes that the source of the imprecision cannot be determined.

Analyte	SAA34		SAA35		RPD
	Sample Conc. (ug/kg)	Sample QL (ug/kg)	Sample Conc. (ug/kg)	Sample QL (ug/kg)	
aldrin	10.0	1.9	40	1.8	120

X. SENSITIVITY CHECK

A. OBJECTIVE

Although most CLP SOWs do not incorporate the analysis of sensitivity checks, many EPA methods do require that a Method Detection Limit (MDL) study be performed prior to sample analysis and/or that a Laboratory Fortified Blank (LFB) be analyzed at the time of sample analysis. The MDL study generates statistically-based detection limits and can be used to assess method sensitivity, laboratory precision, and method bias for specific analytes within an analytical method on a specific instrument and column. An LFB, a type of Laboratory Control Sample, is a reagent blank spiked with several or all of the target analytes at or below their quantitation limits. LFB data can be used to assess laboratory sensitivity and bias for specific analytes at the quantitation limit within an analytical method on a specific instrument and column at the time of sample preparation and analysis. To determine sample qualification, the MDL study is evaluated prior to the LFB data.

Region I routinely uses MDL studies as a pre-qualification check to verify the laboratory's ability to meet the technical specification/method requirements prior to contract award and field sample receipt. Region I also routinely includes LFB analyses to document the method sensitivity and bias associated with the day-to-day preparation and analysis of field samples.

B. CRITERIA

The Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses should be used to validate all Region I Organic data. The CLP-Pesticide/PCB method QC acceptance criteria listed in Appendix F should be used as the default criteria when none exist for the Pesticide/PCB analytical method utilized and when similar QC parameters are required by the non-CLP method and acceptance criteria have not been specified. Deviations, modifications or non-CLP method-specific QC acceptance criteria may be used but must be explicitly defined in tabular format in the site-specific EPA approved QAPP/SAP or amendment to the QAPP/SAP.

1. Method Detection Limit (MDL) Study

a. The method detection limit (MDL) for each target analyte must be established in accordance with the specified method and the Code of Federal Regulations (40 CFR Part 136, App.B). A minimum of seven replicates must be analyzed for each matrix of interest.

b. Surrogates must be spiked into each MDL sample as specified in the method. Recoveries and %RSDs for surrogates and target analytes must meet the criteria specified in the method. If the method does not specify recovery and/or replicate %RSD criteria, then the %RSD for the seven replicates should be less than or equal to 25% and the mean recovery for target analytes and surrogates should be between 80-120%.

c. Samples must be analyzed on the same instrument under the same conditions as was used for the MDL study.

d. The MDL study must be performed within one year prior to the start of the preparation and/or analysis of the samples.

e. The MDL for each target analyte must be less than one half the target analyte method-required quantitation limit.

2. Laboratory Fortified Blank (LFB)

a. Verification of laboratory accuracy at the quantitation level requires the routine analysis of an LFB spiked with target analytes at the quantitation limit and surrogate analytes spiked at the concentrations specified in the method. The stock solution used for spiking the LFB must be prepared from a source other than the source used for preparing the initial and continuing calibration standards.

b. One LFB containing all the target analytes at the quantitation limit must be analyzed immediately prior to sample analysis but after instrument calibration. Subsequently, an LFB must be analyzed every 12 hours. One LFB must be extracted with each sample delivery group of pesticide/PCB samples, or whenever pesticide/PCB samples are extracted, whichever is more frequent.

c. Method QC acceptance criteria must be met for surrogates and target analytes. If the method does not specify recovery QC acceptance criteria for the LFB, then the recovery for target analytes should be between 60-140%. Surrogate analytes for the LFB must meet validation criteria as per Section VI of this document.

C. EVALUATION/ D. ACTION

C. EVALUATION	D. ACTION
Qualification of data should be based on a combined evaluation of both the MDL study and LFB results. To determine appropriate sample qualification, the MDL Study should be evaluated first and then the LFB results.	All potential impacts on the sample data resulting from MDL and/or LFB study anomalies should be noted in the Data Validation Memorandum. The validator should also document and justify all technical decisions made based on professional judgment in the Data Validation Memorandum.
1. Method Detection Limit (MDL) Study	**1. Method Detection Limit (MDL) Study**
a. Verify that the MDL study was generated in accordance with the method and 40 CFR Part 136 App. B, and that a minimum of seven replicates for each matrix of interest were prepared and analyzed.	a. If the required MDL study was not performed at all or was not performed according to the 40 CFR Part 136 App. B criteria, then the validator should evaluate the LFB data, if available, to determine the action to be taken. See Tables Pest/PCB-X-1, Pest/PCB-X-2, and Pest/PCB-X-3 for the appropriate action. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.

C. EVALUATION	D. ACTION
1. b. Verify that the %RSD for each target analyte and surrogate is less than or equal to 25% for all seven replicates of the MDL study.	1. b. If the MDL target analyte and surrogate%RSD criteria are exceeded, then the validator should evaluate initial calibration %RSDs to assess instrument precision and linearity. The validator should use professional judgment to assess the impact of laboratory precision on analytical sensitivity and data quality.
c. Compare all seven replicates of the MDL study to verify that the mean recovery for each target analyte and surrogate is within the 80-120% criteria for each replicate in the MDL Study.	c. If the mean percent recovery for a target analyte and/or surrogate is greater than 120%, then the validator should: - Use professional judgment to estimate (J) positive detects for that analyte in all samples associated with that MDL study, taking into consideration the LFB results. - Accept the non-detects. If the mean percent recovery for a target analyte and/or surrogate is less than 80% but greater than or equal to 10%, then the validator should: - Use professional judgment to estimate (J) positive detects for that analyte in all samples associated with that MDL study, taking into consideration the LFB results. - Use professional judgment to estimate (UJ) the non-detects for that analyte in all samples associated with that MDL study taking into consideration the LFB results. If the mean percent recovery for a target analyte and/or surrogate is less than 10%, then the validator should estimate (J) positive detects for that analyte and reject (R) the non-detects for that analyte in all samples associated with that MDL study.

C. EVALUATION	D. ACTION
*1. d. Check and recalculate the %RSDs and % recoveries for at least three analytes per MDL study. Verify that the recalculated values agree within ± 10% of the reported results.	1. d. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
e. Verify that the samples were analyzed on the same instruments and under the same conditions as was used for the MDL study.	e. If the samples were not analyzed on the same instruments or under the same conditions as the MDL study, then the validator should contact the laboratory to obtain a correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
f. Verify that the matrix for the MDL Study is the same as that of the samples.	f. If the MDL Study is not the same matrix as the samples, then the validator should contact the laboratory to obtain a correct MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.

C. EVALUATION	D. ACTION
1. g. Compare the date of the MDL study to the dates of all associated sample analyses to verify that the MDL study was performed within one year prior to the start of the first sample prepared and/or analyzed in the sample delivery group.	1. g. If the MDL study was not submitted or was not performed within one year of the start of preparation and/or analysis of the first sample in the SDG, then the validator should contact the laboratory to obtain a current MDL study. If an acceptable MDL study is unavailable, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should evaluate the lowest standard of the initial calibration and the daily continuing calibration standard data and use professional judgment to assess the impact of analytical sensitivity on data quality.
h. Verify that all MDLs are not more than one half of the method-required quantitation limits.	h. If the MDL study reveals that a target analyte has a detection limit greater than one half the method-required quantitation limit, then the validator should evaluate the LFB data. If no LFB data are available, then the validator should: i. Elevate the quantitation limit for that target analyte in all samples associated with that MDL study to the lowest concentration calibration standard analyzed or to the laboratory reported MDL, whichever is higher. ii. Estimate (J) positive detects which were below the elevated quantitation limit for that target analyte and/or in all samples associated with that MDL study. iii. The validator should evaluate the elevated quantitation limits in relation to the required quantitation limits in the site DQO's. The validator should discuss the impact of the elevated quantitation limits on the site objectives and whether or not the data are usable for the site objectives in the Data Validation Memorandum.

C. EVALUATION	D. ACTION
1. i. Check and recalculate the MDL value for at least one analyte per MDL Study. Verify that the recalculated values agree within ± 10% of the reported results.	1. i. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.
If the LFB criteria are not met, then laboratory performance related to method bias and method/instrument sensitivity is questionable. **2. Laboratory Fortified Blank (LFB)**	**2. Laboratory Fortified Blank (LFB)**
* a. Check the standards preparation logs to verify that the stock standard used to prepare the LFB was from a source independent from the initial and continuing calibration standards.	a. If the LFB was not prepared from a source independent from the initial and continuing calibration standards, then the laboratory performance related to method bias and method/instrument sensitivity is questionable. The validator should review other calibration verification checks, i.e., Performance Evaluation Sample (PES) analyses to ensure the calibration accuracy. Professional judgment should be used to qualify sample quantitation limits.
b. Verify that an LFB was prepared and/or analyzed at the proper frequency and that it was spiked with the correct analytes at their quantitation limits.	b. If an LFB analysis was not performed or the LFB was not analyzed for the correct analytes at the proper frequency and concentration, then the validator should use professional judgment to assess the impact of analytical sensitivity on data quality.
c. Verify that the reported recoveries for all LFB spike analytes are within the method QC acceptance criteria.	c. Sample data should be qualified based on the number and type of analytes that are recovered outside the method QC acceptance criteria and based on the degree that analyte recoveries exceed the criteria.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. i. If any of the LFB analyte recoveries are outside the method QC acceptance criteria, then the LFB results should be used to qualify sample data for the specific analytes that are included in the LFB solution. The validator should use professional judgment to qualify sample data for analytes not included in the LFB,taking into account the analyte's chemical class, analyte recovery efficiency, and any analytical problems historically associated with the analyte or that were encountered by the laboratory. ii. If an LFB analyte recovery is greater than 140%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential high bias. - Accept the quantitation limit of the affected analyte in any sample associated with that LFB. iii. If more than half of the LFB analyte recoveries are greater than 140%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential high bias. - Accept <u>all</u> quantitation limits for non-detects in all samples associated with that LFB.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. iv. If an LFB analyte recovery is less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential low bias. - Estimate (UJ) the quantitation limit of the affected analyte in any sample associated with that LFB to indicate potential low bias. v. If more than half of the LFB analyte recoveries are less than 60% but greater than or equal to 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential low bias. - Estimate (UJ) <u>all</u> quantitation limits for non-detects in all samples associated with that LFB to indicate potential low bias. vi. If an LFB analyte recovery is less than 10%, then the validator should: - Estimate (J) the affected analyte when detected in any sample associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limit of the affected analyte in any samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives.

C. EVALUATION	D. ACTION
2. c. Continued from above.	2. c. vii. If more than half of the LFB analyte recoveries less than 10%, then the validator should: - Estimate (J) <u>all</u> positive detects in all samples associated with that LFB to indicate potential low bias. - Reject (R) the quantitation limits for <u>all</u> non-detects in all samples associated with that LFB to indicate that the data are unusable due to the possibility of false negatives. viii. If more than half of the LFB analyte recoveries are outside the method QC acceptance limits in one LFB with some low recoveries and some high recoveries, then the validator should use professional judgment to qualify or reject a particular analyte, class of analytes or the entire fraction for samples associated with that LFB. ix. Action on non-compliant surrogate recoveries should follow the guidance provided in Section VI. Professional judgment should be used to evaluate the impact that a non-compliant LFB surrogate recovery has on the sample data.

C. EVALUATION	D. ACTION
*2. d. Check and recalculate the % recovery for at least one target analyte per LFB. Verify that the recalculated value agrees within ± 10% of the reported result.	2 d. If any transcription and/or calculation errors are detected, perform a more comprehensive review to determine the magnitude of the problem. If the problem is extensive, then the validator should have the laboratory requantitate and resubmit all corrected raw data and forms. If a discrepancy remains unresolved, the validator must use professional judgment to decide which value is more accurate. Under these circumstances, the validator may determine that the sample data should be qualified or rejected. A discussion of the rationale for data qualification and the qualifiers used should be documented in the Data Validation Memorandum.

*** Note: The following subsections are applicable only to a Tier III data validation:**

C.1.d, C.2.a, C.2.d

Table Pest/PCB-X-1:

QUALIFICATION OF ORGANIC ANALYTES BASED ON MDL STUDY RESULTS

Sample Results	Mean % Recovery			
	%Rec < 10%	10% ≤ %Rec < 80%	80% ≤ %Rec ≤ 120%	%Rec > 120%
Detects	J	Professional Judgment*	A	Professional Judgment*
Non-Detects	R	Professional Judgment*	A	A

Sample Results	% RSD	
	> 25%	≤ 25%
Detects	Professional Judgment**	A
Non-detects	Professional Judgment**	A

* Taking into consideration LFB results.
** Taking into consideration initial calibration %RSDs.

Table Pest/PCB-X-2:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: ≤ ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
Detects	J	J	A	J
Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

Table Pest/PCB-X-3:

QUALIFICATION OF ORGANIC ANALYTES BASED ON LFB* RECOVERIES WHERE: > ONE-HALF OF LFB ANALYTES OUTSIDE UPPER OR LOWER ACCEPTANCE LIMITS**

Sample Results	% Recovery			
	%Rec < 10%	10% ≤ %Rec < 60%	60% ≤ %Rec ≤ 140%	%Rec > 140%
All Detects	J	J	A	J
All Non-detects	R	UJ	A	A

* LFB = Laboratory fortified blank spiked with several or all of the method target analytes and/or Aroclors at or below the quantitation limit.

** Professional judgment should be used when a combination of low recoveries and high recoveries are obtained.

E. EXAMPLES

Example #1: (Low LFB recoveries for several analytes)

Low concentration water samples were analyzed under CLP SOW OLC03.2, however, no MDL study data was available. LFB analytes dieldrin, endrin, and endosulfan sulfate recovered below QC acceptance criteria but greater than 10%, i.e., 25%, 30%, and 18%, respectively. The validator estimates (J) the positive detects for dieldrin, endrin, and endosulfan sulfate in all the field samples associated with the LFB to indicate potential low bias and estimates (UJ) the quantitation limits for the dieldrin, endrin, and endosulfan sulfate non-detects in all the field samples associated with the LFB to indicate a decrease in sensitivity and the possibility of false negatives. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #2: (High LFB recoveries for more than 50% of pesticide analytes)

Low concentration water samples were analyzed under CLP SOW OLC03.2, however no MDL study data was available. LFB analytes gamma-chlordane, endrin, dieldrin, and heptachlor epoxide recovered above the QC acceptance criteria, i.e., 150%, 160%, 135%, and 173%, respectively. The validator notes that more than 50% of the LFB analytes exceeded the QC criteria. Therefore, the validator estimates (J) all positive detects in all samples associated with the LFB and all non-detects were accepted. The validator reports the qualified results on the Data Summary Table and notes this in the Data Validation Memorandum.

Example #3: (Low MDL recoveries for non-LFB analytes; Acceptable LFB results)

The analytical method used did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) to evaluate the MDL data. The MDL study submitted by the laboratory did not meet the default criteria for mean % recovery for 4,4'-DDD and methoxychlor (45% and 9%, respectively). The validator examines the LFB results submitted with the sample results and determines that QC acceptance criteria were met for all LFB analytes. However, the validator notes that 4,4'-DDD and methoxychlor were not LFB analytes. The validator uses professional judgment to estimate (J) the positive 4,4'-DDD detects, estimate (UJ) the 4,4'-DDD non-detects, estimate (J) the positive methoxychlor detects, and reject the methoxychlor non-detects. The validator reports the qualified results on the Data Summary Table and notes the sample qualifications in the Data Validation Memorandum.

Example #4: (High LFB recovery for two analytes; High MDL %RSDs for two analytes)

The analytical method used for sample analysis did not specify QC acceptance criteria for the MDL study. The validator uses the default criteria for mean % recoveries (80-120%) and % RSDs to evaluate the MDL data. The MDL study submitted by the laboratory did not meet default (25%) %RSD criteria for endrin and dieldrin (32% and 29%, respectively). The validator reviews the initial calibration %RSDs and determines that endrin and dieldrin met the initial calibration %RSD acceptance criteria. In addition, the analytical method used did not specify QC acceptance criteria for the LFB. The validator uses the default recovery criteria of 60-140% to evaluate LFB results. The validator examines the LFB submitted with the analytical results and determines that dieldrin and endrin also exceeded the LFB % recovery criteria of 140% (159% and 160%, respectively). Since the initial calibration %RSDs were acceptable, the high MDL %RSDs were not utilized to qualify sample data. Based upon the LFB recoveries, the validator uses professional judgment to estimate (J) the positive dieldrin and endrin detects to indicate potential high bias for these two analytes and accept the quantitation limits for dieldrin and endrin non-detects in all field samples

associated with the LFB. The validator reports the qualified results on the Data Summary Table and notes the sample qualifications in the Data Validation Memorandum.