

August 20002

SAIC
DATA VALIDATION REPORT (FORM REVIEW*)
CLP PESTICIDE/PCB ORGANICS (Multi-Media, Multi-Concentration)

Project: Palos Verdes

SOW: SW846 8081A

Lot/SDG No:

Laboratory: Woods Hole Env. Labs

ETR No: 0203040

No. of Samples/Matrix: 3 Sediment

The above data package has been reviewed and the quality assurance and performance data have been summarized. The general criteria used to determine the quality of the data were based on an examination of:

- Holding Times
- GC/ECD Instrument Performance Check
- Calibration (Initial, Continuing)
- Blanks (Lab, Field, Rinsate)
- Surrogate Spike
- Matrix Spike/Matrix Spike Duplicate
- Laboratory Control Sample
- Field Duplicates
- Pesticide Cleanup Checks
- Target Compound Identification
- Compound Quantitation and Reported CRQLs

Overall Comments:

4,4-DDE in LUU14-8 was considered an approximated quantity "NJ."

Summary of Exceeded Criteria:

- A. 0 analyses outside of holding times.
- B. 0 extractions outside of holding times.
- C. 0 of 24 surrogate spikes recoveries were outside of the control limits. (for both columns)
- D. Not performed MS/MSD recoveries were outside of the control limits.
- E. Not performed MS/MSD RPDs were outside of the control limits.
- F. 0 compounds were qualified as rejected.
- G. 0 compounds were qualified due to field QC results.
- H. 0 compounds were qualified due to method blank results.

*- Per the Project QAPP this lot does not required recalculation from the raw data. CLP Forms were reviewed to ensure that the QC results fell within the appropriate control limits and any resulting data validation qualifiers were applied.


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Summary of Data Validation Qualifiers Applied:

SAIC Sample ID	Laboratory ID No.	Compound	Data Validation Qualifier	Reason for Qualification
LUUT14-8	0203040-13	4,4'-DDE	NJ	

Definition of Qualifiers:

- U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
- NJ- The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ- The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality criteria. The presence or absence of the analyte cannot be verified.

Validated by: _____ 

Date: 8/6/02

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All criteria were met

I. Holding Times

- Water sample should be extracted within 7 days from sample collection and analyzed within 40 days.
- Soil sample should be extracted within 14 days if not frozen and up to 1 year if frozen from sample collection and analyzed within 40 days.

SAIC Sample No.	Laboratory ID No.	Field Sample No.	Depth ft.	Date Collected	Date Extracted	Extraction Holding Time	Date Analyzed	Analysis Holding Time	Qualifier Applied
LUUT14-6	0203040-11	NA	0	3/11/2002	6/13/2002	94	6/14/2002	1	None
LUUT14-7	0203040-12	NA	0	3/11/2002	6/13/2002	94	6/14/2002	1	None
LUUT14-8	0203040-13	NA	0	3/11/2002	6/13/2002	94	6/18/2002	5	None

Actions:

1. If the technical holding times are exceeded, qualify positive results with "J" and non-detected semivolatile target compounds with "UJ" and document that holding times were exceeded.
2. If technical holding times are grossly exceeded, the reviewer may determine that the positive results or the associated quantitation limits are approximate and should be qualified with "J" or "UJ", respectively. The reviewer may determine that non-detected data are unusable.

Comments:

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Not Applicable

II. GC/ECD Instrument Performance Check

1 Resolution Check Mixture (REM)

- REM was analyzed at the beginning of each initial calibration sequence on each column and instrument. Yes or No

- The depth of the valley between two adjacent peaks in the REM is $\leq 60\%$ of the height of the shortest peak. Yes or No

Deviations:

Compound	Deviation	SAIC Sample No.	Laboratory ID No.	Qualifiers Applied

Action:

If resolution criteria are not met the quantitative results may not be accurate due to inadequate resolution. Detected target compounds which were not adequately resolved should be qualified with "J". Qualitative identifications may also be questionable if coelution exists. Non-detects with retention times in the co-elution may not be valid, depending on the extent of the problem. Professional judgement should be used to determine the need to qualify data as unusable "R".

Comments:

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II. GC/ECD Instrument Performance Check (continued)

2 Performance Evaluation Mixture (PEM)

- PEM was analyzed at the proper frequency and position sequence.
- The resolution between adjacent peaks in initial and continuing calibration is $\geq 90\%$ in both GC columns.
- The absolute RTs for pesticides in each analysis are within the calculated RT windows base on the mean RT from the three-point calibration.
- The percent difference between the calculated amount and the nominal amount for each pesticides and surrogates in both PEM is $\geq -25\%$ and $\leq 25\%$.
- The individual breakdowns for 4,4'-DDT and endrin $\leq 20\%$. *15*

Yes or No
NA Yes or No
 Yes or No
NA Yes or No
 Yes or No

Deviations:

Compound	Deviation	SAIC Sample No.	Laboratory ID No.	Qualifiers Applied
4,4'-DDT	%Break=20%	LUUT14-8	0203040-13	4,4'-DDE NJ

Action:

1. If PEM was not analyzed at the required frequency, then professional judgment should be used if the associated sample data should be qualified.
2. If RT of the pesticides in the PEM are outside windows, professional judgment should be used. Sample data that are potentially affected for standards not meeting the RT windows should be noted in the data review narrative.
3. If percent difference criteria are not met, qualify all associated positive results generated during the analytical sequence with "J" and the sample quantitation limits for non-detected target compounds with "UJ".
4. If the breakdown of DDT is $>15\%$, then qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE are detected, then qualify the detection limit for DDT as unusable "R". Positive DDD and/or DDE as presumptively present at an approximated quantity "NJ".
5. If endrin breakdown is $>20\%$, qualify all positive results for endrin with "J". If endrin was not detected but, endrin aldehyde and endrin ketone are detected, then qualify the quantitation limit for endrin as unusable "R". Qualify positive results for endrin aldehyde and endrin ketone as presumptively present at an approximated quantity "NJ".
6. If the combined 4,4'-DDT and endrin breakdown is $>30\%$ qualify all positive results for 4,4'-DDT and endrin with "J". If DDT was not detected, but DDD and DDE are detected, then qualify the quantitation limit for DDT as unusable "R". If endrin was not detected, but endrin aldehyde and endrin ketone are detected, then qualify the quantitation limit for endrin as unusable "R". Qualify positive results for DDD, DDE, and /or endrin aldehyde and endrin ketone presumptively present at an approximated quantity "NJ".

Comments: 4,4'-DDT was detected.

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All criteria were met

III. Initial Calibration

- The resolution between adjacent peaks in the midpoint concentration of Individual Mixtures (ISM) A and B in the initial calibration is $\geq 90\%$ in both columns. Yes or No *NA*
- The %RSD of the calibration factors (CF) for each of the single component compounds in ISM A and B is $\leq 20\%$. The %RSD for the two surrogates is $\leq 30\%$ (up to two single component target pesticides may exceed the 20% limit, but must be below 30%.) Yes or No

Deviations:

Compound	%RSD	Resolution	Samples Affected		Qualifiers Applied
			SAIC Sample No.	Laboratory ID No.	

Actions:

1. If the the initial calibration sequence was not followed as required, professional judgment must be used to qualify the data.
2. If resolution criteria are not met, then positive sample results for compounds that were not adequately resolved should be qualified with "J". Qualitative identifications may be questionable if coelution exist. Professional judgement should be used to qualify data as unusable "R".
3. If the %RSD criteria are not met for the compounds being quantified, qualify all associated positive results with "J" and non-detected target compounds with "UJ".

Comments :

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All criteria were met

IV. Continuing Calibration

- An instrument blank and the PEM bracketed one end of a 12-h period during which samples are analyzed, and a second instrument blank and the midpoint concentrations of ISM A and B bracketed the other end of the 12-h period.

Yes or No

- The resolution between two adjacent peaks in the midpoint concentration of ISM A and B is $\geq 90\%$.

Yes or No

- The absolute RT for each single component pesticide and surrogate in the midpoint of the ISM A and B are within the RT windows determined from the initial calibration.

Yes or No

- The %D between the calculated amount and the true value amount for each of the pesticides and surrogates in the midpoint concentration of the ISM A and B does not exceed $\pm 15\%$.

Yes or No

Deviations:

Compound	Resolution	%D	RT	Samples Affected		Qualifiers Applied
				SAIC Sample No.	Laboratory ID	

Actions:

- If the the continuing calibration sequence was not followed as required, professional judgment must be used to qualify the data.
- If resolution criteria are not met, then positive sample results for compounds that were not adequately resolved should be qualified with "J". Qualitative identifications may be questionable if coelution exist. Professional judgment should be used to qualify data as unusable "R".
- If the %D criteria are not met for the compounds being quantified, qualify all associated positive results with "J" and non-detected target compounds with "UJ".
- Professional judgment must be used to qualify data which does not meet the RT criteria.

Comments :

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All criteria were met

V. Blanks

The contamination in the blanks are listed below.

Laboratory Blanks (e.g., method, instrument blanks, and sulfur cleanup blanks)

Date Analyzed	Laboratory ID	Compound	Concentration (ppb)	Action Level

Associated Field QC Blanks (e.g., field blanks, equipment rinsates): Tool Rinsate #3.

Collection Date	SAIC Sample ID	Laboratory ID	Compound*	Concentration (ppb)	Action Level
No Detects					0

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All criteria were met

V. Blanks (continued)

Actions:

Action levels are based on 5 times the highest concentration of contaminant in any associated blank for all pesticide/PCB compounds.

Sample weights, volumes, and dilution factors are to be taken into account when applying the "5X" criteria so that a comparison of the total amount of contamination can be made. Sample results are to be reported unqualified when the concentration of the contaminant exceeds the action level. When the concentration of the contaminant is detected below the CRQL in the sample, then the CRQL should be reported as non-detected with "U". The sample CRQL should be elevated to the concentration detected in the sample and reported as non-detected "U", when the contaminant is detected in the sample at a concentration less the action level, but greater than the CRQL.

If gross contamination exists (i.e., saturated peaks) all affected compounds in the associated samples should be qualified as unusable "R" due to interference.

Blank ID	Compound	Maximum Concentration Detected. ppb	Action Level	Sample Affected		Qualifiers Applied
				SAIC Sample No.	Laboratory ID No.	
			0			

Comments:

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All criteria were met

VI. Surrogate Spikes

Surrogate Spike Recovery Criteria (advisory limits)

<u>Pesticide/PCB</u>	<u>Water (%R)</u>	<u>Soil (%R)</u>
Tetrachloro-m-xylene (TCX)	30-150	30-150
Decachlorobiphenyl (DCB)	30-150	30-150

- The RTs of both of the surrogates in the PEM, ISMs, and samples are within the calculated RT windows. TCX is within ±0.05 minutes and DCB is within ±0.10 minutes of the mean RT determined from the initial calibration.

Yes or No

- All surrogate spike recoveries reported on Form II were checked and compared to the control limits.

Yes or No

Deviations:

SAIC Sample ID	Laboratory ID No.	Matrix	TCX	DCB	Qualifiers Applied

Actions:

- If low recoveries (i.e., between 10 and 30 percent), associated detected compounds should be qualified "J" and quantitation limit "UJ".
- If high recoveries (i.e., > 150%) are obtained, this may be an indication of a high bias co-eluting interference. Qualify associated detected compound data with "J", non-detected compounds do not require qualification.
- If any recovery falls between 0% and 10% associated detected compounds should be qualified "J" and non-detected target compounds should be qualified as unusable "R".
- Professional judgment should be used to qualify sample data when the surrogate recovery is zero. If the surrogate is not present, qualify all non-detected target compounds as unusable "R".

Comments :

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Not Performed

VII. Matrix Spike/Matrix Spike Duplicate

Sample IDs:

MS/MSD Criteria

	Water		Soil/Sediment	
	%R	%RPD	%R	%RPD
4,4'-DDE	75-125	30	75-125	30
4,4'-DDD	75-125	30	75-125	30
4,4'-DDT	75-125	30	75-125	30

- For every 20 samples collected, one field sample of each type was spiked for MS/MSD analysis.

Yes or No

Deviations:

Compounds	Matrix	%R		RPD	Sample Affected		Qualifiers
		MS	MSD		SAIC Sample No.	Laboratory ID No.	

Actions:

- 1 No action is taken on MS/MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with the other QC criteria and determine the need for some qualification of the data.

Comments :

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All criteria were met

VII. Laboratory Control Sample

- LCS was prepared, analyzed, and reported once per SDG. Yes or No

LCS Criteria

	<u>Water</u>	<u>Soil/Sediment</u>
4,4'-DDE	75-125	75-125
4,4'-DDD	75-125	75-125
4,4'-DDT	75-125	75-125

- LCS percent recoveries must be within the QC limits. The LCS must meet the recovery criteria for sample data to be accepted.

Deviations:

Compounds	QC limits	%R	Sample Affected		Qualifiers Applied
			SAIC Sample No.	Laboratory ID No.	

Actions:

- If the LCS recovery is greater than the upper acceptance limit, then positive results for the affected compound(s) should be qualified with a "J".
- If the LCS recovery is less than the lower acceptance limit, detected target compounds should be qualified "J" and all associated non-target compounds should be qualified unusable "R".
- If more than the half of the compounds in the LCS are not within the recovery criteria, then all associated target compounds should be qualified "J" and all associated non-detected detected target compounds should be qualified "R".
- Professional judgment should be used to qualify data for compounds other than those compounds that are included in the LCS. Professional judgment to qualify non-LCS compounds should take into account the compound class, recovery efficiency, analytical problems associated with each compound, and comparability in performance of the compound to the non-LCS compound.

Comments :

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Not analyzed with this ETR

VIII. Field Duplicates

- The following field duplicate set was analyzed with this lot:

- Field duplicates were collected at the rate of 1 per every 10 or fewer investigative samples.

Yes or No

Sample ID	Field Duplicate ID	Matrix	Compound	RPD

- There are no required criteria for field duplicate analyses comparability.
- Any evaluation of the field duplicates should be provided within the data reviewer's narrative.

Comments:

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Not Applicable

IX. Pesticide Cleanup Checks

- The Florisil Cartridge Check has percent recoveries for all pesticide and surrogate compounds within 80 to 120% and the recovery of 2,4,5-trichlorophenol is < 5%, and there are no peaks interfering with the detected target analytes. Yes or No
- The Gel Permeation Chromatography (GPC) check has percent recoveries within 80 to 110% for the pesticide compounds and aroclor patterns meet criteria. Yes or No
- A GPC blank was analyzed after each GPC calibration and none of the target compounds detected exceed one half of the CRQL. Yes or No
- The calibration of the GPC is checked once every 7 days. Yes or No

Deviation:

Check Sample ID	Cleanup Check ID	Compound	Sample Affected		Qualifier Applied
			SAIC Sample ID	Laboratory ID No.	

Actions:

- 1 If Florisil Cartridge Check and GPC criteria are not met, professional judgment should be used in qualifying the data.
- 2 If zero recovery was obtained for the pesticide compounds and surrogates during either check, then the non-detected target compounds may be suspect and the data may be qualified unusable "R".
- 3 If high recoveries (i.e., >120%) were obtained for the pesticide and surrogates during either check, use professional judgment to qualify detected target compounds. Non-detected target compounds do not required qualification.

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CLP SEMIVOLATILE ORGANICS (Multi-Media, Multi-Concentration)

All criteria were met

X. Compound Quantitation and Reported CRQLs

1. The method quantitation limit reported by the laboratory was less than or equal to the CRQL (unless dilution required).

Yes or No

2. The detection limit for each sample has been adjusted to reflect all sample dilutions and dry weight factors.

Yes or No

Deviations on CRQL :

SAIC Sample ID	Laboratory ID No.	Compound	Sample Detection Limit	CRQL	Qualifier Applied

4. Samples were diluted where appropriate : Yes or No

The following samples were diluted : DF=5, 10, or 20

Deviations on dilutions :

SAIC Sample ID	Laboratory ID No.	Compound	Reported Result	Qualifier Applied

Comments:

F O U R
Pesticides by GC/ECD



Client: **Science Application International Corp.**
 Project: **Palos Verdes**
 Case: **N/A** SDG: **N/A**
 Client ID: **LUU14-6**
 Matrix: **Sediment**

Lab Code: **M-MA030**
 ETR: **0203040**
 Lab ID: **0203040-11**
 Associated Blank: **PS0613B1**
 Concentration Units: **µg/Kg**

Date Collected	Date Received	Date Extracted	Date Analyzed	Percent Solid	Sample Amount (g)	Final Volume (ml)	Dilution Factor	Analyst
03/11/02	03/12/02	06/13/02	06/14/02	76.1	10.22	10	10	DMB

Parameter	Result
4,4'-DDE	840

DV QUALIFIERS

8/15/02
CS

Surrogate	% Recovery	Acceptance Range (%)	N/A - Not Applicable
Tetrachloro-meta-xylene	86	30-150	
Decachlorobiphenyl	103	30-150	



**Form 1
Pesticides by GC/ECD**

Client: **Science Application International Corp.**
 Project: **Palos Verdes**
 Case: **N/A** SDG: **N/A**
 Client ID: **LUU14-7**
 Matrix: **Sediment**

Lab Code: **M-MA030**
 ETR: **0203040**
 Lab ID: **0203040-12**
 Associated Blank: **PS0613B1**
 Concentration Units: **µg/Kg**

Date Collected	Date Received	Date Extracted	Date Analyzed	Percent Solid	Sample Amount (g)	Final Volume (ml)	Dilution Factor	Analyst
03/11/02	03/12/02	06/13/02	06/14/02	71.8	10.30	10	5	DMB

Parameter	Result
4,4'-DDE	1000

DV QUALIFIERS

9/5/02
CS

Surrogate	% Recovery	Acceptance Range (%)
Tetrachloro-meta-xylene	81	30-150
Decachlorobiphenyl	94	30-150

N/A - Not Applicable

FORM 1
Pesticides by GC/ECD



Client: **Science Application International Corp.**
 Project: **Palos Verdes**
 Case: **N/A** SDG: **N/A**
 Client ID: **LUU14-8**
 Matrix: **Sediment**

Lab Code: **M-MA030**
 ETR: **0203040**
 Lab ID: **0203040-13**
 Associated Blank: **PS0613B1**
 Concentration Units: **µg/Kg**

Date Collected	Date Received	Date Extracted	Date Analyzed	Percent Solid	Sample Amount (g)	Final Volume (ml)	Dilution Factor	Analyst
03/11/02	03/12/02	06/13/02	06/18/02	69.7	10.27	10	20	DMB

Parameter	Result
4,4'-DDE	2600

- NJG

DV QUALIFIERS

8/5/02
CS

Surrogate	% Recovery	Acceptance Range (%)
Tetrachloro-meta-xylene	106	30-150
Decachlorobiphenyl	127	30-150

N/A - Not Applicable