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APPENDIX 1: Analytical Methods

ANALYTICAL METHODS

Table 1 shows an overview of all constituents, the methods of analyses, the analyzing agencies, the minimum detection and rating limits, and the used instrumentation. Water column toxicity and sediment toxicity methods are described separately below.

Toxicity Testing Methods

The Department Fish and Game Aquatic Toxicology Laboratory and AQUA-Science Laboratory ran the water column toxicity testing. Acute toxicity testing was conducted using the invertebrate *Ceriodaphnia dubia* and the larval fathead minnow *Pimephales promelas* according to standard method EPA 821/R-02-012. To identifying toxicity caused by herbicides, 96-hour tests with the green algae *Selenastrum capricornutum* were conducted according to standard method EPA 821/R-02-013.

Sediment Pesticide analysis

The extraction method for the sediment was a modification of USEPA Method #3550, Sonication Extraction for low concentrations of organics and pesticides. Approximately 20 g (±1.0 g) of sediment were removed, spiked with 50ng each of surrogates, dibromooctoflourobiphenyl (DBOFB) and decachlorobiphenyl (DCBP) and dried with anhydrous magnesium sulfate. In case of high sulfur content sediment, 2g of activated copper metal were added to remove sulfur residue. The sample was sonicated with 50 ml of 50:50 methylene chloride:acetone (v/v) for 5 minutes in 3 s pulse mode using a high intensity ultrasonic processor (Model VCX 400, Sonics and Materials Inc., Newtown, CT, USA), decanted and filtered through a Whatman No. 41 filter paper filled with anhydrous magnesium sulfate. This procedure was repeated twice more with a sonication time of 3 minutes. The extract was then collected in an evaporative tube and reduced in volume to approximately 5 ml, under a stream of nitrogen in a TurboVap II evaporator (Zymark, Hopkinton, MA). After cooling, the extract was solvent exchanged with hexane and the volume further reduced to 2 ml.

A deactivated Florisil column was used to remove interference from the extract. The column was packed with 10g Florisil partially deactivated by mixing with distilled water (6% w/v) and a 1cm layer of anhydrous sodium sulfate was used to cap the Florisil. After the concentrated extract was transferred into the Florisil column, pesticides were eluted from the column with 50 mL of 30% diethyl ether in hexane solution (v/v). The eluent was concentrated, dissolved in 2 mL of hexane and transferred to clean screw-cap vials, sealed with a Teflon lined lid and stored at -4°C until analysis on the GC. Additional dilution steps may have been needed for some field-collected agricultural samples due to elevated pesticide concentrations.

Table 1. Laboratory Detection and Reporting Limit Requirements

Table 1. Laboratory Detection and Reporting Limit Requirements

MediumName	MethodName	AnalyteName	FractionName	Units	ChemAgency Code	DL	RL	INSTRUMENTATION
GENERAL PARAMETERS								
samplewater	QC 10308001A	Color	None	Color Units	DFG-WPCL	5	10	FIA
samplewater	QC 10308001A	Color	None	Color Units	DFG-WPCL	5	5	Nessler
samplewater	SM 2130B	Turbidity low level	None	NTU	DFG-WPCL	0.05	0.15	Nephelometer
samplewater	SM 2130B	Turbidity mid level	None	NTU	DFG-WPCL	0.2	1.0	Nephelometer
samplewater	SM 2130B	Turbidity high level	None	NTU	DFG-WPCL	10	10	Nephelometer
samplewater	SM 2540C	Dissolved Solids	Total	mg/L	DFG-WPCL	10	10	
samplewater	EPA 415.1	Total Organic Carbon	Total	mg/L	DFG-WPCL	0.2	1.0	
TRACE ELEMENTS								
samplewater	EPA 1638	Arsenic	Dissolved/Total	µg/L	MPSL-DFG	0.30	0.50	ICP-MS
samplewater	EPA 1638	Boron	Dissolved/Total	µg/L	MPSL-DFG	0.0002	0.0006	ICP-MS
samplewater	EPA 1638	Cadmium	Dissolved/Total	µg/L	MPSL-DFG	0.01	0.02	ICP-MS
samplewater	EPA 1638	Copper	Dissolved/Total	µg/L	MPSL-DFG	0.02	0.04	ICP-MS
samplewater	EPA 1638	Lead	Dissolved/Total	µg/L	MPSL-DFG	0.003	0.02	ICP-MS
samplewater	EPA 1638	Nickel	Dissolved/Total	µg/L	MPSL-DFG	0.03	0.06	ICP-MS
samplewater	EPA 1638	Phosphorous	Dissolved/Total	µg/L	MPSL-DFG	2.0	6.0	ICP-MS
samplewater	EPA 1638	Selenium	Dissolved/Total	µg/L	MPSL-DFG	0.20	0.50	ICP-MS
samplewater	EPA 1638	Zinc	Dissolved/Total	µg/L	MPSL-DFG	0.05	0.20	ICP-MS
INORGANIC (CONVENTIONAL ANALYTES)								
samplewater	SM 4500 NH3 D	Ammonia as N	None	mg/L	DFG-WPCL	0.04	0.100	ISE
samplewater	QC 10107041B	Nitrate+Nitrite as N	None	mg/L	DFG-WPCL	0.010	0.0200	FIA
samplewater	QC 10107041B	Nitrite as N	None	mg/L	DFG-WPCL	0.0020	0.0050	FIA
samplewater	QC 10115011M	Phosphate as P, Ortho	None	mg/L	DFG-WPCL	0.0050	0.0100	FIA
ORGANOCHLORINE PESTICIDES								
samplewater	EPA 8081BM	DDD(o,p')	None	µg/L	DFG-WPCL	0.001	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	DDD(p,p')	None	µg/L	DFG-WPCL	0.001	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	DDE(o,p')	None	µg/L	DFG-WPCL	0.001	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	DDE(p,p')	None	µg/L	DFG-WPCL	0.001	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	DDT(o,p')	None	µg/L	DFG-WPCL	0.001	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	DDT(p,p')	None	µg/L	DFG-WPCL	0.002	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	Dicofol	None	µg/L	DFG-WPCL	0.05	0.1	GC-ECD/GC-MS

samplewater	EPA 8081BM	Dieldrin	None	µg/L	DFG-WPCL	0.001	0.002	GC-ECD/GC-MS
samplewater	EPA 8081BM	Endrin	None	µg/L	DFG-WPCL	0.002	0.005	GC-ECD/GC-MS
samplewater	EPA 8081BM	Methoxychlor	None	µg/L	DFG-WPCL	0.001	0.002	GC-ECD/GC-MS
HERBICIDES								
samplewater	EPA 619Mod	Atrazine	None	µg/L	DFG-WPCL	0.02	0.05	GC-NPD/GC-MS
samplewater	EPA 619Mod	Cyanazine	None	µg/L	DFG-WPCL	0.02	0.05	GC-NPD/GC-MS
samplewater	EPA 619Mod	Molinate	None	µg/L	DFG-WPCL	0.1	0.2	GC-NPD/GC-MS
samplewater	EPA 619Mod	Paraquat dichloride	None	µg/L	DFG-WPCL	0.2	0.5	HPLC-MS
samplewater	EPA 619Mod	Simazine	None	µg/L	DFG-WPCL	0.02	0.05	GC-NPD
samplewater	EPA 619Mod	Thiobencarb	None	µg/L	DFG-WPCL	0.1	0.2	GC-NPD/GC-MS
CARBAMATE PESTICIDES/HERBICIDES								
samplewater	EPA 632 Mod	Aldicarb	None	µg/L	DFG-WPCL	0.01	0.02	HPLC-MS
samplewater	EPA 632 Mod	Captan	None	µg/L	DFG-WPCL	0.05	0.1	HPLC-MS
samplewater	EPA 632 Mod	Carbaryl	None	µg/L	DFG-WPCL	0.01	0.02	HPLC-MS
samplewater	EPA 632 Mod	Carbofuran	None	µg/L	DFG-WPCL	0.01	0.02	HPLC-MS
samplewater	EPA 632 Mod	Diuron	None	µg/L	DFG-WPCL	0.002	0.005	HPLC-MS
samplewater	EPA 632 Mod	Linuron	None	µg/L	DFG-WPCL	0.002	0.005	HPLC-MS
samplewater	EPA 632 Mod	Methiocarb	None	µg/L	DFG-WPCL	0.15	0.25	HPLC-MS
samplewater	EPA 632 Mod	Methomyl	None	µg/L	DFG-WPCL	0.01	0.02	HPLC-MS
PYRETHROID PESTICIDES								
samplewater	EPA 8081BM	Bifenthrin	None	µg/L	DFG-WPCL	0.001	0.002	GC-ECD/GC-MS
samplewater	EPA 8081BM	Cyfluthrin	None	µg/L	DFG-WPCL	0.002	0.004	GC-ECD/GC-MS
samplewater	EPA 8081BM	Cypermethrin	None	µg/L	DFG-WPCL	0.002	0.004	GC-ECD/GC-MS
samplewater	EPA 8081BM	Deltamethrin	None	µg/L	DFG-WPCL	0.002	0.004	GC-ECD/GC-MS
samplewater	EPA 8081BM	Esfenvalerate/Fenvalerate	None	µg/L	DFG-WPCL	0.001	0.002	GC-ECD/GC-MS
samplewater	EPA 8081BM	Fenpropathrin	None	µg/L	DFG-WPCL	0.002	0.004	GC-ECD/GC-MS
samplewater	EPA 8081BM	*Lambda*-cyhalothrin	None	µg/L	DFG-WPCL	0.001	0.002	GC-ECD/GC-MS
samplewater	EPA 8081BM	Permethrin	None	µg/L	DFG-WPCL	0.003	0.005	GC-ECD/GC-MS
ORGANOPHOSPHATE PESTICIDES								
samplewater	EPA 8141AM	Azinphos-Methyl	None	µg/L	DFG-WPCL	0.03	0.05	GC-FPD
samplewater	EPA 8141AM	Chlorpyrifos	None	µg/L	DFG-WPCL	0.003	0.005	GC-FPD
samplewater	EPA 8141AM	Diazinon	None	µg/L	DFG-WPCL	0.003	0.005	GC-FPD
samplewater	EPA 8141AM	Dimethoate	None	µg/L	DFG-WPCL	0.03	0.05	GC-FPD
samplewater	EPA 8141AM	Disulfoton	None	µg/L	DFG-WPCL	0.01	0.05	GC-FPD
samplewater	EPA 8141AM	Malathion	None	µg/L	DFG-WPCL	0.03	0.05	GC-FPD

samplewater	EPA 8141AM	Methidathion	None	µg/L	DFG-WPCL	0.03	0.05	GC-FPD
samplewater	EPA 8141AM	Methyl Parathion	None	µg/L	DFG-WPCL	0.01	0.05	GC-FPD
samplewater	EPA 8141AM	Parathion	None	µg/L	DFG-WPCL	0.01	0.02	GC-FPD
samplewater	EPA 8141AM	Phorate	None	µg/L	DFG-WPCL	0.05	0.2	GC-FPD
samplewater	EPA 8141AM	Phosmet	None	µg/L	DFG-WPCL	0.05	0.2	GC-FPD

Sediment samples were analyzed for the following pesticides: alpha-, beta-, delta-, and gamma-BHC, alpha- and gamma-chlordane, aldrin, endosulfan I and II, endosulfan sulfate, dieldrin, endrin, endrin aldehyde, endrin ketone, heptachlor, methoxychlor, heptachlor epoxide, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, chlorpyrifos, *cis* and *trans* – permethrin, bifenthrin, esfenvalerate lambda-cyhalothrin, cypermethrin, cyfluthrin, and deltamethrin. The detection limit for all analytes was 1 ng/g (or less if determined achievable in preliminary tests). The method validation was conducted with control sediment spiked with each of the target pesticides. Analyses were conducted using a Hewlett Packard 6890 Series Gas Chromatograph System (HP6890GC) equipped with an electron capture detector (ECD).

Grain Size analysis

The sediment was washed on a series of stacked brass or stainless steel sieves (1000, 500, 250, 125, and 63 μm), and the material passing through the smallest sieve collected in a large stainless steel bowl. The contents of each sieve were transferred to an aluminum pan, dried at 100^{o}C overnight and weighed. The contents of the bowl (representing the silt and clay fraction) were allowed to settle for 24-48 hr, the overlying water poured off, and the particles transferred to an aluminum pan for drying and weighing.

Toxicity testing

Sediment toxicity was assessed using a 10-day survival and growth test with *Hyalella azteca* (EPA 600/R-99/064). U.S. EPA, as a standard test for sediment toxicity testing has promulgated this test.

CNH analysis

Inorganic carbon was removed from the sample by: 1) drying at 100^{o}C overnight; 2) grinding the sample with a mortar and pestle; 3) exposure to hydrochloric acid vapors overnight); 4) driving off re-adsorbed water by drying at 100^{o}C for 2-4 hr; and 5) storage of sample at –20^{o}C or in a dessicator until analysis. CHN analyses were done by the Horn Point Environmental Laboratory, University of Maryland, Cambridge, MD using a CE-440 Elemental Analyzer from Exeter Analytical.

APPENDIX 2: Field Methods

FIELD METHODS

Discrete water samples were collected for analysis of various organic compounds (pesticides), trace elements (metals / hardness), inorganic compounds (nutrients, TOC etc.), water column toxicity, and physical parameters (pH, EC, etc). Trihalomethane samples (THM's) were only collected during the 2004 irrigation season. Due to their almost negligible detections, sampling for this class of constituent was suspended. Table 2 presents a summary of all constituents, sample container, volume, initial preservation and holding time recommendations.

Table 2. Summary of Sample Container, Volume, Initial Preservation and Holding Time Recommendations for Water and Sediment Samples

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Physical Parameters[1]				
Color	1 liter glass or polyethylene	500 ml	Cool to 4°C, dark	48 hours at 4°C, dark
Turbidity	"	150 ml	"	48 hours at 4°C, dark
Dissolved Solids	"	1000 ml	"	7 days at 4°C, dark
Nutrients[1]				
Ortho-phosphate ($O\text{-}PO_4$)	Trace clean and certified polyethylene	100 ml	Cool to 4°C, dark	48 hours at 4°C, dark
Nitrate + Nitrite ($NO_3 + NO_2$)	"	150 ml	"	Recommend 48 hours at 4°C, dark or If preserved, H_2SO_4 pH<2 28 days, either one at 4°C, dark
Nitrite (NO_2)	"	150 ml	"	48 hours at 4°C, dark
Ammonia (NH_3)	"	500 ml	"	Recommend 48 hours at 4°C, dark or If preserved, H_2SO_4 pH<2 Recommend: 7 days Maximum: 28 days Either one at 4°C, dark
(1) NOTE: The volume of water necessary to collect in order to analyze for the above constituents is typically combined in multiple 1-liter polyethylene bottles, which also allows enough volume for possible re-analysis and for conducting lab spike duplicates. This is possible since the same laboratory is conducting all of the above analyses; otherwise, individual volumes apply.				
TOC and THMs in Drinking Water and Surface Water				
Trihalomethanes (chloroform, bromoform, dibromochloromethane, bromodichloromethane)	40 ml VOA vials	120 ml (three VOA vials)	Cool to 4°C, dark	14 days at 4°C, dark

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Total Organic Carbon (TOC)	40 ml glass vial	40 ml (one vial)	Cool to 4°C, dark	28 days at 4°C, dark
Trace Elements in Water Samples				
TOTAL ELEMENTS (As, B, Cd, Cu, K, Ni, P, Pb, Se, Zn)	60 ml polyethylene bottle, pre-cleaned in lab using HNO_3	60 ml (one bottle)	Cool to 4°C, dark. Acidify in lab within 48 hrs, with ultra-pure HNO_3 for pH<2.	Once sample is acidified, can store up to 6 months at room temperature
HARDNESS	200 ml polyethylene or glass bottle	200 ml (one bottle)	Cool to 4°C, dark	48 hours at 4°C, dark
Synthetic Organic Compounds in Water Samples				
PESTICIDES & HERBICIDES* □Organophosphate Pesticides □Organochlorine Pesticides □Carbamates □Pyrethroids □Herbicides	1-L I-Chem 200-series certified trace clean amber glass bottle, with Teflon lid-liner (per each sample type)	1000 ml (one container) *Each sample type requires 1000 ml in a separate container	Cool to 4°C, dark If chlorine is present, add 0.1g sodium thiosulfate	Keep at 4°C, dark, up to 7 days. Extraction must be performed within the 7 days; analysis must be performed within 40 days of extraction.
Toxicity Testing - Water Samples				
TOXICITY IN WATER	Two-Four 2.25 L I-Chem 200-series certified amber glass bottles	9000 ml	Cool to 4°C, dark	36 hours at 4°C, dark
Sediment Toxicity - Sediment Samples				
TOXICITY IN SEDIMENT	Four L I-Chem 200-series certified clear glass jugs	3000 ml	Cool to 4°C, dark	One week at 4°C, dark

The water samples were collected following the Standard Operating Procedures included in the Quality Assurance Project Plan developed for the Irrigated Lands Monitoring Program. Samples were categorized as either grab or integrated grab samples. Grab samples were a single sample taken from one location. Integrated samples were a single sample taken from a composite of three different locations, for example across a bridge.
Sediment for sediment toxicity was collected following the Standard Operating Procedure by Don Weston using the metal scoop method.

The water and sediment samples were put on ice immediately after collection and kept on ice until delivered to the different laboratories. The Water Column Toxicity samples were delivered to either AQUA-Science Laboratory (Davis, CA) or CA Department Fish and Game Aquatic

Toxicology Laboratory, the metal samples were acidified by UCD AEAL upon return from the field and then sent to the Department of Fish & Game Marine Pollution Studies Laboratory in Moss Landing. TOC samples were analyzed, in part, by the UC Davis Department of Civil and Environmental Engineering, and in part by the Department of Fish and Game Water Pollution Control Laboratory. All other samples were analyzed at the Department of Fish and Game Water Pollution Control Laboratory in Rancho Cordova. The sediment samples were picked up by Don Weston at UCD AEAL and analyzed at UC Berkeley.

Air and water temperature, pH, electrical conductivity (EC) and dissolved oxygen (DO) were measured using Oakton pH/Con 10 Multiparameter Meter and Oakton Accumet Dissolved Oxygen Meter. Field measurements, weather and water conditions were noted on field sheets as well as the sampling time, the number of collected environmental and quality control samples.

Velocity was measured either by using a bridgeboard or by wading. Four different current meters were used to determine the stream velocities: USGS Price Type AA Current Meter for low and normal velocities, Swoffer Current Velocity Meter Model 2100 or Marsh-McBurney Velocity Meter FLO-MATE Model 2000. Velocity values for some sites were obtained from gauges. Discharge was not measured at all during the 2006 dormant season and during the 2007 sampling efforts as it was not called for.

Discharge was measured following the standard method described in USDA Technical Report RM-245. For velocity that was measured in a channel, the currently recommended mid-section method by the U.S. Geological Survey was used to compute discharge (Harrelson 1994). The failure to measure discharge with every sampling event was due to one of several reasons 1) malfunction of equipment 2) flow was too high or fast for wading 3) water level or flow was too low to take discharge measurement 4) discharge was not required.

Some sites had culverts rather than bridges. Frequently culvert discharges were at high enough velocities to render the bridge method ineffective due to the inability to keep the line vertical on the velocity meter. Also, during periods of high discharge, wading velocity measurements from these culverts can be quite dangerous.

It was determined that velocity did not vary across the width of the culvert at the downstream end at a given depth. Velocity did vary across depth at the downstream end. Given these factors it was determined that it would be possible to accurately estimate discharge by taking velocity measurements at several depths and applying those velocities to horizontal sections of the water column at those depths. Rather than taking many velocity measurements across the width of the culvert, it was possible to use just one measurement at each depth. In this way field technicians could reduce risk since they were not required to reach across the width of the culvert, and they could take just three measurements instead of 20 or more.

Culvert flows were calculated by estimating the cross-section area of the water at the point where it leaves the culvert, then multiplying this area by the velocity of the water. The water was divided into three sections by depth, with velocities taken at one point in each depth range. The depth ranges were bottom of water column to 70% depth of water (DoW); 70% DoW to 40% DoW; and 40% DoW to water surface. The velocity was recorded at 80% DoW, 60% DoW, and 20% DoW. The depths for velocity measurements were chosen based on USGS protocol for velocity estimation in a channel.

APPENDIX 3: Quality Assurance Procedures

QUALITY ASSURANCE PROCEDURES

Quality assurance samples were collected and analyzed to guarantee that the data generated during the analytical phase of the project fulfill Quality Control specifications for precision, accuracy, representativeness, comparability and completeness (PARC). Three types of field quality assurance samples were evaluated: field blanks, field duplicates and matrix spike samples.

Field Quality Control

Field QC samples are used to assess the influence of sampling procedures and equipment used in sampling. They are also used to characterize matrix heterogeneity. For basic water quality analyses, quality control samples to be prepared in the field will consist of field blanks, field duplicates and matrix spikes (when applicable). The number of field duplicates and field blanks are set to achieve an overall rate of at least 5% of all analyses for a particular parameter or one per batch. The external QA samples are rotated among sites and events to achieve the overall rate of 5% field duplicate samples and 5% field blanks (as appropriate for specific analyses).

Field Blanks

Field blanks were generated to demonstrate that neither the sampling procedures nor atmospheric exposure resulted in contaminated samples. Field blanks were collected at a rate of 5% of the total number of samples along with the associated environmental sample. Field blanks were assigned randomly to sampling sites and were distinguished from the environmental sample through a time offset of 1 minute. Water used for the blanks consisted of de-ionized water from the Aquatic Biology and Environmental Sciences Building at UC Davis for all blanks except the blanks established for metals and water column toxicity. UCD Vet- Med Central Services MilliQ water was used for the metal samples, and blank water from DFG-ATL and Aqua-SCIENCE Laboratory for the toxicity samples.

Field Duplicates

Field duplicate samples demonstrate the precision of the analytical process. Duplicates were collected in rapid succession and in an identical manner to the associated environmental sample. Duplicates were collected at a rate of 5% of the total samples and were assigned randomly to sample sites. Duplicates were distinguished from the environmental sample through a time offset of 3 minutes. For cases where contaminants were detected in both samples, the assessment of the difference in concentration between the environmental sample and the paired replicate was determined by calculating the relative percent difference between the two values, which is defined as:

$$RPD = (([C_{env} - C_{rep}] / ([C_{env} + C_{rep}]/2)) * 100$$

RPD = the relative percent difference
C_{env} = concentration of pesticide in environmental sample
C_{rep} = concentration of pesticide in replicate sample.

If an RPD greater than 25% is confirmed by reanalysis, the environmental results were flagged with the appropriate qualifier code.

Matrix Spikes and Matrix Spike Duplicates

The purpose of analyzing matrix spikes (MS) and matrix spike duplicates (MSD) was to demonstrate the performance of the analytical method in a particular sample matrix. Matrix spike and matrix spike duplicate samples were collected at a rate of 5% or one pair per sample batch, assigned randomly to sites and labeled with a time offset of 9 minutes. Each matrix spike and matrix spike duplicate consisted of an aliquot of laboratory-fortified environmental samples. Recovery is the accuracy of an analytical test measured against a known analyte addition to a sample.

Recovery is calculated as follows:

Recovery = ((Matrix plus spike result – Matrix result) * 100) / expected Matrix plus spike result

If matrix spike recovery of any analyte was outside of the acceptable range, the result was determined to have failed the acceptance criteria (75-125%) the data will be flagged with the appropriate qualifier code.

Laboratory Quality Control

Laboratory QC was necessary to control the analytical process within method and project specifications, and to assess the accuracy and precision of analytical results.
For basic water quality analyses, quality control samples prepared in the laboratory(s) typically consisted of equipment blanks, method blanks, laboratory control samples, laboratory duplicates and a surrogate added to each sample (organic analysis).

Method Blanks

The purpose of analyzing method blanks was to demonstrate that the analytical procedures did not result in sample contamination. Method blanks (MB) were prepared and analyzed by the laboratory at a rate of at least one for each analytical batch.

Laboratory Control Samples

The purpose of analyzing laboratory control samples (LCS) was to demonstrate the accuracy of the analytical method. Laboratory control samples were analyzed at the rate of one per sample batch.

Laboratory Duplicates

The purpose of analyzing laboratory duplicates (LCSD) was to demonstrate the precision of the analytical method. Laboratory duplicates were analyzed at the rate of one pair per sample batch.

APPENDIX 4: Data summary tables

4.1. FIELD MEASUREMENTS

Table 3. Field Measurements summary

FIELD MEASUREMENTS (2004-2007)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Discharge (cfs)	83.32	11.98	0.00	1457.00	237	237	100
Oxygen, Dissolved (mg/L)	7.74	8.00	0.10	23.10	578	578	100
pH	7.46	7.39	4.48	11.40	580	580	100
Specific Conductivity (µS)	332.8	195.0	3.0	2680.0	579	579	100
Temperature (°C)	18.0	19.8	6.6	36.5	580	580	100

FIELD MEASUREMENS (IRRIGATION 2004)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Discharge (cfs)	29.86	10.54	0.00	390.00	93	93	100
Oxygen, Dissolved (mg/L)	6.75	7.00	0.10	19.40	129	129	100
pH	7.47	7.29	6.28	9.43	130	130	100
Specific Conductivity (µS)	379.0	261.5	3.0	1392.0	130	130	100
Temperature (°C)	23.2	23.0	16.8	36.5	130	130	100
FIELD MEASUREMENTS (Not Applicable 2004)	**Mean**	**Median**	**Minimum**	**Maximum**	**Count**	**Count of Detection**	**% Frequency**
Discharge (cfs)	3.30	3.30	3.30	3.30	1	1	100
Oxygen, Dissolved (mg/L)	8.69	8.76	6.20	11.10	3	3	100
pH	7.70	7.78	7.11	8.20	3	3	100
Specific Conductivity (µS)	649.5	701.0	139.6	1108.0	3	3	100
Temperature (°C)	17.7	17.8	15.5	19.9	3	3	100

FIELD MEASUREMENTS (DORMANT 2005)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Discharge (cfs)	251.22	17.45	0.00	1457.00	54	54	100
Oxygen, Dissolved (mg/L)	9.17	9.60	1.20	18.70	131	131	100
pH	7.48	7.46	6.56	11.40	133	133	100
Specific Conductivity (µS)	415.4	187.0	53.3	2680.0	134	134	100
Temperature (°C)	11.2	10.9	7.1	20.2	134	134	100

FIELD MEASUREMENTS (WET 2005)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Discharge (cfs)	80.82	2.44	0.00	538.00	12	12	100
Oxygen, Dissolved (mg/L)	8.52	8.60	2.90	23.10	29	29	100
pH	7.52	7.40	6.63	9.03	29	29	100
Specific Conductivity (µS)	491.5	197.0	92.0	2330.0	29	29	100
Temperature (°C)	9.8	9.2	6.6	15.2	29	29	100

FIELD MEASUREMENTS (IRRIGATION 2005)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Discharge (cfs)	31.57	14.01	0.00	238.00	77	77	100
Oxygen, Dissolved (mg/L)	6.91	7.00	0.60	16.20	123	123	100
pH	7.36	7.31	5.78	9.45	122	122	100
Specific Conductivity (µS)	212.2	173.2	24.5	1049.0	122	122	100
Temperature (°C)	21.8	21.8	13.7	32.4	122	122	100
FIELD MEASUREMENTS (NOT APPLICABLE 2005)	**Mean**	**Median**	**Minimum**	**Maximum**	**Count**	**Count of Detection**	**% Frequency**
Oxygen, Dissolved (mg/L)	7.26	7.00	6.20	8.90	5	5	100
pH	7.79	7.86	7.12	8.51	5	5	100
Specific Conductivity (µS)	426.56	421.00	197.80	607.00	5	5	100
Temperature (°C)	18.6	19.0	16.6	19.7	5	5	100

FIELD MEASUREMENTS (DORMANT 2006)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Oxygen, Dissolved (mg/L)	9.14	9.23	2.27	11.94	38	38	100
pH	7.67	7.63	4.48	8.80	38	38	100
Specific Conductivity (µS)	388.7	258.0	74.0	1666.0	38	38	100
Temperature (°C)	11.0	10.5	7.7	18.4	38	38	100

FIELD MEASUREMENTS (IRRIGATION 2007)	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Oxygen, Dissolved (mg/L)	7.46	7.75	1.67	11.50	120	120	100
pH	7.46	7.41	6.52	8.65	120	120	100
Specific Conductivity (µS)	243.6	198.6	44.8	836.0	118	118	100
Temperature (°C)	20.5	20.9	8.1	25.6	119	119	100

"Count of Detection" for Discharge includes "no flow" events (0 cfs) as well as actual numerical results

4.2. INORGANICS AND TRIHALOMETHANES

Table 4. Inorganics and Trihalomethanes summary table

INORGANICS (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.25	0.09	0.04	0.25	541	315	58
Bromodichloromethane (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Bromoform (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Chloroform (µg/L)	0.16	0.10	0.09	0.30	70	3	4
Color (color units)	27.57	16.00	1.90	200.00	541	519	96
Dibromochloromethane (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Dissolved Solids (mg/L)	219.64	129.00	20.00	1540.00	541	541	100
Hardness as CaCO3 (mg/L)	115.20	79.70	8.00	1250.00	541	541	100
Nitrate + Nitrite as N, mg/L	0.90	0.22	0.01	18.20	540	473	88
Nitrite as N (mg/L)	0.04	0.02	0.00	0.38	541	320	59
OrthoPhosphate as P (mg/L)	0.13	0.05	0.01	4.78	541	538	99
Total Organic Carbon (mg/L)	5.70	3.60	0.60	47.46	524	518	99
Turbidity (NTU)	32.45	32.45	32.45	32.45	538	538	100

INORGANICS (IRRIGATION 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.21	0.08	0.04	0.04	123	72	59
Bromodichloromethane (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Bromoform (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Chloroform (µg/L)	0.16	0.10	0.09	0.30	70	3	4
Color (color units)	22.92	16.20	2.60	113.00	123	123	100
Dibromochloromethane (µg/L)	0.00	0.00	0.00	0.00	70	0	0
Dissolved Solids (mg/L)	239.41	164.00	20.00	902.00	123	123	100
Hardness as CaCO3 (mg/L)	117.63	86.50	8.00	390.00	123	123	100
Nitrate + Nitrite as N, mg/L	0.91	0.15	0.01	8.34	123	102	83
Nitrite as N (mg/L)	0.05	0.04	0.01	0.33	123	64	52
OrthoPhosphate as P (mg/L)	0.14	0.07	0.01	0.77	123	122	99
Total Organic Carbon (mg/L)	11.05	7.96	0.64	47.46	117	113	97
Turbidity (NTU)	19.43	9.20	0.65	155.00	122	122	100

INORGANICS (DORMANT 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.48	0.13	0.04	12.30	120	73	61
Color (color units)	48.18	26.00	2.00	200.00	120	117	98
Dissolved Solids (mg/L)	284.63	134.00	45.00	1540.00	120	120	100
Hardness as CaCO3 (mg/L)	151.20	79.50	19.40	936.00	120	120	100
Nitrate + Nitrite as N, mg/L	0.68	0.39	0.01	6.20	120	118	98
Nitrite as N (mg/L)	0.03	0.02	0.01	0.17	120	79	66
OrthoPhosphate as P (mg/L)	0.17	0.09	0.01	4.78	120	119	99
Total Organic Carbon (mg/L)	4.43	3.67	0.60	19.73	117	117	100
Turbidity (NTU)	40.03	23.50	1.00	250.00	120	120	100

INORGANICS (WET 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.33	0.18	0.04	1.02	28	13	46
Color (color units)	38.11	19.00	4.00	160.00	27	27	100
Dissolved Solids (mg/L)	344.71	167.00	66.00	1390.00	28	28	100
Hardness as CaCO3 (mg/L)	220.20	85.85	40.80	1250.00	28	28	100
Nitrate + Nitrite as N, mg/L	0.54	0.42	0.01	2.34	28	25	89
Nitrite as N (mg/L)	0.02	0.01	0.01	0.07	28	18	64
OrthoPhosphate as P (mg/L)	0.10	0.08	0.01	0.51	28	28	100
Total Organic Carbon (mg/L)	4.45	4.11	1.68	8.30	26	26	100
Turbidity (NTU)	20.09	12.00	0.80	85.00	27	27	100

INORGANICS (IRRIGATION 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.21	0.07	0.04	1.81	108	68	63
Color (color units)	24.21	15.00	1.90	170.00	108	104	96
Dissolved Solids (mg/L)	144.65	102.50	21.00	688.00	108	108	100
Hardness as CaCO3 (mg/L)	73.10	67.65	8.50	238.00	108	108	100
Nitrate + Nitrite as N, mg/L	0.66	0.08	0.01	7.46	108	90	83
Nitrite as N (mg/L)	0.05	0.03	0.01	0.22	108	42	39
OrthoPhosphate as P (mg/L)	0.16	0.05	0.01	1.76	108	108	100
Total Organic Carbon (mg/L)	4.69	3.02	0.80	23.09	107	105	98
Turbidity (NTU)	22.44	8.10	0.80	390.00	108	108	100

INORGANICS (NOT APPLICABLE 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.08	0.09	0.05	0.11	5	5	100
Color (color units)	23.60	23.00	14.00	32.00	5	5	100
Dissolved Solids (mg/L)	278.80	266.00	130.00	414.00	5	5	100
Hardness as CaCO3 (mg/L)	149.88	163.00	74.40	192.00	5	5	100
Nitrate + Nitrite as N, mg/L	0.09	0.08	0.08	0.11	4	3	75
Nitrite as N (mg/L)	0.01	0.01	0.01	0.01	5	1	20
OrthoPhosphate as P (mg/L)	0.09	0.11	0.03	0.14	5	5	100
Turbidity (NTU)	12.90	12.00	6.50	19.00	5	5	100

INORGANICS (DORMANT 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.11	0.09	0.04	0.42	37	30	81
Color (color units)	21.82	18.50	6.00	48.00	38	38	100
Dissolved Solids (mg/L)	300.43	152.00	63.00	1480.00	37	37	100
Hardness as CaCO3 (mg/L)	137.85	119.00	28.20	566.00	37	37	100
Nitrate + Nitrite as N, mg/L	0.80	0.28	0.02	13.20	37	37	100
Nitrite as N (mg/L)	0.03	0.01	0.00	0.38	37	34	92
OrthoPhosphate as P (mg/L)	0.14	0.05	0.01	1.47	37	37	100
Total Organic Carbon (mg/L)	5.56	4.18	1.47	18.20	37	37	100
Turbidity (NTU)	160.26	39.50	2.60	1300.00	36	36	100

INORGANICS (IRRIGATION 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Ammonia as N (mg/L)	0.14	0.07	0.04	1.01	120	54	45
Color (color units)	12.93	11.00	5.00	80.00	120	105	88
Dissolved Solids (mg/L)	145.33	115.00	30.00	553.00	120	120	100
Hardness as CaCO3 (mg/L)	81.65	78.95	13.30	263.00	120	120	100
Nitrate + Nitrite as N, mg/L	1.54	0.25	0.01	18.20	120	98	82
Nitrite as N (mg/L)	0.02	0.01	0.02	0.38	120	82	68
OrthoPhosphate as P (mg/L)	0.08	0.03	0.01	1.70	120	119	99
Total Organic Carbon (mg/L)	3.12	2.50	0.90	24.40	120	120	100
Turbidity (NTU)	12.36	6.30	0.90	192.00	120	120	100

4.3. ACARICIDES

Table 5. Acaricides summary table

Acaricides (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Propargite (µg/L)	0.742	0.250	0.020	2.000	111	9	8

Acaricides (IRRIGATION 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Propargite (µg/L)	0.742	0.250	0.020	2.000	111	9	8

4.4. CARBAMATE PESTICIDES

Table 6. Carbamate pesticides summary table

CARBAMATES (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Aldicarb (µg/L)	5.043	5.000	0.410	9.720	402	3	0.7
Carbaryl (µg/L)	0.594	0.250	0.061	3.600	402	9	2.2
Carbofuran (µg/L)	0.071	0.024	0.015	0.316	403	9	2.2
Methomyl (µg/L)	0.510	0.360	0.015	2.250	403	15	3.7

CARBAMATES (Irrigation 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Aldicarb (µg/L)	7.360	7.360	5.000	9.720	130	2	1.5
Carbaryl (µg/L)	0.223	0.245	0.146	0.256	130	4	3.1
Carbofuran (µg/L)	0.080	0.015	0.015	0.316	130	6	4.6
Methomyl (µg/L)	0.595	0.169	0.015	2.250	130	2	1.7
CARBAMATES (Not Applicable 2004)							
Analyte	**Mean**	**Median**	**Minimum**	**Maximum**	**Count**	**Count of Detection**	**% Frequency**
Aldicarb (µg/L)	0.41	0.41	0.41	0.41	4	1	25.0
Methomyl (µg/L)	0.695	0.695	0.695	0.695	4	1	25.0

CARBAMATES (Irrigation 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Carbaryl (µg/L)	1.098	0.315	0.160	3.600	111	4	3.6
Methomyl (µg/L)	0.442	0.370	0.054	1.000	111	6	5.4

CARBAMATES (Irrigation 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Carbaryl (µg/L)	0.061	0.061	0.061	0.061	119	1	0.8
Carbofuran (µg/L)	0.053	0.024	0.024	0.111	120	3	2.5
Methomyl (µg/L)	0.143	0.143	0.143	0.143	120	1	0.8

4.5. FUNGICIDES

Table 7. Fungicides summary table

Fungicides (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Captan (µg/L)	0.00	0.00	0.00	0.00	382	0	0

4.6. HERBICIDES

Table 8. Herbicides summary table

Herbicides (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.082	0.035	0.020	0.740	111	63	57
Cyanazine (µg/L)	0.113	0.079	0.028	0.264	484	4	1
Diuron (µg/L)	0.410	0.153	0.004	5.600	403	71	18
Linuron (µg/L)	0.397	0.376	0.014	0.824	403	4	1
Metolachlor (µg/L)	0.496	0.217	0.012	3.370	111	27	24
Molinate (µg/L)	0.151	0.040	0.035	1.260	484	31	6
Norflurazon (µg/L)	0.162	0.166	0.095	0.256	111	7	6
Oxyfluorfen (µg/L)	0.131	0.080	0.022	0.447	111	8	7
Prometryn (µg/L)	0.039	0.039	0.039	0.039	111	1	1
Propanil (µg/L)	3.726	0.622	0.116	23.170	111	7	6
Propazine (µg/L)	0.037	0.026	0.013	0.115	111	9	8
Prowl (µg/L)	0.179	0.179	0.034	0.325	111	2	2
Simazine (µg/L)	0.785	0.278	0.024	5.400	484	93	19
Thiobencarb (µg/L)	5.474	0.122	0.035	150.000	484	29	6
Trifluralin (µg/L)	0.186	0.089	0.010	0.643	111	20	18

Herbicides (Irrigation 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.037	0.035	0.035	0.052	130	27	21
Diuron (µg/L)	0.198	0.120	0.004	0.950	130	29	22
Molinate (µg/L)	0.073	0.035	0.035	0.550	130	17	13
Simazine (µg/L)	0.043	0.035	0.035	0.078	130	12	9
Thiobencarb (µg/L)	0.077	0.035	0.035	0.250	130	16	12

Herbicides (Not Applicable 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.170	0.087	0.035	0.470	4	4	100

Herbicides (Dormant 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.146	0.090	0.020	0.740	65	21	32
Cyanazine (µg/L)	0.113	0.079	0.028	0.264	65	4	6
Simazine (µg/L)	1.230	0.636	0.030	5.400	65	44	68
Thiobencarb (µg/L)	0.389	0.389	0.280	0.498	65	2	3

Herbicides (Wet 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.038	0.038	0.038	0.038	10	1	10
Simazine (µg/L)	1.608	1.530	0.606	3.000	10	6	60

Herbicides (Irrigation 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.062	0.072	0.035	0.080	111	3	3
Diuron (µg/L)	0.196	0.080	0.030	0.692	111	11	10
Metolachlor (µg/L)	0.496	0.217	0.012	3.370	111	27	24
Molinate (µg/L)	0.176	0.060	0.040	0.760	111	9	8
Norflurazon (µg/L)	0.162	0.166	0.095	0.256	111	7	6
Oxyfluorfen (µg/L)	0.131	0.080	0.022	0.447	111	8	7
Prometryn (µg/L)	0.039	0.039	0.039	0.039	111	1	1
Propanil (µg/L)	3.726	0.622	0.116	23.170	111	7	6
Propazine (µg/L)	0.037	0.026	0.013	0.115	111	9	8
Prowl (µg/L)	0.179	0.179	0.034	0.325	111	2	2
Simazine (µg/L)	0.539	0.121	0.024	2.500	111	6	5
Thiobencarb (µg/L)	26.010	1.330	0.300	150.000	111	6	5
Trifluralin (µg/L)	0.186	0.089	0.010	0.643	111	20	18

Herbicides (Dormant 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.027	0.024	0.020	0.045	38	5	13
Diuron (µg/L)	0.896	0.410	0.011	5.600	38	21	55
Linuron (µg/L)	0.687	0.687	0.550	0.824	38	2	5
Simazine (µg/L)	0.266	0.130	0.024	1.320	38	19	50

Herbicides (Irrigation 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Atrazine (µg/L)	0.041	0.041	0.029	0.052	121	2	2
Diuron (µg/L)	0.239	0.157	0.038	0.864	120	10	8
Linuron (µg/L)	0.108	0.108	0.014	0.201	120	2	2
Molinate (µg/L)	0.371	0.170	0.100	1.260	121	5	4
Simazine (µg/L)	0.070	0.040	0.034	0.225	121	6	5
Thiobencarb (µg/L)	0.138	0.137	0.105	0.188	121	5	4

4.7. ORGANOCHLORINE PESTICIDES

Table 9. Orangochlorine pesticides summary table

Organochlorines (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDD(o,p') (µg/L)	0.005	0.004	0.002	0.010	561	14	2.5
DDD(p,p') (µg/L)	0.007	0.007	0.003	0.010	561	4	0.7
DDE(o,p') (µg/L)	0.003	0.003	0.002	0.005	561	3	0.5
DDE(p,p') (µg/L)	0.012	0.005	0.001	0.060	561	25	4.5
DDT(o,p') (µg/L)	0.009	0.010	0.005	0.011	561	3	0.5
DDT(p,p') (µg/L)	0.017	0.009	0.004	0.051	561	8	1.4
Dieldrin (µg/L)	0.003	0.003	0.001	0.010	561	24	4.3

Organochlorines (Irrigation 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDD(o,p')(µg/L)	0.004	0.003	0.003	0.005	130	3	2.3
DDD(p,p')(µg/L)	0.007	0.007	0.003	0.010	130	4	3.1
DDE(o,p')(µg/L)	0.004	0.004	0.003	0.005	130	2	1.5
DDE(p,p')(µg/L)	0.014	0.006	0.003	0.060	130	13	10.0
DDT(o,p')(µg/L)	0.012	0.010	0.004	0.027	130	3	2.3
DDT(p,p')(µg/L)	0.015	0.010	0.004	0.027	130	5	3.8
Dieldrin(µg/L)	0.006	0.006	0.002	0.010	130	6	4.6

Organochlorines (Not Applicable 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDE(p,p') (µg/L)	0.016	0.016	0.016	0.016	3	1	33.3
DDT(p,p') (µg/L)	0.005	0.005	0.005	0.005	3	1	33.3
Dieldrin (µg/L)	0.002	0.002	0.002	0.002	3	1	33.3

Organochlorines (Dormant 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDE(p,p') (µg/L)	0.004	0.004	0.002	0.007	127	6	4.7
DDT(p,p') (µg/L)	0.005	0.005	0.005	0.005	127	1	0.8

Organochlorines (Wet 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDD(o,p') (µg/L)	0.002	0.002	0.002	0.002	30	1	3.3

Organochlorines (Irrigation 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDD(o,p') (µg/L)	0.005	0.005	0.005	0.005	111	1	0.9

Organochlorines (Dormant 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDE(p,p') (µg/L)	0.030	0.030	0.009	0.050	38	2	5.3
DDT(p,p') (µg/L)	0.051	0.051	0.051	0.051	38	1	2.6

Organochlorines (Irrigation 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
DDD(o,p') (µg/L)	0.004	0.002	0.002	0.008	121	3	2.5
DDE(p,p') (µg/L)	0.004	0.005	0.001	0.005	121	3	2.5
Dieldrin (µg/L)	0.003	0.002	0.001	0.006	121	17	14.0

4.8. ORGANOPHOSPHATE PESTICIDES

Table 10. Organophosphate pesticides summary table

Organophosphates (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Azinphos methyl (µg/L)	0.126	0.055	0.040	0.330	564	5	0.9
Chlorpyrifos (µg/L)	0.054	0.025	0.004	2.200	564	162	28.7
Diazinon (µg/L)	0.137	0.042	0.004	2.000	565	182	32.2
Dimethoate (µg/L)	0.307	0.087	0.030	1.840	565	60	10.6
Disulfoton (µg/L)	0.071	0.030	0.010	0.418	565	55	9.7
Malathion (µg/L)	1.885	0.050	0.032	46.000	565	26	4.6
Methidathion (µg/L)	0.076	0.048	0.032	0.272	565	29	5.1
Parathion, Methyl (µg/L)	0.041	0.030	0.014	0.188	565	17	3.0
Phorate (µg/L)	0.125	0.125	0.125	0.125	565	2	0.4
Phosmet (µg/L)	0.398	0.398	0.398	0.398	565	1	0.2

Organophosphates (Irrigation 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Azinphos methyl (µg/L)	0.040	0.040	0.040	0.040	130	2	1.5
Chlorpyrifos (µg/L)	0.046	0.024	0.004	0.400	130	53	40.8
Diazinon (µg/L)	0.025	0.011	0.004	0.250	130	28	21.5
Dimethoate (µg/L)	0.216	0.087	0.040	1.200	130	33	25.4
Disulfoton (µg/L)	0.061	0.030	0.030	0.260	130	19	14.6
Malathion (µg/L)	0.072	0.040	0.040	0.182	130	8	6.2
Parathion, Methyl (µg/L)	0.031	0.030	0.030	0.045	130	11	8.5
Phorate (µg/L)	0.125	0.125	0.125	0.125	130	2	1.5

Organophosphates (Not Applicable 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Chlorpyrifos (µg/L)	0.014	0.014	0.012	0.015	4	2	50.0
Diazinon (µg/L)	0.013	0.013	0.006	0.020	4	2	50.0
Dimethoate (µg/L)	0.085	0.085	0.084	0.085	4	2	50.0
Disulfoton (µg/L)	0.030	0.030	0.030	0.030	4	3	75.0

Organophosphates (Dormant 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Chlorpyrifos (µg/L)	0.025	0.024	0.006	0.072	127	58	45.7
Diazinon (µg/L)	0.195	0.083	0.012	1.610	127	94	74.0
Dimethoate (µg/L)	0.070	0.070	0.053	0.087	127	2	1.6
Disulfoton (µg/L)	0.101	0.031	0.011	0.418	127	20	15.7
Malathion (µg/L)	0.062	0.053	0.033	0.101	127	10	7.9
Methidathion (µg/L)	0.076	0.047	0.032	0.272	127	28	22.0

Organophosphates (Wet 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Chlorpyrifos (µg/L)	0.023	0.025	0.011	0.030	30	6	20.0
Diazinon (µg/L)	0.135	0.084	0.040	0.430	30	17	56.7
Disulfoton (µg/L)	0.111	0.130	0.062	0.140	30	3	10.0

Organophosphates (Irrigation 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Azinphos methyl (µg/L)	0.248	0.248	0.165	0.330	111	2	1.8
Chlorpyrifos (µg/L)	0.151	0.039	0.018	2.200	111	28	25.2
Diazinon (µg/L)	0.519	0.030	0.015	2.000	111	4	3.6
Dimethoate (µg/L)	0.369	0.063	0.031	1.200	111	15	13.5
Disulfoton (µg/L)	0.015	0.015	0.013	0.017	111	2	1.8
Malathion (µg/L)	15.386	0.125	0.032	46.000	111	3	2.7
Parathion, Methyl (µg/L)	0.068	0.044	0.016	0.188	111	5	4.5

Organophosphates (Not Applicable 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Chlorpyrifos (µg/L)	0.046	0.046	0.046	0.046	6	1	16.7
Diazinon (µg/L)	0.056	0.056	0.004	0.107	6	2	33.3

Organophosphates (Dormant 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Chlorpyrifos (µg/L)	0.047	0.028	0.007	0.138	38	7	18.4
Diazinon (µg/L)	0.037	0.022	0.004	0.162	38	31	81.6
Disulfoton (µg/L)	0.027	0.027	0.021	0.032	38	2	5.3
Malathion (µg/L)	0.063	0.063	0.037	0.090	38	2	5.3

Organophosphates (Irrigation 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Azinphos methyl (µg/L)	0.055	0.055	0.055	0.055	118	1	0.8
Chlorpyrifos (µg/L)	0.006	0.004	0.004	0.014	118	7	5.9
Diazinon (µg/L)	0.047	0.045	0.012	0.088	119	4	3.4
Dimethoate (µg/L)	0.677	0.527	0.030	1.840	119	8	6.7
Disulfoton (µg/L)	0.039	0.042	0.010	0.063	119	6	5.0
Malathion (µg/L)	0.508	0.093	0.050	1.380	119	3	2.5
Methidathion (µg/L)	0.071	0.071	0.071	0.071	119	1	0.8
Parathion, Methyl (µg/L)	0.014	0.014	0.014	0.014	119	1	0.8
Phosmet (µg/L)	0.398	0.398	0.398	0.398	119	1	0.8

4.9. PYRETHROID PESTICIDES

Table 11. Pyrethroid pesticides summary table

PYRETHROIDS (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Bifenthrin (µg/L)	0.020	0.016	0.012	0.037	562	4	0.7
Cyhalothrin, lambda, total (µg/L)	0.004	0.003	0.001	0.007	141	7	5.0
Cypermethrin, total (µg/L)	0.018	0.018	0.005	0.030	255	2	0.8
Cypermethrin-1 (µg/L)	0.234	0.234	0.234	0.234	307	1	0.3
Cypermethrin-2 (µg/L)	0.199	0.199	0.199	0.199	307	1	0.3
Cypermethrin-3 (µg/L)	0.215	0.215	0.215	0.215	307	1	0.3
Cypermethrin-4 (µg/L)	0.160	0.160	0.160	0.160	307	1	0.3
Esfenvalerate/Fenvalerate, total (µg/L)	0.126	0.004	0.002	0.450	255	5	2.0
Esfenvalerate/Fenvalerate-1 (µg/L)	0.009	0.009	0.009	0.009	307	1	0.3
Permethrin, total (µg/L)	0.006	0.006	0.006	0.006	255	1	0.4
Permethrin-1 (µg/L)	0.100	0.072	0.012	0.216	307	3	1.0
Permethrin-2 (µg/L)	0.096	0.024	0.016	0.390	307	6	2.0

PYRETHROIDS (Irrigation 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Bifenthrin (µg/L)	0.015	0.015	0.012	0.018	131	2	1.5
Cypermethrin, total (µg/L)	0.030	0.030	0.030	0.030	131	1	0.8

PYRETHROIDS (Not Applicable 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Esfenvalerate/Fenvalerate, total (µg/L)	0.450	0.450	0.450	0.450	3	1	33.3

PYRETHROIDS (Irrigation 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Bifenthrin (µg/L)	0.037	0.037	0.037	0.037	111	1	0.9
Esfenvalerate/Fenvalerate-1 (µg/L)	0.009	0.009	0.009	0.009	111	1	0.9

PYRETHROIDS (Dormant 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Cypermethrin-1 (µg/L)	0.234	0.234	0.234	0.234	38	1	2.6
Cypermethrin-2 (µg/L)	0.199	0.199	0.199	0.199	38	1	2.6
Cypermethrin-3 (µg/L)	0.215	0.215	0.215	0.215	38	1	2.6
Cypermethrin-4 (µg/L)	0.160	0.160	0.160	0.160	38	1	2.6
Permethrin-1 (µg/L)	0.100	0.072	0.012	0.216	38	3	7.9
Permethrin-2 (µg/L)	0.096	0.024	0.016	0.390	38	6	15.8

PYRETHROIDS (Irrigation 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Bifenthrin (µg/L)	0.014	0.014	0.014	0.014	121	1	0.8
Cyhalothrin, lambda, total (µg/L)	0.004	0.003	0.001	0.007	121	7	5.8
Cypermethrin, total (µg/L)	0.005	0.005	0.005	0.005	121	1	0.8
Esfenvalerate/Fenvalerate, total (µg/L)	0.045	0.004	0.002	0.172	121	4	3.3
Permethrin, total (µg/L)	0.006	0.006	0.006	0.006	121	1	0.8

4.10. WATER COLUMN TOXICITY

Table 12. Water column toxicity summary table

Toxicity (2004-2007)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	9.393	0.000	29	425	6.8
Pimephales promelas, Survival (%)	82.041	20.000	15	424	3.5
Selenastrum capricornutum, Total Cell Count	64.046	3.000	102	381	26.8

Toxicity (IS 2004)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	10.833	0.000	6	123	4.9
Pimephales promelas, Survival (%)	84.210	84.210	1	123	0.8
Selenastrum capricornutum, Total Cell Count	69.219	43.140	62	123	50.4

Toxicity (Dormant 2005)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	14.567	0.000	6	28	21.4
Selenastrum capricornutum, Total Cell Count	37.333	6.800	12	28	42.9

Toxicity (Irrigation 2005)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	0.385	0.000	13	107	12.1
Pimephales promelas, Survival (%)	90.000	85.000	4	107	3.7
Selenastrum capricornutum, Total Cell Count	3.000	3.000	1	107	0.9

Toxicity (Not Applicable 2005)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Selenastrum capricornutum, Total Cell Count	34.390	21.910	5	5	100.0

Toxicity (Dormant 2006)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	0.000	0.000	1	37	2.7
Pimephales promelas, Survival (%)	83.150	79.500	2	37	5.4
Selenastrum capricornutum, Total Cell Count	55.856	22.620	7	37	18.9

Toxicity (Irrigation 2007)

Species	Mean (%Control)	Magnitude (%Control)	Count of Toxic Samples	Total Count	Frequency (%)
Ceriodaphnia dubia, Survival (%)	38.333	0.000	3	120	2.5
Pimephales promelas, Survival (%)	77.513	20.000	8	120	6.7
Selenastrum capricornutum, Total Cell Count	81.817	31.286	15	77	19.5

Mean = Mean survival or % growth based on control for all toxic samples

Magnitude = Greatest survival (% control) for *Ceriodaphnia dubia* and *Pimephales promelas or* least % difference in growth based on control for *Selenastrum capricornutum* for all toxic samples

4.11. METALS

Table 13. Metals summary table

METALS (2004-2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	2.80	1.62	0.13	54.00	448	445	99.3
Boron (µg/L)	145.23	14.60	2.70	1690.00	449	447	99.6
Cadmium (µg/L)	0.06	2.47	0.01	2.01	448	320	71.4
Copper (µg/L)	16.85	2.14	0.54	4403.00	435	435	100.0
Lead (µg/L)	1.13	1.84	0.04	41.80	445	445	100.0
Nickel (µg/L)	8.81	2.01	0.02	1245.00	441	439	99.5
Phosphorus as P (µg/L)	212.25	2.00	1.47	2326.00	445	442	99.3
Selenium (µg/L)	1.16	1.70	0.10	6.88	446	319	71.5
Zinc (µg/L)	9.36	1.79	0.14	328.00	448	445	99.3

METALS (IRRIGATION 2004)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	3.63	1.84	0.49	23.8	123	123	100.0
Boron (µg/L)	207.80	62.70	4.8	1690	121	107	88.4
Cadmium (µg/L)	0.04	0.02	0.005	0.9	123	91	74.0
Copper (µg/L)	4.12	2.96	0.64	17.9	119	119	100.0
Lead (µg/L)	0.71	0.38	0.04	4.3	123	123	100.0
Nickel (µg/L)	4.04	2.16	0.02	22.8	121	121	100.0
Phosphorus as P (µg/L)	213.14	96.80	1.47	1902	123	123	100.0
Selenium (µg/L)	1.05	0.58	0.118	3.48	123	95	77.2
Zinc (µg/L)	4.59	2.75	0.27	25.8	123	123	100.0

METALS (DORMANT 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	2.90	1.98	0.99	10.80	52	52	100.0
Boron (µg/L)	287.25	173.00	11.10	1,580.00	52	52	100.0
Cadmium (µg/L)	0.06	0.05	0.01	0.14	52	51	98.1
Copper (µg/L)	9.62	9.24	3.65	24.90	49	49	100.0
Lead (µg/L)	1.82	1.48	0.15	4.85	50	50	100.0
Nickel (µg/L)	9.59	8.48	0.50	51.50	51	51	100.0
Phosphorus as P (µg/L)	344.08	245.00	60.60	1,417.00	51	51	100.0
Selenium (µg/L)	1.90	1.17	0.16	6.88	51	51	100.0
Zinc (µg/L)	22.04	18.25	2.43	59.60	52	52	100.0

METALS (WET 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	3.41	2.17	1.25	11.20	13	13	100.0
Boron (µg/L)	372.27	233.50	15.90	1210.00	12	12	100.0
Cadmium (µg/L)	0.04	0.04	0.01	0.13	13	13	100.0
Copper (µg/L)	5.99	5.25	2.18	13.30	12	12	100.0
Lead (µg/L)	0.96	0.69	0.26	2.10	13	13	100.0
Nickel (µg/L)	5.40	5.59	1.53	10.80	12	12	100.0
Phosphorus as P (µg/L)	327.45	186.50	78.60	1145.00	12	12	100.0
Selenium (µg/L)	2.04	1.53	0.20	5.08	12	12	100.0
Zinc (µg/L)	10.38	7.81	1.43	28.60	13	13	100.0

METALS (IRRIGATION 2005)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	1.91	1.06	0.13	7.27	108	108	100.0
Boron (µg/L)	67.15	22.95	2.70	538.00	108	108	100.0
Cadmium (µg/L)	0.04	0.02	0.01	0.32	108	56	51.9
Copper (µg/L)	6.18	2.99	0.54	115.00	105	105	100.0
Lead (µg/L)	0.97	0.43	0.04	22.20	107	107	100.0
Nickel (µg/L)	5.13	1.18	0.10	173.00	106	106	100.0
Phosphorus as P (µg/L)	227.40	88.60	2.23	2065.00	107	105	98.1
Selenium (µg/L)	1.01	0.64	0.10	5.63	108	94	87.0
Zinc (µg/L)	7.56	2.32	0.14	183.00	108	105	97.2

METALS (DORMANT 2006)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	5.99	2.18	0.18	54.00	37	35	94.6
Boron (µg/L)	150.03	32.50	4.70	814.00	36	36	100.0
Cadmium (µg/L)	0.14	0.04	0.01	2.01	37	35	94.6
Copper (µg/L)	13.76	6.61	0.69	106.00	35	35	100.0
Lead (µg/L)	3.93	1.04	0.07	41.80	37	37	100.0
Nickel (µg/L)	54.03	5.20	0.40	1245.00	36	36	100.0
Phosphorus as P (µg/L)	306.99	140.00	5.48	2326.00	37	37	100.0
Selenium (µg/L)	1.48	1.13	0.43	5.45	37	22	59.5
Zinc (µg/L)	27.66	7.29	0.38	328.00	37	37	100.0

METALS (IRRIGATION 2007)

Analyte	Mean	Median	Minimum	Maximum	Count	Count of Detection	% Frequency
Arsenic (µg/L)	1.66	1.55	0.30	5.15	115	114	99.1
Boron (µg/L)	67.77	33.35	3.76	373.00	120	120	100.0
Cadmium (µg/L)	0.07	0.03	0.01	0.23	115	74	64.3
Copper (µg/L)	44.93	2.71	0.69	4403.00	115	115	100.0
Lead (µg/L)	0.54	0.39	0.06	7.65	115	115	100.0
Nickel (µg/L)	2.99	1.31	0.09	66.70	115	113	98.3
Phosphorus as P (µg/L)	95.50	2.70	2.50	1540.00	115	114	99.1
Selenium (µg/L)	0.50	0.39	0.20	1.36	115	45	39.1
Zinc (µg/L)	4.38	2.36	0.25	65.90	115	115	100.0

Total Metal and Irrigation 2007 results do not reflect five samples collected on 11/28/07. A complete list of results is on the CD.

APPENDIX 5: Precision and Accuracy / QC data summary tables

5.1. CHEMISTRY

Precision is assessed by calculating the relative percent difference (RPD) between a sample and a duplicate sample. This can be assessed by laboratory control spikes (LCS) and laboratory control spike duplicates (LCSD), matrix spikes (MS) and matrix spike duplicates (MSD), and/or an environmental sample that is split in the laboratory and run as two samples. Accuracy is assessed by spiking water with a known concentration and calculating the percent recovery (PR) of the spiked analyte(s). Samples used to assess accuracy include LCS samples, MS samples and/or certified reference materials (CRMs). Matrix spike samples are also used to assess matrix interference which could result in low recoveries. Contamination is assessed with laboratory blanks which are laboratory water that undergoes the same process (preparation, extraction and analysis) as the other samples in the batch.

To assess contamination in the field, field blanks are collected and analyzed (except for sediment). Field duplicates are collected to assess field precision and can also provide information on the homogeneity of the water column or sediment. Both laboratory and field quality control (QC) analysis are assessed in this section for the entire Ag Waiver Phase II project.

For constituents such as color, turbidity, total dissolved solids, and metals the values in the environmental sample may exceed the amount that the detector can detect and therefore requires a dilution. The result reported is the amount found in the diluted sample multiplied by the dilution factor to represent the amount of the analyte present in the original sample. The dilution factor is recorded and the reporting limit (RL) is generally increased by multiplying the RL for that analyte by the dilution factor. There are times that the RL is increased higher than this value based on method requirements. Therefore, for each dilution that occurs, there is a corresponding increase in the limit of quantification.

All results are reported in table format on an attached CD. Each result is flagged if it does not meet data quality objectives (acceptability criteria) using SWAMP codes and can also be found in the SWAMP comparable database posted on the UC Davis Center for Environmental Data Exchange Network (CEDEN) ftp site (ftp://aeal-FTP.ucdavis.edu). A review of the number of samples analyzed for both water and sediment analysis and the percentage per analyte that meets acceptability criteria are listed in the tables following this section. A brief overview is listed below to assess overall precision and accuracy per analyte (all pesticides are grouped and discussed together).

- Ammonia as N: Eighty-nine percent of field duplicates and 86% of field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the MDL. Certified reference materials, matrix spikes and matrix spike duplicates were within acceptability criteria for all batches meeting requirements of accuracy and precision.

- Color: Ninety-six percent of all field blanks and 89% of field duplicates met acceptability criteria. Certified reference materials and lab blanks were run with each color batch and all met laboratory QC criteria. Lab duplicates were recorded by the laboratory to access precision and 100% had RPDs less than 25.

- Dissolved Solids (TDS): One hundred percent of field blanks and field duplicates met acceptance criteria. Lab blanks were run with every batch and were less than the RL for 100% of samples. Laboratory duplicates were analyzed with each batch and met acceptance criteria for 100% of samples. Lab control spikes and matrix spikes cannot be performed for TDS.

- Hardness: One hundred percent of field blanks were less than the MDL. One hundred percent of field duplicates had RPDs less than 25. Certified reference materials, matrix spikes, matrix spike duplicates, lab duplicates and lab blanks were run with every batch and all met precision and accuracy requirements.

- Nitrate + Nitrite as N: One hundred percent of field duplicates and field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the MDL for all samples. Lab control spikes and laboratory duplicates were within acceptability criteria for all batches. Matrix spikes were performed in each batch with 100% meeting acceptability requirements and matrix spike duplicates met 100% of acceptability requirement for precision.

- Nitrite as N: One hundred percent of field duplicates and field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the MDL. Lab control spikes, matrix spikes and matrix spike duplicates were within acceptability criteria for all batches meeting requirements of accuracy and precision.

- Orthophosphate as P: One hundred percent of field duplicates and 100% of field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the MDL. Certified reference materials, matrix spikes and matrix spike duplicates were within acceptability criteria for all batches meeting requirements of accuracy and precision.

- Phosphorus as P: Ninety-six percent of field duplicates and 75% of field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the RL. Certified reference materials, laboratory duplicates, matrix spikes and matrix spike duplicates were within acceptability criteria for all batches meeting requirements of accuracy and precision.

- Turbidity: One hundred percent of field duplicates and 81% of field blanks met acceptance criteria. Lab blanks were run with every batch and were less than the MDL. Laboratory duplicates were analyzed with each batch and 99% met acceptance criteria. Certified reference materials were run with every batch and met acceptability criteria for 100% of samples. Matrix spikes cannot be performed for turbidity.

- Total Organic Carbon (TOC): For water samples, 78% of field duplicates and 63% of field blanks collected met acceptance criteria. Lab blanks were run with every batch and were less than the MDL. Lab control spikes laboratory duplicates were within acceptability criteria for 100% and 83% of all batches. Matrix spikes were performed in

each batch with 100% meeting acceptability requirements and matrix spike duplicates met 95% of acceptability requirement for precision.

For sediment samples, 80% of field duplicates collected met acceptance criteria. Lab blanks were run with every batch, with 0% less than the MDL. One hundred percent of certified reference materials were within acceptability criteria for accuracy. All laboratory duplicates were within acceptability criteria for precision. Matrix spikes were not performed for sediment TOC.

- Total Metals: The tables and analysis do not include the results of five samples (CS34, LSAC29, CS31, D04, and NSJ31) collected on 11/28/07 that were provided after the draft report was created. Overall eighty-six percent of the metals analyzed in the field duplicates met acceptability criteria. Specifically, 95% of field duplicates for copper, 93% for arsenic, boron, lead, nickel and zinc, 91% for cadmium and 80% for selenium has RPDs less than 25. Overall 49% of field blanks met acceptance criteria (< MDL) in water samples. Only arsenic met field blank criteria for all field blanks collected. In 2007 it was determined that the water used for the field blanks was not appropriate for trace level metals and had detectable levels of metals prior to being used by this project. Laboratory blanks were run for each metals batch and 100% of samples met acceptability criteria. Laboratory control spikes were within acceptable recovery limits for 100% of samples run. Matrix spikes and matrix spike duplicates met acceptable criteria for 99% of the samples. To meet batch requirements a lab duplicate may also have been performed on a sample other than a matrix spike. Ninety-one percent of lab duplicates met acceptability criteria.

  For sediment data, laboratory blanks were run for each metals batch and 100% of samples met acceptability criteria. Laboratory control spikes were within acceptable recovery limits for 100% of samples run. Matrix spike and matrix spike duplicate recovery was within control limits for 100% of samples and met both precision and accuracy requirements. One hundred percent of lab duplicates met acceptability criteria.

- Pesticides: Pesticides have been broken up into seven groups: pyrethroids (EPA 8081BM and EPA 1660M), organochlorines (EPA 8081AM and EPA 8081BM), organophosphates (EPA 8141AM), carbamates (EPA 632M), fungicides (EPA 632M), acaricides (EPA 619M), and herbicides (EPA 619, EPA 619M, EPA 632M, WPCL Method 42). Sediment samples were analyzed for pyrethroids (EPA 8081AM). For water samples, 89% of field blanks and 99% of field duplicates collected met acceptability. For sediment samples, 88% of field duplicates met acceptability criteria. No field blanks were taken for sediment samples. Lab blanks were performed for each batch and met acceptability criteria for contamination for all analysis in both water and sediment samples. Matrix spikes and lab control spikes were performed for each batch to assess precision and accuracy as well as possible matrix interference. Either a matrix spike duplicate and/or a lab control spike duplicate were performed per batch to assess precision. As per method requirements, surrogates were run for each analysis. Surrogate recoveries were within specific acceptance criteria for 97% of both water and sediment samples. Laboratory control spikes and laboratory control spike duplicates were within

acceptability criteria for 89% of all analytes for water samples, and 95% of sediment samples. Ninety-five percent of laboratory control spike duplicates and 92% of matrix spike duplicates for analytes in water samples. Ninety-seven percent of matrix spike duplicates met acceptability criteria in sediment samples. All batches with laboratory QCs outside of acceptability criteria have been flagged. If a surrogate was outside of recovery, the surrogate was flagged as well as the associated analytes. Batches are approved by evaluating all measures of precision and accuracy such that although a single QC sample may be outside of acceptability criteria, the entire batch may be accepted due to other QCs performed within that batch meeting acceptability criteria.

Ninety-one percent of water samples and 92% of sediment samples met hold time acceptability requirements for chemistry analysis.

5.2. TOXICITY

- Water Column Toxicity: Five percent of all samples collected were field duplicates and were tested for *Ceriodaphnia, Selenastrum, and Pimephales*. For these three species RPDs for all field duplicates were within acceptability criteria (RPD < 25). 95% of tests met holding time requirements (<36 hrs), water quality requirements and control requirements (as listed in the EPA method guidelines). Field blanks were also collected and all met acceptability criteria of not being significantly different than the control.

- Sediment Toxicity: Eight field duplicates were collected during the 2004 and 2005 sampling seasons and all were within acceptability criteria. One hundred percent of the sediment samples had laboratory controls within acceptability criteria. Sixty-two percent of sediment samples met holding time criteria.

Table 14. Summary of field blank quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	<3x MDL or <RL	21	21	100.00
Carbamates	Carbaryl	EPA 632M	<3x MDL or <RL	21	21	100.00
Carbamates	Carbofuran	EPA 632M	<3x MDL or <RL	21	21	100.00
Carbamates	Methiocarb	EPA 632M	<3x MDL or <RL	21	21	100.00
Carbamates	Methomyl	EPA 632M	<3x MDL or <RL	21	21	100.00
Fungicides	Captan	EPA 632M	<3x MDL or <RL	21	21	100.00
Acaricides	Propargite	EPA 619M	<3x MDL or <RL	6	5	83.33
Herbicides	Alachlor	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Ametryn	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Atraton	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Atrazine	EPA 619	<3x MDL or <RL	1	1	100.00
Herbicides	Atrazine	EPA 619M	<3x MDL or <RL	24	23	95.83
Herbicides	Cyanazine	EPA 619	<3x MDL or <RL	1	1	100.00
Herbicides	Cyanazine	EPA 619M	<3x MDL or <RL	24	24	100.00
Herbicides	Diuron	EPA 632M	<3x MDL or <RL	21	20	95.24
Herbicides	Linuron	EPA 632M	<3x MDL or <RL	21	21	100.00
Herbicides	Metolachlor	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Molinate	EPA 619M	<3x MDL or <RL	24	24	100.00
Herbicides	Molinate	WPCL Method 42	<3x MDL or <RL	1	1	100.00
Herbicides	Norflurazon	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Oxyfluorfen	EPA 619M	<3x MDL or	6	6	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
			<RL			
Herbicides	Prometon	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Prometryn	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Propanil	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Propazine	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Prowl	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Secbumeton	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Simazine	EPA 619	<3x MDL or <RL	1	1	100.00
Herbicides	Simazine	EPA 619M	<3x MDL or <RL	24	23	95.83
Herbicides	Simetryn	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Terbuthylazine	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Terbutryn	EPA 619M	<3x MDL or <RL	6	6	100.00
Herbicides	Thiobencarb	EPA 619M	<3x MDL or <RL	24	24	100.00
Herbicides	Thiobencarb	WPCL Method 42	<3x MDL or <RL	1	1	100.00
Herbicides	Trifluralin	EPA 619M	<3x MDL or <RL	6	6	100.00
Organochlorines	DDD(o,p')	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	DDD(o,p')	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	DDE(o,p')	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	DDE(o,p')	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	<3x MDL or <RL	24	23	95.83
Organochlorines	DDE(p,p')	EPA 8081BM	<3x MDL or <RL	7	6	85.71

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDT(o,p')	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	DDT(o,p')	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	DDT(p,p')	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	Dicofol	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	Dicofol	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	Dieldrin	EPA 8081AM	<3x MDL or <RL	24	23	95.83
Organochlorines	Dieldrin	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	Endrin	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	Endrin	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organochlorines	Methoxychlor	EPA 8081AM	<3x MDL or <RL	24	24	100.00
Organochlorines	Methoxychlor	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Organophosphates	Azinphos methyl	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Chlorpyrifos	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Diazinon	EPA 8141AM	<3x MDL or <RL	30	29	96.67
Organophosphates	Dimethoate	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Disulfoton	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Malathion	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Methidathion	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Parathion, Ethyl	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Phorate	EPA 8141AM	<3x MDL or <RL	30	30	100.00
Organophosphates	Phosmet	EPA 8141AM	<3x MDL or	30	30	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
			<RL			
Pyrethroids	Bifenthrin	EPA 1660M	<3x MDL or <RL	25	25	100.00
Pyrethroids	Bifenthrin	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Cyfluthrin-1	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cyfluthrin-2	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cyfluthrin-3	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cyfluthrin-4	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	<3x MDL or <RL	1	1	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Cypermethrin, total	EPA 1660M	<3x MDL or <RL	7	7	100.00
Pyrethroids	Cypermethrin, total	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Cypermethrin-1	EPA 1660M	<3x MDL or <RL	18	17	94.44
Pyrethroids	Cypermethrin-2	EPA 1660M	<3x MDL or <RL	18	17	94.44
Pyrethroids	Cypermethrin-3	EPA 1660M	<3x MDL or <RL	18	17	94.44
Pyrethroids	Cypermethrin-4	EPA 1660M	<3x MDL or <RL	18	17	94.44
Pyrethroids	Deltamethrin	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Deltamethrin	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	<3x MDL or <RL	7	7	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	<3x MDL or <RL	18	18	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Permethrin, total	EPA 1660M	<3x MDL or <RL	7	7	100.00
Pyrethroids	Permethrin, total	EPA 8081BM	<3x MDL or <RL	7	7	100.00
Pyrethroids	Permethrin-1	EPA 1660M	<3x MDL or <RL	18	18	100.00
Pyrethroids	Permethrin-2	EPA 1660M	<3x MDL or <RL	18	18	100.00
Inorganics	Ammonia as N	EPA 350.3	<MDL	21	17	80.95
Inorganics	Ammonia as N	SM 4500 NH3 D	<MDL	7	7	100.00
Inorganics	Color	QC 10308001A	<MDL	7	7	100.00
Inorganics	Color	SM 2120 B	NA	NA	NA	NA
Inorganics	Color	SM 2120 BM	<MDL	20	19	95.00
Inorganics	Dissolved Solids	SM 2540 C	<MDL	27	27	100.00
Inorganics	Hardness as CaCO3	EPA 130.1	<MDL	3	3	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	<MDL	7	7	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	<MDL	12	12	100.00
Inorganics	Nitrate + Nitrite as N	EPA 353.2	<MDL	21	21	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	<MDL	7	7	100.00
Inorganics	Nitrite as N	EPA 353.2	<MDL	21	21	100.00
Inorganics	Nitrite as N	QC 10107041B	<MDL	7	7	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	<MDL	20	20	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	<MDL	8	8	100.00
Inorganics	Total Organic Carbon	EPA 415.1	<MDL	17	9	52.94
Inorganics	Total Organic Carbon	EPA 415.1M	<MDL	10	8	80.00
Inorganics	Turbidity	SM 2130 B	<MDL	27	22	81.48
THMs	Bromodichloromethane	EPA 8260	<MDL	4	4	100.00
THMs	Bromoform	EPA 8260	<MDL	4	4	100.00
THMs	Chloroform	EPA 8260	<MDL	4	4	100.00
THMs	Dibromochloromethane	EPA 8260	<MDL	4	4	100.00
Metals	Arsenic	EPA 1638	<MDL	4	4	100.00
Metals	Arsenic	EPA 1638M	<MDL	18	18	100.00
Metals	Boron	EPA 1638	<MDL	3	0	0.00
Metals	Boron	EPA 1638M	<MDL	19	1	5.26
Metals	Cadmium	EPA 1638	<MDL	4	4	100.00
Metals	Cadmium	EPA 1638M	<MDL	18	15	83.33

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Copper	EPA 1638	<MDL	4	2	50.00
Metals	Copper	EPA 1638M	<MDL	18	1	5.56
Metals	Lead	EPA 1638	<MDL	4	4	100.00
Metals	Lead	EPA 1638M	<MDL	18	10	55.56
Metals	Nickel	EPA 1638	<MDL	4	3	75.00
Metals	Nickel	EPA 1638M	<MDL	18	3	16.67
Metals	Phosphorus as P	EPA 1638	<MDL	4	4	100.00
Metals	Phosphorus as P	EPA 1638M	<MDL	18	9	50.00
Metals	Selenium	EPA 1638	<MDL	4	3	75.00
Metals	Selenium	EPA 1638M	<MDL	18	12	66.67
Metals	Zinc	EPA 1638	<MDL	4	2	50.00
Metals	Zinc	EPA 1638M	<MDL	18	2	11.11
			Total	**1850**	**1717**	**92.81**

Table 15. Summary of field duplicate quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	RPD<25	9	9	100.00
Carbamates	Carbaryl	EPA 632M	RPD<25	9	9	100.00
Carbamates	Carbofuran	EPA 632M	RPD<25	9	9	100.00
Carbamates	Methiocarb	EPA 632M	RPD<25	9	9	100.00
Carbamates	Methomyl	EPA 632M	RPD<25	9	9	100.00
Fungicides	Captan	EPA 632M	RPD<25	9	9	100.00
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619M	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619M	RPD<25	9	9	100.00
Herbicides	Diuron	EPA 632M	RPD<25	9	9	100.00
Herbicides	Linuron	EPA 632M	RPD<25	9	9	100.00
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	RPD<25	9	9	100.00
Herbicides	Molinate	WPCL Method 42	NA	NA	NA	NA
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619M	RPD<25	9	9	100.00
Herbicides	Simazine	EPA 619M	NA	NA	NA	NA
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	RPD<25	9	9	100.00
Herbicides	Thiobencarb	WPCL Method 42	NA	NA	NA	NA
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA	RPD<25	2	2	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
		8081AM				
Organochlorines	DDD(o,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	DDE(o,p')	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	DDE(o,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	DDE(p,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	DDT(o,p')	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	DDT(o,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	DDT(p,p')	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	Dicofol	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	Dicofol	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	Dieldrin	EPA 8081BM	RPD<25	7	6	85.71
Organochlorines	Endrin	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	Endrin	EPA 8081BM	RPD<25	7	7	100.00
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	2	2	100.00
Organochlorines	Methoxychlor	EPA 8081BM	RPD<25	7	7	100.00
Organophosphates	Azinphos methyl	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Chlorpyrifos	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Diazinon	EPA 8141AM	RPD<25	9	8	88.89

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organophosphates	Dimethoate	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Disulfoton	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Malathion	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Methidathion	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Parathion, Ethyl	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Phorate	EPA 8141AM	RPD<25	9	9	100.00
Organophosphates	Phosmet	EPA 8141AM	RPD<25	9	9	100.00
Pyrethroids	Bifenthrin	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Bifenthrin	EPA 8081BM	RPD<25	7	7	100.00
Pyrethroids	Cyfluthrin-1	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cyfluthrin-2	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cyfluthrin-3	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cyfluthrin-4	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	RPD<25	7	7	100.00
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cypermethrin, total	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin, total	EPA 8081BM	RPD<25	7	6	85.71
Pyrethroids	Cypermethrin-1	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cypermethrin-2	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cypermethrin-3	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Cypermethrin-4	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Deltamethrin	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Deltamethrin	EPA 8081BM	RPD<25	7	7	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	RPD<25	7	7	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	RPD<25	2	2	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Permethrin, total	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Permethrin, total	EPA 8081BM	RPD<25	7	7	100.00
Pyrethroids	Permethrin-1	EPA 1660M	RPD<25	2	2	100.00
Pyrethroids	Permethrin-2	EPA 1660M	RPD<25	2	2	100.00
Inorganics	Ammonia as N	EPA 350.3	RPD<25	2	2	100.00
Inorganics	Ammonia as N	SM 4500 NH3 D	RPD<25	7	6	85.71
Inorganics	Color	QC 10308001A	RPD<25	7	6	85.71
Inorganics	Color	SM 2120 B	NA	NA	NA	NA
Inorganics	Color	SM 2120 BM	RPD<25	2	2	100.00
Inorganics	Dissolved Solids	SM 2540 C	RPD<25	9	9	100.00
Inorganics	Hardness as CaCO3	EPA 130.1	RPD<25	2	2	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	RPD<25	7	7	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	NA	NA	NA	NA
Inorganics	Nitrate + Nitrite as N	EPA 353.2	RPD<25	2	2	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	RPD<25	7	7	100.00
Inorganics	Nitrite as N	EPA 353.2	RPD<25	2	2	100.00
Inorganics	Nitrite as N	QC 10107041B	RPD<25	7	7	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	RPD<25	1	1	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	RPD<25	8	8	100.00
Inorganics	Total Organic Carbon	EPA 415.1	NA	NA	NA	NA
Inorganics	Total Organic Carbon	EPA 415.1M	RPD<25	9	7	77.78
Inorganics	Turbidity	SM 2130 B	RPD<25	9	9	100.00
THMs	Bromodichloromethane	EPA 8260	NA	NA	NA	NA
THMs	Bromoform	EPA 8260	NA	NA	NA	NA
THMs	Chloroform	EPA 8260	NA	NA	NA	NA
THMs	Dibromochloromethane	EPA 8260	NA	NA	NA	NA
Metals	Arsenic	EPA 1638	RPD<25	2	2	100.00
Metals	Arsenic	EPA 1638M	RPD<25	23	20	86.96
Metals	Boron	EPA 1638	RPD<25	2	2	100.00
Metals	Boron	EPA 1638M	RPD<25	23	20	86.96

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Cadmium	EPA 1638	RPD<25	2	2	100.00
Metals	Cadmium	EPA 1638M	RPD<25	23	19	82.61
Metals	Copper	EPA 1638	RPD<25	2	2	100.00
Metals	Copper	EPA 1638M	RPD<25	23	21	91.30
Metals	Lead	EPA 1638	RPD<25	2	2	100.00
Metals	Lead	EPA 1638M	RPD<25	23	20	86.96
Metals	Nickel	EPA 1638	RPD<25	2	2	100.00
Metals	Nickel	EPA 1638M	RPD<25	23	20	86.96
Metals	Phosphorus as P	EPA 1638	RPD<25	2	2	100.00
Metals	Phosphorus as P	EPA 1638M	RPD<25	23	21	91.30
Metals	Selenium	EPA 1638	RPD<25	2	2	100.00
Metals	Selenium	EPA 1638M	RPD<25	23	14	60.87
Metals	Zinc	EPA 1638	RPD<25	2	2	100.00
Metals	Zinc	EPA 1638M	RPD<25	23	20	86.96
			TOTAL	**677**	**638**	**94.24**

Table 16. Summary of field duplicate quality control sample evaluations for sediment samples

AgGrouping	AnalyteName	MethodName	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Bifenthrin	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Chlorpyrifos	EPA 8081AM	RPD<25	5	4	80
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Cypermethrin, total	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	DDD(p,p')	EPA 8081AM	RPD<25	5	4	80
Pyrethroids	DDE(p,p')	EPA 8081AM	RPD<25	5	1	20
Pyrethroids	DDT(p,p')	EPA 8081AM	RPD<25	5	2	40
Pyrethroids	Deltamethrin	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Dieldrin	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Endrin	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Methoxychlor	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Permethrin, total	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Permethrin-1	EPA 8081AM	RPD<25	5	5	100
Pyrethroids	Permethrin-2	EPA 8081AM	RPD<25	5	5	100
Inorganics	Total Organic Carbon	KahnM	RPD<25	5	4	80
Metals	Arsenic	EPA 200.8	NA	NA	NA	NA
Metals	Cadmium	EPA 200.8	NA	NA	NA	NA
Metals	Copper	EPA 200.8	NA	NA	NA	NA
Metals	Lead	EPA 200.8	NA	NA	NA	NA
Metals	Nickel	EPA 200.8	NA	NA	NA	NA
Metals	Selenium	EPA 200.8	NA	NA	NA	NA
			TOTAL	**80**	**70**	**87.5**

Table 17. Summary of method blank quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	<3xMDL or <RL	26	26	100
Carbamates	Carbaryl	EPA 632M	<3xMDL or <RL	26	26	100
Carbamates	Carbofuran	EPA 632M	<3xMDL or <RL	26	26	100
Carbamates	Methiocarb	EPA 632M	<3xMDL or <RL	26	26	100
Carbamates	Methomyl	EPA 632M	<3xMDL or <RL	26	26	100
Fungicides	Captan	EPA 632M	<3xMDL or <RL	25	25	100
Acaricides	Propargite	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Alachlor	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Ametryn	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Atraton	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Atrazine	EPA 619	<3xMDL or <RL	1	1	100
Herbicides	Atrazine	EPA 619M	<3xMDL or <RL	36	36	100
Herbicides	Cyanazine	EPA 619	<3xMDL or <RL	1	1	100
Herbicides	Cyanazine	EPA 619M	<3xMDL or <RL	36	36	100
Herbicides	Diuron	EPA 632M	<3xMDL or <RL	26	26	100
Herbicides	Linuron	EPA 632M	<3xMDL or <RL	26	26	100
Herbicides	Metolachlor	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Molinate	EPA 619M	<3xMDL or <RL	36	36	100
Herbicides	Molinate	WPCL Method 42	<3xMDL or <RL	1	1	100
Herbicides	Norflurazon	EPA 619M	<3xMDL or <RL	6	6	100

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Herbicides	Oxyfluorfen	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Prometon	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Prometryn	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Propanil	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Propazine	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Prowl	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Secbumeton	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Simazine	EPA 619	<3xMDL or <RL	1	1	100
Herbicides	Simazine	EPA 619M	<3xMDL or <RL	36	36	100
Herbicides	Simetryn	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Terbuthylazine	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Terbutryn	EPA 619M	<3xMDL or <RL	6	6	100
Herbicides	Thiobencarb	EPA 619M	<3xMDL or <RL	36	36	100
Herbicides	Thiobencarb	WPCL Method 42	<3xMDL or <RL	1	1	100
Herbicides	Trifluralin	EPA 619M	<3xMDL or <RL	6	6	100
Organochlorines	DDD(o,p')	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	DDD(o,p')	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	DDD(p,p')	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	DDD(p,p')	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	DDE(o,p')	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	DDE(o,p')	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	DDE(p,p')	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	DDE(p,p')	EPA 8081BM	<3xMDL or	10	10	100

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
			<RL			
Organochlorines	DDT(o,p')	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	DDT(o,p')	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	DDT(p,p')	EPA 8081AM	<3xMDL or <RL	35	35	100
Organochlorines	DDT(p,p')	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	Dicofol	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	Dicofol	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	Dieldrin	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	Dieldrin	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	Endrin	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	Endrin	EPA 8081BM	<3xMDL or <RL	10	10	100
Organochlorines	Methoxychlor	EPA 8081AM	<3xMDL or <RL	26	26	100
Organochlorines	Methoxychlor	EPA 8081BM	<3xMDL or <RL	10	10	100
Organophosphates	Azinphos methyl	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Chlorpyrifos	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Diazinon	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Dimethoate	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Disulfoton	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Malathion	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Methidathion	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Parathion, Ethyl	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Parathion, Methyl	EPA 8141AM	<3xMDL or <RL	38	38	100
Organophosphates	Phorate	EPA 8141AM	<3xMDL or <RL	38	38	100

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organophosphates	Phosmet	EPA 8141AM	<3xMDL or <RL	38	38	100
Pyrethroids	Bifenthrin	EPA 1660M	<3xMDL or <RL	26	26	100
Pyrethroids	Bifenthrin	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Cyfluthrin-1	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cyfluthrin-2	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cyfluthrin-3	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cyfluthrin-4	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	<3xMDL or <RL	1	1	100
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cypermethrin, total	EPA 1660M	<3xMDL or <RL	8	8	100
Pyrethroids	Cypermethrin, total	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Cypermethrin-1	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cypermethrin-2	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cypermethrin-3	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Cypermethrin-4	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Deltamethrin	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Deltamethrin	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	<3xMDL or <RL	8	8	100
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Esfenvalerate/Fenvalerate-	EPA 1660M	<3xMDL or	18	18	100

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
	2		<RL			
Pyrethroids	Permethrin, total	EPA 1660M	<3xMDL or <RL	8	8	100
Pyrethroids	Permethrin, total	EPA 8081BM	<3xMDL or <RL	10	10	100
Pyrethroids	Permethrin-1	EPA 1660M	<3xMDL or <RL	18	18	100
Pyrethroids	Permethrin-2	EPA 1660M	<3xMDL or <RL	18	18	100
Inorganics	Ammonia as N	EPA 350.3	Blanks<MDL	45	45	100
Inorganics	Ammonia as N	SM 4500 NH3 D	Blanks<MDL	10	10	100
Inorganics	Color	QC 10308001A	Blanks<MDL	15	15	100
Inorganics	Color	SM 2120 B	Blanks<MDL	3	3	100
Inorganics	Color	SM 2120 BM	Blanks<MDL	73	73	100
Inorganics	Dissolved Solids	SM 2540 C	Blanks<MDL	57	57	100
Inorganics	Hardness as CaCO3	EPA 130.1	Blanks<MDL	4	4	100
Inorganics	Hardness as CaCO3	QC 10301311B	Blanks<MDL	10	10	100
Inorganics	Hardness as CaCO3	SM 2340 C	Blanks<MDL	38	38	100
Inorganics	Nitrate + Nitrite as N	EPA 353.2	Blanks<MDL	39	39	100
Inorganics	Nitrate + Nitrite as N	QC 10107041B	Blanks<MDL	11	11	100
Inorganics	Nitrite as N	EPA 353.2	Blanks<MDL	77	77	100
Inorganics	Nitrite as N	QC 10107041B	Blanks<MDL	17	17	100
Inorganics	OrthoPhosphate as P	EPA 365.1M	Blanks<MDL	75	75	100
Inorganics	OrthoPhosphate as P	QC 10115011M	Blanks<MDL	20	20	100
Inorganics	Total Organic Carbon	EPA 415.1	Blanks<MDL	20	20	100
Inorganics	Total Organic Carbon	EPA 415.1M	Blanks<MDL	14	14	100
Inorganics	Turbidity	SM 2130 B	Blanks<MDL	211	211	100
THMs	Bromodichloromethane	EPA 8260	Blanks<MDL	12	12	100
THMs	Bromoform	EPA 8260	Blanks<MDL	12	12	100
THMs	Chloroform	EPA 8260	Blanks<MDL	12	12	100
THMs	Dibromochloromethane	EPA 8260	Blanks<MDL	12	12	100
Metals	Arsenic	EPA 1638	Blanks<MDL	4	4	100
Metals	Arsenic	EPA 1638M	Blanks<MDL	21	21	100
Metals	Boron	EPA 1638	Blanks<MDL	3	3	100
Metals	Boron	EPA 1638M	Blanks<MDL	24	24	100
Metals	Cadmium	EPA 1638	Blanks<MDL	4	4	100
Metals	Cadmium	EPA 1638M	Blanks<MDL	21	21	100
Metals	Copper	EPA 1638	Blanks<MDL	4	4	100
Metals	Copper	EPA 1638M	Blanks<MDL	21	21	100
Metals	Lead	EPA 1638	Blanks<MDL	4	4	100

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Lead	EPA 1638M	Blanks<MDL	21	21	100
Metals	Nickel	EPA 1638	Blanks<MDL	4	4	100
Metals	Nickel	EPA 1638M	Blanks<MDL	21	21	100
Metals	Phosphorus as P	EPA 1638	Blanks<MDL	4	4	100
Metals	Phosphorus as P	EPA 1638M	Blanks<MDL	21	21	100
Metals	Selenium	EPA 1638	Blanks<MDL	4	4	100
Metals	Selenium	EPA 1638M	Blanks<MDL	21	21	100
Metals	Zinc	EPA 1638	Blanks<MDL	4	4	100
Metals	Zinc	EPA 1638M	Blanks<MDL	21	21	100
			TOTAL	**2676**	**2676**	**100**

Table 18. Summary of method blank quality control sample evaluations for sediment samples

AG Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Bifenthrin	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Chlorpyrifos	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Cypermethrin, total	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	DDD(p,p')	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	DDE(p,p')	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	DDT(p,p')	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Deltamethrin	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Dieldrin	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Endrin	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Methoxychlor	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Permethrin, total	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Permethrin-1	EPA 8081AM	<MDL	9	9	100.00
Pyrethroids	Permethrin-2	EPA 8081AM	<MDL	9	9	100.00
Inorganics	Total Organic Carbon	KahnM	<MDL	12	0	0.00
Metals	Arsenic	EPA 200.8	<MDL	4	4	100.00
Metals	Cadmium	EPA 200.8	<MDL	4	4	100.00
Metals	Copper	EPA 200.8	<MDL	4	4	100.00
Metals	Lead	EPA 200.8	<MDL	4	4	100.00
Metals	Nickel	EPA 200.8	<MDL	4	4	100.00
Metals	Selenium	EPA 200.8	<MDL	4	4	100.00
			TOTAL	**171**	**159**	**92.98**

Table 19. Summary of lab control spike and certified reference material quality control sample envaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	PR 80-120	36	30	83.33
Carbamates	Carbaryl	EPA 632M	PR 80-120	36	32	88.89
Carbamates	Carbofuran	EPA 632M	PR 80-120	36	35	97.22
Carbamates	Methiocarb	EPA 632M	PR 80-120	36	34	94.44
Carbamates	Methomyl	EPA 632M	PR 80-120	36	34	94.44
Fungicides	Captan	EPA 632M	PR 80-120	39	16	41.03
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	PR 80-120	2	2	100.00
Herbicides	Atrazine	EPA 619M	PR 80-120	41	35	85.37
Herbicides	Cyanazine	EPA 619	PR 80-120	2	2	100.00
Herbicides	Cyanazine	EPA 619M	PR 80-120	41	31	75.61
Herbicides	Diuron	EPA 632M	PR 80-120	36	35	97.22
Herbicides	Linuron	EPA 632M	PR 80-120	36	31	86.11
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	PR 80-120	41	36	87.80
Herbicides	Molinate	WPCL Method 42	PR 80-120	2	2	100.00
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619	PR 80-120	2	2	100.00
Herbicides	Simazine	EPA 619M	PR 80-120	41	34	82.93
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	PR 80-120	41	38	92.68
Herbicides	Thiobencarb	WPCL Method 42	PR 80-120	2	2	100.00
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA 8081AM	PR 80-120	24	22	91.67

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(p,p')	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	DDE(o,p')	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	PR 80-120	24	23	95.83
Organochlorines	DDT(o,p')	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	Dicofol	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	Dieldrin	EPA 8081AM	PR 80-120	24	24	100.00
Organochlorines	Endrin	EPA 8081AM	PR 80-120	24	22	91.67
Organochlorines	Methoxychlor	EPA 8081AM	PR 80-120	24	23	95.83
Organochlorines	DDD(o,p')	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	PR 80-120	20	19	95.00
Organochlorines	DDE(o,p')	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	DDE(p,p')	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	DDT(o,p')	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	DDT(p,p')	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	Dicofol	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	Dieldrin	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	Endrin	EPA 8081BM	PR 80-120	20	20	100.00
Organochlorines	Methoxychlor	EPA 8081BM	PR 80-120	20	18	90.00
Organophosphates	Azinphos methyl	EPA 8141AM	PR 80-120	45	43	95.56
Organophosphates	Chlorpyrifos	EPA 8141AM	PR 80-120	45	45	100.00
Organophosphates	Diazinon	EPA 8141AM	PR 80-120	45	45	100.00
Organophosphates	Dimethoate	EPA 8141AM	PR 80-120	45	35	77.78
Organophosphates	Disulfoton	EPA 8141AM	PR 80-120	45	17	37.78
Organophosphates	Malathion	EPA 8141AM	PR 80-120	45	43	95.56
Organophosphates	Methidathion	EPA 8141AM	PR 80-120	45	43	95.56
Organophosphates	Parathion, Ethyl	EPA 8141AM	PR 80-120	50	46	92.00
Organophosphates	Parathion, Methyl	EPA 8141AM	PR 80-120	45	42	93.33
Organophosphates	Phorate	EPA 8141AM	PR 80-120	45	36	80.00
Organophosphates	Phosmet	EPA 8141AM	PR 80-120	45	40	88.89
Pyrethroids	Bifenthrin	EPA 1660M	PR 80-120	26	25	96.15
Pyrethroids	Bifenthrin	EPA 8081BM	PR 80-120	20	14	70.00
Pyrethroids	Cyfluthrin-1	EPA 1660M	PR 80-120	17	16	94.12
Pyrethroids	Cyfluthrin-2	EPA 1660M	PR 80-120	17	15	88.24
Pyrethroids	Cyfluthrin-3	EPA 1660M	PR 80-120	17	16	94.12
Pyrethroids	Cyfluthrin-4	EPA 1660M	PR 80-120	17	14	82.35
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	PR 80-120	2	2	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	PR 80-120	20	15	75.00
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	PR 80-120	17	17	100.00
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	PR 80-120	17	15	88.24
Pyrethroids	Cypermethrin, total	EPA 1660M	PR 80-120	9	8	88.89
Pyrethroids	Cypermethrin, total	EPA 8081BM	PR 80-120	20	18	90.00
Pyrethroids	Cypermethrin-1	EPA 1660M	PR 80-120	17	16	94.12

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin-2	EPA 1660M	PR 80-120	17	16	94.12
Pyrethroids	Cypermethrin-3	EPA 1660M	PR 80-120	17	16	94.12
Pyrethroids	Cypermethrin-4	EPA 1660M	PR 80-120	17	15	88.24
Pyrethroids	Deltamethrin	EPA 1660M	PR 80-120	17	14	82.35
Pyrethroids	Deltamethrin	EPA 8081BM	PR 80-120	20	12	60.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	PR 80-120	9	9	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	PR 80-120	20	18	90.00
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	PR 80-120	17	15	88.24
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	PR 80-120	17	15	88.24
Pyrethroids	Permethrin, total	EPA 1660M	PR 80-120	9	9	100.00
Pyrethroids	Permethrin, total	EPA 8081BM	PR 80-120	20	15	75.00
Pyrethroids	Permethrin-1	EPA 1660M	PR 80-120	17	17	100.00
Pyrethroids	Permethrin-2	EPA 1660M	PR 80-120	17	17	100.00
Inorganics	Ammonia as N	EPA 350.3	PR 80-120	45	45	100.00
Inorganics	Ammonia as N	SM 4500 NH3 D	PR 80-120	10	10	100.00
Inorganics	Color	QC 10308001A	PR 80-120	15	15	100.00
Inorganics	Color	SM 2120 B	PR 80-120	3	3	100.00
Inorganics	Color	SM 2120 BM	PR 80-120	74	74	100.00
Inorganics	Dissolved Solids	SM 2540 C	PR 80-120	57	57	100.00
Inorganics	Hardness as CaCO3	EPA 130.1	PR 80-120	4	4	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	PR 80-120	10	10	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	PR 80-120	38	38	100.00
Inorganics	Nitrate + Nitrite as N	EPA 353.2	PR 80-120	39	…….	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	PR 80-120	11	11	100.00
Inorganics	Nitrite as N	EPA 353.2	PR 80-120	77	77	100.00
Inorganics	Nitrite as N	QC 10107041B	PR 80-120	17	17	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	PR 80-120	75	75	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	PR 80-120	20	20	100.00
Inorganics	Total Organic Carbon	EPA 415.1	PR 75-125	5	5	100.00
Inorganics	Total Organic Carbon	EPA 415.1M	PR 75-125	22	22	100.00
Inorganics	Turbidity	SM 2130 B	PR 80-120	212	212	100.00
THMs	Bromodichloromethane	EPA 8260	NA	NA	NA	NA
THMs	Bromoform	EPA 8260	NA	NA	NA	NA
THMs	Chloroform	EPA 8260	NA	NA	NA	NA
THMs	Dibromochloromethane	EPA 8260	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Arsenic	EPA 1638	PR 80-120	6	6	100.00
Metals	Arsenic	EPA 1638M	PR 80-120	22	22	100.00
Metals	Boron	EPA 1638	PR 80-120	5	5	100.00
Metals	Boron	EPA 1638M	PR 80-120	23	23	100.00
Metals	Cadmium	EPA 1638	PR 80-120	6	6	100.00
Metals	Cadmium	EPA 1638M	PR 80-120	22	22	100.00
Metals	Copper	EPA 1638	PR 80-120	6	6	100.00
Metals	Copper	EPA 1638M	PR 80-120	22	22	100.00
Metals	Lead	EPA 1638	PR 80-120	6	6	100.00
Metals	Lead	EPA 1638M	PR 80-120	22	22	100.00
Metals	Nickel	EPA 1638	PR 80-120	6	6	100.00
Metals	Nickel	EPA 1638M	PR 80-120	22	22	100.00
Metals	Phosphorus as P	EPA 1638	PR 80-120	1	1	100.00
Metals	Phosphorus as P	EPA 1638M	PR 80-120	22	22	100.00
Metals	Selenium	EPA 1638	PR 80-120	6	6	100.00
Metals	Selenium	EPA 1638M	PR 80-120	22	22	100.00
Metals	Zinc	EPA 1638	PR 80-120	6	6	100.00
Metals	Zinc	EPA 1638M	PR 80-120	22	22	100.00
			TOTAL	**2857**	**2618**	**91.63**

Table 20. Summary of lab control spike quality control sample evaluations for sediment samples

AG Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Bifenthrin	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Chlorpyrifos	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Cypermethrin, total	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	DDD(p,p')	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	DDE(p,p')	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	DDT(p,p')	EPA 8081AM	PR 50-150	2	1	50.00
Pyrethroids	Deltamethrin	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Dieldrin	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Endrin	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081AM	PR 50-150	2	1	50.00
Pyrethroids	Methoxychlor	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Permethrin, total	EPA 8081AM	PR 50-150	2	2	100.00
Pyrethroids	Permethrin-1	EPA 8081AM	PR 50-150	NA	NA	NA
Pyrethroids	Permethrin-2	EPA 8081AM	PR 50-150	NA	NA	NA
Inorganics	Total Organic Carbon	KahnM	PR 50-150	13	13	100.00
Metals	Arsenic	EPA 200.8	PR75-125	2	2	100.00
Metals	Cadmium	EPA 200.8	PR75-125	2	2	100.00
Metals	Copper	EPA 200.8	PR75-125	2	2	100.00
Metals	Lead	EPA 200.8	PR75-125	2	2	100.00
Metals	Nickel	EPA 200.8	PR75-125	2	2	100.00
Metals	Selenium	EPA 200.8	PR75-125	2	2	100.00
			TOTAL	**51**	**49**	**96.08**

Table 21. Summary of lab control spike duplicate and certified reference material duplicate quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	RPD<25	11	10	90.91
Carbamates	Carbaryl	EPA 632M	RPD<25	11	11	100.00
Carbamates	Carbofuran	EPA 632M	RPD<25	11	11	100.00
Carbamates	Methiocarb	EPA 632M	RPD<25	11	11	100.00
Carbamates	Methomyl	EPA 632M	RPD<25	11	11	100.00
Fungicides	Captan	EPA 632M	RPD<25	16	15	93.75
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	RPD<25	1	1	100.00
Herbicides	Atrazine	EPA 619M	RPD<25	11	11	100.00
Herbicides	Cyanazine	EPA 619	RPD<25	1	1	100.00
Herbicides	Cyanazine	EPA 619M	RPD<25	11	9	81.82
Herbicides	Diuron	EPA 632M	RPD<25	11	11	100.00
Herbicides	Linuron	EPA 632M	RPD<25	11	10	90.91
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	RPD<25	11	11	100.00
Herbicides	Molinate	WPCL Method 42	RPD<25	1	1	100.00
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619	RPD<25	1	1	100.00
Herbicides	Simazine	EPA 619M	RPD<25	11	10	90.91
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	RPD<25	11	11	100.00
Herbicides	Thiobencarb	WPCL Method 42	RPD<25	1	1	100.00
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	DDD(o,p')	EPA 8081BM	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDE(o,p')	EPA 8081AM	RPD<25	12	11	91.67
Organochlorines	DDE(o,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	12	11	91.67
Organochlorines	DDE(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDT(o,p')	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	DDT(o,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	12	11	91.67
Organochlorines	DDT(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Dicofol	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	Dicofol	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	Dieldrin	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Endrin	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	Endrin	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	12	12	100.00
Organochlorines	Methoxychlor	EPA 8081BM	NA	NA	NA	NA
Organophosphates	Azinphos methyl	EPA 8141AM	RPD<25	11	10	90.91
Organophosphates	Chlorpyrifos	EPA 8141AM	RPD<25	11	11	100.00
Organophosphates	Diazinon	EPA 8141AM	RPD<25	11	10	90.91
Organophosphates	Dimethoate	EPA 8141AM	RPD<25	11	10	90.91
Organophosphates	Disulfoton	EPA 8141AM	RPD<25	11	10	90.91
Organophosphates	Malathion	EPA 8141AM	RPD<25	11	11	100.00
Organophosphates	Methidathion	EPA 8141AM	RPD<25	11	11	100.00
Organophosphates	Parathion, Ethyl	EPA 8141AM	RPD<25	16	15	93.75
Organophosphates	Parathion, Methyl	EPA 8141AM	RPD<25	11	11	100.00
Organophosphates	Phorate	EPA 8141AM	RPD<25	11	11	100.00
Organophosphates	Phosmet	EPA 8141AM	RPD<25	11	9	81.82
Pyrethroids	Bifenthrin	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Bifenthrin	EPA 8081BM	RPD<25	10	7	70.00
Pyrethroids	Cyfluthrin-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-3	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-4	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	RPD<25	10	9	90.00
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cypermethrin, total	EPA 8081BM	RPD<25	10	9	90.00
Pyrethroids	Cypermethrin-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin-2	EPA 1660M	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin-3	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin-4	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Deltamethrin	EPA 8081BM	RPD<25	10	8	80.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	RPD<25	10	9	90.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Permethrin, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Permethrin, total	EPA 8081BM	RPD<25	10	9	90.00
Pyrethroids	Permethrin-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Permethrin-2	EPA 1660M	NA	NA	NA	NA
Inorganics	Ammonia as N	EPA 350.3	NA	NA	NA	NA
Inorganics	Ammonia as N	SM 4500 NH3 D	NA	NA	NA	NA
Inorganics	Color	QC 10308001A	NA	NA	NA	NA
Inorganics	Color	SM 2120 B	NA	NA	NA	NA
Inorganics	Color	SM 2120 BM	NA	NA	NA	NA
Inorganics	Dissolved Solids	SM 2540 C	NA	NA	NA	NA
Inorganics	Hardness as CaCO3	EPA 130.1	NA	NA	NA	NA
Inorganics	Hardness as CaCO3	QC 10301311B	NA	NA	NA	NA
Inorganics	Hardness as CaCO3	SM 2340 C	NA	NA	NA	NA
Inorganics	Nitrate + Nitrite as N	EPA 353.2	NA	NA	NA	NA
Inorganics	Nitrate + Nitrite as N	QC 10107041B	NA	NA	NA	NA
Inorganics	Nitrite as N	EPA 353.2	NA	NA	NA	NA
Inorganics	Nitrite as N	QC 10107041B	NA	NA	NA	NA
Inorganics	OrthoPhosphate as P	EPA 365.1M	NA	NA	NA	NA
Inorganics	OrthoPhosphate as P	QC 10115011M	NA	NA	NA	NA
Inorganics	Total Organic Carbon	EPA 415.1	NA	NA	NA	NA
Inorganics	Total Organic Carbon	EPA 415.1M	RPD<25	4	4	100.00
Inorganics	Turbidity	SM 2130 B	NA	NA	NA	NA
THMs	Bromodichloromethane	EPA 8260	NA	NA	NA	NA
THMs	Bromoform	EPA 8260	NA	NA	NA	NA
THMs	Chloroform	EPA 8260	NA	NA	NA	NA
THMs	Dibromochloromethane	EPA 8260	NA	NA	NA	NA
Metals	Arsenic	EPA 1638	RPD<25	1	1	100.00
Metals	Arsenic	EPA 1638M	RPD<25	8	8	100.00
Metals	Boron	EPA 1638	RPD<25	1	1	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Boron	EPA 1638M	RPD<25	6	6	100.00
Metals	Cadmium	EPA 1638	RPD<25	1	1	100.00
Metals	Cadmium	EPA 1638M	RPD<25	8	8	100.00
Metals	Copper	EPA 1638	RPD<25	1	1	100.00
Metals	Copper	EPA 1638M	RPD<25	8	8	100.00
Metals	Lead	EPA 1638	RPD<25	1	1	100.00
Metals	Lead	EPA 1638M	RPD<25	8	8	100.00
Metals	Nickel	EPA 1638	RPD<25	1	1	100.00
Metals	Nickel	EPA 1638M	RPD<25	8	8	100.00
Metals	Phosphorus as P	EPA 1638	RPD<25	8	8	100.00
Metals	Phosphorus as P	EPA 1638M	RPD<25	8	8	100.00
Metals	Selenium	EPA 1638	RPD<25	1	1	100.00
Metals	Selenium	EPA 1638M	RPD<25	8	8	100.00
Metals	Zinc	EPA 1638	RPD<25	1	1	100.00
Metals	Zinc	EPA 1638M	RPD<25	8	8	100.00
			TOTAL	554	529	95.49

Table 22. Summary of matrix spike quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	RPD<25	23	19	82.61
Carbamates	Carbaryl	EPA 632M	RPD<25	23	20	86.96
Carbamates	Carbofuran	EPA 632M	RPD<25	23	22	95.65
Carbamates	Methiocarb	EPA 632M	RPD<25	23	22	95.65
Carbamates	Methomyl	EPA 632M	RPD<25	23	22	95.65
Fungicides	Captan	EPA 632M	RPD<25	16	14	87.50
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619M	RPD<25	26	24	92.31
Herbicides	Cyanazine	EPA 619	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619M	RPD<25	26	20	76.92
Herbicides	Diuron	EPA 632M	RPD<25	23	21	91.30
Herbicides	Linuron	EPA 632M	RPD<25	23	21	91.30
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	RPD<25	26	21	80.77
Herbicides	Molinate	WPCL Method 42	NA	NA	NA	NA
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619	NA	NA	NA	NA
Herbicides	Simazine	EPA 619M	RPD<25	26	23	88.46
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	RPD<25	26	23	88.46
Herbicides	Thiobencarb	WPCL Method 42	NA	NA	NA	NA
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	DDD(o,p')	EPA 8081BM	RPD<25	9	9	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDE(o,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDE(o,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDE(p,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDT(o,p')	EPA 8081AM	RPD<25	20	18	90.00
Organochlorines	DDT(o,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	DDT(p,p')	EPA 8081BM	RPD<25	9	8	88.89
Organochlorines	Dicofol	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Dicofol	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	Dieldrin	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Endrin	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Endrin	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Methoxychlor	EPA 8081BM	RPD<25	9	8	88.89
Organophosphates	Azinphos methyl	EPA 8141AM	RPD<25	32	29	90.63
Organophosphates	Chlorpyrifos	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Diazinon	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Dimethoate	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Disulfoton	EPA 8141AM	RPD<25	32	30	93.75
Organophosphates	Malathion	EPA 8141AM	RPD<25	32	30	93.75
Organophosphates	Methidathion	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Parathion, Ethyl	EPA 8141AM	RPD<25	16	16	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Phorate	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Phosmet	EPA 8141AM	RPD<25	32	31	96.88
Pyrethroids	Bifenthrin	EPA 1660M	RPD<25	25	22	88.00
Pyrethroids	Bifenthrin	EPA 8081BM	RPD<25	9	7	77.78
Pyrethroids	Cyfluthrin-1	EPA 1660M	RPD<25	17	15	88.24
Pyrethroids	Cyfluthrin-2	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cyfluthrin-3	EPA 1660M	RPD<25	17	17	100.00
Pyrethroids	Cyfluthrin-4	EPA 1660M	RPD<25	17	15	88.24
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	RPD<25	9	8	88.89
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cypermethrin, total	EPA 1660M	RPD<25	8	6	75.00
Pyrethroids	Cypermethrin, total	EPA 8081BM	RPD<25	9	8	88.89
Pyrethroids	Cypermethrin-1	EPA 1660M	RPD<25	17	14	82.35
Pyrethroids	Cypermethrin-2	EPA 1660M	RPD<25	17	15	88.24

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin-3	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cypermethrin-4	EPA 1660M	RPD<25	17	13	76.47
Pyrethroids	Deltamethrin	EPA 1660M	RPD<25	17	13	76.47
Pyrethroids	Deltamethrin	EPA 8081BM	RPD<25	9	7	77.78
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	RPD<25	8	8	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	RPD<25	9	9	100.00
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	RPD<25	17	11	64.71
Pyrethroids	Permethrin, total	EPA 1660M	RPD<25	8	7	87.50
Pyrethroids	Permethrin, total	EPA 8081BM	RPD<25	9	6	66.67
Pyrethroids	Permethrin-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Permethrin-2	EPA 1660M	RPD<25	17	14	82.35
Inorganics	Ammonia as N	EPA 350.3	RPD<25	51	51	100.00
Inorganics	Ammonia as N	SM 4500 NH3 D	RPD<25	10	10	100.00
Inorganics	Color	QC 10308001A	RPD<25	15	15	100.00
Inorganics	Color	SM 2120 B	RPD<25	2	2	100.00
Inorganics	Color	SM 2120 BM	RPD<25	75	75	100.00
Inorganics	Dissolved Solids	SM 2540 C	NA	NA	NA	NA
Inorganics	Hardness as CaCO3	EPA 130.1	RPD<25	3	3	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	RPD<25	11	11	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	NA	NA	NA	NA
Inorganics	Nitrate + Nitrite as N	EPA 353.2	RPD<25	40	40	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	RPD<25	12	12	100.00
Inorganics	Nitrite as N	EPA 353.2	RPD<25	76	76	100.00
Inorganics	Nitrite as N	QC 10107041B	RPD<25	17	17	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	RPD<25	76	76	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	RPD<25	20	20	100.00
Inorganics	Total Organic Carbon	EPA 415.1	RPD<25	5	4	80.00
Inorganics	Total Organic Carbon	EPA 415.1M	RPD<25	14	14	100.00
Inorganics	Turbidity	SM 2130 B	NA	NA	NA	NA
THMs	Bromodichloromethane	EPA 8260	RPD<25	4	4	100.00
THMs	Bromoform	EPA 8260	RPD<25	4	3	75.00
THMs	Chloroform	EPA 8260	RPD<25	4	4	100.00
THMs	Dibromochloromethane	EPA 8260	RPD<25	4	4	100.00
Metals	Arsenic	EPA 1638	RPD<25	6	6	100.00
Metals	Arsenic	EPA 1638M	RPD<25	27	27	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Boron	EPA 1638	RPD<25	5	5	100.00
Metals	Boron	EPA 1638M	RPD<25	27	27	100.00
Metals	Cadmium	EPA 1638	RPD<25	6	6	100.00
Metals	Cadmium	EPA 1638M	RPD<25	27	27	96.30
Metals	Copper	EPA 1638	RPD<25	6	6	100.00
Metals	Copper	EPA 1638M	RPD<25	27	27	100.00
Metals	Lead	EPA 1638	RPD<25	6	6	100.00
Metals	Lead	EPA 1638M	RPD<25	27	27	100.00
Metals	Nickel	EPA 1638	RPD<25	6	6	100.00
Metals	Nickel	EPA 1638M	RPD<25	27	27	100.00
Metals	Phosphorus as P	EPA 1638	RPD<25	6	6	100.00
Metals	Phosphorus as P	EPA 1638M	RPD<25	27	26	100.00
Metals	Selenium	EPA 1638	RPD<25	6	6	100.00
Metals	Selenium	EPA 1638M	RPD<25	27	25	96.30
Metals	Zinc	EPA 1638	RPD<25	6	6	100.00
Metals	Zinc	EPA 1638M	RPD<25	27	27	100.00
			Total	**2031**	**1924**	**94.73**

Table 23. Summary of matrix spike quality sample evaluations for sediment samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	Bifenthrin	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	Chlorpyrifos	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	Cyhalothrin, lambda, total	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	Cypermethrin, total	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	PR 50-150	16	12	75.00
Organochlorines	DDT(p,p')	EPA 8081AM	PR 50-150	16	14	87.50
Organochlorines	Deltamethrin	EPA 8081AM	PR 50-150	16	15	93.75
Organochlorines	Dieldrin	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	Endrin	EPA 8081AM	PR 50-150	16	15	93.75
Organochlorines	Esfenvalerate/Fenvalerate, total	EPA 8081AM	PR 50-150	16	25	156.25
Organochlorines	Methoxychlor	EPA 8081AM	PR 50-150	16	16	100.00
Organochlorines	Permethrin, total	EPA 8081AM	PR 50-150	16	14	87.50
Organochlorines	Permethrin-1	EPA 8081AM	PR 50-150	NA	NA	NA
Organochlorines	Permethrin-2	EPA 8081AM	PR 50-150	NA	NA	NA
Inorganics	Total Organic Carbon	KahnM	NA	NA	NA	NA
Metals	Arsenic	EPA 200.8	PR 75-125	4	4	100.00
Metals	Cadmium	EPA 200.8	PR 75-125	4	4	100.00
Metals	Copper	EPA 200.8	PR 75-125	4	4	100.00
Metals	Lead	EPA 200.8	PR 75-125	4	4	100.00
Metals	Nickel	EPA 200.8	PR 75-125	4	4	100.00
Metals	Selenium	EPA 200.8	PR 75-125	4	4	100.00
			TOTAL	**232**	**231**	**99.57**

Table 24. Summary of matrix spike duplicate quality control sample evaluations for water samples

Included in the following table are NONAG matrix spikes for batch quality assurance purposes. Evaluations are sorted by analyte grouping, analyte, and method.

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	RPD<25	23	19	82.61
Carbamates	Carbaryl	EPA 632M	RPD<25	23	20	86.96
Carbamates	Carbofuran	EPA 632M	RPD<25	23	22	95.65
Carbamates	Methiocarb	EPA 632M	RPD<25	23	22	95.65
Carbamates	Methomyl	EPA 632M	RPD<25	23	22	95.65
Fungicides	Captan	EPA 632M	RPD<25	16	14	87.50
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619M	RPD<25	26	24	92.31
Herbicides	Cyanazine	EPA 619	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619M	RPD<25	26	20	76.92
Herbicides	Diuron	EPA 632M	RPD<25	23	21	91.30
Herbicides	Linuron	EPA 632M	RPD<25	23	21	91.30
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	RPD<25	26	21	80.77
Herbicides	Molinate	WPCL Method 42	NA	NA	NA	NA
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619	NA	NA	NA	NA
Herbicides	Simazine	EPA 619M	RPD<25	26	23	88.46
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	RPD<25	26	23	88.46
Herbicides	Thiobencarb	WPCL Method 42	NA	NA	NA	NA
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(o,p')	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	DDD(o,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDD(p,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDE(o,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDE(o,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	DDE(p,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDT(o,p')	EPA 8081AM	RPD<25	20	18	90.00
Organochlorines	DDT(o,p')	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	DDT(p,p')	EPA 8081BM	RPD<25	9	8	88.89
Organochlorines	Dicofol	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Dicofol	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	20	20	100.00
Organochlorines	Dieldrin	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Endrin	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Endrin	EPA 8081BM	RPD<25	9	9	100.00
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	20	19	95.00
Organochlorines	Methoxychlor	EPA 8081BM	RPD<25	9	8	88.89
Organophosphates	Azinphos methyl	EPA 8141AM	RPD<25	32	29	90.63
Organophosphates	Chlorpyrifos	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Diazinon	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Dimethoate	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Disulfoton	EPA 8141AM	RPD<25	32	30	93.75
Organophosphates	Malathion	EPA 8141AM	RPD<25	32	30	93.75
Organophosphates	Methidathion	EPA 8141AM	RPD<25	32	31	96.88
Organophosphates	Parathion, Ethyl	EPA 8141AM	RPD<25	16	16	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Phorate	EPA 8141AM	RPD<25	32	32	100.00
Organophosphates	Phosmet	EPA 8141AM	RPD<25	32	31	96.88
Pyrethroids	Bifenthrin	EPA 1660M	RPD<25	25	22	88.00
Pyrethroids	Bifenthrin	EPA 8081BM	RPD<25	9	7	77.78
Pyrethroids	Cyfluthrin-1	EPA 1660M	RPD<25	17	15	88.24
Pyrethroids	Cyfluthrin-2	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cyfluthrin-3	EPA 1660M	RPD<25	17	17	100.00
Pyrethroids	Cyfluthrin-4	EPA 1660M	RPD<25	17	15	88.24
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	RPD<25	9	8	88.89
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cypermethrin, total	EPA 1660M	RPD<25	8	6	75.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin, total	EPA 8081BM	RPD<25	9	8	88.89
Pyrethroids	Cypermethrin-1	EPA 1660M	RPD<25	17	14	82.35
Pyrethroids	Cypermethrin-2	EPA 1660M	RPD<25	17	15	88.24
Pyrethroids	Cypermethrin-3	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Cypermethrin-4	EPA 1660M	RPD<25	17	13	76.47
Pyrethroids	Deltamethrin	EPA 1660M	RPD<25	17	13	76.47
Pyrethroids	Deltamethrin	EPA 8081BM	RPD<25	9	7	77.78
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	RPD<25	8	8	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	RPD<25	9	9	100.00
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	RPD<25	17	11	64.71
Pyrethroids	Permethrin, total	EPA 1660M	RPD<25	8	7	87.50
Pyrethroids	Permethrin, total	EPA 8081BM	RPD<25	9	6	66.67
Pyrethroids	Permethrin-1	EPA 1660M	RPD<25	17	16	94.12
Pyrethroids	Permethrin-2	EPA 1660M	RPD<25	17	14	82.35
Inorganics	Ammonia as N	EPA 350.3	RPD<25	51	51	100.00
Inorganics	Ammonia as N	SM 4500 NH3 D	RPD<25	10	10	100.00
Inorganics	Color	QC 10308001A	RPD<25	15	15	100.00
Inorganics	Color	SM 2120 B	RPD<25	2	2	100.00
Inorganics	Color	SM 2120 BM	RPD<25	75	75	100.00
Inorganics	Dissolved Solids	SM 2540 C	NA	NA	NA	NA
Inorganics	Hardness as CaCO3	EPA 130.1	RPD<25	3	3	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	RPD<25	11	11	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	NA	NA	NA	NA
Inorganics	Nitrate + Nitrite as N	EPA 353.2	RPD<25	40	40	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	RPD<25	12	12	100.00
Inorganics	Nitrite as N	EPA 353.2	RPD<25	76	76	100.00
Inorganics	Nitrite as N	QC 10107041B	RPD<25	17	17	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	RPD<25	76	76	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	RPD<25	20	20	100.00
Inorganics	Total Organic Carbon	EPA 415.1	RPD<25	5	4	80.00
Inorganics	Total Organic Carbon	EPA 415.1M	RPD<25	14	14	100.00
Inorganics	Turbidity	SM 2130 B	NA	NA	NA	NA
THMs	Bromodichloromethane	EPA 8260	RPD<25	4	4	100.00
THMs	Bromoform	EPA 8260	RPD<25	4	3	75.00
THMs	Chloroform	EPA 8260	RPD<25	4	4	100.00
THMs	Dibromochloromethane	EPA 8260	RPD<25	4	4	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Arsenic	EPA 1638	RPD<25	6	6	100.00
Metals	Arsenic	EPA 1638M	RPD<25	27	27	100.00
Metals	Boron	EPA 1638	RPD<25	5	5	100.00
Metals	Boron	EPA 1638M	RPD<25	27	27	100.00
Metals	Cadmium	EPA 1638	RPD<25	6	6	100.00
Metals	Cadmium	EPA 1638M	RPD<25	27	27	96.30
Metals	Copper	EPA 1638	RPD<25	6	6	100.00
Metals	Copper	EPA 1638M	RPD<25	27	27	100.00
Metals	Lead	EPA 1638	RPD<25	6	6	100.00
Metals	Lead	EPA 1638M	RPD<25	27	27	100.00
Metals	Nickel	EPA 1638	RPD<25	6	6	100.00
Metals	Nickel	EPA 1638M	RPD<25	27	27	100.00
Metals	Phosphorus as P	EPA 1638	RPD<25	6	6	100.00
Metals	Phosphorus as P	EPA 1638M	RPD<25	27	26	100.00
Metals	Selenium	EPA 1638	RPD<25	6	6	100.00
Metals	Selenium	EPA 1638M	RPD<25	27	25	96.30
Metals	Zinc	EPA 1638	RPD<25	6	6	100.00
Metals	Zinc	EPA 1638M	RPD<25	27	27	100.00
			Total	**2031**	**1924**	**94.73**

Table 25. Summary of matrix spike duplicate quality control sample evaluations for sediment samples

Included in the following table are NONAG matrix spikes for batch quality assurance purposes. Evaluations are sorted by analyte grouping, analyte, and method.

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	Bifenthrin	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Chlorpyrifos	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Cyhalothrin, lambda, total	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Cypermethrin, total	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Deltamethrin	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Endrin	EPA 8081AM	RPD<25	7	6	85.71
Organochlorines	Esfenvalerate/Fenvalerate, total	EPA 8081AM	RPD<25	7	6	85.71
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	7	7	100.00
Organochlorines	Permethrin, total	EPA 8081AM	RPD<25	7	6	85.71
Organochlorines	Permethrin-1	EPA 8081AM	NA	NA	NA	NA
Organochlorines	Permethrin-2	EPA 8081AM	RPD<25	NA	NA	NA
Inorganics	Total Organic Carbon	KahnM	NA	NA	NA	NA
Metals	Arsenic	EPA 200.8	RPD<25	2	2	100.00
Metals	Cadmium	EPA 200.8	RPD<25	2	2	100.00
Metals	Copper	EPA 200.8	RPD<25	2	2	100.00
Metals	Lead	EPA 200.8	RPD<25	2	2	100.00
Metals	Nickel	EPA 200.8	RPD<25	2	2	100.00
Metals	Selenium	EPA 200.8	RPD<25	2	2	100.00
			TOTAL	**103**	**100**	**97.09**

Table 26. Summary of lab duplicate quality control sample evaluations for water samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	NA	NA	NA	NA
Carbamates	Carbaryl	EPA 632M	NA	NA	NA	NA
Carbamates	Carbofuran	EPA 632M	NA	NA	NA	NA
Carbamates	Methiocarb	EPA 632M	NA	NA	NA	NA
Carbamates	Methomyl	EPA 632M	NA	NA	NA	NA
Fungicides	Captan	NA	NA	NA	NA	NA
Acaricides	Propargite	NA	NA	NA	NA	NA
Herbicides	Alachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Ametryn	EPA 619M	NA	NA	NA	NA
Herbicides	Atraton	EPA 619M	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619	NA	NA	NA	NA
Herbicides	Atrazine	EPA 619M	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619	NA	NA	NA	NA
Herbicides	Cyanazine	EPA 619M	NA	NA	NA	NA
Herbicides	Diuron	EPA 632M	NA	NA	NA	NA
Herbicides	Linuron	EPA 632M	NA	NA	NA	NA
Herbicides	Metolachlor	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	EPA 619M	NA	NA	NA	NA
Herbicides	Molinate	WPCL Method 42	NA	NA	NA	NA
Herbicides	Norflurazon	EPA 619M	NA	NA	NA	NA
Herbicides	Oxyfluorfen	EPA 619M	NA	NA	NA	NA
Herbicides	Prometon	EPA 619M	NA	NA	NA	NA
Herbicides	Prometryn	EPA 619M	NA	NA	NA	NA
Herbicides	Propanil	EPA 619M	NA	NA	NA	NA
Herbicides	Propazine	EPA 619M	NA	NA	NA	NA
Herbicides	Prowl	EPA 619M	NA	NA	NA	NA
Herbicides	Secbumeton	EPA 619M	NA	NA	NA	NA
Herbicides	Simazine	EPA 619	NA	NA	NA	NA
Herbicides	Simazine	EPA 619M	NA	NA	NA	NA
Herbicides	Simetryn	EPA 619M	NA	NA	NA	NA
Herbicides	Terbuthylazine	EPA 619M	NA	NA	NA	NA
Herbicides	Terbutryn	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	EPA 619M	NA	NA	NA	NA
Herbicides	Thiobencarb	WPCL Method 42	NA	NA	NA	NA
Herbicides	Trifluralin	EPA 619M	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDD(o,p')	EPA 8081BM	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(p,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDD(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDE(o,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDE(o,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDE(p,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDE(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDT(o,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDT(o,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	DDT(p,p')	EPA 8081AM	NA	NA	NA	NA
Organochlorines	DDT(p,p')	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Dicofol	EPA 8081AM	NA	NA	NA	NA
Organochlorines	Dicofol	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Dieldrin	EPA 8081AM	NA	NA	NA	NA
Organochlorines	Dieldrin	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Endrin	EPA 8081AM	NA	NA	NA	NA
Organochlorines	Endrin	EPA 8081BM	NA	NA	NA	NA
Organochlorines	Methoxychlor	EPA 8081AM	NA	NA	NA	NA
Organochlorines	Methoxychlor	EPA 8081BM	NA	NA	NA	NA
Organophosphates	Azinphos methyl	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Chlorpyrifos	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Diazinon	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Dimethoate	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Disulfoton	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Malathion	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Methidathion	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Parathion, Ethyl	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Phorate	EPA 8141AM	RPD<25	1	1	100.00
Organophosphates	Phosmet	EPA 8141AM	RPD<25	1	1	100.00
Pyrethroids	Bifenthrin	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Bifenthrin	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-3	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyfluthrin-4	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Cypermethrin, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Cypermethrin-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin-2	EPA 1660M	NA	NA	NA	NA

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin-3	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Cypermethrin-4	EPA 1660M	NA	NA	NA	NA
	Deltamethrin	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Deltamethrin	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	NA	NA	NA	NA
Pyrethroids	Permethrin, total	EPA 1660M	RPD<25	1	1	100.00
Pyrethroids	Permethrin, total	EPA 8081BM	NA	NA	NA	NA
Pyrethroids	Permethrin-1	EPA 1660M	NA	NA	NA	NA
Inorganics	Ammonia as N	EPA 350.3	RPD<25	23	23	100.00
Inorganics	Ammonia as N	SM 4500 NH3 D	RPD<25	10	10	100.00
Inorganics	Color	QC 10308001A	RPD<25	14	14	100.00
Inorganics	Color	SM 2120 B	RPD<25	3	3	100.00
Inorganics	Color	SM 2120 BM	RPD<25	27	27	100.00
Inorganics	Dissolved Solids	SM 2540 C	RPD<25	57	57	100.00
Inorganics	Hardness as CaCO3	EPA 130.1	RPD<25	5	5	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	RPD<25	11	11	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	RPD<25	42	42	100.00
Inorganics	Nitrate + Nitrite as N	EPA 353.2	RPD<25	21	21	100.00
Inorganics	Nitrate + Nitrite as N	QC 10107041B	RPD<25	12	12	100.00
Inorganics	Nitrite as N	EPA 353.2	RPD<25	33	33	100.00
Inorganics	Nitrite as N	QC 10107041B	RPD<25	17	17	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	RPD<25	29	29	100.00
Inorganics	OrthoPhosphate as P	QC 10115011M	RPD<25	20	20	100.00
Inorganics	Total Organic Carbon	EPA 415.1	RPD<25	8	5	62.50
Inorganics	Total Organic Carbon	EPA 415.1M	RPD<25	10	10	100.00
Inorganics	Turbidity	SM 2130 B	RPD<25	212	210	99.06
THMs	Bromodichloromethane	EPA 8260	RPD<25	1	1	100.00
THMs	Bromoform	EPA 8260	RPD<25	1	1	100.00
THMs	Chloroform	EPA 8260	RPD<25	1	1	100.00
THMs	Dibromochloromethane	EPA 8260	RPD<25	1	1	100.00
Metals	Arsenic	EPA 1638	RPD<25	8	8	100.00
Metals	Boron	EPA 1638	RPD<25	7	5	71.43

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Metals	Cadmium	EPA 1638	RPD<25	8	8	100.00
Metals	Copper	EPA 1638	RPD<25	8	7	87.50
Metals	Lead	EPA 1638	RPD<25	8	8	100.00
Metals	Nickel	EPA 1638	RPD<25	8	8	100.00
Metals	Phosphorus as P	EPA 1638	RPD<25	8	8	100.00
Metals	Selenium	EPA 1638	RPD<25	8	6	75.00
Metals	Zinc	EPA 1638	RPD<25	8	8	100.00
Metals	Arsenic	EPA 1638M	RPD<25	38	38	100.00
Metals	Boron	EPA 1638M	RPD<25	37	29	78.38
Metals	Cadmium	EPA 1638M	RPD<25	38	34	89.47
Metals	Copper	EPA 1638M	RPD<25	38	33	86.84
Metals	Lead	EPA 1638M	RPD<25	38	37	97.37
Metals	Nickel	EPA 1638M	RPD<25	38	35	92.11
Metals	Phosphorus as P	EPA 1638M	RPD<25	38	38	100.00
Metals	Selenium	EPA 1638M	RPD<25	38	30	78.95
Metals	Zinc	EPA 1638M	RPD<25	38	35	92.11
			Total	986	944	95.74

Table 27. Summary of lab duplicate quality control sample evaluations for sediment samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	Bifenthrin	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Chlorpyrifos	EPA 8081AM	RPD<25	1	0	0.00
Organochlorines	Cyhalothrin, lambda, total	EPA 8081AM	RPD<25	1	0	0.00
Organochlorines	Cypermethrin, total	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	DDD(p,p')	EPA 8081AM	RPD<25	1	0	0.00
Organochlorines	DDE(p,p')	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	DDT(p,p')	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Deltamethrin	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Dieldrin	EPA 8081AM	RPD<25	1	0	0.00
Organochlorines	Endrin	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Esfenvalerate/Fenvalerate, total	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Methoxychlor	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Permethrin, total	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Permethrin-1	EPA 8081AM	RPD<25	1	1	100.00
Organochlorines	Permethrin-2	EPA 8081AM	RPD<25	1	1	100.00
Inorganics	Total Organic Carbon	KahnM	RPD<25	23	23	100.00
Metals	Arsenic	EPA 200.8	RPD<25	4	4	100.00
Metals	Cadmium	EPA 200.8	RPD<25	4	4	100.00
Metals	Copper	EPA 200.8	RPD<25	4	4	100.00
Metals	Lead	EPA 200.8	RPD<25	4	4	100.00
Metals	Nickel	EPA 200.8	RPD<25	4	4	100.00
Metals	Selenium	EPA	RPD<25	4	4	100.00

		200.8				
			TOTAL	62	58	93.55

Table 28. Summary of surrogate recovery quality control sample evaluations for water samples

AnalyteName	MethodName	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
BDMNMC, 4,3,5(Surrogate)	EPA 632M	PR 75-125	152	113	74.34
BDMNMC, 4,3,5(Surrogate)	EPA 632M	PR 75-125	30	24	80.00
Bromofluorobenzene, 4-(Surrogate)	EPA 8260	PR 75-125	78	78	100.00
Bromofluorobenzene, 4-(Surrogate)	EPA 8260	PR 75-125	12	12	100.00
DBOFB(Surrogate)DB-608	EPA 8081AM	PR 75-125	149	147	98.66
DBOFB(Surrogate)DB-608	EPA 8081AM	PR 75-125	17	17	100.00
DBOFB(Surrogate)HP-5	EPA 8081AM	PR 75-125	149	145	97.32
DBOFB(Surrogate)HP-5	EPA 8081AM	PR 75-125	17	15	88.24
Decachlorobiphenyl(Surrogate)DB-608	EPA 8081AM	PR 75-125	149	148	99.33
Decachlorobiphenyl(Surrogate)DB-608	EPA 8081AM	PR 75-125	17	17	100.00
Decachlorobiphenyl(Surrogate)HP-5	EPA 8081AM	PR 75-125	149	147	98.66
Decachlorobiphenyl(Surrogate)HP-5	EPA 8081AM	PR 75-125	17	17	100.00
Dibromofluoromethane(Surrogate)	EPA 8260	PR 75-125	78	78	100.00
Dibromofluoromethane(Surrogate)	EPA 8260	PR 75-125	12	12	100.00
Dibromooctafluorobiphenyl(Surrogate)	EPA 1660M	PR 75-125	537	522	97.21
Dibromooctafluorobiphenyl(Surrogate)	EPA 1660M	PR 75-125	54	53	98.15
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081AM	PR 75-125	525	510	97.14
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081AM	PR 75-125	51	49	96.08
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081BM	PR 75-125	306	304	99.35
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081BM	PR 75-125	60	56	93.33
Dichloroethane-d4, 1,2-(Surrogate)	EPA 8260	PR 75-125	78	78	100.00
Dichloroethane-d4, 1,2-(Surrogate)	EPA 8260	PR 75-125	12	12	100.00
Toluene-d8(Surrogate)	EPA 8260	PR 75-125	78	78	100.00
Toluene-d8(Surrogate)	EPA 8260	PR 75-125	12	12	100.00
Triphenyl phosphate(Surrogate)	EPA 619M	PR 75-125	580	574	98.97
Triphenyl phosphate(Surrogate)	EPA 619M	PR 75-125	78	75	96.15
Triphenyl phosphate(Surrogate)	EPA 8141AM	PR 75-125	689	678	98.40
Triphenyl phosphate(Surrogate)	EPA 8141AM	PR 75-125	85	81	95.29
		Total	**4171**	**4052**	**97.15**

Table 29. Summary of surrogate recovery quality control sample evaluations for sediment samples

Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
BDMNMC, 4,3,5(Surrogate)	EPA 632M	PR 75-125	182	137	75.27
Bromofluorobenzene, 4-(Surrogate)	EPA 8260	PR 75-125	90	90	100.00
DBOFB(Surrogate)DB-608	EPA 8081AM	PR 75-125	166	164	98.80
DBOFB(Surrogate)HP-5	EPA 8081AM	PR 75-125	166	160	96.39
Decachlorobiphenyl(Surrogate)DB-608	EPA 8081AM	PR 75-125	166	165	99.40
Decachlorobiphenyl(Surrogate)HP-5	EPA 8081AM	PR 75-125	166	164	98.80
Dibromofluoromethane(Surrogate)	EPA 8260	PR 75-125	90	90	100.00
Dibromooctafluorobiphenyl(Surrogate)	EPA 1660M	PR 75-125	591	575	97.29
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081AM	PR 75-125	576	559	97.05
Dibromooctafluorobiphenyl(Surrogate)	EPA 8081BM	PR 75-125	366	360	98.36
Dichloroethane-d4, 1,2-(Surrogate)	EPA 8260	PR 75-125	90	90	100.00
Toluene-d8(Surrogate)	EPA 8260	PR 75-125	90	90	100.00
Triphenyl phosphate(Surrogate)	EPA 619M	PR 75-125	658	649	98.63
Triphenyl phosphate(Surrogate)	EPA 8141AM	PR 75-125	774	759	98.06
		TOTAL	**4171**	**4052**	**97.15**

Table 30. Summary of water sample holding time evaluations for environmental, field blank, field duplicate and matrix spike samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Carbamates	Aldicarb	EPA 632M	<7 days	468	443	94.66
Carbamates	Carbaryl	EPA 632M	<7 days	468	443	94.66
Carbamates	Carbofuran	EPA 632M	<7 days	469	443	94.46
Carbamates	Methiocarb	EPA 632M	<7 days	469	443	94.46
Carbamates	Methomyl	EPA 632M	<7 days	469	443	94.46
Fungicides	Captan	EPA 632M	<7 days	439	414	94.31
Acaricides	Propargite	EPA 619M	<7 days	123	123	100.00
Herbicides	Alachlor	EPA 619M	<7 days	123	123	100.00
Herbicides	Ametryn	EPA 619M	<7 days	123	123	100.00
Herbicides	Atraton	EPA 619M	<7 days	123	123	100.00
Herbicides	Atrazine	EPA 619	<7 days	6	6	100.00
Herbicides	Atrazine	EPA 619M	<7 days	555	475	85.59
Herbicides	Cyanazine	EPA 619	<7 days	6	6	100.00
Herbicides	Cyanazine	EPA 619M	<7 days	555	475	85.59
Herbicides	Diuron	EPA 632M	<7 days	469	443	94.46
Herbicides	Linuron	EPA 632M	<7 days	469	443	94.46
Herbicides	Metolachlor	EPA 619M	<7 days	123	123	100.00
Herbicides	Molinate	EPA 619M	<7 days	555	475	85.59
Herbicides	Molinate	WPCL Method 42	<7 days	6	6	100.00
Herbicides	Norflurazon	EPA 619M	<7 days	123	123	100.00
Herbicides	Oxyfluorfen	EPA 619M	<7 days	123	123	100.00
Herbicides	Prometon	EPA 619M	<7 days	123	123	100.00
Herbicides	Prometryn	EPA 619M	<7 days	123	123	100.00
Herbicides	Propanil	EPA 619M	<7 days	123	123	100.00
Herbicides	Propazine	EPA 619M	<7 days	123	123	100.00
Herbicides	Prowl	EPA 619M	<7 days	123	123	100.00
Herbicides	Secbumeton	EPA 619M	<7 days	123	123	100.00
Herbicides	Simazine	EPA 619	<7 days	6	6	100.00
Herbicides	Simazine	EPA 619M	<7 days	555	475	85.59
Herbicides	Simetryn	EPA 619M	<7 days	123	123	100.00
Herbicides	Terbuthylazine	EPA 619M	<7 days	123	123	100.00
Herbicides	Terbutryn	EPA 619M	<7 days	123	123	100.00
Herbicides	Thiobencarb	EPA 619M	<7 days	555	475	85.59
Herbicides	Thiobencarb	WPCL Method 42	<7 days	6	6	100.00
Herbicides	Trifluralin	EPA 619M	<7 days	123	123	100.00
Organochlorines	DDD(o,p')	EPA 8081AM	<7 days	506	435	85.97
Organochlorines	DDD(o,p')	EPA 8081BM	<7 days	506	435	85.97

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	DDD(p,p')	EPA 8081AM	<7 days	506	435	85.97
Organochlorines	DDD(p,p')	EPA 8081BM	<7 days	506	435	85.97
Organochlorines	DDE(o,p')	EPA 8081AM	<7 days	506	435	85.97
Organochlorines	DDE(o,p')	EPA 8081BM	<7 days	506	435	85.97
Organochlorines	DDE(p,p')	EPA 8081AM	<7 days	506	435	85.97
Organochlorines	DDE(p,p')	EPA 8081BM	<7 days	506	435	85.97
Organochlorines	DDT(o,p')	EPA 8081AM	<7 days	506	435	85.97
Organochlorines	DDT(o,p')	EPA 8081BM	<7 days	506	435	85.97
Organochlorines	DDT(p,p')	EPA 8081AM	<7 days	144	126	87.50
Organochlorines	DDT(p,p')	EPA 8081BM	<7 days	144	126	87.50
Organochlorines	Dicofol	EPA 8081AM	<7 days	144	126	87.50
Organochlorines	Dicofol	EPA 8081BM	<7 days	144	126	87.50
Organochlorines	Dieldrin	EPA 8081AM	<7 days	144	126	87.50
Organochlorines	Dieldrin	EPA 8081BM	<7 days	144	126	87.50
Organochlorines	Endrin	EPA 8081AM	<7 days	144	126	87.50
Organochlorines	Endrin	EPA 8081BM	<7 days	144	126	87.50
Organochlorines	Methoxychlor	EPA 8081AM	<7 days	144	126	87.50
	Methoxychlor	EPA 8081BM	<7 days	144	126	87.50
Organophosphates	Azinphos methyl	EPA 8141AM	<7 days	657	655	99.70
Organophosphates	Chlorpyrifos	EPA 8141AM	<7 days	657	655	99.70
Organophosphates	Diazinon	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Dimethoate	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Disulfoton	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Malathion	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Methidathion	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Parathion, Ethyl	EPA 8141AM	<7 days	642	642	100.00
Organophosphates	Parathion, Methyl	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Phorate	EPA 8141AM	<7 days	658	656	99.70
Organophosphates	Phosmet	EPA 8141AM	<7 days	658	656	99.70
Pyrethroids	Bifenthrin	EPA 1660M	<7 days	513	439	85.58
Pyrethroids	Bifenthrin	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Cyfluthrin-1	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cyfluthrin-2	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cyfluthrin-3	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cyfluthrin-4	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cyhalothrin, lambda, total	EPA 1660M	<7 days	25	25	100.00
Pyrethroids	Cyhalothrin, lambda, total	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Cyhalothrin, lambda-1	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cyhalothrin, lambda-2	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cypermethrin, total	EPA 1660M	<7 days	156	156	100.00
Pyrethroids	Cypermethrin, total	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Cypermethrin-1	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cypermethrin-2	EPA 1660M	<7 days	357	283	79.27

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Pyrethroids	Cypermethrin-3	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Cypermethrin-4	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Deltamethrin	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Deltamethrin	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 1660M	<7 days	156	156	100.00
Pyrethroids	Esfenvalerate/Fenvalerate, total	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Esfenvalerate/Fenvalerate-1	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Esfenvalerate/Fenvalerate-2	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Permethrin, total	EPA 1660M	<7 days	156	156	100.00
Pyrethroids	Permethrin, total	EPA 8081BM	<7 days	144	126	87.50
Pyrethroids	Permethrin-1	EPA 1660M	<7 days	357	283	79.27
Pyrethroids	Permethrin-2	EPA 1660M	<7 days	357	283	79.27
Inorganics	Ammonia as N	EPA 350.3	48 hours	513	511	99.61
Inorganics	Ammonia as N	SM 4500 NH3 D	48 hours	144	144	100.00
Inorganics	Color	QC 10308001A	48 hours	149	147	98.66
Inorganics	Color	SM 2120 B	48 hours	10	10	100.00
Inorganics	Color	SM 2120 BM	48 hours	531	530	99.81
Inorganics	Dissolved Solids	SM 2540 C	48 hours	598	596	99.67
Inorganics	Hardness as CaCO3	EPA 130.1	48 hours	64	64	100.00
Inorganics	Hardness as CaCO3	QC 10301311B	48 hours	150	150	100.00
Inorganics	Hardness as CaCO3	SM 2340 C	48 hours	390	390	100.00
Inorganics	Nitrate + Nitrite as N	EPA 353.2	48 hours	497	92	18.51
Inorganics	Nitrate + Nitrite as N	QC 10107041B	48 hours	151	146	96.69
Inorganics	Nitrite as N	EPA 353.2	48 hours	533	533	100.00
Inorganics	Nitrite as N	QC 10107041B	48 hours	156	156	100.00
Inorganics	OrthoPhosphate as P	EPA 365.1M	48 hours	511	504	98.63
Inorganics	OrthoPhosphate as P	QC 10115011M	48 hours	181	181	100.00
Inorganics	Total Organic Carbon	EPA 415.1	28 days	399	382	95.74
Inorganics	Total Organic Carbon	EPA 415.1M	28 days	229	227	99.13
Inorganics	Turbidity	SM 2130 B	48 hours	598	592	99.00
THMs	Bromodichloromethane	EPA 8260	<14 days	81	81	100.00
THMs	Bromoform	EPA 8260	<14 days	81	81	100.00
THMs	Chloroform	EPA 8260	<14 days	81	81	100.00

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
THMs	Dibromochloromethane	EPA 8260	<14 days	81	81	100.00
Metals	Arsenic	EPA 1638	<6 months	86	86	100.00
Metals	Arsenic	EPA 1638M	<6 months	79	79	100.00
Metals	Boron	EPA 1638	<6 months	86	86	100.00
Metals	Boron	EPA 1638M	<6 months	86	86	100.00
Metals	Cadmium	EPA 1638	<6 months	86	86	100.00
Metals	Cadmium	EPA 1638M	<6 months	86	86	100.00
Metals	Copper	EPA 1638	<6 months	86	86	100.00
Metals	Copper	EPA 1638M	<6 months	86	86	100.00
Metals	Lead	EPA 1638	<6 months	86	86	100.00
Metals	Lead	EPA 1638M	<6 months	442	399	90.27
Metals	Nickel	EPA 1638	<6 months	448	405	90.40
Metals	Nickel	EPA 1638M	<6 months	442	399	90.27
Metals	Phosphorus as P	EPA 1638	<6 months	442	399	90.27
Metals	Phosphorus as P	EPA 1638M	<6 months	442	399	90.27
Metals	Selenium	EPA 1638	<6 months	442	399	90.27
Metals	Selenium	EPA 1638M	<6 months	442	399	90.27
Metals	Zinc	EPA 1638	<6 months	442	399	90.27
Metals	Zinc	EPA 1638M	<6 months	442	399	90.27
			Total	40440	36797	90.99

Table 31. Summary of sediment sample holding time evaluations for environmental, field blank, field duplicate and matrix spike samples

Ag Grouping	Analyte Name	Method Name	Data Quality Objective	Number of Samples	Samples Within Control Limits	Percent Samples Acceptable
Organochlorines	Bifenthrin	EPA 8081AM	<7 days	137	133	97.08
Organochlorines	Chlorpyrifos	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	Cyhalothrin, lambda, total	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	Cypermethrin, total	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	DDD(p,p')	EPA 8081AM	<7 days	134	130	97.01
Organochlorines	DDE(p,p')	EPA 8081AM	<7 days	131	127	96.95
Organochlorines	DDT(p,p')	EPA 8081AM	<7 days	137	133	97.08
Organochlorines	Deltamethrin	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	Dieldrin	EPA 8081AM	<7 days	134	130	97.01
Organochlorines	Endrin	EPA 8081AM	<7 days	131	127	96.95
Organochlorines	Esfenvalerate/Fenvalerate, total	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	Methoxychlor	EPA 8081AM	<7 days	131	127	96.95
Organochlorines	Permethrin, total	EPA 8081AM	<7 days	136	132	97.06
Organochlorines	Permethrin-1	EPA 8081AM	<7 days	127	127	100.00
Organochlorines	Permethrin-2	EPA 8081AM	<7 days	127	127	100.00
Inorganics	Total Organic Carbon	KahnM	<7 days	121	121	100.00
Metals	Arsenic	EPA 200.8	<6 months	22	1	4.55
Metals	Cadmium	EPA 200.8	<6 months	22	1	4.55
Metals	Copper	EPA 200.8	<6 months	22	1	4.55
Metals	Lead	EPA 200.8	<6 months	22	1	4.55
Metals	Nickel	EPA 200.8	<6 months	22	1	4.55
Metals	Selenium	EPA	<6 months	22	1	4.55

		200.8				
			TOTAL	2258	2080	92.12

Table 32. Summary of water toxicity field duplicate sample evaluations

Toxicity Species	Method	Data Quality Objective (DQO)	Total Field Duplicate Samples	Total Number Sample Within DQO	Percent Samples Within Acceptable Criteria
Ceriodaphnia dubia	EPA 821/R-02-012	RPD<25	19	19	100
Ceriodaphnia dubia	EPA 821/R-02-012 mod	RPD<25	3	3	100
Hyalella azteca	EPA 600/R-99-064	RPD<25	8	8	100
Pimephales promelas	EPA 821/R-02-012	RPD<25	19	19	100
Pimephales promelas	EPA 821/R-02-012 mod	RPD<25	3	3	100
Selenastrum capricornutum	EPA 821/R-02-013	RPD<25	16	16	100
Selenastrum capricornutum	EPA 821/R-02-013 mod	RPD<25	3	3	100
		Total	**71**	**71**	**100.00**

Table 33. Summary of water toxicity field blank quality control sample evaluations

Toxicity Species	Method	Data Quality Objective (DQO)	Total Field Blank QC Samples	Total Number Sample Within DQO	Percent Samples Within Acceptable Criteria
EPA 821/R-02-012	Ceriodaphnia dubia	Not significantly different than the control	19	19	100.00
EPA 821/R-02-012	Pimephales promelas	Not significantly different than the control	19	19	100.00
EPA 821/R-02-012 mod	Ceriodaphnia dubia	Not significantly different than the control	3	3	100.00
EPA 821/R-02-012 mod	Pimephales promelas	Not significantly different than the control	3	3	100.00
EPA 821/R-02-013	Selenastrum capricornutum	Not significantly different than the control	16	16	100.00
EPA 821/R-02-013 mod	Selenastrum capricornutum	Not significantly different than the control	3	3	100.00
		Total	**63**	**63**	**100.00**

Table 34. Summary of water toxicity sample holding time evaluations

Toxicity Species	Method	Data Quality Objective (DQO)	Total Toxicity Samples	Total Number Sample Within DQO	Percent Samples Within Acceptable Criteria
Ceriodaphnia dubia	EPA 821/R-02-012	<36 hours	466	466	100.00
Ceriodaphnia dubia	EPA 821/R-02-012 mod	<36 hours	109	109	100.00
Hyalella azteca	EPA 600/R-99-064	<36 hours	202	116	57.43
Pimephales promelas	EPA 821/R-02-012	<36 hours	464	464	100.00
Pimephales promelas	EPA 821/R-02-012 mod	<36 hours	109	109	100.00
Selenastrum capricornutum	EPA 821/R-02-013	<36 hours	406	402	99.01
Selenastrum capricornutum	EPA 821/R-02-013 mod	<36 hours	109	109	100.00
		Total	**1865**	**1775**	**95.17**

Table 35. Summary of sediment toxicity field duplicate sample evaluations

Toxicity Species	Method	Data Quality Objective (DQO)	Total Field Duplicate Samples	Total Number Sample Within DQO	Percent Samples Within Acceptable Criteria
EPA 600/R-99-064	Hyalella azteca	RPD<25	8	8	100.00

Table 36. Summary of sediment toxicity sample holding time evaluations

Toxicity Species	Method	Data Quality Objective (DQO)	Total Toxicity Samples	Total Number Sample Within DQO	Percent Samples Within Acceptable Criteria
EPA 600/R-99-064	Hyalella azteca	<14 days	226	141	62.39

APPENDIX 6: AquaScience Environmental Toxicology Consultants. Identification of Causes of Toxicity to *Ceriodaphnia dubia* and Green Algae (*Selenastrum capricornutum*) in Agriculture-Dominated Discharge Samples from the San Joaquin Watershed, Northern California

Identification of Causes of Toxicity to *Ceriodaphnia dubia a*nd Green Algae *(Selenastrum capricornutum)* in Agriculture-Dominated Discharge Samples from the San Joaquin Watershed, Northern California

FINAL REPORT

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Identification of Causes of Toxicity to *Ceriodaphnia dubia a*nd Green Algae *(Selenastrum capricornutum)* in Agriculture-Dominated Discharge Samples from the San Joaquin Watershed, Northern California

1.0 EXECUTIVE SUMMARY

A total of 150 samples collected from the San Joaquin River water shed, Northern California, during two winter events (January-February 2005) and four irrigation events (May-August 2005) were screened for toxicity to green algae *(Selenastrum capricornutun)*, the invertebrate *Ceriodaphnia dubia*, and larval fathead minnows *(Pimephales promelas)*. Toxicity was detected in 11 of the algae tests (7%), 20 of the *C. dubia* tests (13%), and 3 (2%) of the fathead minnow tests. Toxicity Identification Evaluations (TIEs) were conducted on 3 samples that caused algal chronic toxicity and 16 samples that caused acute *C. dubia* toxicity to identify the cause(s) of the toxicity. The TIEs identified the chemical(s) causing the majority of *C. dubia* toxicity in 14 of 16 (88%) of the samples evaluated. The chemicals causing toxicity included chlorpyrifos (11 samples), diazinon (6 samples), carbaryl (3 samples) and malathion (2 samples). However, the algae TIEs failed to identify the chemical(s) responsible for the majority of the toxicity in the three samples evaluated. Overall, TIEs were effective in identifying the causes of acute *C. dubia* toxicity in the majority of samples evaluated. However, there is need to develop TIE profiles for many pesticides commonly detected in agricultural discharges, to develop information on toxic interactions for the chemicals that co-occur in agricultural discharges, and to refine analytical procedures for identification of the cause(s) of algal toxicity.

2.0 INTRODUCTION

AQUA-Science (A-S) was retained under contract to the University of California, Davis to conduct aquatic toxicity tests and toxicity identification evaluations (TIEs) on agricultural-dominated discharges collected in response to monitoring requirements of the Conditional Waiver of Waste Discharge for Irrigated Lands (Ag Waiver). A total of 150 samples collected during two storm events (January-February, 2006), and four irrigation events (May-August, 2005) were tested using three-species toxicity tests with the cladoceran (*Ceriodaphnia dubia*), green algae (*Selenastrum capricornutum*), and larval fathead minnows (*Pimephales promelas*). TIEs were conducted on 3 samples that demonstrated toxicity to algae and 16 samples that demonstrated toxicity *C. dubia*. Results of these analyses are reported herein.

3.0 materials and methods

3.1 Test Samples

Test samples (4 gallon/site) were subsurface grabs obtained by UC Davis sampling personnel under the direction of Dr. Mike Johnson. Sample locations are shown in Table 1. Field measurements included temperature, dissolved oxygen (DO), conductivity, and flow. Samples were placed in ice chests with sufficient wet ice to maintain sample temperature at ≤6 °C. Upon arrival at A-S, temperature, DO, conductivity, hardness, alkalinity, and pH were measured. Samples were stored in the dark at 4 °C until tested, within 24 hours of collection.

Table 1. Location of TIE Samples

Sample Code	*Sample Location*	*GPS Coordinates*
CS01	Tributary Home Colony Canal	39.78425, -122.19659
CS12	Unnamed Drain Walker Creek on Country Road 28	39.66846, -122.22385
CS15	Spring Creek at Walnut Creek	39.11975, -122.19318
CS23	Spring Creek at East Camp Road	39.10878, -122.21082
CS24	Drain to Walker Creek at County Road F	39.67449, -122.23312
SS05A	North Main Canal at Sankey Road	38.77978, -121.53259
FT24	Elk Bayou at Road 96	36.12429, -119.35671
NSJ31	Calaveras River at Pezzi Road	38.04536, -121.19982
NSJ32	Bear Creek at Alpine Road	38.07402, -121.21093
SS06	Winters Canal at Road 86A	38.66366, -122.01609
SSJ03	Willow Slough at Road 99	38.60471, -121.78422
SSJ03	Willow Slough at Road 99	38.60471, -121.78422
SSJ04	Unnamed Ditch at SW corner of Levee Road and Riego Road	38.75116, -121.49370
SSJ07	West Adams Canal Road 89	38.70488, -121.96093

3.2 Toxicity Test Design

Acute toxicity tests with *C. dubia* and fathead minnows were conducted according to USEPA (2002a) acute protocols, while algae toxicity tests utilized the USEPA (2002b) chronic protocol. Initially, samples were tested without dilution. If toxicity was exhibited in the screening tests, a

dilution series test consisting of 5-7 test concentrations was conducted using lab water as the diluent. Lab water was reverse-osmosis and carbon-treated well water amended with dry salts to USEPA moderately hard (EPAMH) specifications (pH 7.4-7.8, hardness 80-100 mg/L, alkalinity 60-70 mg/L). Sites that exhibited toxicity were resampled and tested for toxicity to the affected species within 2 days of completion of the screening tests.

3.3 Algae Toxicity Tests

Each test concentration included four replicate 125-mL flasks containing 50 mL of test sample. The flasks, containing algal assay media without EDTA, were inoculated with 1 x 10^4 cells/mL of a culture of *S. capricornutum* in log phase growth (University of Texas Algae Type Collection, Austin, TX, USA). Flasks were placed on a shaker table (100 rpm) in an environmental chamber at 25 ± 1 °C with continuous lighting (400 ± 40 ft-c_ and were randomized twice daily. After the 96-hour test period, cells were enumerated with an electronic particle counter (Model Z1 Coulter Counter™, Beckman Coulter Inc., Brea, CA, USA). Tests were considered invalid if the final control cell count was < 2 x 10^5 cells/mL or the percent coefficient of variation (%CV) in the control was >20%.

3.4 *Ceriodaphnia dubia* Toxicity Tests

Test organisms were <24-hour neonate *C. dubia* collected within an 8-hour period from A-S cultures. Each test sample was tested using four replicates of 5 neonates each in 20-mL glass scintillation vials containing 18 mL of test solution. *C. dubia* were fed a mixture of green algae (*S. capricornutum*) and YTC (a mixture of yeast, organic alfalfa and trout chow) two hours prior to daily renewal of test solutions. Tests were conducted at 25 ± 2 °C with a 16-hour light:8-hour dark photoperiod. Mortality was noted daily.

3.5 Fathead Minnow Toxicity Tests

Fathead minnows were obtained from Aquatox, Inc. (Hot Springs, AK, USA) and maintained in EPAMH lab water until tested when 6-10 days old. Fish were fed *Artemia* nauplii once daily 2 hours prior to sample renewal. Test chambers were in 400 mL glass beakers containing 250 mL of test solution. Tests were conducted at 25 ± 2 °C with a 16-hour light:8-hour dark photoperiod. Test samples were renewed daily and mortality was noted. Due to the absence of or very low mortality observed in the ambient samples, no dilution series tests or TIEs were conducted on fathead minnows.

3.6 Chemicals and Reagents

Pesticide analytical standards (≥99% pure) obtained from AccuStandard (New Haven, CT) were diluted in HPLC-grade methanol. All other reagents and chemicals were obtained from Fisher Scientific (Pittsburg, PA) and were the highest purity available.

3.7 Water Quality Measurements

Meter calibration/verification and water quality measurements followed the recommended procedures described by the SWRCB QAMP[1]. Temperature was measured in initial and 48-hour test solutions at change-out with a calibrated digital thermometer (Central Co., Friendswood, TX). Temperature was continuously recorded in all bioassay test chambers with a Dickson pen recorder (Model ICT855, Addison, IL). D.O. (YSI Model 550A, Yellow Springs, OH), pH (Beckman 240, Fulton, CO), and conductivity (WTW Model 330, Ft. Myers, FL) were measured in the initial and 48-hour test solutions at change-out. Alkalinity and hardness were measured with Hach colorimetric tests (Hach Co., Loveland, CO). Ammonia was measured in the initial sample using a Hach DR-700 Colorimeter (Method 8038).

3.8 Statistical Analysis

Each test sample was subjected to statistical analysis using ToxCalc v. 5.23 (Tidepool Scientific, McKinleyville, CA, USA) according to USEPA procedures (USEPA 2002a,b) to determine if the observed effect was statistically different ($p<0.05$) from the control. In the TIEs, mortality data from the dilution series tests were used to estimate EC_{25}/IC_{25} values, e.g., the calculated concentration of the test sample that results a 25% effect on the test endpoint. Toxic units (TUs) were calculated from the LC_{25}/IC_{25} values ($100/IC_{25}$ or EC_{25}).

3.9 Chemical Analysis

All test samples were subjected to chemical analysis for target constituents using gas-liquid chromatography/mass spectroscopy (GC/MS) and/or high performance liquid chromatography/mass spectroscopy (HPLC/MS) at the California Department of Fish and Game Water Pollution Control Laboratory (CDFG-WPCL; Rancho Cordova, CA, Appendix I).

[1] SWRCB. 2002. Quality Assurance Management Plan for the State of California's Surface Water Ambient Monitoring Program. Division of Water Quality. Sacramento, CA.

3.10 Toxicity Identification Evaluations (TIEs)

The purpose of the Phase I TIE is to identify the chemical class of the toxicant(s) (USEPA 1991). The purpose of the Phase II TIE is to gain the identity of the material(s) responsible for the sample toxicity (USEPA 1993a). The purpose of the Phase III TIE is to determine if there is a robust relationship between the concentrations of the suspected toxicant(s) identified in Phase II, and the amount of toxicity measured in the test sample (USEPA 1993b).

3.10.1 Algae Phase I TIEs

Algae Phase I TIEs consisted of a dilution series toxicity test to determine the toxic units (TUs; $100/EC_{25}$), solid phase extraction (SPE) column treatment for non-polar organic chemicals (NPOs), and ethylenediamine tetrachloroacetic acid (EDTA) addition for cationic metals. In some TIEs, samples were aerated for detection of volatile toxicants. The SPE column was eluted three times with 1-mL of methanol. Because algae are affected by even low concentrations of organic solvent (Miller et al, 2005), the acetonitrile (ACN) eluate from the SPE column was reduced to dryness using a gentle stream of nitrogen and reconstituted in the SPE through-column sample at 1X or 1.5X. The algae Phase I TIE flow chart is shown in Figure 1 (Miller et al, 2005).

3.10.2 *C. dubia* Phase I TIEs

The Phase I *C. dubia* TIE incorporated EDTA addition, SPE column treatment, and piperonyl butoxide (PBO) addition. PBO binds *in vivo* with mixed-function oxidase enzymes that metabolize non-polar organic chemicals such as organophosphorous (OP) and pyrethroid insecticides. With OPs, PBO prevents the metabolism to their toxic oxone form, decreasing/preventing toxicity (Ankley et al, 1991). With pyrethroids, PBO prevents metabolism to less toxic forms, increasing/prolonging toxicity (Wheelock et al, 2005). The *C. dubia* Phase I TIE flow chart is shown in Figure 2.

3.10.3 Phase II TIEs

Test samples that exhibited reduced toxicity following SPE treatment were subjected to fractionation to facilitate chemical identification (USEPA 1993a). Algae and *C. dubia* Phase II TIEs consisted of concentration of the SPE column methanol eluate that was obtained in Phase I, followed by fraction of this eluate by high performance liquid chromatography (HPLC) as shown in Figures 3 and 4, respectively. A total of 30 1-mL fractions were collected for each eluate and

tested for toxicity at 1.5X. The toxic HPLC fractions were chemically analyzed by the CDFG-WPCL to identify the chemical toxicants.

3.10.4 Phase III TIEs

In the Phase III TIE, the sample toxic units (TUs; 100/ EC_{25}) measured in the dilution series toxicity test is compared with the TU calculated from the detected concentration of the suspect toxicants [(predicted TUs = measured conc (µg/L)/suspect chemical EC_{25} or EC_{50}); USPEA 1993b]. If more than one suspect toxicant was present, the TUs were summed if there was evidence that they act jointly, e.g., diazinon and chlorpyrifos (Bailey et al, 1997). Although the USEPA TIE guidance has not established a minimum level of agreement between measured TU and predicted TU in a sample, our experience indicates that these values should agree within ±40% [1-(predicted TU/measured TU x 100)], or the sample should be subjected to further Phase II TIE evaluation. The Phase III TIE requires reliable information on the toxicity of candidate toxicants to the test species. Unfortunately, query of the ECOTOX database (http://cfpub.epa.gov/ecotox/) and a search of the scientific literature did not, in most cases, provide toxicity information for the chemicals of interest. Therefore, we conducted toxicity tests on algae and *C. dubia* with herbicides and insecticides, respectively, for which no toxicity information could be located as described in Section 3.3 and 3.4, respectively.

4.0 results and discussion

4.1 Phase I TIEs

The results of toxicity tests and Phase I TIEs on the 19 test samples that demonstrated toxicity to algae or *C. dubia* are summarized in Table 2.

Table 2. Summary of Phase I TIE Results on Samples that Caused Toxicity to Algae or *C. dubia*.

Test Species	*Sample No.*	*TU*[a]	*Effect on Sample Toxicity*					*Toxicant Chemical Class(es)*[f]
			Decreased by SPE[b]	*Recovered in SPE Add-Back*[c]	*Decreased by PBO*[d]	*Decreased by EDTA*[e]	*Decreased by Aeration*	
Algae	1	3.8	Yes	Yes	--[g]	No	--	NPO
	2	5.4	Yes	Partial	--	No	--	NPO
	3	10.7	Yes	Yes	--	No	--	NPO
C. dubia	4	1.3	Yes	Partial[h]	Yes	No	No	OP
	5	2.7	Yes	Yes	Majority[i]	No	Partial	OP + NPO
	6	10.7	Yes	Yes	Majority	No	No	OP
	7	1.3	Yes	Yes	Yes	--	--	OP
	8	5.3	Yes	Yes	Yes	No	No	OP
	9	21.3	Yes	Yes	Majority	No	No	OP
	10	2.7	Yes	Yes	Partial	--	--	OP + NPO
	11	22.7	Yes	Yes	Majority	No	--	OP
	12	1.3	Yes	Yes	Yes	--	--	OP
	13	1.0	Yes	Yes	Yes	--	--	OP
	14	~1.0	Yes	No	Yes	--	--	OP
	15	1.3	Yes	No	Synergized	--	--	Pyrethroid
	16	2.7	Yes	Yes	Yes	--	--	OP
	17	4.7	Yes	Partial	Yes	--	--	OP
	18	5.3	Yes	Yes	Yes	--	--	OP
	19	1.3	Yes	Partial	Yes	--	--	OP

a TU = 100/EC_{25} (algae) or 100/LC_{25} (*C. dubia*) determined by dilution series toxicity test
b SPE = solid-phase extraction column
c SPE add-back = SPE column eluted with 3 x 1-mL methanol and added back to lab water at 1X or 1.5X
d PBO = piperonyl butoxide
e EDTA = ethylene diaminetetrachloracetic acid
f NPO = non-polar organic(s); OP = metabolically-activated organophosphorous insecticide
g -- = indicated TIE treatment not tested
h Toxicity decreased but not eliminated by TIE treatment
i Majority of toxicity eliminated by PBO addition

4.1.1 Algae TIEs

Algae Phase I TIEs were conducted on Samples 1-3 that contained 3.8-10.8 TUc (Table 2). In all three samples, the toxicity was completely removed by SPE column treatment, and the toxicity was either fully or partially recovered in the SPE column eluate add-back. PBO

treatment was not used in the algae TIEs because algae (lacking a nervous system) are insensitive to insecticides. Collectively, the results suggest that one or more non-polar organics (NPOs) were responsible for the algal toxicity of these samples.

4.1.2 *C. dubia* TIEs

C. dubia Phase I TIEs were conducted on sixteen samples (Table 2). The toxicity of these samples ranged from ~1 to 27.6 TUc. SPE column treatment removed all the detectable toxicity in each of the samples, and the toxicity was fully or partially recovered in the SPE column eluate add-back at 1X or 1.5X. The results suggest NPOs were responsible for the toxicity of all of the samples examined. Furthermore, PBO addition prevented all or most of the toxicity in all but one sample (Sample 15), suggesting that OP insecticides, which require metabolic activation (e.g., phosphothioates such as diazinon and chlorpyrifos), were responsible for the toxicity of these samples (Ankley et al, 1991). The reason for substantial but not complete protection of toxicity in Samples 6, 9 and 11 by PBO is that PBO can only protect 6-10 TUc of OP toxicity (Bailey et al, 1996). PBO increased the toxicity of Sample 15, suggesting that one or more pyrethroid insecticides were present in the sample since PBO synergizes the toxicity of this class of chemical (Wheelock et al, 2004). PBO addition to Sample 5, 10 and 11 delayed but did not prevent the onset of toxicity, suggesting the sample contained both OPs and NPOs.

4.2 Phase III TIEs

Phase III TIEs require three types of information: 1) the measured toxicity of the sample using a dilution series toxicity test, e.g., TUs; 2) the identity and concentration of potential toxicant(s) in the test sample; and 3) the toxicity of the potential toxicant(s) to the test species. Each of the toxic samples was analyzed by the DFG WPCL for selected OP insecticides, carbamates and herbicides (see Appendix I) for the list of analytes. However, in many cases there was no published information on the toxicity of the chemicals detected in the samples to the species of interest. Without this information it is impossible to ascertain if the measured concentration(s) of the suspected toxicant(s) in the test samples can account for the sample TUs. Therefore, we conducted toxicity tests on the detected insecticides and herbicides using *C. dubia* and algae, respectively (Table 3).

Table 3. Summary of Toxicity of Selected Insecticides and Herbicides

Chemical Category (Test Species)	*Chemical*	*96-hr Endpoint Value*[a]			*96-hr ECOTOX LC_{50} Values (µg/L)*
		NOEC	*LC_{25}/EC_{25}*	*LC_{50}/EC_{50}*	
Insecticide (*C. dubia*)	Azinphos methyl	0.56	0.67	0.78	n/a[b]
	Carbaryl	3.2	2.8	3.9	8.3-9.7
	Chlorpyrifos	0.025	0.040	0.08	0.05-0.06
	Diazinon	0.10	0.18	0.38	0.32-0.40
	Methomyl	5.6	6.3	7.6	n/a
	Malathion	0.56	0.78	1.0	n/a
	Methyl parathion	0.56	0.78	1.0	n/a
Herbicide (*S. capricornutum*)	Chlorathalonil	1.0	21.4	53.5	n/a
	Diuron	1.0	3.4	7.0	n/a
	Metolachlor	100	> 100	> 100	n/a
	Oxyfluofen	< 1	1.0	2.6	n/a
	Pendimethalin	1.0	21.4	53.5	n/a
	Trifuralin	1.0	0.7	3.7	n/a

a Values are the results of one or more toxicity tests with the indicated species and chemical
b n/a = Value not available in the ECOTOX database (http://cfpub.epa.gov/ecotox/)

The insecticides had 96-hour acute *C. dubia* EC_{50} values ranging over two orders of magnitude from 0.080 to 7.6 µg/L. Among the insecticides, chlorpyrifos was the most toxic, followed by diazinon, azinphos methyl, methyl parathion, malathion, carbaryl and methomyl. The herbicides had 96-hour chronic *S. capricornutum* IC_{50} values that ranged from 3.7 to >100 µg/L, with oxyfluofen being the most toxic, followed by diuron, chlorathalonil, pendimethalin and metolachlor.

To validate the EC_{50} values generated in this study, we queried the USEPA ECOTOX database for the toxicity of each insecticide and herbicide of interest to *C. dubia* and algae, respectively. 96-hour *C. dubia* LC_{50} values were only available for three insecticides: carbaryl, chlorpyrifos and diazinon. The ECOTOX LC_{50} values matched those developed in this study within a factor of approximately 2 or less in all cases (Table 3). There were no *S. capricornutum* 96-hour IC_{50} values reported by ECOTOX for any of the herbicides. We anticipate that toxicity values developed in this work will be useful to others conducting TIEs because they were developed under the same toxicity test conditions that are used in many ambient monitoring programs, such as those conducted by the agricultural discharger coalitions, the CVRWQCB and other Regional Boards.

4.2.1 Algae Phase III TIE

Table 4 shows the Phase III analysis for the three samples that caused toxicity to algae. For Sample 1, chemicals detected in the sample accounted for 1.0 of the 3.8 (26%) of the TUc detected in the sample. Similarly, chemicals detected in Sample 2 accounted for only 20% of the measured toxicity. No chemicals were quantitated by the CDFG-WPCL in Sample 3, which was puzzling since this sample produced 10.8 TUc. All three samples were fractionated by HPLC (Figure 3) and toxic fractions were analyzed by the CDFG-WPCL in an effort to identify other toxicants and/or refine estimate of toxicant concentrations.

Table 4. Algae Phase III TIE Analyses

Test Sample (Date) Sample No.	*Chemical Detected*	*Conc. (µg/L)*[a]	*Pred. TUc*[b]	*Total Pred. TUc*[c]	*Measured TUc*[d]	*TUc Accounted for*[e]
CS15 (2/16/05) #1	Diuron	0.200	0.10	1.0	3.8	26%
	Trifuralin	0.010	< 0.01			
	Metolachlor	0.145	< 0.01			
	Pendimethalin	0.197	< 0.01			
	Oxyfluofen	0.880	0.9			
SS06 (2/16/05) #2	Diuron	8.000	0.9	1.1	5.4	20%
	Simazine	0.072	< 0.01			
	Metolachlor	0.063	< 0.01			
	Oxyfluofen	0.200	0.2			
CS01 (7/11/05) #3	No herbicides detected[f]	n/a	n/a	n/a	10.8	0%

a Samples analyzed by CDFG-WPCL
b Concentration detected in sample (µg/L) / IC_{25} of suspected toxicant (µg/L)
c Sum of all predicted TUc
d TUc measured in dilution series toxicity tests
e Total predicted TUc / Measured TUc x 100
f Six herbicides were detected but none were quantitated

4.2.2 *C. dubia* Phase III TIE

Table 5 shows the Phase III analysis for the sixteen samples that produced toxicity to *C. dubia*. In this sample set, TUa ranged from ~1-27.5 TUa.

Table 5 ***C. dubia*** **Phase III TIE Analysis**

Test Sample (Date) Sample No.	*Chemical Detected*[a]	*Conc. (µg/L)*	*Pred. TUa*[b]	*Total Pred. TUa*[c]	*Measured TUa*[d]	*TUa Accounted for by Suspect Toxicants*
CS15 (1/26/05) 4	Diazinon Methidathion	0.418 0.043	1.1 < 0.1	1.1	1.3	85%
SSJ03 (1/27/05) 5	Diazinon Chlorpyrifos Disulfoton	0.562 0.035 0.023	1.5 0.4 < 0.1	1.9	2.7	70%
CS15 (2/16/05) 6	Diazinon	4.060	10.7	10.7	10.7	100%
CS12 (6/13/05) 7	Chlorpyrifos Carbaryl	0.083 0.330	1.0 0.1	1.1	1.3	85%
CS23 (6/13/05) 8	Diazinon	2.0	5.3	5.3	5.3	100%
SS05A (6/14/05) 9	Malathion Diazinon	46.0 0.020	21.9 0.1	22.0	21.3	103%
SSJ03 (7/7/05) 10	Chlorpyrifos	0.260	3.3	3.3	2.7	122%
SSJ04 (7/7/05) 11	Chlorpyrifos Dimethoate Malathion	2.20 0.030 0.125	27.5 < 0.1 0.1	27.6	22.7	122%
SSJ07A (7/7/05) 12	Chlorpyrifos Dimethoate Malathion	0.105 0.010 0.020	1.3 < 0.1 < 0.1	1.3	1.3	100%
NSJ31 (7/13/05) 13	Azinphos methyl Methyl parathion	0.330 0.188	0.5 0.24	0.74	1.3	57%
SSJ04 (7/20/05) 14	Chlorpyrifos Dimethoate Methomyl	0.098 0.044 0.640	1.2 < 0.1 0.1	1.3	1.0	130%

Table 5 ***C. dubia*** **Phase III TIE Analysis (continued)**

Test Sample (Date) Sample No.	*Chemical Detected*[a]	*Conc. (µg/L)*	*Pred. TUa*[b]	*Total Pred. TUa*[c]	*Measured TUa*[d]	*TUc Accounted for by Suspect Toxicants*
CS12	Chlorpyrifos	0.040	0.5	0.6	1.3	48%
(7/25/05)	Carbaryl	0.330	0.1			
15						
CS24	Chlorpyrifos	0.043	0.5	1.9	2.7	70%
(7/25/05)	Diazinon	0.025	0.1			
16	Carbaryl	3.60	1.3			
NSJ32	Azinphos methyl	0.165	0.2	2.9	4.7	62%
(7/27/05)	Chlorpyrifos	0.214	2.7			
17	Dimethoate	0.059	< 0.01			
	Methyl parathion	0.016	< 0.01			
FT24	Chlorpyrifos	0.027	0.3	0.3	5.3	6%
(8/1/05)	Dimethoate	0.046	< 0.01			
18	Methomyl	0.052	< 0.01			
SSJ04	Chlorpyrifos	0.098	1.2	1.2	1.3	92%
(8/3/05)	Dimethoate	0.044	< 0.01			
19						

Several of the sample sites exhibited toxicity to *C. dubia* on more than one occasion - CS12, CS15 and SSJ03 on two occasions, and SSJ04 on three occasions. For 12 of the 16 samples analyzed, the detected insecticides accounted for 70-130% of the measured TUs. For the four other samples (Samples 13, 15 and 17), the suspect toxicants accounted for 46-62% of the measured TUs. For Sample 18, only 6% of the measured toxicity was accounted for by the insecticides detected in the sample. This sample, along with Samples 13 and 15, were subjected to Phase II TIE fractionation and the toxic HPLC fractions were analyzed by the CDFG-WPCL to identify other toxicants.

Table 6 shows chemicals in the samples that were detected at concentrations equivalent to ≥0.1TUc. These chemicals included chlorpyrifos (11 samples), diazinon (6 samples), carbaryl (3 samples), malathion (2 samples), and azinphos methyl, methomyl, and methyl parathion (1 sample each). Three samples exhibited exceptionally high toxicity; Sample 6 contained 10.7 TUa of diazinon, Sample 9 had 21.9 TUa of malathion, and Sample 11 contained 27.5 TUa of chlorpyrifos. In each of these samples, the TUa predicted from the measured insecticide concentrations closely matched the TUa measured in the toxicity tests. Other chemicals that

were detected in the toxic samples but were less than 0.1 TUc included methidathion, dimethoate, methyl parathion and disulfoton. Multiple insecticides were usually detected in each of the toxic samples. For example, two insecticides were detected in 6 samples, while three or more insecticides were detected in 7 samples.

Table 6. Frequency and TUa of Chemicals Detected in Samples Causing *C. dubia* Toxicity

Detected Insecticide	***No. Toxic Samples Containing the Insecticide (% of toxic samples)***	***Range of TUa in Toxic Samples***
Chlorpyrifos	11 (69%)	0.3-27.5
Diazinon	6 (38%)	0.1-10.7
Carbaryl	3 (19%)	0.1-1.3
Malathion	2 (13%)	0.1-21.9
Azinphos methyl	1 (6%)	0.2
Methomyl	1 (6%)	0.1
Methyl parathion	1 (6%)	0.24

5.0 CONCLUSIONS

- A total 150 samples of agricultural-dominated discharge collected during two winter events (January-February 2005) and four irrigation events (May-August 2005) were tested using three-species toxicity tests with algae, *C. dubia* and larval fathead minnows.

- Toxicity was detected in 11 of the algae tests (7%), 20 of the *C. dubia* tests (13%), and 3 (2%) of the fathead minnow tests.

- TIEs were conducted on 3 samples that caused algal toxicity and 16 samples that caused *C. dubia* toxicity to identify the cause(s) of the toxicity.

- The TIEs identified the chemical(s) causing *C. dubia* toxicity (within ± 40% of the TUs) in 12 of 16 (75%) of samples evaluated.

- Chemicals responsible for the majority of the *C. dubia* toxicity included chlorpyrifos (11 samples), diazinon (6 samples), carbaryl (3 samples) and malathion (2 samples).

- The TIEs failed to identify the chemicals responsible for the majority of the algal toxicity.

- The majority of toxic samples contained multiple toxicants. More information is needed on the toxic interaction of pesticides detected in agricultural discharges.

- Three of the samples toxic to algae and four of the samples toxic to *C. dubia* were fractionated by HPLC and the toxic fractions analyzed to identify the chemicals responsible for the observed toxicity. The chemical analyses of the fractions by the CDFG-WPCL failed to identify chemicals in the fractions that could account for the sample toxicity.

- Overall, this work demonstrated that proper application of the 3-phase TIE process was effective in identifying the causes of acute *C. dubia* toxicity in the majority of samples evaluated. However, there is need to develop TIE profiles for many commonly detected pesticides and to refine analytical procedures for herbicides. Additional recommendations are discussed below.

6.0 Recommendations

- *Streamline the TIE profile development process.* TIE profiles for only a few agricultural chemicals have been published (Bailey et al, 1996). The present work has identified a need to implement a more streamlined process for development of TIE profiles for chemicals frequently detected at toxic concentrations in the ambient samples. Using the standard Phase I and II TIE process, each chemical is subjected to two SPE column concentration steps and then fractionated by HPLC into 30 or more individual fractions. Toxicity tests are conducted on each of the fractions to determine which fraction(s) are toxic e.g. the test organism is the chemical detector, then the toxic fractions are subjected to chemical analysis, typically by HPLC/MS and/or GC/MS, to confirm recovery at each step. This process can take weeks to months, depending on the turn-around for the analytical chemistry. Considerable time and expense can be saved by using a more automated approach. For example, using an HPLC/MS system with an automated sample injector, multiple HPLC runs using varying

conditions e.g. HPLC column types and gradient systems, can be conducted rapidly to optimize separation and recovery of the individual chemicals. We are working with the CDFG-WPCL to develop and test this new approach.

- *Investigate toxicity of mixtures of pesticides and degradates.* Identification of the cause(s) of toxicity using the TIE process is complicated by the simultaneous occurrence of multiple pesticides and their degradates in ambient samples. In the National Water Quality Assessment (NAWQA) ambient monitoring studies, mixtures of pesticides were detected in more than 90% of the samples analyzed, with three or more pesticides detected more than 70% of the time (USGS, 2006). Yet, most research has evaluated the effects of pesticides as if they occurred alone. The assessment of the causes of ambient toxicity is further complicated by the presence of pesticide degradates resulting from biotic and abiotic transformation of parent pesticides in the environment. Many of the degradates are more persistent in the environment than the parent compounds, and many are more mobile, as well (Boxall et al, 2004). In most cases, there is a paucity of toxicological information on pesticide degradates. However, a recent review of pesticide degradates for which some toxicity information is available, reported that 39% of the degradates had similar toxicity as the parent chemical while 20% were more than three times as toxic and 10% were more than 10 times as toxic (Sinclair and Boxall, 2003). Similar patterns are apparent for eight pesticides frequently detected by NAWQA with 23% of the degradates being more toxic to fish and 21% being more toxic to daphnids than the parent chemical (USGS, 2006). It is clear from these reports that more toxicity information is needed on major pesticide degradates and that ambient monitoring programs should include analyses of at least the most toxic of these chemicals.

- *Investigate toxic interaction of multiple pesticides.* Because pesticides and their degradates are more commonly detected in the environment than single chemicals, assessment of their role in the toxicity of ambient samples must account for the combined effects of each of the chemicals present. Research on pesticide mixtures has demonstrated a wide array of possible interactions, including independent, additive, antagonistic and synergistic. Generally, pesticides within the same chemical class have a similar mode of action and produce additive toxicity, while pesticides from different classes have more varied and unpredictable effects. For example, mixtures of diazinon and chlorpyrifos exhibit directly-additive effects to *C. dubia* (Bailey et al, 1996), while mixtures of diazinon and esfenvalerate produce greater than additive effects on fathead minnows (Denton et al, 2003). Mixtures of non-toxic levels of atrazine with diazinon produced a synergistic effect, e.g., up to 400% greater toxicity than

expected (Pape-Lindstrom and Lydy, 1997). Conversely, diazinon and ammonia in combination are apparently antagonistic (Bailey et al, 2001). Further research is needed before the toxicity of samples containing multiple chemicals can be accurately predicted. For example, even with a mixture of 5 chemicals, there are 25 possible combinations of pairs and several hundred combinations of pairs, triples, and so on. Obviously, the large number of possible combinations of environmental chemicals and varying exposure scenarios make this task extremely difficult. Certainly, it will be necessary to prioritize testing of chemical combinations, perhaps based on toxicity of individual chemicals and frequency of detection to develop toxicity information which may then be incorporated into models to predict the toxicity of pesticide mixtures.

- *Develop centers for analytical support of TIEs.* Reliable and efficient identification of chemical cause(s) of ambient toxicity in TIEs is essential for subsequent source identification and remediation activities. However this step is often problematic due to limitations in analytical chemistry. It has been previously suggested that success in identification of the chemicals causes of toxicity would be substantially enhanced through the creation and continued funding of regional analytical centers primarily devoted to the support of TIE investigations (Norberg-King et al, 2005). In these centers, highly experienced analytical chemists with access to state-of-the-art instrumentation including high resolution mass spectrometry (MS), gas chromatography/MS/MS (GC/MS/MS), HPLC/MS/MS, nuclear magnetic resonance (NMR), and inductively coupled plasma/MS (ICP-MS), would work closely with TIE practitioners on samples for which competently conducted TIEs have failed to identify the cause(s) of toxicity. Analytical approaches used in the chemical identification process along with spectral information could be made available via the Internet and through frequent presentations, workshops, and publications. Clearly, despite the considerable procedural development that will be required and issues of securing funding (for both creation and continued support), personnel, site selection, and logistics that will likely be challenging, development of such TIE support centers should, in our view, be considered a high priority for those entities involved in ambient monitoring.

7.0 acknowledgments

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Figure 1. Phase I TIE Flowchart Modified for Algae

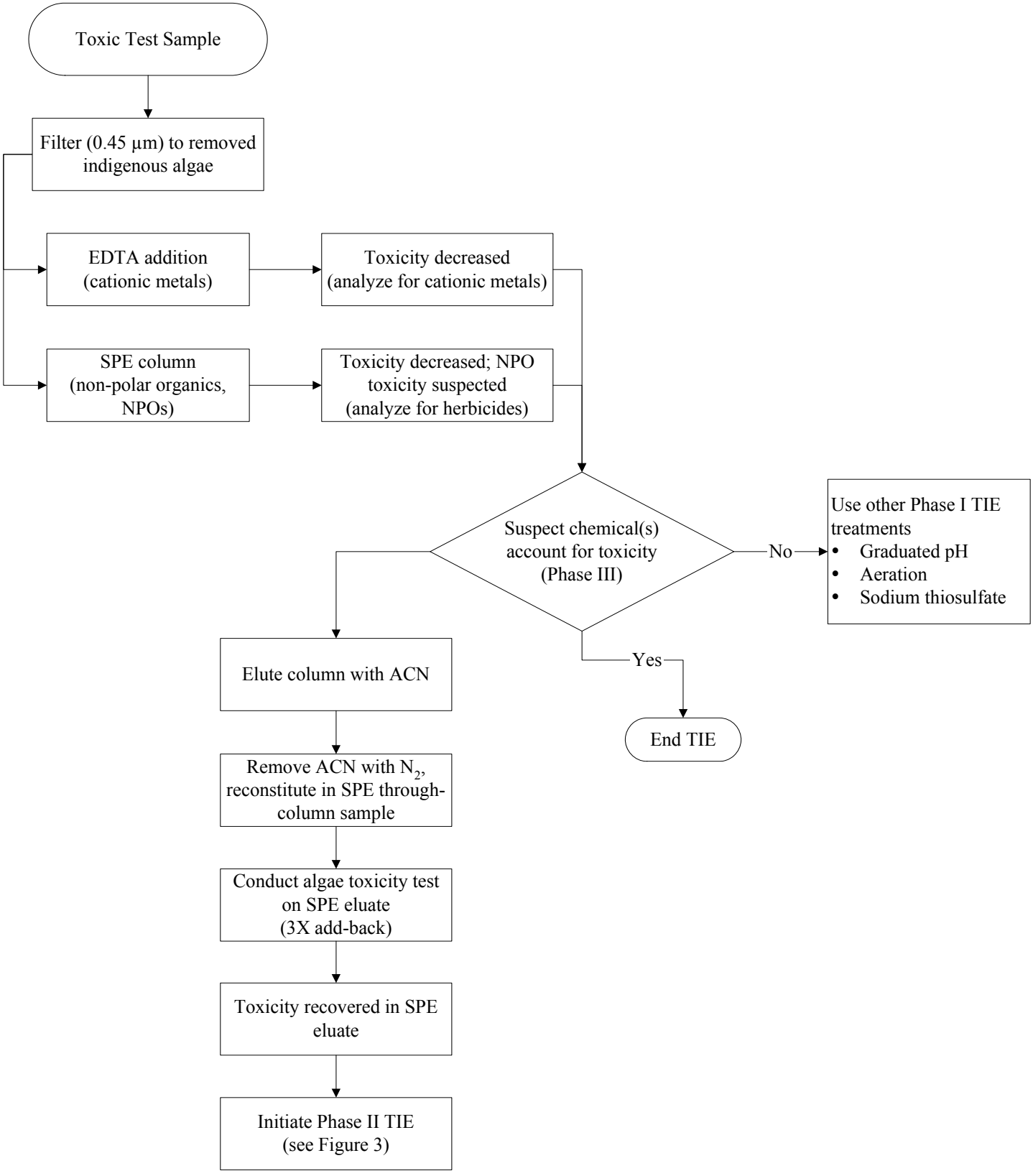


Figure 2. Phase I TIE Flowchart for *C. dubia*

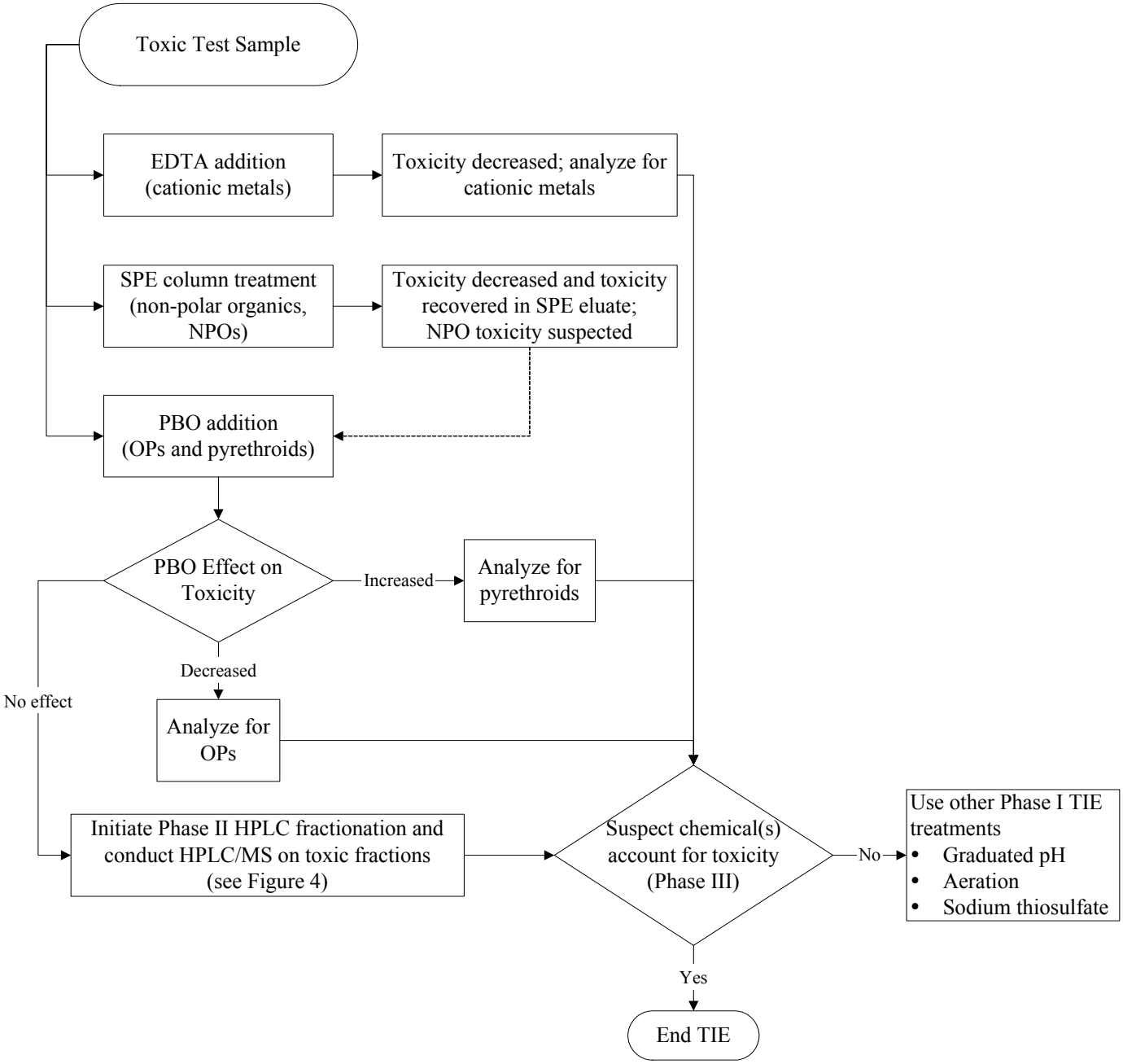


Figure 3. Phase II TIE Flow Chart for Identification of Non-Polar Organic Toxicity to Algae

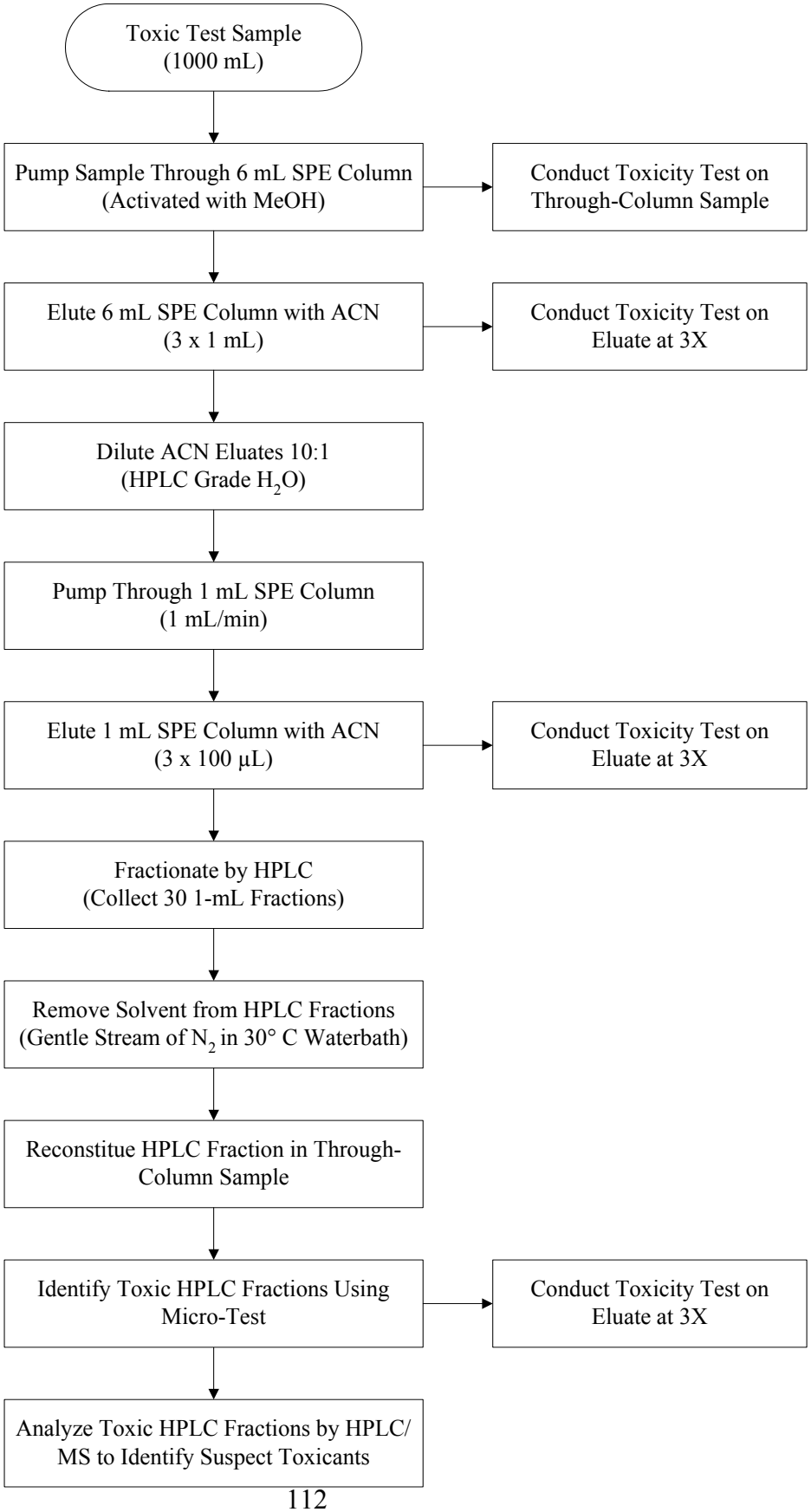
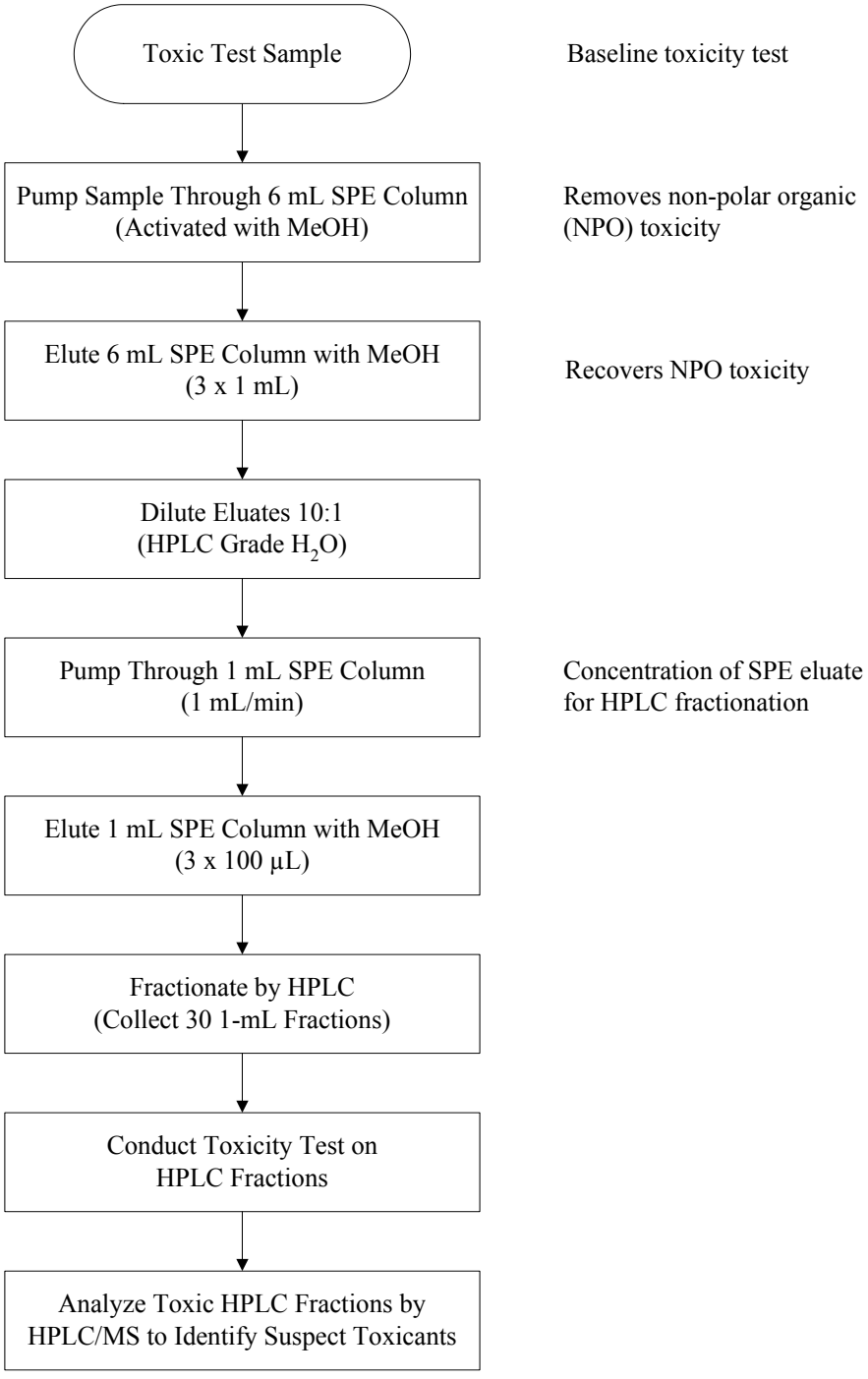


Figure 4. Phase II TIE Flow Chart for Identification of Non-Polar Organic Toxicity to *C. dubia*



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APPENDIX 7: D. Weston. Sediment toxicity in agricultural areas of California and the role of hydrophobic pesticides

Sediment toxicity in agricultural areas of California and the role of hydrophobic pesticides.

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Abstract

In order to assess the impact of agricultural pesticides on sediment quality, 200 sediment samples were collected in agriculture-affected water bodies throughout California's Central Valley. Most of these samples were tested for acute toxicity to the amphipod Hyalella azteca, and analyzed for pesticides including pyrethroids, chlorpyrifos and organochlorines. Twenty-seven percent of the samples exhibited acute toxicity, and in 67% of these instances, the measured analytes were in sufficient concentration to explain it. Pyrethroids (most notably bifenthrin, lambda-cyhalothrin and esfenvalerate) reached concentrations associated with H., azteca toxicity in 55% of the toxic samples, or 61% if toxicity of compounds within the class is assumed to be additive. Chlorpyrifos reached acutely toxic concentrations in 20% of the samples. Organochlorines, while frequently present, rarely, if ever, contributed to acute toxicity. Evidence from toxicity unit analysis, sediment dilution testing, and Toxicity Identification Evaluation procedures are in close agreement and all indicate a role of these pyrethroids and chlorpyrifos as causative agents for the toxicity. Small agricultural drains nearest the points of pesticide use are more impacted than the major water bodies to which they flow, and the pesticides appear to be entering these drains by way of irrigation runoff during the summer months.

Keywords: Pesticides, Hyalella, Sediments, Toxicity, Pyrethroids, Chlorpyrifos

1. Introduction

About 40% of California is located in the Central Valley, a region of highly productive agricultural land. The area produces an exceptionally wide variety of crops, with the dominant commodities including alfalfa, hay, corn, rice, tomatoes, lettuce, citrus fruits, peaches, plums, almonds, nuts and grapes. The Central Valley provides nearly all commercial U.S. production of almonds, walnuts, figs, kiwifruit, nectarines, olives, pistachios, prunes, and raisins.

Maintaining the 7,000,000 acres of irrigated agricultural land within the Central Valley requires a complex network of water courses to supply water and carry irrigation runoff (known as tailwater) to the region's major rivers. A total of 32,000 km of channels have been constructed, and there is an additional 2,400 km of natural creeks and rivers, many of which have been modified and are heavily used for agricultural supply water or drainage (CVRWQCB, 1992).

Habitat quality within this network of constructed and natural water courses is affected by the 57 million kg of pesticides used in the Valley annually (2004 usage data: http://www.cdpr.ca.gov/docs/pur/pur04rep/04_pur.htm). Since the early 1990s, rivers and other water bodies receiving tailwater in the Central Valley have been frequently found to be acutely toxic to a standard testing species, the cladoceran Ceriodaphnia dubia (Kuivila and Foe, 1995; Werner et al., 2000). In most cases when the causative agent could be identified, it was found to be one of the organophosphate pesticides, often diazinon or chlorpyrifos. These findings led to extensive water quality monitoring for the organophosphates, development of management practices to reduce organophosphate runoff, and widespread grower education efforts. Agricultural organophosphate use is now less than half of what it was in the early 1990s, and the frequency of C. dubia toxicity has decreased significantly.

Sediment quality in the Central Valley, on the other hand, has until recently, received little attention. An assessment done in 2002 and 2003 indicated widespread toxicity to the amphipod Hyalella azteca, and less frequently, to Chironomus tentans (Weston et al., 2004). About 28% of the sediment samples collected were acutely toxic to H. azteca. Based on a comparison of the pyrethroid concentrations occurring in the samples to estimates of the concentrations in sediment likely to cause toxicity, pyrethroids were believed to contribute to the toxicity in the majority of the cases.

The Central Valley of California is unique in the amount of environmental data available on pyrethroid pesticides in sediments. The compounds are widely used in agriculture, yet there has been little or no monitoring for the compounds in other agricultural areas throughout the world, and only limited data are available from other locations within California (Anderson et al. ,2006). Thus, a close assessment of the Central Valley provides the opportunity to determine if environmental residues are present in aquatic habitats, whether they are present at toxic levels, and which specific compounds within the class most often contribute to toxicity. The present study is intended to re-evaluate and build upon the findings of Weston et al. (2004). In the intervening years there have been several improvements in our ability to assess sediment quality in the Central Valley. First, the database is considerably larger. While Weston et al. (2004) was based on 70 samples, the current study is based on 200, and the Central Valley counties with available data have increased from 10 to 17 (of the 19 within the Valley). Secondly, in the former study it was necessary to estimate the concentrations of pyrethroids that would be acutely toxic to H. azteca, since precise measurement had not been made for the most members of the class. However, such data are now available (Amweg et al., 2004) making it possible to more confidently establish when pyrethroids may be contributing to observed toxicity. Several other approaches are also now possible to help identify the causative agent of toxicity.

2. Materials and methods

2.1 Overall sampling design

The available data represents 133 sites throughout the Central Valley, 117 of which have complete toxicity and chemistry data (the remainder with only toxicity data). Most of these sites (81) were sampled on one occasion, 42 were sampled twice, six were sampled three times, and four were sampled four to five times. This effort yielded 200 samples, 180 of which have complete chemistry and toxicity data (Appendix 1). The 70 samples of Weston et al. (2004) are included within this total.

About two-thirds of the samples were provided by a study of sediment quality in waterways throughout the Valley receiving agricultural tailwater. Most of these waterways were of moderate size and intended to be representative of regional inputs rather than one or a few farms. Sites were selected to obtain even geographic coverage across the Valley, without regard to crop type or pesticides use in the vicinity. The remaining third of the samples were located in areas of high pyrethroid use. For this latter subset of samples, sites were located in ten of the Central Valley counties with the highest annual agricultural pyrethroid use, as determined by the California Department of Pesticide Regulation's Pesticide Use Reporting (PUR) database (http://www.cdpr.ca.gov/docs/pur/purmain.htm). These sites were roughly evenly divided between the counties in the northern half of the Valley (Sacramento River watershed) and the southern counties (San Joaquin River watershed).

Samples were collected from March 2003 through April 2006. Most samples were collected either at the end of the rainy season before agricultural irrigation begins when sediment quality would be expected to be influenced by stormwater runoff (March-April) or the end of the irrigation season when tailwater return provides the primary route for transport of hydrophobic pesticides (mostly August samples, with a few in July and September).

2.2 Sampling methodology

Sampling efforts were focused on fine-grained sediments given their higher organic carbon content, and therefore higher affinity for hydrophobic pesticides (DiToro et al., 1991). Even in those waterways dominated by gravel or hardpan clay, it was usually possible to find soft sediment deposits from which to collect the samples. When possible, samples were composited over a reach of at least 30 m, though sometimes a shorter segment was sampled because of limited soft sediment availability or access difficulties.

Sediments were collected from the bank or by wading in to shallow water, using a stainless steel scoop to skim the upper 1-2 cm of the sediment column. A sample consisted of a dozen or more such scoops, composited in a solvent-cleaned 4 L glass jar. The sediment was held on ice until return to the laboratory, where it was homogenized by hand mixing in a stainless steel bowl. Approximately 4% of the samples contained gravel, vegetation or other debris requiring removal by sieving on a 1 mm screen to obtain homogeneous material. Subsamples were taken from the mixing bowl for pesticide and total organic carbon analysis (both held at -20°C until analysis) and toxicity testing and grain size analysis (both held at 4°C).

2.3 Toxicity testing

Sediments were tested for toxicity using the amphipod, Hyalella azteca, using standard protocols (U.S. E.P.A., 2000). The only significant departures from these protocols were use of a slightly smaller amount of sediment (75 ml instead of 100 ml) and use of only the mortality endpoint (growth was measured in some samples but data are not presented here). Briefly, 400 ml beakers were filled with 75 ml sediment and 250 ml moderately hard water, reconstituted by addition of salts to Milli-Q purified deionized water (Millipore Corp., Billerica, MA, USA). Each batch of test sediments was accompanied by a control sediment, using material either from the American River at Folsom Lake, CA, San Pablo Dam

Reservoir, El Sobrante, CA, or Lake Anza, Berkeley, CA. Ten individuals of H. azteca, 7-10 days of age, were added to eight replicates for each sediment, or five replicates in about 18% of the samples. Tests were conducted for 10 d, at 23°C, with a 16: 8 hr light:dark cycle, and feeding 1 ml YCT (yeast, cerophyll, trout food) per beaker per day. A few of the samples required gentle aeration to keep dissolved oxygen levels within test limits. Water was changed at the rate of two volume additions daily (total 500 ml) by an automatic water delivery system. Ammonia, hardness, alkalinity, and pH were measured at the start and end of the test; temperature and dissolved oxygen were monitored regularly throughout the test. Water quality data are not presented but were always within permissible limits of the standard protocols. After 10 d, the sediment was sieved on a 425 µm screen and the surviving animals enumerated.

A few samples exhibiting high mortality were tested in a dilution series using control sediment as the diluent, concentration steps of a factor of two (e.g., 50%, 25%, 12%, 6%), and three replicates per concentration. Control sediment and test sediment were thoroughly mixed by hand, and the test initiated 24 hr later.

Test data were analyzed using ToxCalc software (Tidepool Scientific Software, McKinleyville, CA, USA). Test sediments were compared to control using Dunnett's procedure when parametric assumptions were met, with arcsine squareroot transformation when necessary. Steel's Many-One Rank test was used when parametric assumptions were not met. Lethal concentrations for half the test organisms (LC50) were determined in the sediment dilution series by maximum likelihood regression using probit transformation.

2.4 Chemical analysis

All the sediment samples were analyzed for four pyrethroids: bifenthrin, esfenvalerate, lambda-cyhalothrin and permethrin. Three additional pyrethroids, cyfluthrin, cypermethrin, and deltamethrin, were added to the analyte list later, and were analyzed for in two-thirds of the samples. the pyrethroid fenpropathrin was assessed in only a single sample. Organochlorine pesticides analyzed included alpha-, beta-, delta-, and gamma-BHC, heptachlor, heptachlor epoxide, alpha- and gamma-chlordane, alpha- and beta-endosulfan, endosulfan sulfate, *p,p'*- DDE, *p,p'*- DDD, *p,p'*- DDT, aldrin, dieldrin, endrin, endrin aldehyde, endrin ketone, and methoxychlor. Chlorpyrifos was the only organophosphate insecticide quantified.

Analysis was performed on an Agilent 6890 series gas chromatograph equipped with an Agilent 7683 autosampler and an electron capture detector (Agilent Technologies, Palo Alto, CA). Two columns from Agilent, a HP-5MS (30m x 0.25mm; 0.25µm film thickness) and a DB-608 (30m x 0.25mm; 0.25µm film thickness) were used. Five external standards solutions ranged from 5 to 250 ng/ml were used for calibration. The calibration curves were linear within this concentration range. Qualitative identity was established using a retention window of 1% with confirmation on a second column.

Prior to analysis, frozen sediment was thawed, centrifuged to remove excess water and homogenized. The extraction and cleanup methods were developed and validated in an earlier study (You et al., 2004). Two surrogates, 4,4'-dibromooctafluorobiphenyl and decachlorobiphenyl, were added to the sediment prior to the extraction to verify extraction and cleanup efficiency. Approximately 20 g of sediment (wet weight) was mixed with anhydrous $MgSO_4$ and sonicated with 50 ml of 50:50 acetone: methylene chloride (v/v) for 3

minutes using a high intensity ultrasonic processor (Sonics and Materials Model VCX 400, Newtown, CT). The extract was centrifuged, decanted and filtered. This procedure was repeated twice more. Extracts were combined, solvent exchanged with hexane and the volume reduced to 2 ml. Adsorption chromatography with Florisil, deactivated by mixing with distilled water (6% w/v), was used for extract cleanup. The pesticides were eluted from the column with 50 ml of 30% diethyl ether in hexane (v/v). The elute was evaporated, redissolved in 2 ml of hexane and analyzed on the gas chromatograph. Additional dilution steps were needed for some field-collected samples due to elevated pesticide concentrations. With method detection limits of 0.22-0.85 ng/g dry weight, the method reporting limits were set at 1 ng/g for all the analytes.

Quality control measures included re-analysis of pyrethroid pesticides in five samples by an independent laboratory using gas chromatography-mass spectroscopy (GC-MS) and blind analysis of 13 spiked sediment samples. Qualitative agreement with the GC-MS analysis was excellent, with the GC-MS confirming GC-ECD-derived compound identity in all cases. Quantitative agreement was good, with GC-MS and GC-ECD quantitations having a median relative percent difference of 25%, and equal instances of the GC-MS yielding higher and lower values than the GC-ECD. Analysis of the 13 blind samples produced pyrethroid recoveries that were nearly always 50-120% of nominal values (median = 79%). Chlorpyrifos recoveries in the blind spikes were 33-104% (median = 66%) indicating the concentrations reported herein may sometimes be lower than actual. Organochlorine recoveries from the blind spikes were usually 60-100% (median = 74%).

Total organic carbon was measured using a CE-440 elemental analyzer (Exeter Analytical, Chelmsford, MA, USA) after acid vapor treatment to remove inorganic carbon. Grain size was determined by wet sieving, with silt and clay combined in the <64 μm fraction.

3.0 Results and discussion

3.1 Sediment properties

The sediments sampled were deliberately chosen to represent the finest-grained material available at each site, in which hydrophobic pesticides would be more likely to be present at measurable levels. The percentage of silt and clay particles within the samples ranged from 7-97% by weight, with a median of 42%. Eighty percent of the sites contained >25% silt and clay. Total organic carbon of the sediment samples ranged from 0.1-7.4%, with a median value of 1.1%. Seventy percent of the samples fell within the range of 0.5-2.5% organic carbon.

3.2 Toxicity testing

Control survival was good, with a median value of 94% across all tests, and was never below 86%. Test sediments, however, frequently caused acute mortality to H. azteca. Overall, a total of 53 out of the 200 samples (27%) exhibited toxicity, and 39 out of the 133 sites (29%) were toxic on at least one sampling occasion. The later statistic, while accurate, is somewhat distorted by those sites that were sampled on multiple occasions, when they may have only been toxic once. If the percentage of sites exhibiting toxicity is calculated only the basis of the first sampling event at each site, regardless of findings in later events, (a

more reasonable approximation of the frequency of toxicity if measured at a single point in time) the percentage of toxic sites decreases to 23%.

Toxicity, however, was not uniformly distributed throughout the Central Valley (Figure 1). Sites in the southern half of the Valley within the San Joaquin River watershed, were twice as likely to show toxicity as those in the northern half within the Sacramento River watershed (37% of the southern sites vs. 19% in the north). In particular, the northwestern portion of the San Joaquin watershed, comprised of San Joaquin and Stanislaus Counties, was an area of frequent sediment toxicity, with 15 out of 34 sites (44%) in these two counties causing toxicity. Fresno County, at the southern end of the Valley, also had a high frequency of toxicity, with 47% of the sites toxic on at least one occasion.

Of the various water body types within the Valley, unnamed drains showed the most frequent toxicity (Table 1). These drains are entirely constructed water bodies, and because they serve a relatively small number of farms, are unnamed and do not appear on regional maps. Forty-one percent of the sampling sites in these drains showed toxicity, the highest of any water body type, as might be expected given the close proximity of these drains to the points of pesticide application, and the fact that water flow to the drains consists entirely of field runoff. The frequency of toxicity is reduced by nearly half in named drains (e.g., Island Field Drain, Colusa Drain, Button Ditch), water bodies serving larger watersheds and more critical to regional irrigation systems. Creeks showed a surprisingly high frequency of toxicity, with 40% of the creek sites toxic on at least one occasions. The creeks generally originate around the periphery of the Central Valley, with their headwaters in the surrounding mountains, and are natural water bodies though their flow is highly managed for irrigation purposes in their agricultural reaches. The high frequency of toxicity in creeks is, however, somewhat distorted by the numerous creeks in western Stanislaus and San Joaquin counties that have consistently been found to contain toxic sediments. Excluding this region to obtain a more representative picture of the Central Valley, creek toxicity is reduced to 26% of the sites. Three of 11 river sites (27%) showed sediment toxicity (Calaveras River, Kaweah River, and the San Joaquin River near the town of Vernalis, CA).

Nearly all sites were sampled either at the end of the winter rainy season (March/April) or late in the summer irrigation season (July/August/September). The timing of the sampling did not have a great effect on the frequency of sediment toxicity observed. In the late winter 29% of the samples showed acute toxicity, whereas in the late summer that proportion was 21%.

3.3 Contributors to sediment toxicity

One approach to identifying likely contributors to sediment toxicity is the use of toxicity units (TU) normalized to sediment organic carbon (o.c.; Weston et al., 2004) defined as:

$$TU = \frac{\text{Actual sediment concentration of the analyte on o.c. basis}}{\text{Reported 10-d } LC_{50} \text{ concentration of the analyte on an o.c. basis}} \quad (1)$$

Application of the TU approach to explaining H. azteca toxicity requires that 10-d sediment LC50 values for the species be available for all toxicants of interest. These values

have been published for all pyrethroids regularly analyzed in this study (cypermethrin 10-d LC50 = 0.38 µg/g o.c., lambda-cyhalothrin = 0.45 µg/g o.c., bifenthrin = 0.52 µg/g o.c., deltamethrin = 0.79 µg/g o.c., cyfluthrin = 1.08 µg/g o.c, esfenvalerate = 1.54 µg/g o.c., permethrin = 10.83 µg/g o.c. (Amweg et al., 2004; Maund, et al., 2002). The chlorpyrifos LC50 has been determined in two different sediments in our laboratory (Weston et al., in preparation), and averages 3.07 µg/g o.c. The sediment LC50 of fenpropathrin to H. azteca has not been determined. However in water-only exposures to aquatic life, the 10^{th} percentile LC50 of fenpropathrin is 1.3 times that of permethrin (240 ng/L vs. 180 ng/L; Solomon et al., 2001), and this ratio was applied to the permethrin sediment LC50 to derive an estimated fenpropathrin sediment LC50 of 14.4 µg/g o.c.

Since H. azteca mortality of 50% would be expected at 1 TU, a value of 0.5 TU is herein used as an approximation of the concentration at which mortality would first appear, and a threshold above which the given toxicant is considered potentially responsible for mortality when observed (Weston et al., 2004). The 0.5 TU value is arbitrary but suggests the compound is on the verge of reaching acutely toxic concentrations if not already surpassing them.

Pyrethroid pesticides are implicated by the TU analysis as a probable cause of the toxicity in the majority of cases. Since pyrethroids all have similar modes of neurotoxic action, the most probably interaction between members of the group is additivity. Assuming additivity of pyrethroid TUs, 61% of the acutely toxic samples (31 out of 51 samples; excluding two toxic samples with no chemistry data) contain at least 0.5 TU of total pyrethroids (Figure 2). The assumption of additivity, while reasonable, has not been proven specifically for pyrethroids, but it does not substantially affect the analysis. Even without the assumption of additivity, 55% of the acutely toxic samples contain at least 0.5 TU of at least one individual pyrethroid. Chlorpyrifos concentrations reached 0.5 TU in 20% of the toxic samples. Organochlorines almost never reached concentrations expected to be toxic to H. azteca. Estimated organochlorine LC50 values are available for gamma-BHC, endosulfan, DDE, DDT, DDD, dieldrin, endrin, and methoxychlor (Weston et al., 2004). Not a single toxic sample contained at least 0.5 TU of any organochlorine. There was only one instance of an organochlorine (endrin) present at 0.5 TU and this sample showed no acute toxicity.

After accounting for all analytes for which TU values could be calculated, there remained 33% of the toxic samples for which none of the measured analytes exceeded 0.5 TU, even if assuming additivity of TU within the pesticide classes. Toxicity in these cases may have been due to one of the many agricultural pesticides used in the Central Valley which are not among the analytes of this or any monitoring program, or it may have been the result of sediment properties that enhanced bioavailability and toxicity of the measured analytes above that expected based on organic carbon normalization alone.

The TU approach, even when considering pyrethroid TUs alone, was highly predictive of toxicity to H. azteca (Figure 3). Below about 0.5 TU of pyrethroids, toxicity was rarely seen, and when present tended to be fairly modest (<40% mortality). The three samples in the upper-left of Figure 3 (>80% mortality but less than 0.01 TU of pyrethroids) can be explained by the presence of chlorpyrifos which exceeded 0.8 TU in all three of these samples. Above 0.5 pyrethroid TUs, mortality rate climbed rapidly, as would be expected if pyrethroids were the primary causative agent. Above about 3 TU of pyrethroids, there was total or near total mortality in all samples. The seven samples with surprisingly low toxicity (1-4 TU but <30% mortality) tended to be from coarse sands: all but one of these samples were among the third of the samples with the lowest proportion of silt and clay. Previous

work (Amweg et al., 2006a) has also reported an overestimate of pyrethroid toxicity by the TU approach in similar sediments. While there are a few data points that deviate slightly from the expected TU:mortality relationship of Figure 3, the relationship is remarkably good, and consistent with pyrethroids being the causative agent for much of the observed toxicity.

A second line of evidence to help infer causality for the toxicity is provided by dilutions of samples exhibiting high H. azteca mortality. A parameter referred to as "observed TUs" was calculated based on the toxicity test dilutions as:

$$\text{Observed TU} = 100/\text{Observed LC50 of test sediment determined by dilution} \quad (2)$$

The observed TU derived by toxicity testing can then be compared to the expected TU previously shown in equation 1, calculated based on chemical concentrations and literature-derived LC50 concentrations. Close agreement of the observed TU with the expected TU provides evidence that the compounds used to calculate the expected TU are indeed responsible for the toxicity.

This approach is commonly used in a Toxicity Identification Evaluation context for water samples (U.S. E.P.A, 1989), but a mathematical adjustment is necessary when applying it to hydrophobic toxicants in sediment. The bioavailability, and hence toxicity, of such materials is highly dependent upon the sediment organic carbon content (DiToro et al., 1991), but rarely will the sediment used for dilution be of equal organic content to the test sediment. Dilution with a control sediment high in organic carbon will yield a higher LC50 estimate than if the control sediment diluent contained little organic carbon. If the organic carbon content of the test sediment and control sediment diluent are both known, it is possible to calculate the organic content of the diluted sediment when at its LC50 concentration, and then use this value to express the observed LC50 on an organic carbon adjusted basis as:

$$\text{Observed } LC50_{oc} = \text{Observed LC50} \times \frac{\text{o.c. of undiluted test sediment}}{\text{o.c. of diluted sediment at the LC50}} \quad (3)$$

The observed $LC50_{oc}$ can then be used in a manner analogous to equation 2 to obtain an observed TU_{oc}. The approach assumes the LC50 is linearly related to the organic carbon content of the sediment, and expresses that LC50 as if the organic carbon at the LC50 concentration was equivalent to that of the original test sediment.

There are 11 test sediments that were tested in dilution series, and for which it is possible to compare the observed TU_{oc} with the expected TU of either pyrethroids or chlorpyrifos (Table 2). In only a couple cases was there precise agreement between the observed and expected TU, but in nearly every case the two approaches to derive TUs agreed within a factor of two. Variation in LC50s of this magnitude are common when testing multiple sediments (Maund et al., 2002; Amweg et al., 2005), and thus the expected TUs, derived using generalized LC50s, could easily incorporate a factor of two error when applied to specific sediments. Taking this potential variability in to account there was good agreement between observed and expected TUs, regardless of whether the putative toxicant was esfenvalerate, lambda-cyhalothrin, bifenthrin, fenpropathrin, or chlorpyrifos, further supporting the role of these compounds in causing the observed toxicity.

One complicating factor in this analysis is the nature of the toxicological interaction of pyrethroids and chlorpyrifos. For a few of the sediments in Table 2, additivity of pyrethroid TUs was assumed, which is a reasonable assumption given the similar mode of action for all the pyrethroids. However, pyrethroid and chlorpyrifos TUs were calculated independently as the two groups have different modes of neurotoxicity. There are at least two studies indicating toxicity of pyrethroids and organophosphates is at least additive and potentially synergistic (Denton et al., 2003; Belden and Lydy, 2006), but since such an interaction is not widely established, no implicit assumptions of their interaction was made here. Thus, for a few sediments, such as CS15 and CS12 samples both of Aug. 9, when there could be substantial toxicity due to both pyrethroids and chlorpyrifos, the actual expected TUs when including both pesticides remains uncertain.

Finally, the third line of evidence for causality comes from newly developed Toxicity Identification Evaluation techniques for bulk sediments. Pyrethroids are atypical in that they become more toxic as the temperature decreases, whereas chlorpyrifos toxicity to H. azteca is temperature independent (Weston et al, in preparation). Seven sediments from the current study were tested with H. azteca at reduced temperatures (Hospital, Del Puerto, and Spring Creeks, and the unnamed drains of SED11, CS12, FT19, and SED15). In every case the sediments were more toxic. Those samples in which chlorpyrifos was suspected to be contributing to the toxicity showed a statistically significant but only slight temperature response, whereas those containing pyrethroids showed a strong temperature response, with typically an increase in toxicity of a factor of 2 or more (Weston, et al., in preparation). Five sediments from the current study (Del Puerto and Spring Creeks, and the unnamed drains of CS12, FT19, and SED15) were also tested with piperonyl butoxide (PBO) in the overlying water. PBO enhances the toxicity of pyrethroids (Amweg et al., 2006b; Weston et al., 2006) but lessens the toxicity of chlorpyrifos (Bailey et al., 1996). In four sediments in which pyrethroids were believed to be substantial contributors to toxicity based on TU calculations, PBO enhanced toxicity supporting the suspected role of pyrethroids (Weston, et al., in preparation). In one sediment sample containing only chlorpyrifos, toxicity was diminished by addition of PBO.

3.4 Patterns of pesticide use and sediment contamination

This study included analysis of 28 pesticides, the majority of which did not appear to play a significant role in determining toxicity to H. azteca. Only eight pesticides reached concentrations of at least half their estimated sediment LC50 to the species (Table 3). Of these eight, three pesticides reached this 0.5 TU threshold at more than 5% of the sites: bifenthrin, lambda-cyhalothrin and chlorpyrifos.

The distribution of these three pesticides that most often exceeded the 0.5 TU threshhold in Central Valley sediments is shown in Figures 4, 5, and 6. Their concentration in the sediment is shown on a ng/g basis, but with the concentration categories corresponding to the toxicity units of the respective compounds (undetected; less than 0.5 TU; 0.5 to 2 TU; greater than 2 TU – with all TUs calculated assuming a typical 1% sediment o.c.). To put the concentrations in the context of patterns of pesticide use, these maps also indicate the intensity of agricultural use of the given pesticide in each Central Valley county based on 2004 data from the California Department of Pesticide Regulation's PUR database (http://www.cdpr.ca.gov/docs/pur/purmain.htm). This database provides statistics on the mass of pesticide used in each county, however, the counties differ

dramatically in size, the amount of their land area committed to agriculture, and the amount of agricultural land likely to have few or no pesticides applied (e.g. rangeland). Therefore, the usage in each county has been adjusted to the area of harvested cropland, using 2002 acreage figures from the California Department of Finance (http://www.dof.ca.gov/HTML/FS_DATA/STAT-ABS/tables/g8.pdf), and the maps indicate the amount of the given pesticide used annually in each county to produce a hectare of harvested crop. Thus, "high use" counties on these maps may not use a large amount of the pesticide on an absolute basis, but do use a relatively large amount within their land area of harvested cropland. Finally, these maps also illustrate the seasonal patterns of use for the given pesticide, as such information has ramifications for appropriate mitigation practices.

Bifenthrin was detected in 23% of the samples and reached the 0.5 TU level on at least one occasion at 16% of the sampling sites (i.e., one out of six Central Valley sites contained acutely toxic concentrations of bifenthrin in at least one sampling event). Concentrations of bifenthrin in sediments that would be potentially toxic to H. azteca are limited almost entirely to the southern Central Valley counties (San Joaquin County and southward). Similarly, five of the eight southern counties use relatively large amounts of bifenthrin per unit cropland, and comparable amounts are applied in only a single northern county (Sutter). Every instance of very high bifenthrin concentrations in sediments (>10.4 ng/g, or >2 TUs assuming 1% o.c.) occurred in these southern high use counties. Bifenthrin use in the Central Valley is limited entirely to the summer months, with nearly all of the compound applied in June, July and August.

Lambda-cyhalothrin exceeded 0.5 TU in 9% of the sampling sites. As was the case for bifenthrin, highest sediment concentrations of lambda-cyhalothrin are largely limited to the southern counties, with a high frequency of potentially toxic concentrations in Stanislaus and Fresno counties. Lambda-cyhalothrin use, however, is more equitably distributed between the northern and southern counties. Four of the six high use counties are to the north, indicating other factors besides use (e.g., soil type and potential for erosion, irrigation practices) play a significant role in determining the potential for contamination of surface water bodies. The greatest monthly use of lambda-cyhalothrin occurs in March when it is applied to alfalfa. Substantial quantities are also used in the May through August growing period on a variety of other row crops.

Chlorpyrifos approached or exceeded concentrations toxic to H. azteca in 8% of the sampling sites. Compared to the pyrethroids, chlorpyrifos showed a greater frequency of detectable but non-toxic concentrations in both the southern and northern counties (36% frequency of detection). Potentially toxic concentrations were scattered throughout San Joaquin, Fresno, Tulare and Kings Counties in the south, and Colusa County in the north. There is a small amount of chlorpyrifos used as a dormant spray on orchards in the winter months, but the vast majority is applied during the growing season, particularly in July and August.

Esfenvalerate approached or exceeded acutely toxic concentrations at 4% of the sites. Three of these sites were in San Joaquin County (Little John Creek and two unnamed drains), one in neighboring Stanislaus County (Del Puerto Creek), and one in Tulare County (Knestric Ditch). Use of esfenvalerate in the Central Valley is nearly equally split between winter and summer months. Fifty-five percent of the annual use of esfenvalerate occurs in April through October, with the remainder applied during winter months, largely on almond and stone fruit orchards (e.g. plums, peaches).

Permethrin is the most heavily used of the pyrethroids in Central Valley agriculture, and was often detected in the sediment samples (40% frequency of detection). However, it is one of the least toxic of the pyrethroids to aquatic life (Solomon et al., 2001), and approached toxic concentrations at only two sites; Root Creek in Madera County in an area dominated by pistachio orchards and an unnamed drain in San Joaquin County. Cypermethrin use in urban areas as a termiticide is far greater than its use in agriculture in California. It was detected in only 3% of the samples and reached the 0.5 TU threshhold in only two agricultural drains in the southern Central Valley (Fresno and Tulare Counties). Fenpropathrin was not among the analytes typically measured in this study. It was analyzed in only one sample when Toxicity Identification Evaluation procedures suggested the presence of a pyrethroid, though none of the regularly quantified pyrethroid analytes were present. Further analysis of this single sample indicated the presence of 52 ng/g fenpropathrin.

Of all the pesticides measured, the most frequently detected were DDT and its degradates, DDE and DDD (frequency of detection 91%, 73%, and 34%, respectively). Maximum concentrations seen were 177 ng/g, 225 ng/g, and 15 ng/g, respectively. Even at these highest concentrations, these compounds were unlikely to significantly contribute to the observed H. azteca toxicity. The highest concentration of DDT corresponded to only 0.1 TU, and no other sample exceeded 0.02 TU of DDT. While DDT and its degradates may be of concern in the watershed for other reasons (e.g., bioaccumulation and trophic transfer), the compounds at the concentrations now prevailing in Central Valley sediments appear to have little potential for acute toxicity, at least to H. azteca, and, based on more limited data, to Chironomus tentans (Weston et al., 2004).

For the remainder of the pesticide analytes, detections were infrequent, and when present were at low concentrations not expected to contribute to the observed toxicity based on estimate toxicity thresholds (Weston et al., 2004), though measured thresholds are lacking for many of the compounds. The frequency of detection (at 1 ng/g reporting limit) and maximum concentration observed were: alpha-BHC (3%, 37 ng/g), beta-BHC (7%, 7 ng/g), gamma-BHC (1% 2 ng/g), delta-BHC (3%, 4 ng/g), heptachlor (4%, 3 ng/g), heptachlor epoxide (1%, 1 ng/g), aldrin (1%, 6 ng/g), alpha-chlordane (5%, 3 ng/g), gamma-chlordane (4%, 2 ng/g), endosulfan I (7%, 35 ng/g), endosulfan II (9%, 23 ng/g), endosulfan sulfate (11%, 14 ng/g), endrin aldehyde (4%, 11 ng/g), endrin ketone (5%, 138 ng/g), methoxychlor (16%, 190 ng/g).

3.5 Persistence of toxicity and sediment-associated pesticides

As this study was intended to assess toxicity across California's Central Valley, the emphasis of the design was on broad geographic distribution of sampling sites, and the majority of sites were sampled on only one or two occasions. However, a few sites were sampled repeatedly over several years, providing an opportunity to assess the persistence of sediment toxicity and the pesticide responsible for it (Figure 7). Such a field-based approach to studying persistence provides no control of environmental variables, thus it should be recognized that a decrease in chemical concentration may be the result of chemical degradation, burial of contaminated material beneath cleaner sediments, or transport of the contaminated material to more downstream sites by irrigation or storm-derived flow. The patterns discussed below, however, are not likely to be due to spatial heterogeneity, as triplicate samples were collected on one occasion from three sites discussed below (Sites

CS15, SED24, SED15), and negligible differences were found in chemical concentration among the replicates.

Site CS15, located in Spring Creek, Colusa County, CA, exhibited a dramatic increase in sediment toxicity in August of 2005, and an accompanying increase in sediment chlorpyrifos concentration, presumably due to summertime use of the compound. However, there was a 73% reduction in chlorpyrifos concentration by November 2005, and the intervening fall months since the peak concentrations in August were a period of minimal irrigation flow and rainfall, suggesting degradation in place. By the following spring the site contained no chlorpyrifos and was no longer toxic, at least in part due to sediment transport during the winter rains, and particularly heavy rainfall in March 2006.

Toxicity appeared in Stinson Canal (Site SED24) in the summer of 2005, most likely due to the presence of bifenthrin. The high toxicity and elevated bifenthrin concentrations persisted through November 2005, but were substantially diminished by the following spring. This site contained water only during the summer irrigation season, and thus persistence patterns observed in the winter months may not be indicative of aquatic systems.

SED15, an unnamed drain in Kings County, consistently showed high toxicity on every sampling occasion, due to a variety of pesticides including bifenthrin, chlorpyrifos and lambda-cyhalothrin. All these pesticides reached peak concentrations in August 2005, and chlorpyrifos in particular had been applied to an adjacent field within a couple weeks prior to the August sampling. By November of 2005, bifenthrin concentrations had declined by 74%, chlorpyrifos by 97%, and lambda-cyhalothrin by 100%, though based on toxicity units the concentration of bifenthrin was still high enough even after the decrease to account for mortality of H. azteca. In the intervening three months between the August and November samples there was minimal irrigation flow and no storm runoff capable of eroding sediments (<0.5 cm rainfall from August to November, 2005 in Visalia, CA; http://cdec.water.ca.gov/cgi-progs/queryMonthly?VSL), suggesting contaminant degradation as the reason for the reduction in concentrations.

The San Joaquin River near Vernalis was sampled every spring or summer from 2002 to 2005 (not shown in Figure 7). Toxicity was seen in 2002, possibly due to esfenvalerate, but no toxicity was observed in subsequent years. In 2004 there was 1.2 TU of bifenthrin in the sediments at this site, though without observed toxicity.

Overall, there were 31 instances when toxicity was seen and another sample taken at the same location in a subsequent sampling event 2-12 months later. When toxicity was seen in the first occasion, there was a 45% chance of observing it in the subsequent sample. In the study overall, there was a 27% frequency of toxicity, thus there is a substantially greater tendency to find toxicity at a site if it has historically shown toxicity. Persistence of toxicity may indicate slow contaminant degradation, minimal sediment transport, or on-going pesticide inputs from surrounding farmland. However, for slightly more than half the sites exhibiting sediment toxicity, toxicity is relatively short-lived and not observed if tested a few months later.

The field data gives the overall impression of less environmental persistence of the pyrethroids than is indicated by the very limited published data. Laboratory-based studies are available only for permethrin and bifenthrin, but indicate half-lives in sediments usually in the range of 6 months to a couple years (Gan et al., 2005). Comparison between the disparate field and lab persistence estimates is complicated by the difficulties noted above in deriving persistence from field data, but the data certainly indicate that further study of pyrethroid persistence in sediments is warranted.

4.0 Conclusions

Sediment toxicity is widespread in agricultural areas of the Central Valley, occurring in 29% of the sites. It is twice as common in constructed agricultural drains as in natural (though often highly modified) creeks and rivers. Nevertheless, even in these natural surface waters, about one-quarter of the sites showed acute sediment toxicity to H. azteca.

H. azteca is widely used throughout the U.S. for sediment toxicity assessment. The integration of sediment chemistry data with measured toxicity thresholds, as incorporated in the toxicity unit approach, proved highly effective in predicting which samples were likely to be toxic and identifying potential causative agents. Despite the necessary simplifying assumptions (the arbitrary 0.5 TU threshold for the onset of toxicity, the reasonable but untested assumption of the additivity of pyrethroid toxicity, the implied independence of pyrethroid and organophospate toxicity, the use of total organic carbon used as the sole normalizing factor for bioavailability), the toxicity unit approach when applied to pyrethroids and chlorpyrifos was successful in predicting toxicity with 84% accuracy (observing toxicity in samples >0.5 TU of either pyrethroids or chlorpyrifos, and not observing it when below that threshold). In 9% of the samples the approach underestimated the likelihood of toxicity, with toxicity observed despite <0.5 TU pyrethroids or chlorpyrifos, presumably due in part to unmeasured contaminants in the sediments. In 7% of the samples the approach overestimated the potential for toxicity (non-toxic despite >0.5 TU), often in cases of very coarse, low organic carbon sediments in which unquantified factors appeared to influence bioavailability.

There is strong evidence that pyrethroids, most notably bifenthrin and lambda-cyhalothrin (and secondarily, esfenvalerate), are responsible for much of the observed toxicity. Their role was implicated by the toxicity unit analysis, the dilution series data, and both temperature and PBO TIE procedures. Approximately one out of four Central Valley samples contained bifenthrin, and it was acutely toxic in one out of six sites. One out of six sediments contained lambda-cyhalothrin, and it was acutely toxic in one out of twelve sites. These compounds are not the most used pyrethroids in California agriculture, falling in 6th and 5th place, respectively, on a statewide basis (led by, in decreasing order, permethrin, fenpropathrin, esfenvalerate, and zeta-cypermethrin). Their contribution to H. azteca toxicity is attributable to a high sensitivity of the species to these pyrethroids (Amweg et al., 2005), or could be a consequence of greater environmental persistence leading to sediment concentrations out of proportion to their use.

While this study focused on agriculture-dominated water bodies, it is important to recognize that other surface waters in the Central Valley and elsewhere can also be affected by urban pyrethroid use. Non-agricultural bifenthrin use in California (primarily for pest control around homes and other structures) is 2.5 times greater than agricultural use, and the compound is a frequent contributor to H. azteca toxicity in urban creeks (Weston et al., 2005; Amweg et al., 2006a). The amount of lambda-cyhalothrin used in agriculture in California is only slightly greater than its non-agricultural use.

The only non-pyrethroid found to be a significant contributor to H. azteca toxicity was the organophosphate, chlorpyrifos. It contributed to toxicity about half as often as bifenthrin, and with comparable frequency as lambda-cyhalothrin. However, the use of chlorpyrifos in Central Valley agriculture is 100-fold greater than for these pyrethroids. The comparatively low incidence of sediment toxicity is probably due in part to a lower

hydrophobicity of the compound, allowing for greater dispersal and dilution of dissolved phase residues.

Historically, water quality concerns related to agricultural pesticides in the Central Valley have been greatest in the winter months, when organophosphates are applied to orchards, and heavy rains wash the residues in to surface water bodies (Werner et al., 2000). However, for the pesticides that appear to present a threat to sediment quality, the summer months are of greater concern. The compounds that contributed most to H. azteca toxicity were used largely (lambda-cyhalothrin ,esfenvalerate, chlorpyrifos) or entirely (bifenthrin) in the summer months. The sediments of the water bodies studied had higher concentrations of all these compounds in the summer months in nearly two-thirds of the cases. At least for the pyrethroids, half-lives for residues in aerobic soils, as in farm fields, are on the order of 1-2 months (Laskowski, 2002). Thus, in the five months from peak use (July) to the first heavy winter rains (usually December), it is likely that much of the pesticide has been degraded.

The greater summer use of the contaminants of concern, the higher concentrations in sediment usually observed during summer, and the relatively short persistence in farm soils all suggest that summer irrigation return flows, rather than winter storms, are likely to be the more important mechanism for transporting contaminated soils to the drains and creeks on which this study focused. Winter rains, and the accompanying high flows, may play a significant role in further downstream transport, moving the contaminated sediments in to the major rivers. If irrigation return flows are in fact the principal mechanism for transport to aquatic systems, this finding has important implications for management practices, since control of irrigation return flow and its associated eroded soil is more feasible than control of winter storm runoff. Measures developed for agricultural erosion control (e.g. polyacrylamide addition to irrigation water (McCutchan et al., 1993), vegetated ditches or filter strips (Moore et al., 2001)), particularly if focused on the finer particle sizes, ought to be effective in controlling entry of the pesticides of concern in to surface water bodies.

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Table 1. Frequency of H. azteca sediment toxicity in various water body types within the Central Valley.

Water Body Type	Number of sites	Proportion of sites with toxicity (%)
Unnamed drains	34	41
Named drains	17	24
Canals	8	13
Sloughs	28	11
Creeks	35	40
Creeks excluding westside[a]	27	26
Rivers	11	27

[a] "Westside" is a local designation for the area on the west side of the San Joaquin River in portions of Stanislaus and San Joaquin Counties. It includes sampling sites in Hospital, Ingram, Del Puerto, and Orestimba Creeks, all of which consistently have had sediments toxic to H. azteca.

Table 2. Comparison of observed TU_{oc}, derived from toxicity testing of a dilution series, with the expected TU, derived from the sediment chemical data. The range in the observed TU_{oc} reflects the 95% confidence interval of the calculated LC50.

Sample site	Sample date	Observed TU_{oc}	Expected pyrethroid TU (specific compound)	Expected chlorpyrifos TU
SED11 (unnamed drain)	Aug. 28, 2004	1.5-2.2	1.0 (esfenvalerate)	0
SED11 (unnamed drain)	Oct. 13, 2004	1.1-1.6	1.1 (esfenvalerate)	0
NSJ18 (Orestimba Creek)	Aug. 12, 2004	2.1-2.4	3.9 (lambda-cyhalothrin)	0.1
SED12 (Hospital Creek)	Oct. 13, 2004	3.6-4.6	5.6 (bifenthrin, lambda-cyhalothrin)	0
SED15 (unnamed drain)	Mar. 24, 2005	2.2-2.9	1.2 (lambda-cyhalothrin)	0.6
SED15 (unnamed drain)	Aug. 18, 2005	19-136	36 (bifenthrin, lambda-cyhalothrin)	24
CS15 (Spring Creek)	Aug. 9, 2005	1.6-2.2	0.9 (bifenthrin)	1.5
CS12 (unnamed drain)	Aug. 9, 2005	1.9-2.5	0.6 (fenpropathrin)	0.8

FT19 (unnamed drain)	Aug 2, 2005	2.8-3.7	0	1.6
FT19 (unnamed drain)	Aug. 19, 2005	3.3-4.9	0	5.3
SED40 (Del Puerto Creek)	Dec. 7, 2005	71-100	47 (bifenthrin)	0

Table 3. Proportion of sites (out of 117 total) with concentrations of one the measured analytes exceeding 0.5 TU. Those sites in bold type had H. azteca toxicity that statistically exceeded control.

Pesticide	% sites at or above 0.5 TU	Water bodies with 0.5 TU exceedances
Bifenthrin	16	3 rivers (San Joaquin, Tule, **Kaweah**) 5 creeks (**Spring, Hospital, Del Puerto**, **Orestimba**, Root) 2 sloughs (**Poso**, Elk Bayou) 1 canal (**Stinson**) 2 named drains (Crescent Ditch, **Boundary Drain**) 4 unnamed drains (FS, **TL**, **MA**, **SED15**)
Lambda-cyhalothrin	9	1 river (San Joaquin) 3 creeks (**Hospital, Del Puerto, Orestimba**) 2 sloughs (**Murphy**, **Poso**) 3 unnamed drains (FS, **MA**, **SED15**)
Chlorpyrifos	8	1 creek (**Spring**) 1 slough (**Poso**) 3 named drains (**Holland Drain**, Button Ditch, **Knestric Ditch**) 4 unnamed drains (**FT19**, **CS12**, **SED15**, **AD2**)
Esfenvalerate	4	2 creeks (**Littlejohn**, **Del Puerto**) 1 named drain (**Knestric Ditch**) 2 unnamed drains (**AD6**, **SED11**)
Cypermethrin	3	1 named drain (**Knestric Ditch**) 1 unnamed drain (**SED23**)
Permethrin	2	1 creek (Root) 1 unnamed drain (**AD5**)
Fenpropathrin[a]	unknown	1 unnamed drain (**CS12**)
Endrin	1	1 named drain (TID#3)

[a] Fenpropathrin was only analyzed in a single sample; thus this table may underestimate its prevalence or contribution to toxicity.

FIGURE LEGENDS

Figure 1. Distribution of H. azteca toxicity among the Central Valley sediment sampling sites. County names are abbreviated as: BU=Butte, CO=Colusa, FR=Fresno, GL=Glenn, KE=Kern, KI=Kings, MA=Madera, ME=Merced, SA=Sacramento, SJ=San Joaquin, SO=Solano, ST=Stanislaus, SU=Sutter, TE=Tehama, TU=Tulare, YO=Yolo, YU=Yuba. The Central Valley counties of Shasta and Placer are not shown as there were no samples taken in those locations. The inset map shows the location of the study area within the state of California.

Figure 2. Proportions of the 53 toxic samples containing at least 0.5 TU of the indicated analytes, suggesting a potential causal relationship for the toxicity.

Figure 3. Relationship between total pyrethroid TU and H. azteca mortality in the same sediment samples data from all 180 samples for which both chemistry and toxicity results are available. A TU value of 0.01 has been arbitrarily used for those samples in which pyrethroids were undetected.

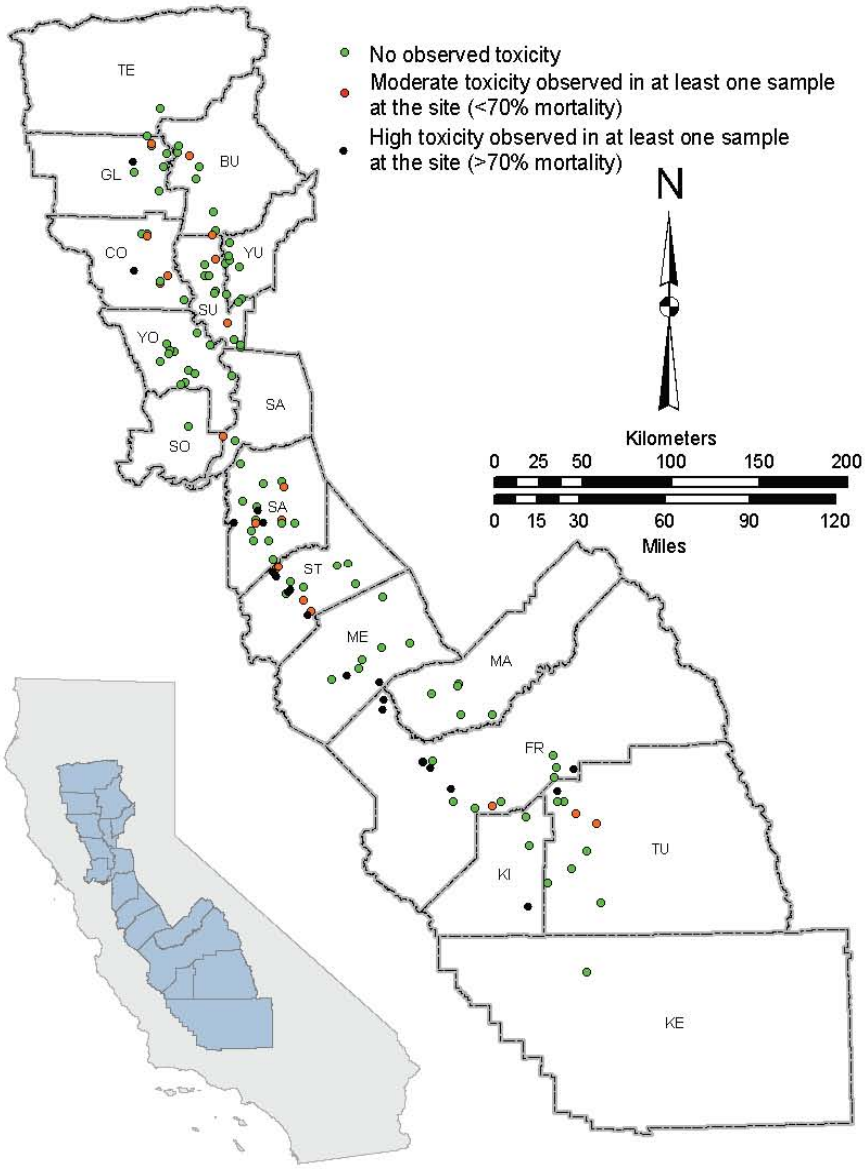
Figure 4. Concentration of bifenthrin at each of the study sites, with the highest concentration shown if the site was sampled on multiple occasions. The breakpoints between the four categories of concentration correspond to specific TU thresholds assuming a generic organic carbon content of 1%: undetected, detectable but acute H. azteca toxicity unlikely (<0.5 TU), toxicity likely (0.5-2 TU), high toxicity likely (>2 TU). The figure also shows the annual usage of bifenthrin within each county, normalized to area of harvested cropland, and the monthly use of bifenthrin in Central Valley agriculture using 2004 data. County abbreviations are defined in Figure 1.

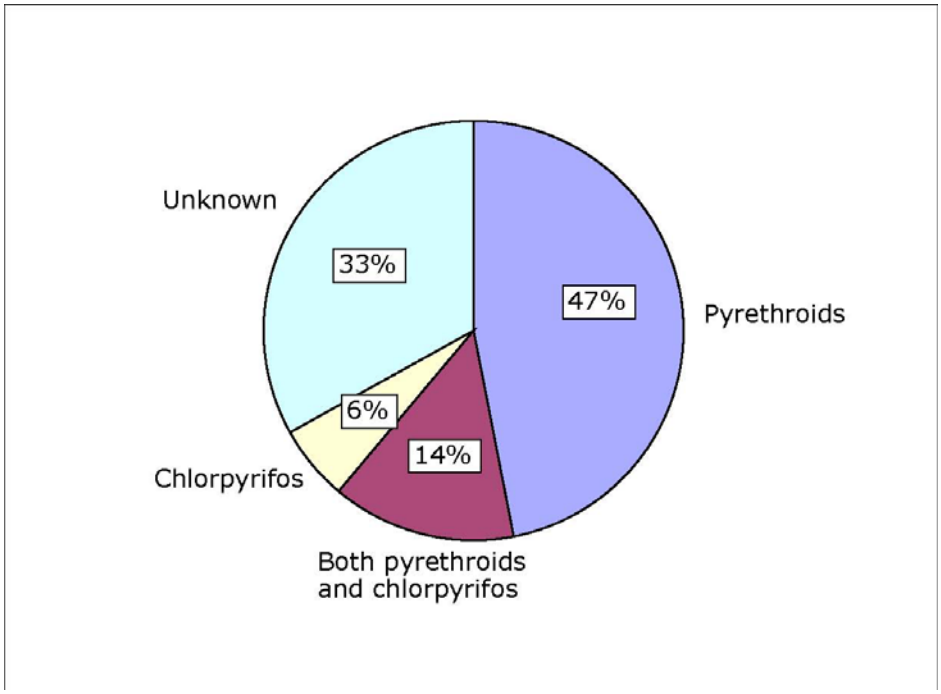
Figure 5. Concentration of lambda-cyhalothrin at each of the study sites, with the highest concentration shown if the site was sampled on multiple occasions. The breakpoints between the four categories of concentration correspond to specific TU thresholds assuming a generic organic carbon content of 1%: undetected, detectable but acute H. azteca toxicity unlikely (<0.5 TU), toxicity likely (0.5-2 TU), high toxicity likely (>2 TU). The figure also shows the annual usage of lambda-cyhalothrin within each county, normalized to area of harvested cropland, and the monthly use of lambda-cyhalothrin in Central Valley agriculture using 2004 data. County abbreviations are defined in Figure 1.

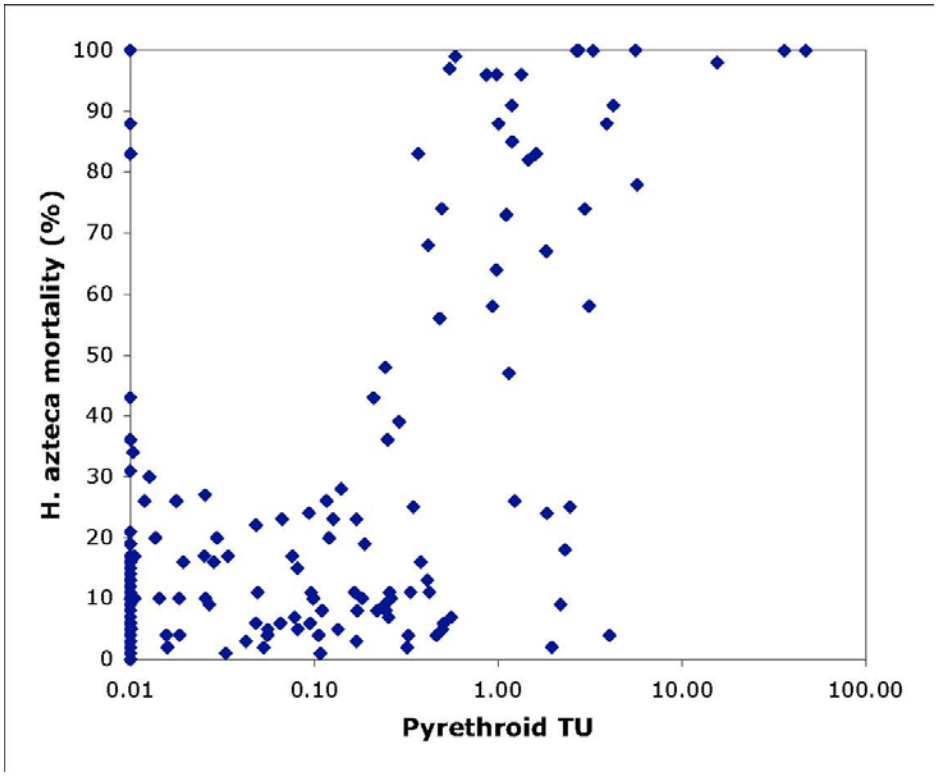
Figure 6. Concentration of chlorpyrifos at each of the study sites, with the highest concentration shown if the site was sampled on multiple occasions. The breakpoints between the four categories of concentration correspond to specific TU thresholds assuming a generic organic carbon content of 1%: undetected, detectable but acute H. azteca toxicity unlikely (<0.5 TU), toxicity likely (0.5-2 TU), high toxicity likely (>2 TU). The figure also shows the annual usage of chlorpyrifos within each county, normalized to area of harvested cropland, and the monthly use of chlorpyrifos in Central Valley agriculture using 2004 data. County abbreviations are defined in Figure 1.

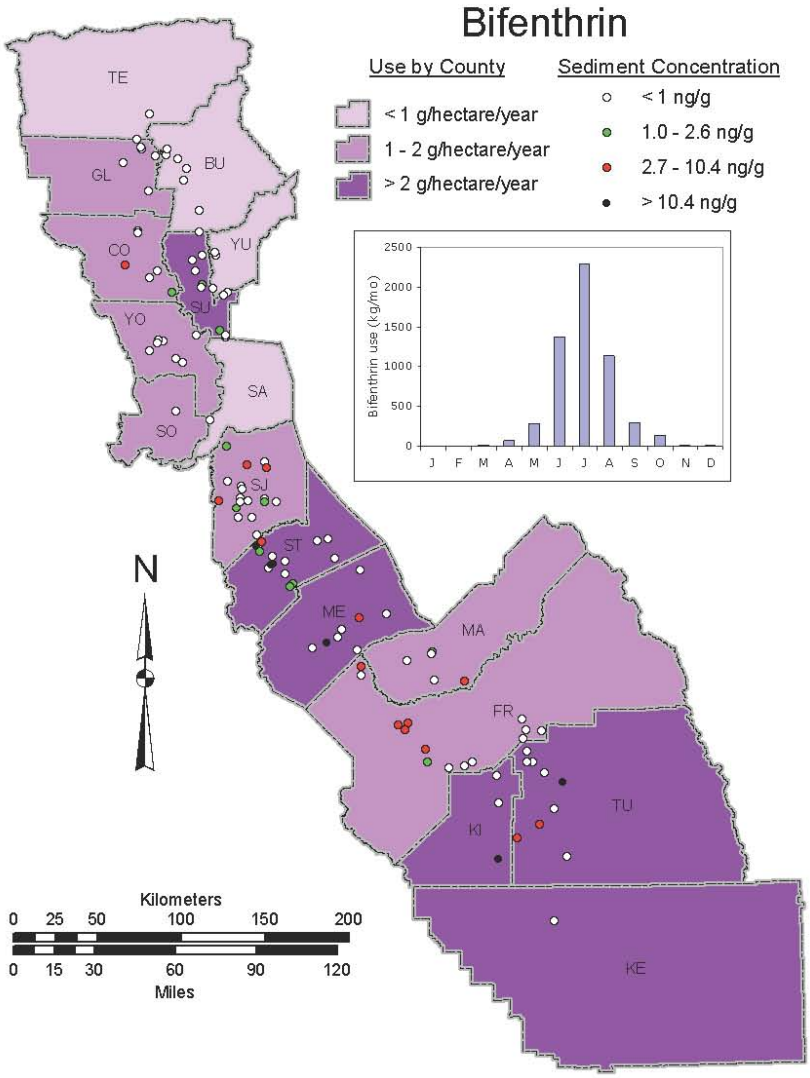
Figure 7. Peristence of toxicity and the pesticides likely contributing to it (based on TU

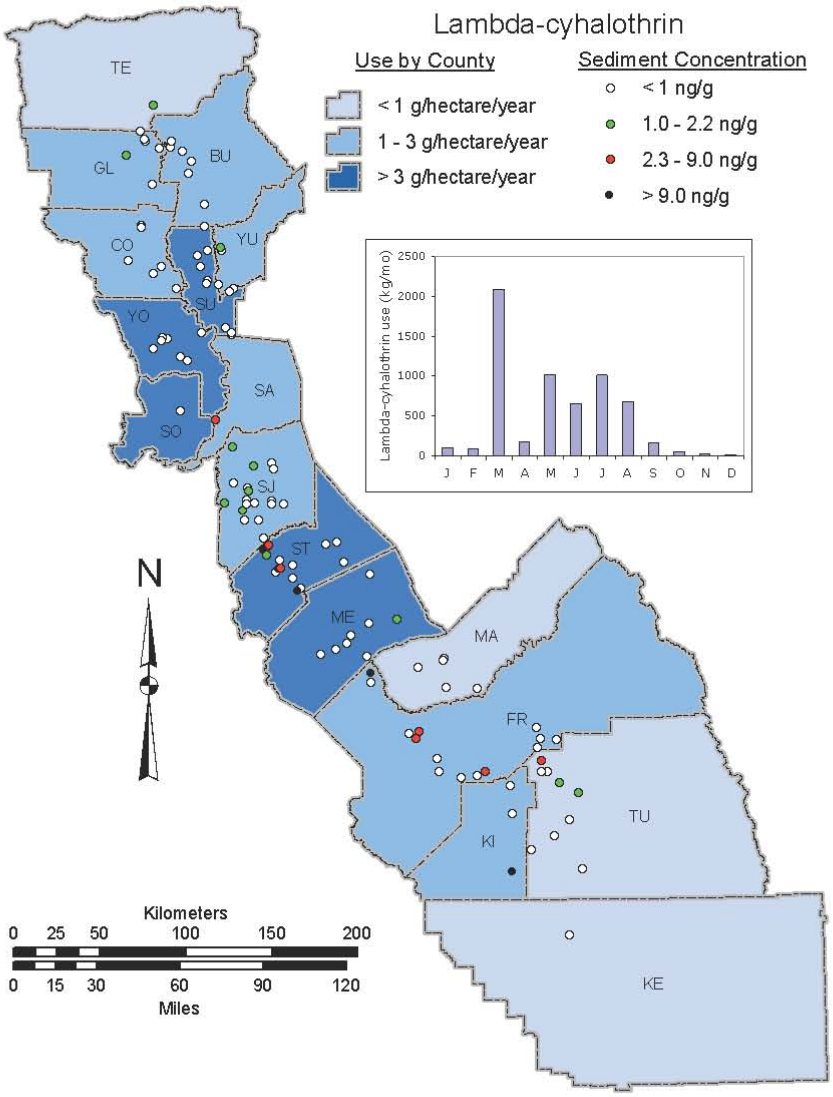
calculations) at three sampling sites at which sediments were repeatedly sampled.

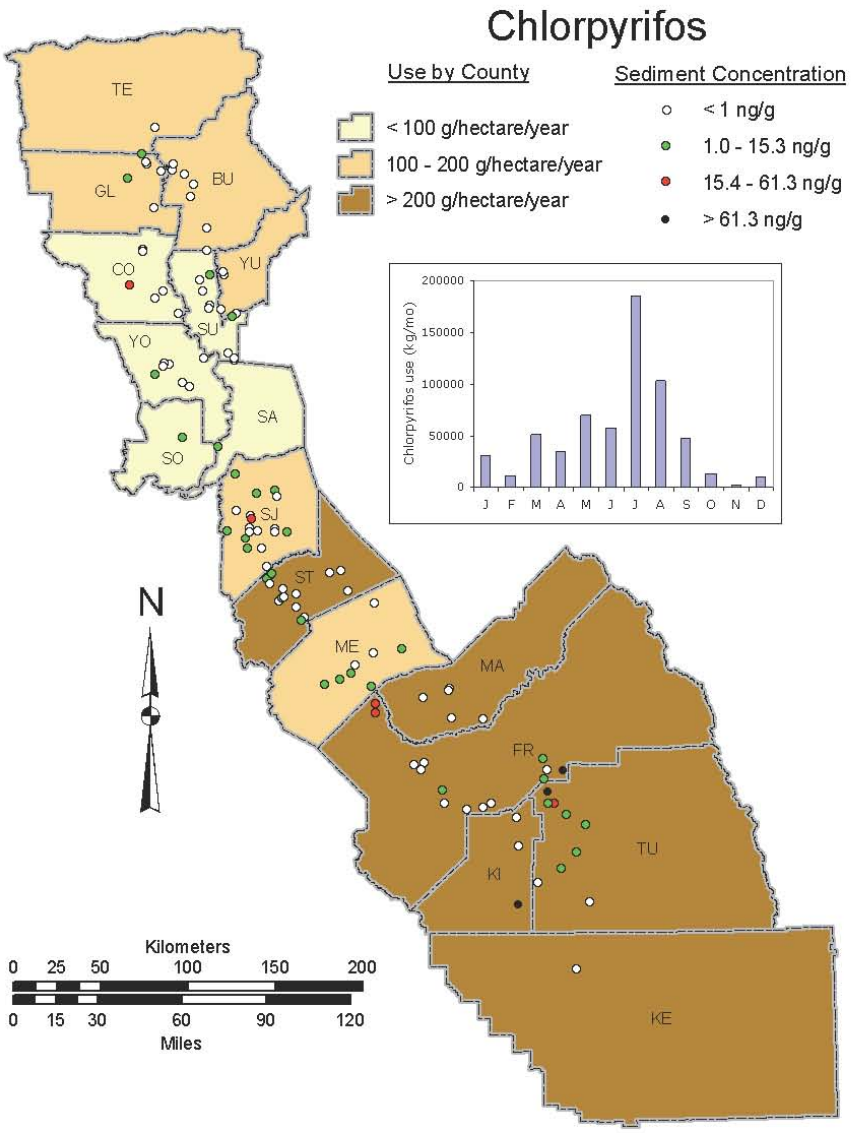


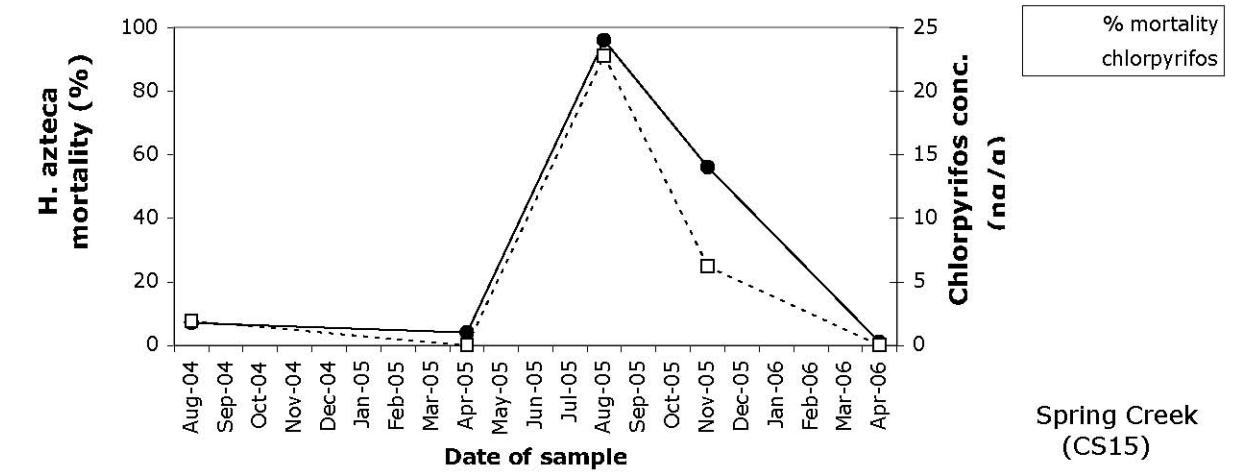

Unknown
33%
47%
Pyrethroids
6%
14%
Chlorpyrifos
Both pyrethroids
and chlorpyrifos

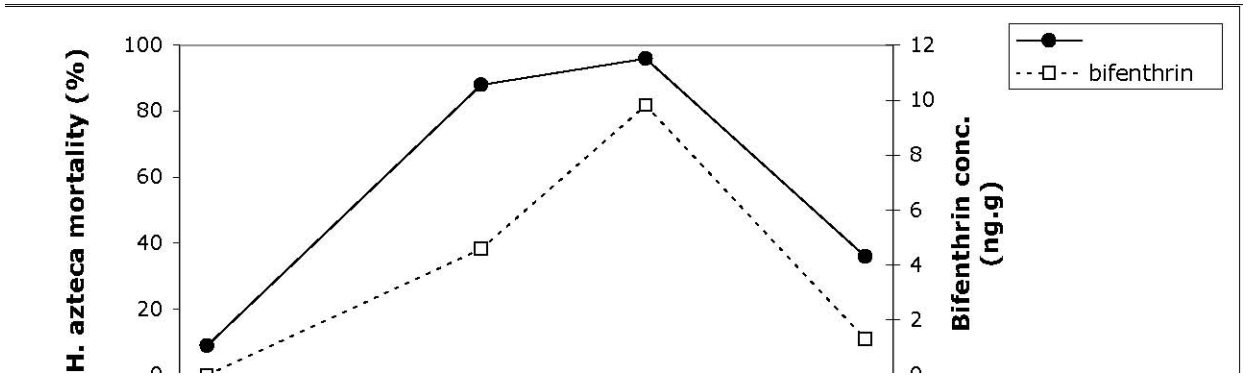

H. azteca mortality (%)
100
90
80
70
60
50
40
30
20
10
0
0.01
0.10
1.00
10.00
100.00
Pyrethroid TU

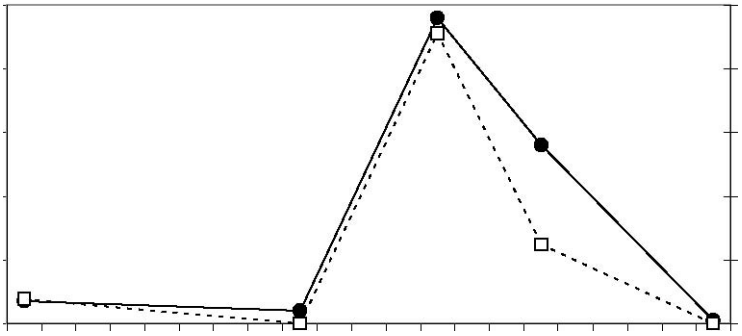

Bifenthrin
Use by County
< 1 g/hectare/year
1 - 2 g/hectare/year
> 2 g/hectare/year
Sediment Concentration
< 1 ng/g
1.0 - 2.6 ng/g
2.7 - 10.4 ng/g
> 10.4 ng/g
Bifenthrin use (kg/mo)
2500
2000
1500
1000
500
0
J F M A M J J A S O N D
TE
BU
GL
CO
YU
SU
YO
SA
SO
SJ
ST
ME
MA
FR
KI
TU
KE
N
Kilometers
0 25 50 100 150 200
0 15 30 60 90 120
Miles

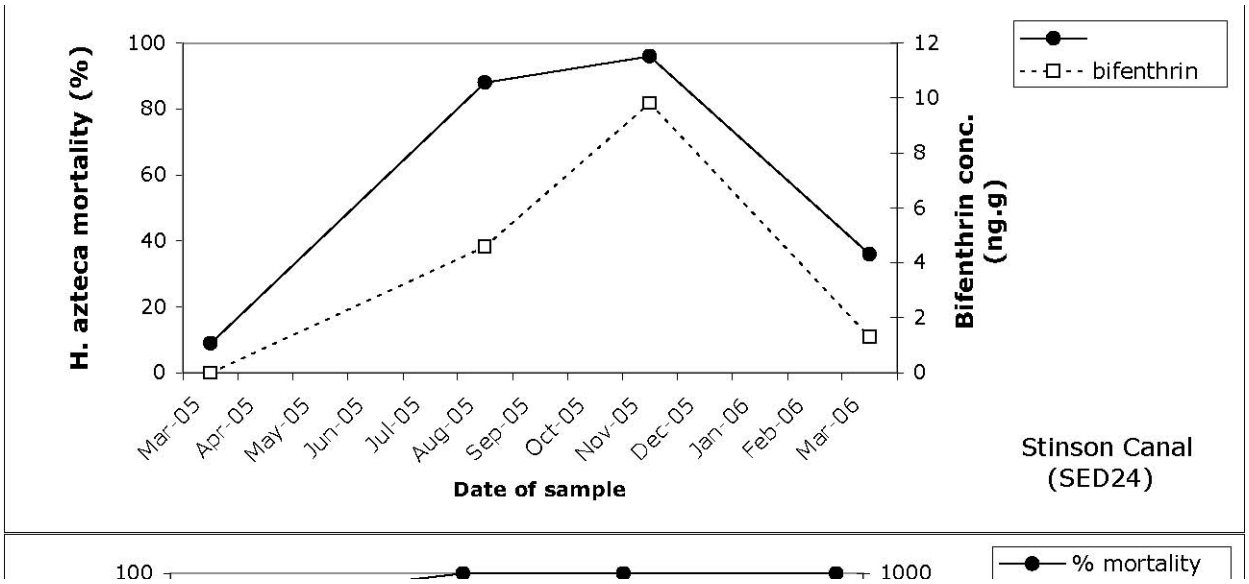

Lambda-cyhalothrin
Use by County
< 1 g/hectare/year
1 - 3 g/hectare/year
> 3 g/hectare/year
Sediment Concentration
< 1 ng/g
1.0 - 2.2 ng/g
2.3 - 9.0 ng/g
> 9.0 ng/g
Lambda-cyhalothrin use (kg/mo)
2500
2000
1500
1000
500
0
J F M A M J J A S O N D
TE
GL
BU
CO
YU
SU
YO
SA
SO
SJ
ST
ME
MA
FR
TU
KI
KE
N
Kilometers
0 25 50 100 150 200
0 15 30 60 90 120
Miles

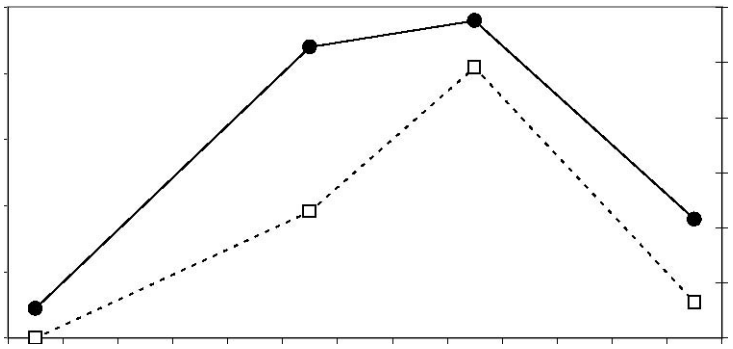

Chlorpyrifos
Use by County
< 100 g/hectare/year
100 - 200 g/hectare/year
> 200 g/hectare/year
Sediment Concentration
< 1 ng/g
1.0 - 15.3 ng/g
15.4 - 61.3 ng/g
> 61.3 ng/g
Chlorpyrifos use (kg/mo)
200000
150000
100000
50000
0
J F M A M J J A S O N D
TE
GL
BU
CO
YU
SU
YO
SA
SO
SJ
ST
ME
MA
FR
KI
TU
KE
N
Kilometers
0 25 50 100 150 200
0 15 30 60 90 120
Miles

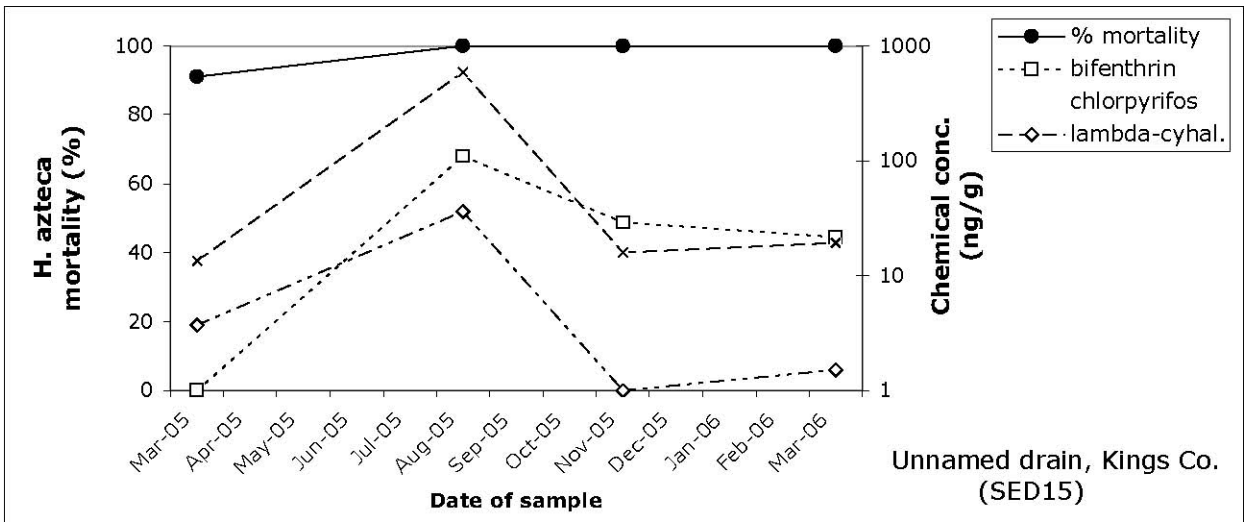

% mortality
chlorpyrifos
H. azteca mortality (%)
Chlorpyrifos conc. (ng/g)
Date of sample
Aug-04
Sep-04
Oct-04
Nov-04
Dec-04
Jan-05
Feb-05
Mar-05
Apr-05
May-05
Jun-05
Jul-05
Aug-05
Sep-05
Oct-05
Nov-05
Dec-05
Jan-06
Feb-06
Mar-06
Apr-06
Spring Creek
(CS15)


bifenthrin
H. azteca mortality (%)
Bifenthrin conc. (ng.g)




H. azteca mortality (%)
Bifenthrin conc. (ng.g)
bifenthrin
Mar-05
Apr-05
May-05
Jun-05
Jul-05
Aug-05
Sep-05
Oct-05
Nov-05
Dec-05
Jan-06
Feb-06
Mar-06
Date of sample
Stinson Canal
(SED24)
% mortality




H. azteca mortality (%)
100
80
60
40
20
0
1000
100
10
1
Chemical conc. (ng/g)
% mortality
bifenthrin
chlorpyrifos
lambda-cyhal.
Mar-05
Apr-05
May-05
Jun-05
Jul-05
Aug-05
Sep-05
Oct-05
Nov-05
Dec-05
Jan-06
Feb-06
Mar-06
Date of sample
Unnamed drain, Kings Co. (SED15)