

Appendix G:

Inter-Laboratory Comparison Sub-Study

Appendix G document:

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Appendix G tables:

Table G1. Analytical Standard Comparison Results

Table G2. Sample Extracts Results Chlorpyrifos (CPF) and Chlorpyrifos-oxon (CPF-oxon)

Table G3. Spiked OVS Tubes Results Chlorpyrifos (CPF) and Chlorpyrifos-oxon (CPF-oxon)

Appendix G Key Terms

- id= identification
- ng= nanogram
- ng/m³= nanograms over cubic meters
- CPF= chlorpyrifos
- CPF total= sum of chlorpyrifos and chlorpyrifos-oxon expressed as the molar equivalent of CPF
- CPF-oxon= chlorpyrifos-oxon
- std dev= standard deviation
- CV = coefficient of variation
- n/a= non applicable

ORGANOPHOSPHORUS PESTICIDE AIR MONITORING PROJECT

Inter-Laboratory Comparison

As a part of the quality control component of this study, the University of Washington has arranged with Randy Segawa at the California Department of Pesticide Regulation (Cal-EPA) for a comparative analysis across laboratories. CDPR analyzed air samples for pesticides, including chlorpyrifos and its oxon, as a part of the 2006 Parlier study.

The CDPR lab uses a GC-MS method for measurement of chlorpyrifos and chlorpyrifos-oxon, and reports a limit of quantification of 0.2 micrograms per sample for both analytes. The UW Environmental Health Laboratory has developed an LC-MS-MS method for analysis of these same compounds, and reports a limit of quantification of 2 nanograms per sample for each analyte.

Inter-Laboratory Comparison Plan

UW will send CDPR samples for analysis as described here and as summarized in the table below. The UW lab will prepare all of these samples in duplicate and will run an identical set of samples so that a direct comparison can be made across laboratories.

Analytical Standards. UW will provide a sample of each of the analytical standards (chlorpyrifos and chlorpyrifos-oxon) that are being used for quantification. These will ensure that we have comparable analytical methods.

Spiked OVS Tubes. UW will spike three tubes at two times the CDPR LOQ, and three tubes at four times the CDPR LOQ with both chlorpyrifos and chlorpyrifos-oxon. Only the front section of the tubes will be spiked, so analysis of the back sections of the tubes will not be necessary. These will allow comparison of both extraction and analysis procedures.

Disguised Standard. One additional calibrant will be sent that is labeled with a laboratory label as a field sample. It will contain both chlorpyrifos and chlorpyrifos-oxon each spiked with an appropriate quantity of the analytical standard used above.

Field Sample Extracts. UW will provide 5 extracts from samples with concentrations of both chlorpyrifos and chlorpyrifos-oxon above the CDPR LOQ of 0.2 micrograms per sample. These will allow comparison of analysis procedures independent of extraction procedures.

Blank OVS tube. One blank sample will be included in the sample batch for quality control purposes

Unused OVS tubes. Ten unused OVS tubes will be provided for internal CDPR quality control procedures.

Retention of sample preparations. UW will retain portions of all samples sent to CA and store at - 80 C.

Sample Type	Number	Analyte(s)	Comments
Analytical standard	1	chlorpyrifos	Standard comparison
Analytical standard	1	Chlorpyrifos-oxon	Standard comparison
Disguised standard	1	chlorpyrifos + oxon	Analytical standard spiked on OVS tube
Spiked OVS tube	3	Chlorpyrifos + oxon	Spike level = 0.4 ug
Spiked OVS tube	3	Chlorpyrifos + oxon	Spike level = 0.8 ug
Field sample extract	5	Chlorpyrifos + oxon	
Blank OVS tube	1	Chlorpyrifos + oxon	
Unused OVS tubes	10		For CDPR QC
Total for analysis	15		

Table G1: Analytical Standard Comparison Results

Analytical Standard Comparison Result:						
	Chlorpyrifos (1000 ng/mL) response			Chlorpyrifos OA (1000 ng/mL) response		
Injection #	UW	CDPR-CDFA	% Difference^a	UW	CDPR-CDFA	% Difference^a
1	58815	63645		33359	36934	
2	58145	65470		36828	36818	
3	62762	55279		32967	30149	
4	63050	58323		31920	31677	
5		68371			39294	
6		58250			28515	
7		59647			31925	
Average	60693	61284	-0.97%	33769	33616	0.45%
STDEV	2573	4655		2128	4045	
%CV	4.24%	7.60%		6.30%	12.03%	
Footnotes						
^a % difference= (Avg UW response -Avg CDPR response)/Avg UW response						

Table G2: Sample Extracts Results Chlorpyrifos (CPF) and Chlorpyrifos-oxon (CPF-oxon)

UW-CDFA lab comparison: Sample extracts^a						
Sample ID	UW CPF Analysis (ng)	CA CPF Analysis (ng)	UW as % of CA	UW CPF-Oxon Analysis (ng)	CA CPF-Oxon Analysis (ng)	UW as % of CA
6150 ^a	979	1240	79%	263	336	78%
6128	1582	1460	108%	273	543	50%
6135	1920	1650	116%	278	550	51%
3623	934	1890	49%	299	744	40%
6068	1424	1320	108%	349	645	54%
6110	2092	1840	114%	382	774	49%
		Mean	99%		Mean	49%
		Std Dev	0.28		Std Dev	0.05
		CV^b	28.3		CV^b	10.6
Footnotes:						
^a all CA sample values were above the highest value of the calibration curve (1,000 ng)						
^a Sample 6150 was a disguised standard; values not included in mean calculation						
^b CV = std dev/mean *100						

Table G3: Spiked OVS Tubes Results Chlorpyrifos (CPF) and Chlorpyrifos-oxon (CPF-oxon)

UW-CDPR Inter-lab comparison: Spiked OVS tubes										
Sample ID	CPF Spike (ng)	UW CPF (ng)	UW as % CPF Spike	CA CPF (ng)	CA % CPF Spike	CPF-Oxon Spike (ng)	UW CPF-Oxon (ng)	UW CPF-oxon % CPF Oxon Spike (%)	CA CPF-Oxon (ng)	CA CPF-Oxon % of CPF-Oxon
1	0	<1	na	<50	na		<1	na		
QC-1	400	380	95%	311	78%	400	312	78%	256	64%
QC-2	400	377	94%	335	84%	400	315	79%	287	72%
QC-3	400	364	91%	327	82%	400	301	75%	314	79%
	Mean	374	93%	324	81%	Mean	309	77%	286	71%
	Std Dev	8.5	0.02	12.2	0.03	Std Dev	7.4	0.02	29.0	0.07
	CV		2.3		3.8	CV		2.4		10.2
QC-4	800	777	97%	609	76%	800	643	80%	586	73%
QC-5	800	771	96%	631	79%	800	638	80%	571	71%
QC-6	800	751	94%	683	85%	800	620	78%	572	72%
	Mean	766	96%	641	80%	Mean	634	79%	576	72%
	Std Dev	13.6	0.02	38.0	0.05	Std Dev	12.1	0.02	8.4	0.01
	CV		1.8		5.9	CV		1.9		1.5