

Pesticide Concentrations in Water and Sediment and Associated Invertebrate
Toxicity in Del Puerto and Orestimba Creeks, California

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November 19, 2009



CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY

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Report 243

ABSTRACT

The San Joaquin Valley is an important agricultural production area in California, where more than 1.5 million pounds of organophosphorous (OP) and pyrethroid insecticides are applied annually. The major river flowing through the valley, the San Joaquin River (SJR), is listed on the 2006 Clean Water Act §303(d) list for pesticide impairment. Several SJR tributaries are also listed, including Orestimba (ORC) and Del Puerto (DPC) Creeks. From December 2007 through June 2008, water and sediment samples were collected from ORC and DPC in Stanislaus County to determine concentrations of OP and pyrethroid insecticides, and to identify related toxicity to *Ceriodaphnia dubia* and *Hyalella azteca*. OPs were detected in almost half (10 of 21) of the water samples, at concentrations from 0.005 to 0.912 $\mu\text{g L}^{-1}$. Diazinon was the most frequently detected OP, followed by chlorpyrifos and dimethoate. Two water samples were toxic to *C. dubia*; based on LC_{50}s , chlorpyrifos was likely the cause of this toxicity. Pyrethroids were detected more frequently in sediment samples (18 detections) than in water samples (three detections). Pyrethroid concentrations in water samples ranged from 0.005 to 0.021 $\mu\text{g L}^{-1}$. These concentrations were well below reported *C. dubia* LC_{50}s and toxicity was not observed in laboratory bioassays. Cyfluthrin, bifenthrin, esfenvalerate, and λ -cyhalothrin were detected in sediment samples at concentrations ranging from 1.0 to 74.4 ng g^{-1} , dry weight. At DPC, all but one sample caused 100% toxicity to *H. azteca*. Based on estimated toxicity units (TUs) calculated from measured pyrethroid concentrations in sediment, bifenthrin was likely responsible for this toxicity; λ -cyhalothrin also probably contributed. At ORC, survival of *H. azteca* was significantly reduced in four of the 11 sediment samples. However, pyrethroids were detected in only two of these samples. Based on TUs, bifenthrin and λ -cyhalothrin likely contributed to toxicity in these two samples.

ACKNOWLEDGEMENTS

Many people have generously given their time to help this study succeed. I would like to thank Sheryl Gill for working with CURES and securing the contracts that were needed before this project could be initiated. I would like to thank the staff at DPR, Environmental Monitoring Branch for assisting in the field sampling. I would like to thank Carissa Ganapathy for sample coordination and organization between DPR and CDFG and to Jessie Ybarra for his help in maintaining DPR's West Sacramento's facility. I extend gratitude to the staff at CDFG for sample analysis and to the staff at UCD ATL for conducting the toxicity studies. Finally, I thank the California State Water Resources Control Board for funding this project through a grant from the Pesticide Research and Investigation of Source and Mitigation (PRISM) Grant Program.

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I. INTRODUCTION

The San Joaquin River (SJR) watershed is an important agricultural production area in the Central Valley of California. The SJR drains about 32,000 square miles through the San Joaquin Valley. In the SJR watershed, applicators apply greater than 22 million lb active ingredient (a.i.) of pesticides yearly (CDPR, 2009a). Pesticides are frequently detected in the SJR and observed to cause toxicity to indicator species. As a result, the Central Valley Regional Water Quality Control Board has placed the SJR on the 2006 Clean Water Act §303(d) list for pesticide impairment (CA EPA, 2009). Orestimba (ORC) and Del Puerto (DPC) Creeks are tributaries of the SJR; these two creeks are also on the §303(d) list due to the presence of organophosphorous (OP) insecticides. In addition, ORC and DPC are listed for sediment toxicity (unknown source) and for pyrethroid insecticides, respectively (Cal/EPA, 2009).

Researchers have routinely detected OPs and pyrethroids in water or sediment samples taken from DPC and ORC. An analysis by Spurlock (2002) showed that between 1991 – 2001 diazinon and chlorpyrifos were routinely monitored and detected in rivers and tributaries in the Central Valley, including ORC. Eleven percent of the water samples from this creek during this timeframe were toxic to *Ceriodaphnia dubia*, although neither diazinon nor chlorpyrifos were likely the sole toxicant. In more recent work, researchers detected chlorpyrifos, dimethoate, diazinon, azinphos-methyl, dichlorvos, methyl parathion, and disulfoton at ORC or DPC; however, no toxicity testing was conducted in these studies (Starner *et al.*, 2003; Kelley and Starner, 2004). Bacey *et al.* (2004) noted that OP concentrations of diazinon and chlorpyrifos were sufficient to account for the observed toxicity to *C. dubia* at DPC. In other studies, OPs have routinely been detected and linked to toxicity of aquatic invertebrates (Hunt, *et al.*, 2003; Werner, *et al.*, 2000).

In the past several years, OP agricultural use has decreased. In place of these insecticides, growers are using more pyrethroids. With this increased use, pyrethroids have been detected in creek water and sediments. Bacey *et al.* (2004) detected the pyrethroid esfenvalerate in the water column and in sediment at DPC. In other studies at DPC, additional pyrethroids (permethrin, esfenvalerate, bifenthrin, λ -cyhalothrin, cyfluthrin) were frequently detected in sediments at concentrations sufficiently high to have caused toxicity to *Hyalella azteca* (Weston *et al.*, 2004; Weston *et al.*, 2008a).

Currently, both OPs and pyrethroids are used to control a myriad of pests. In the 2008 water year (WY)¹, 158,057 and 31,379 lb a.i., respectively, were applied in Stanislaus County (CDPR, 2009a). OPs were primarily used in production agriculture, but pyrethroids were intensively used in both production and non-production agriculture² (Tables 1 and 2). For both classes of insecticides, the heaviest agriculture use is May through September. Chlorpyrifos, dimethoate, and phosmet have the highest OP

¹ The 2008 water year is from October 1, 2007 through September 30, 2008. The 2008 water year is the time-frame of this study.

² Non-production agriculture includes uses as applications to parks and recreation areas, golf courses, cemeteries, roadsides, railway rights-of-way, and structural pest control and is required to be reported in CDPR's PUR database (CDPR, 2009a). Non-agricultural uses that are not reported in the PUR are mostly homeowner and most institutional and industrial uses.

production agricultural use; bifenthrin, esfenvalerate, λ -cyhalothrin, and permethrin have the highest pyrethroid use. However, over half of all pyrethroid applications are for non-production agricultural use. Permethrin and cypermethrin have the largest non-production agriculture use, with permethrin by far having the most use.

Although there have been numerous monitoring studies at both ORC and DPC, there is a lack of recent published monitoring data collected over consecutive months, especially with concurrent toxicity testing. This study had two objectives:

1. Determine the occurrence and concentrations of OPs in water and of pyrethroids in water and sediment from DPC and ORC during both the rainy and irrigation seasons; and
2. Determine the toxicity of creek water and sediments to the representative aquatic invertebrate organisms, *C. dubia* and *H. azteca*, respectively.

II. MATERIALS AND METHODS

Study Area. DPC and ORC run through the west side of Stanislaus County. At DPC, we sampled at Vineyard Avenue near the town of Patterson and at ORC on River Road near the town of Crows Landing (Fig. 1).

Field Sampling. Water and sediment samples were collected monthly from December 2007 through June 2008. In December, January, and February, one additional sample per month was collected during a rain event. Sediment samples could not be collected at every storm event due to high and rapid water flow.

Water samples were collected near midstream directly into 1-L glass amber bottles using an extendable pole. Prior to water collection, bottles were given a native rinse. Bottles were sealed with Teflon®-lined lids. Sediments were collected using a stainless steel trowel or small shovel. Soil sediments (up to a 2 cm depth) were put into clear glass Mason® jars (for chemical analysis) or into pre-cleaned 1-L polypropylene containers (for toxicity studies). Sediments were generally taken from the same areas at both ORC and DPC due to limited sediment accumulation at the selected sampling sites. Both creeks are often scoured clean by rapid water flow; thus the creekbeds are mainly gravel with a few selected spots where sediments build up.

Immediately after sampling, water and sediment samples were stored on wet ice at 4°C for transport. Upon arrival at the laboratory, water samples and sediments for grain size and toxicity testing were refrigerated (4°C) whereas sediments for pyrethroid chemical analysis and for TOC (total organic carbon) analysis were frozen (−20°C). Water samples were analyzed for OPs and pyrethroids and sediment samples were analyzed for pyrethroids. Several other parameters were also analyzed:

- Total suspended solids (TSS) in water samples, following US EPA method (US EPA, 1971) and as described in Kelley and Starner (2004);
- TOC in sediment samples, following method of Gunasekara, 2006;
- Sediment grain size (analyzed by Applied Marine Science, Inc.).

Water Quality Measurements. Dissolved oxygen (DO), electrical conductivity (EC), salinity, pH, and temperature were measured *in situ*. Measurements were taken with a YSI 85 meter, YSI 60 meter, or YSI 6920 V2 meter (YSI Incorporated, Yellow Springs, Ohio). Prior to use, all instruments were calibrated according to manufacturer's recommendations (<http://www.ysi.com>) or CDPR SOPs (<http://www.cdpr.ca.gov/docs/emon/pubs/sop.htm>).

Analytical Chemistry. Chemical analyses were conducted by California Department of Fish and Game (CDFG), Fish and Wildlife Water Pollution Control Laboratory. CDFG employed the following methods (CDPR, 2009b):

- OPs: liquid-liquid extraction and high resolution gas chromatography with Flame Photometric Detector and Thermionic Bead Specific Detector;
- Pyrethroids: liquid-liquid extraction and high resolution gas chromatography with electron capture detector (GC/ECD).

For both procedures, CDFG used gas chromatography/mass spectrometry and ion trap detection (GC/MS-ITD) to confirm detections. Table 3 lists the method detection limits (MDL) and reporting limits (RL) for each chemical analyzed. The MDL is the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The reporting limit is usually one to five times the MDL, dependent on analytical method and matrix (Segawa, 1995). We report the results as:

- nd (not detected, concentrations below the MDL);
- trace (trace detection, where in the chemist's best professional judgment the analyte does exist between the RL and the MDL);
- a numerical concentration in $\mu\text{g L}^{-1}$ (water samples) or ng g^{-1} (dry weight; sediment samples).

Toxicity Testing. In addition to the physical and chemical analysis, UCD Aquatic Toxicity Laboratory (ATL) conducted aquatic toxicity tests with *C. dubia* (water samples) and with *H. azteca* (sediment samples). Within 24 h of collection, water and sediment samples were transported to UCD ATL for toxicity testing. UCD ATL uses standard EPA methods for these toxicity tests (University of California, Davis, 2009). Briefly, for water toxicity tests, five *C. dubia* neonates per replicate (four replicates per treatment) were placed into glass scintillation vials containing 18 mL of the water sample. Scintillation vials were housed in an environmental chamber at 25°C with a 16:8 h light:dark photoperiod. Every 24 h, water was renewed and *C. dubia* was fed daily. After 96 h, the surviving organisms were counted and the percent survival was compared to an untreated control. For sediment studies, 10 - 14 day old *H. azteca* were placed into 300-mL glass beakers containing control water and sampled creek sediments. Beakers were placed in an environmental chamber or water bath maintained at 23°C with a 16:8 hour light:dark photoperiod. Water was renewed twice daily and *H. azteca* was fed after the second daily water change. After 10 days, *H. azteca* was removed from the beakers, dried at 86°C for 16 h, cooled and weighed. Percent survival was also recorded.

QA/QC for Water, Sediment, and Toxicity Samples. Quality control for this study followed the CDPR SOP guidelines on Chemistry Laboratory Quality Control (Segawa, 1995) and the Quality Assurance Project Plan (QAPP) written for this study (CDPR, 2009c). For OP and pyrethroid water analysis, quality control consisted of surrogate analytes (“surrogates”), method blanks, laboratory control samples, and laboratory control sample duplicates. In addition, there were two blind spikes, two field duplicates, and two field blanks (5% of the field samples for each). For pyrethroids in sediments, quality control consisted of two surrogates, method blanks, laboratory control samples, laboratory control sample duplicates, matrix spikes, and matrix spike duplicates. In addition, 5% of the field samples were field duplicates. QA/QC for toxicity testing comprised of field duplicates (10%) and field blanks (5%).

III. RESULTS AND DISCUSSION

Quality Control

Generally, QC was within control limits; exceptions are discussed below (Appendixes 1 – 3)

Holding times were within control limits for all water samples, sediment samples (including grain size and TOC analysis), and samples sent to UCD ATL except:

- Three of 50 water samples exceeded the 40 day holding time by one day (analyzed on day 41; Table A1, Appendix 1);
- Holding times for some of the TSS samples were exceeded (Table A6, Appendix 1).

In water and sediment samples, all 184 surrogates were within control limits³. The RPD (relative percent differences) between surrogates added to laboratory duplicate samples were within the RPD control limit (of $\leq 25\%$ differences as defined in the QAPP written for this study) in 29 of 30 samples. The one sample outside the control limit occurred with the first water analysis and thereafter the RPD were within the control limit (Table A2, Appendix 1; Table A10, Appendix 2).

The recovery of spiked laboratory control samples from OP-analyzed water samples was outside of the control limits in 4% of the analyses. Low recovery of phosmet was the most common of the exceedances; five times it was below the lower control limit (LCU). Also, dimethoate and azinphos-methyl exceeded the upper control limit (UCL) in one analysis. In addition, in the OP-analyzed water samples, 8% of the laboratory duplicates were outside of the RPD control limit. However, because OPs were within analytical control limits in the extraction sets where there were detections or during months of OP use in the watersheds, we assume that the variability in spike recoveries do not reflect on the analytical results (Table A3, Appendix 1).

The recovery of spiked laboratory control samples from pyrethroid-analyzed water samples was outside of the control limits in 7% of the analyses. In most cases,

³ Control limits are defined as 75 - 125% recovery for all surrogate, laboratory control samples, and matrix spikes as defined in CDPR 2009b.

exceedances were due to low recoveries of the pyrethroids, which could have led to underreporting of the pyrethroids in water samples. In addition, 9% of the laboratory duplicates were outside of the RPD control limit (Table A4, Appendix 1).

In pyrethroid sediment samples, the recovery of spiked laboratory control samples or matrix samples was outside of the control limits in 5% of the analysis, with no exceedances of the RPD criterion (Table A11, Appendix 2). However, in one lab duplicate analysis, the RPD of bifenthrin was 34.5% (Table A12, Appendix 2). Other than this incident with bifenthrin, QC problems in the sediment samples were mostly due to low recovery of deltamethrin (Table A11, Appendix 2). Deltamethrin had virtually no use in the Stanislaus County during the course of this study; therefore, it is unlikely that this pyrethroid would have been present in the samples (Table 2).

The final QC problem occurred in one of the field blanks, where diazinon was reported below its RL but above its MDL (Table A5, Appendix 1). Unfortunately, these results were received after the field sampling was complete and additional field blanks could not be collected. The analytical results from the field sample taken at the same time (March at DPC) also reported a trace detection of diazinon; this detection may be an artifact.

Water Samples

Water Quality Measurements. Water temperature, pH, and EC (conductivity and salinity) were significantly higher at DPC than at ORC based on paired t-tests ($p < 0.05$). In addition, DPC water quality parameters were generally more variable than ORC. DPC is a much smaller creek than ORC, with about one-fifth of the flow. Thus, the lower baseline flow at DPC may allow rain or irrigation runoff to have a greater relative impact on DPC water quality. More variable or adverse water quality could potentially have a negative effect on sensitive aquatic species. All of the water quality data can be found in Table A17, Appendix IV.

Total Suspended Sediments. TSS in the water samples ranged from 4.8 mg L^{-1} to 287 mg L^{-1} at DPC and from 7.2 mg L^{-1} to 98.1 mg L^{-1} at ORC, except for the January storm sample at ORC where the TSS concentration was 1766 mg L^{-1} . The TSS concentrations were not significantly different between the two creeks (Appendix IV, Table A18).

Pesticide Detections. We collected 21 water samples for OP and pyrethroid analysis. In these 21 water samples, 12 samples contained OPs (Tables 4 and 5). Diazinon was the most frequently detected OP (six detections) and was detected in the highest concentration of any pesticide in this study ($0.912 \text{ } \mu\text{g L}^{-1}$). Diazinon was detected during the dormant season⁴ (four detections) and during the irrigation season (two detections). In the dormant season, diazinon was detected during rain and regular monthly (non-storm) sampling events.

Chlorpyrifos was the second most commonly detected OP (four detections). Dimethoate was detected twice, but no other OPs were detected. Both chlorpyrifos and dimethoate

⁴ Dormant season sampling occurred in December, January, and February. Irrigation season sampling occurred in March through June. In the 2008 WY, there was no rain after February 20 (CIMIS, 2008).

were only detected during the irrigation season, which is the highest use season for these two OPs.

In both the DPC and ORC watersheds, there was sufficient reported production agriculture use around the time and location of sampling to account for the dimethoate and chlorpyrifos detections. However, the source of the diazinon detections is unknown. Currently, there is no reported diazinon use around the time of the diazinon detections (CDPR, unpublished data⁵).

Only three pyrethroids were detected out of the 21 water samples collected, all at ORC (Tables 4 and 5). Cyfluthrin was detected in January's storm sample; bifenthrin and λ -cyhalothrin were detected in March. As discussed in the Quality Control section, pyrethroid water concentrations may be low-biased due to low recoveries (Table A4, Appendix 1). Only the λ -cyhalothrin detection was consistent with reported production agriculture use (CDPR, unpublished data).

***C. dubia* Toxicity.** UCD ATL tested 21 water samples for toxicity to *C. dubia*. Only two of these samples (May and June 26) from ORC were toxic to *C. dubia* (Tables 4 and 5). Based on the acute LC₅₀ of chlorpyrifos to aquatic invertebrates (0.05 $\mu\text{g L}^{-1}$; US EPA, 2009a), chlorpyrifos concentrations in these two water samples were high enough to cause toxicity to *C. dubia*. Chlorpyrifos was also detected in the June 10 water sample at ORC (trace detection) and in the June 26 water sample at DPC (0.035 $\mu\text{g L}^{-1}$), concentrations not likely to be toxic.

Although no other water samples from either creek caused toxicity to *C. dubia* (Tables 4 and 5), based on the acute LC₅₀ of diazinon to aquatic invertebrates (0.105 $\mu\text{g L}^{-1}$; US EPA, 2009a), the monthly February ORC sample (0.912 $\mu\text{g L}^{-1}$) exceeded the *C. dubia* acute LC₅₀ and both of the January DPC samples were essentially equal to diazinon's acute LC₅₀. In these water samples, the lack of toxicity to *C. dubia* is unexplained. Diazinon was also detected in three other samples from DPC, but at concentrations much lower than its LC₅₀. We also detected dimethoate in two water samples, at 0.074 and 0.190 $\mu\text{g L}^{-1}$. However, at these concentrations it would not likely be toxic to *C. dubia*. The LC₅₀ of dimethoate to aquatic invertebrate organisms is more than two orders of magnitude greater than these detections (21.5 $\mu\text{g L}^{-1}$; US EPA, 2009a).

The three ORC water samples with detectable levels of pyrethroids did not cause any toxicity to *C. dubia*. Although there are no water aquatic benchmarks for pyrethroids (US EPA, 2009a), the US EPA does list LC₅₀ values for the pyrethroids in its Ecotox database (US EPA, 2009b). The pyrethroids were detected at an order of magnitude less than their LC₅₀s, thus the lack of toxicity is consistent with these values. Pyrethroids are much more toxic to *H. azteca* than they are to *C. dubia* (Haver *et al.*, 2008). *H. azteca* may be a better choice for toxicity testing in pyrethroid-containing aquatic systems.

⁵ The 2008 CDPR PUR data is currently in draft form and not available for public use.

Sediment Samples

Grain Size and Total Organic Carbon (TOC). At DPC and ORC, fine grain particles (“fines”: silt and clay particles less than 62.5 μm) ranged from 15.3 to 82.6% and TOC ranged from 0.28 to 1.39% (Table A19, Appendix IV). Sediments with these properties are likely to contain pyrethroids which could be potentially toxic to *H. azteca*. Other work has shown that sediments with a similar composition contained pyrethroids and were toxic to *H. azteca* (Holmes *et al.*, 2008; Weston *et al.*, 2004; You *et al.*, 2008).

Pesticide Detections. Nineteen sediment samples were collected for pyrethroid analysis. Twelve of the 19 samples contained pyrethroids, with some samples containing up to three different pyrethroids. Most of the pyrethroid detections occurred at DPC, where all of the nine sediment samples contained at least one pyrethroid. In all, we detected 14 pyrethroids at DPC (Table 6). ORC sediment was much less contaminated with pyrethroids; only three of the 10 sediment samples contained pyrethroids.

Bifenthrin was the most frequently detected pyrethroid (10 detections) and detected at the highest concentration (74.4 ng g^{-1}). It was detected in all but one of the samples at DPC but was only detected twice at ORC. Bifenthrin was detected in both the dormant and irrigation season with about equal frequency, and was detected at higher concentrations in the irrigation season.

Three other pyrethroids were detected: λ -cyhalothrin (four detections), cyfluthrin (twice) and esfenvalerate (twice) (Table 6). Esfenvalerate was detected in December at both creeks whereas cyfluthrin was detected in the irrigation season at DPC. Lambda-cyhalothrin was detected twice in the dormant season and twice in the irrigation season. There was sufficient reported use around the time and location of sampling to account for the λ -cyhalothrin and esfenvalerate detections. However, this is not the case for the bifenthrin and cyfluthrin detections (CDPR, unpublished data).

***H. azteca* Toxicity.** Due to differing toxicities of pyrethroids, toxicity units (TUs) are commonly used to identify pyrethroids that may contribute to sediment toxicity (Amweg *et al.*, 2005). To calculate TUs, pyrethroid concentration, normalized to organic carbon, are divided by 10-day sediment LC_{50} values (also normalized to organic carbon). As common practice, we used previously published LC_{50} values (Amweg, 2005; Maund, 2002) to calculate TUs. We expect 50% *H. azteca* toxicity with 1 TU, and 0.5 TU can be used as an approximate concentration where toxicity would likely begin to appear (Weston *et al.*, 2008b). TUs for the different pyrethroids are assumed to be additive due to a similar mode of action.

Every sediment sample from DPC caused 100% mortality to *H. azteca* in the UCD ATL toxicity screen, except for the sediment from the December storm sample which caused almost 80% mortality (Table 6). From calculated TUs, bifenthrin was the major contributor of toxicity at DPC. Except for the December sample, bifenthrin was detected in every sediment sample at this creek, containing between 1.5 and 15 TUs. Cyfluthrin, λ -cyhalothrin and esfenvalerate were also detected at DPC, but only λ -cyhalothrin would

have contributed to toxicity, with between 0.6 to 1.8 TUs. Cyfluthrin and esfenvalerate contributed a minor amount, less than 0.5 TUs (Fig. 3).

At ORC, the sediment was not as toxic. Four of the 11 sediment samples caused significant toxicity to *H. azteca*. In these four samples, survivability ranged from 55 to 72% of the control levels (Table 6). Pyrethroids were only detected in December's two sampling dates and in the first June sampling date. In December's storm sample, λ -cyhalothrin had 0.97 TUs and likely caused the toxicity to *H. azteca* (esfenvalerate also had 0.1 TU). The December monthly sample contained 0.44 TU, all due to bifenthrin (Fig. 4). The 0.44 TU could cause some toxicity to *H. azteca*. Amweg *et al.* (2005) found that a majority of sediment samples with TUs greater than 0.4 resulted in 40% or higher *H. azteca* toxicity.

The June 10 sample from ORC contained almost 6 TUs, all due to bifenthrin. However, there was no toxicity associated with this sediment sample. This occurs occasionally; in other research, scientists have reported high TUs with little or no toxicity (Amweg *et al.*, 2005; Amweg *et al.*, 2006; Weston *et al.*, 2008b). This is generally attributed to lack of bioavailability due to sediment factors other than TOC, which has already been factored into the TU analysis. Sediments with medium sands or coarser grain sizes tend to give artificially high TUs with corresponding low toxicity (Amweg *et al.*, 2006; Weston *et al.*, 2008b). However, the June 10 sample contained greater than 50% fines and thus would not explain reduced toxicity. Undetermined factors likely reduced bioavailability of this sediment.

At ORC, no other pyrethroids were detected in any of the other sediment samples, yet significant toxicity to *H. azteca* occurred in February (monthly) and in April. The source of the toxicity is unknown, likely due to some other contaminant or stressor other than pyrethroids.

At ORC, TUs were not predictive of *H. azteca* toxicity (Fig. 5). Approximately 40 to 50% *H. azteca* mortality was associated with anywhere from none to almost 1 TU, and bifenthrin's almost 6 TUs had no *H. azteca* toxicity. However, at DPC, TUs greater than 1 TU were always associated with 100% toxicity of *H. azteca*. In one instance where there was only 0.13 TU, higher than expected toxicity was observed (Fig. 5, point a). Some other contaminant or stressor (*e.g.*, chlorpyrifos or metals), or perhaps a synergist (as PBO), may have caused this higher than expected toxicity. We did not analyze the sediment samples for any of these other stressors.

IV. CONCLUSIONS

The main conclusions were concise but clear. OPs were detected in water; pyrethroids were mainly detected in sediments (although a few were detected in water samples at ORC).

Diazinon and chlorpyrifos were the most frequently detected OPs. Both were detected at concentrations greater or equal to their respective acute aquatic invertebrate LC₅₀s. However, of the 21 collected water samples, only two samples were toxic to *C. dubia*.

These two samples only contained chlorpyrifos at high enough concentrations to cause the toxicity. Diazinon was not associated with toxicity in the *C. dubia* toxicity tests. Dimethoate was also detected, but at concentrations much lower than its reported acute aquatic invertebrate toxicity. Chlorpyrifos and dimethoate were detected during the irrigation season and were associated with reported agricultural production use. Diazinon was detected in the dormant (both storm and non-storm sampling) and during the irrigation season, but in current PUR database records, diazinon had little to no reported production agriculture use in the DPC or ORC watersheds.

Bifenthrin and λ -cyhalothrin were the most commonly detected pyrethroids in sediment, and both (based on TUs) were highly associated with toxicity to *H. azteca*. However, bifenthrin by far was the most commonly detected pyrethroid in sediment. It was detected every month, at the highest concentration of any pyrethroid, had the highest TUs, and likely contributed the most to *H. azteca* toxicity. Cyfluthrin and esfenvalerate were also detected in sediments, albeit less frequently and were not associated with toxicity to *H. azteca*.

When bifenthrin was detected, it was not associated with reported production agriculture use during the sampling period. In the DPC and ORC watersheds, bifenthrin has reported production agriculture use May through September, with no use the remainder of the year. Bifenthrin detections were attributed to the long persistence of this pyrethroid. Esfenvalerate and λ -cyhalothrin detections were associated with reported production agriculture use but, as in the water detections, cyfluthrin detections were not associated with reported use.

Comparing the two creeks, Sediments from DPC were highly contaminated with pyrethroids, whereas sediments from ORC were less contaminated. Thus, at DPC, overall TUs were predictive of toxicity to *H. azteca* but at ORC TUs were not.

V. LITERATURE CITED

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Table 1. Organophosphorous (OP) insecticide use (lb a.i.) in Stanislaus County, California, for the 2008 water year.

OP	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	June	July	Aug	Sep	TOTAL
OP Production Agriculture Use in Stanislaus County													
Azinphos-Methyl	0	0	0	0	0	0	0	236	119	415	35	0	804
Chlorpyrifos	3,946	329	419	917	147	950	1,880	18,003	6,369	25,979	8,879	3,319	71,137
Diazinon	208	13	677	1,603	21	1	54	61	0	139	68	14	2,859
Dimethoate	361	44	0	94	0	1,678	147	240	2,159	12,081	37,125	1,418	55,348
Ethoprop	0	0	0	0	0	0	1,072	4,012	0	0	0	0	5,084
Malathion	0	0	0	0	0	1,540	59	199	172	401	1,301	738	4,411
Methidathion	0	14	119	49	0	0	0	0	0	0	0	0	181
Methyl Parathion	0	0	0	0	0	0	0	1,020	871	868	0	0	2,759
Naled	310	0	0	0	0	0	0	0	0	105	350	3	768
Phosmet	0	0	0	0	0	0	759	3,529	3,495	4,360	1,238	694	14,076
Agriculture Total	4,824	401	1,215	2,662	168	4,170	3,971	27,300	13,185	44,349	48,997	6,187	157,428
OP Non-Production Agriculture Use in Stanislaus County													
Chlorpyrifos	3	0	0	0	0	2	0	0	0	2	1	4	13
Diazinon	0	0	0	0	0	0	0	0	0	1	0	8	9
Dimethoate	0	0	0	0	0	0	1	0	0	0	0	0	1
Malathion	51	6	3	39	34	40	71	96	48	64	6	4	462
Naled	64	12	0	0	0	0	0	0	0	25	24	19	145
Non-Agriculture Total	118	19	3	39	34	42	72	97	48	91	32	36	629
Grand Total	4,942	419	1,218	2,701	201	4,212	4,043	27,397	13,233	44,440	49,028	6,223	158,057

Table 2. Pyrethroid insecticide (lb a.i.) use in Stanislaus County, California, for the 2008 water year.

Pyrethroid	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	June	July	Aug	Sep	Total
Pyrethroid Production Agriculture Use in Stanislaus County													
Bifenthrin	0	1	0	24	2	6	1	35	449	2,533	497	90	3,639
Cyfluthrin ¹	0	9	0	0	0	4	4	14	21	55	29	8	144
Cypermethrin ²	2	4	0	0	0	20	15	31	4	14	5	0	94
Deltamethrin	0	0	0	0	0	0	0	0	2	4	0	0	6
Esfenvalerate	27	0	90	270	70	12	19	630	297	1,201	74	35	2,726
Fenpropathrin	0	0	0	0	0	0	81	7	28	29	81	21	247
Gamma-Cyhalothrin	0	0	0	2	0	0	4	6	5	5	0	0	23
Lambda-Cyhalothrin	30	3	2	60	7	605	47	723	364	935	525	89	3,390
Permethrin	19	3	0	189	0	31	61	744	190	596	33	335	2,202
Agriculture Total	79	20	92	545	78	679	231	2,191	1,361	5,372	1,245	578	12,471
Pyrethroid Non-Production Agriculture Use in Stanislaus County													
Bifenthrin	90	38	24	27	33	38	37	37	52	33	24	119	554
Cyfluthrin ¹	11	8	5	4	4	13	55	102	86	96	115	101	599
Cypermethrin ²	70	131	48	35	175	119	1,041	399	276	501	190	207	3,192
Deltamethrin	2	1	1	2	1	2	2	2	2	3	4	2	23
Esfenvalerate	0	0	1	2	55	3	4	0	0	0	0	0	66
Lambda-Cyhalothrin	5	1	2	2	1	1	2	2	5	8	7	6	41
Permethrin	269	107	64	70	119	87	836	163	218	2,909	4,547	5,042	14,432
Non-Agriculture Total	446	287	144	142	389	264	1,978	704	639	3,550	4,887	5,477	18,908
Grand Total	525	307	236	687	468	943	2,209	2,896	1,999	8,922	6,132	6,055	31,379

¹includes β -cyfluthrin and cyfluthrin

²includes (S)-cypermethrin and cypermethrin

Table 3. Insecticides analyzed by the California Department of Fish and Game in water or sediment, with their method detection and reporting limits.

Compound	Method Detection Limit ($\mu\text{g L}^{-1}$)	Reporting Limit ($\mu\text{g L}^{-1}$)
Organophosphorous in Water (by GC/FPD)		
Azinphos methyl	0.030	0.050
Chlorpyrifos	0.010	0.020
Diazinon	0.005	0.020
Dimethoate	0.030	0.050
Disulfoton	0.010	0.050
Malathion	0.030	0.050
Methidathion	0.030	0.050
Methyl Parathion	0.010	0.050
Phorate	0.030	0.050
Phosmet	0.030	0.050
Pyrethroids in Water (by GC/ECD)		
Bifenthrin	0.001	0.002
Cyfluthrin, total ¹	0.002	0.004
Cypermethrin, total ²	0.002	0.004
Deltamethrin	0.002	0.004
Esfenvalerate, total ³	0.001	0.002
Fenpropathrin	0.002	0.004
λ -Cyhalothrin	0.001	0.002
Permethrin (cis, trans isomers or mixed isomers)	0.003	0.005
Pyrethroids in Sediment (by GC/ECD)	Method Detection Limit (ng g^{-1})	Reporting Limit (ng g^{-1})
Bifenthrin	0.500	1.00
Cyfluthrin, total ¹	2.00	4.00
Cypermethrin, total ²	2.00	4.00
Deltamethrin	2.00	4.00
Esfenvalerate, total ³	1.00	2.00
Fenpropathrin	2.00	4.00
Permethrin, (cis, trans isomers or mixed isomers)	2.00	5.00
λ -Cyhalothrin, total	2.00	4.00

¹contains both cyfluthrin and β -cyfluthrin

²contains both cypermethrin and (S)-cypermethrin

³contains both esfenvalerate and fenvalerate

Table 4. OP and pyrethroid detections and survivability of *C. dubia* in water samples collected at Del Puerto Creek, California.

Month	Type		Azinphos methyl	Chlorpyrifos	Diazinon	Dimethoate	Disulfoton	Malathion	Methidathion	Parathion, methyl	Phorate	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	λ-Cyhalothrin	Permethrin, cis	Permethrin, trans	C. dubia survival
	Storm	Monthly																			
																					-----Concentration (µg L ⁻¹) -----
Dec	X		nd ¹	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Dec		X	Missing values (MV), no water in creek to sample																		MV
Jan	X		nd	nd	0.100	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Jan		X	nd	nd	0.101	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Feb	X		nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Feb		X	nd	nd	trace ²	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Mar		X	nd	nd	trace ³	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Apr		X	nd	nd	nd	0.074	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
May		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
June 10		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
June 26		X	nd	0.035	trace	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100

¹nd, not detected (below the minimum detection limit)

²trace detection (below the reporting limit but above the minimum detection limit)

³A field blank sample also was analyzed as a trace detection of diazinon

Table 5. OP and pyrethroid detections and survivability of *C. dubia* in water samples collected at Orestimba Creek, California.

Month	Type		Azinphos methyl	Chlorpyrifos	Diazinon	Dimethoate	Disulfoton	Malathion	Methidathion	Parathion, methyl	Phorate	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	λ-Cyhalothrin	Permethrin, cis	Permethrin, trans	C. dubia survival
	Storm	Monthly																			
Dec	X		nd ¹	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Dec		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	105
Jan	X		nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.021	nd	nd	nd	nd	nd	nd	nd	95
Jan		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Feb	X		nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Feb		X	nd	nd	0.912	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
Mar		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.005	nd	nd	nd	nd	nd	0.016	nd	nd	100
Apr		X	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	100
May		X	nd	0.079	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0
June 10		X	nd	trace ²	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	96
June 26		X	nd	0.046	nd	0.190	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	65

¹nd, not detected (below the minimum detection limit)

²trace detection (below the reporting limit but above the minimum detection limit)

[‡]Bolded and highlighted cells are significantly different from the control water

Table 6. Grain size, percent organic carbon, pyrethroid detections, and survivability of *H. azteca* in sediments collected at Del Puerto Creek and Orestimba Creeks, California.

Month collected	Collection type		Grain Size (% fines ¹)	Total Organic Carbon (%)	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	Permethrin, cis	Permethrin, trans	λ-Cyhalothrin	H. azteca survival		
	Storm	Non-storm Monthly			----- Concentration (ng g ⁻¹ , dry weight) -----											% of control‡
DPC																
Dec	X		28.8	0.50	nd ²	nd	nd	nd	trace ³	nd	nd	nd	nd	18 / 35 ⁴		
Dec		X	Missing values, no water in creek to sample													
Jan	X		Missing values, current too swift to collect sediment samples													
Jan		X	64.6	1.11	8.57	nd	nd	nd	nd	nd	nd	nd	nd	0		
Feb	X		82.0	1.18	24.4	nd	nd	nd	nd	nd	nd	nd	nd	0		
Feb		X	85.9	0.78	17.4	nd	nd	nd	nd	nd	nd	nd	trace	0		
Mar		X	86.2	0.89	69.06	4.19	nd	nd	nd	nd	nd	nd	3.58	0		
Apr		X	74.6	1.11	62.68	trace	nd	nd	nd	nd	nd	nd	8.98	0		
May		X	81.6	0.96	74.4	nd	nd	nd	nd	nd	nd	nd	nd	0		
June 10		X	66.1	1.20	28.2	nd	nd	nd	nd	nd	nd	nd	nd	0		
June 26		X	79.9	1.39	43.6	nd	nd	nd	nd	nd	nd	nd	nd	0		

Table 6 continued.

Month collected	Collection type		Grain Size (% fines ¹)	% Total Organic Carbon	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	Permethrin, cis	Permethrin, trans	λ-Cyhalothrin	H. azteca survival		
	Storm	Non-storm Monthly			Concentration (ng g ⁻¹ , dry weight)											% of control‡
					ORC											
Dec	X		75.1	1.14	nd ²	nd	nd	nd	trace	nd	nd	nd	5 ⁵	82 / 56 ⁴		
Dec		X	81.1	1.28	2.9	nd	nd	nd	nd	nd	nd	nd	nd	72		
Jan	X		Missing values, current too swift to collect sediment samples													
Jan		X	58.0	0.95	nd	nd	nd	nd	nd	nd	nd	nd	nd	157 ⁶		
Feb	X		24.8	0.28	nd	nd	nd	nd	nd	nd	nd	nd	nd	84		
Feb		X	15.3	0.31	nd	nd	nd	nd	nd	nd	nd	nd	nd	56		
Mar		X	55.7	0.78	nd	nd	nd	nd	nd	nd	nd	nd	nd	91		
Apr		X	61.1	1.29	nd	nd	nd	nd	nd	nd	nd	nd	nd	55		
May		X	65.9	1.16	nd	nd	nd	nd	nd	nd	nd	nd	nd	91		
June 10		X	53.8	1.17	36.3	nd	nd	nd	nd	nd	nd	nd	nd	94		
June 26		X	72.4	1.31	nd	nd	nd	nd	nd	nd	nd	nd	nd	78		

¹Fines are silt and clay particles < 62.5 μ m in size²nd, not detected (below the minimum detection limit)³trace detection (below the reporting limit but above the minimum detection limit)⁴Field sample and field duplicate, respectively⁵Average of two lab analyses.⁶Test did not meet USEPA criteria for control survival

‡Bolded and highlighted cells are significantly different from the control sediment

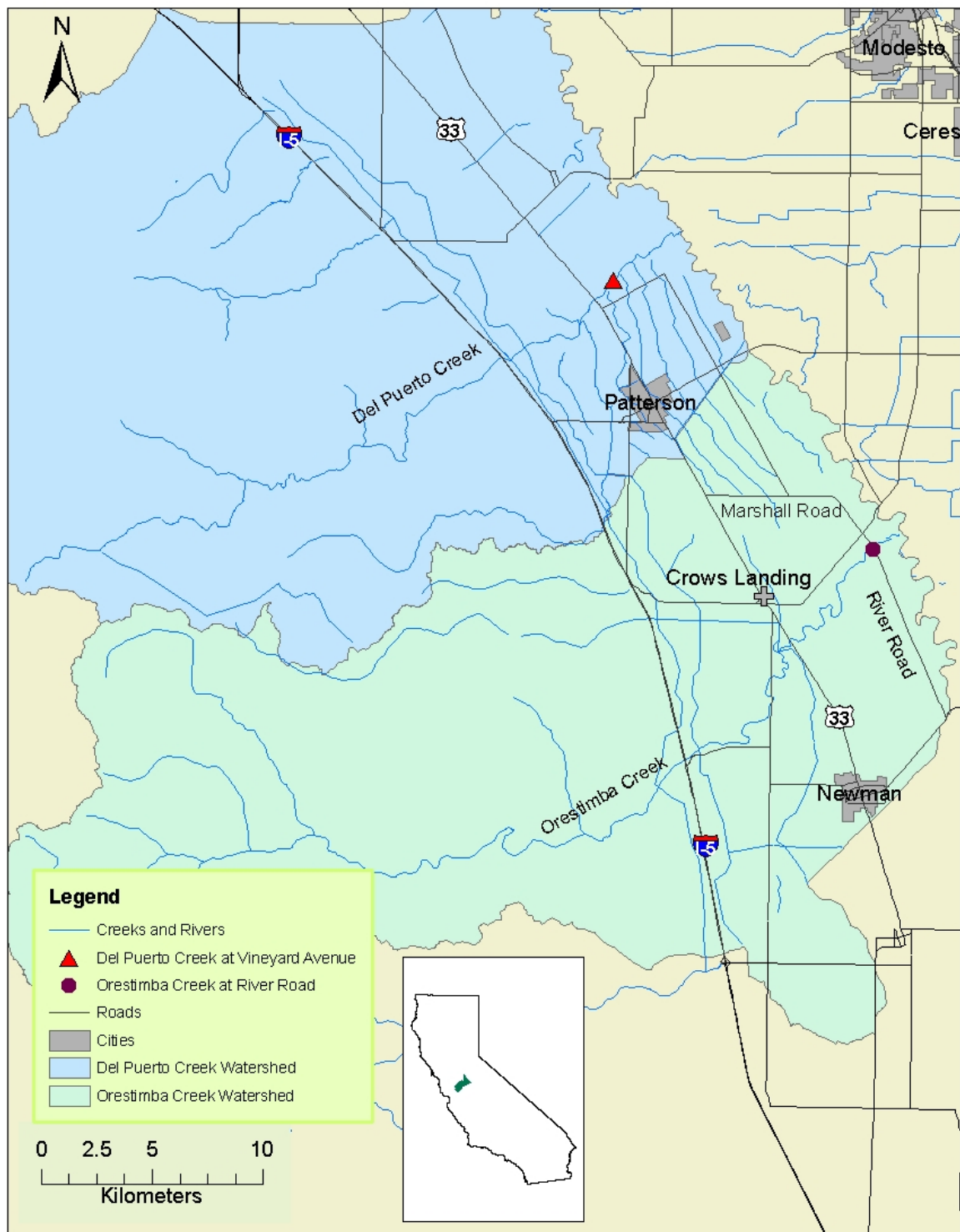


Figure 1. Sampling sites at Orestimba Creek and at Del Puerto Creek, California. GPS Coordinates (NAD83): ORC, N37.41395 W121.01495; DPC, N37.52145 W121.14863. ORC and DPC watersheds have previously been described by Zhang *et al.* (2008).

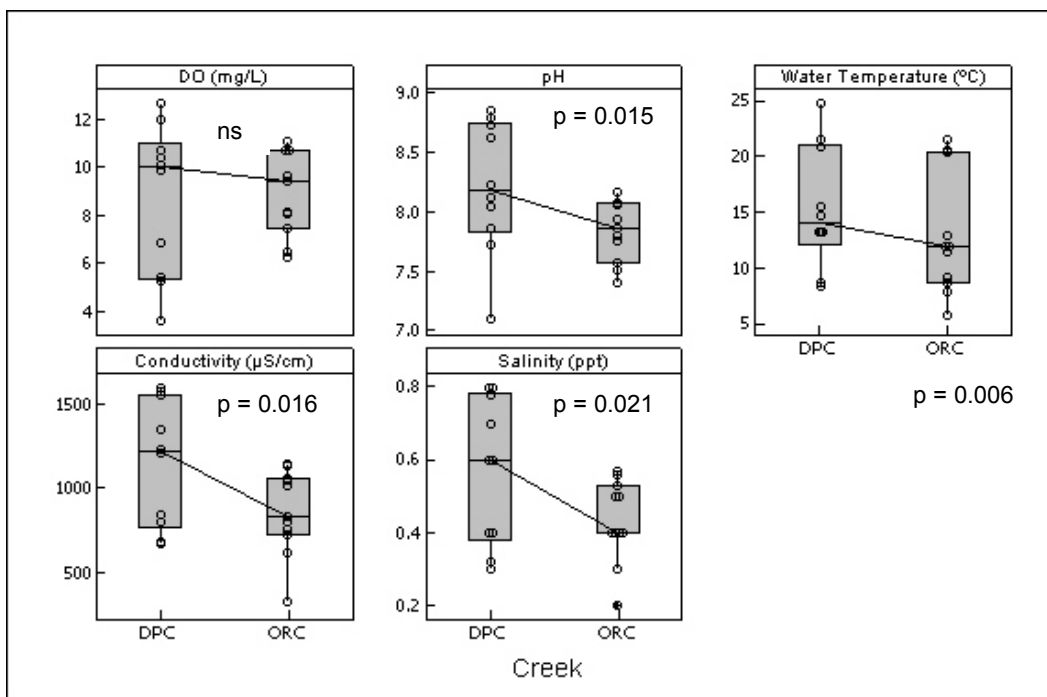


Figure 2. Box plots of water quality measurements at Del Puerto Creek (DPC) and at Orestimba Creek (ORC). P values indicate significant differences at the 5% level in paired *t*-tests of the mean differences between the two creeks (DPC greater than ORC; ns, not significant).

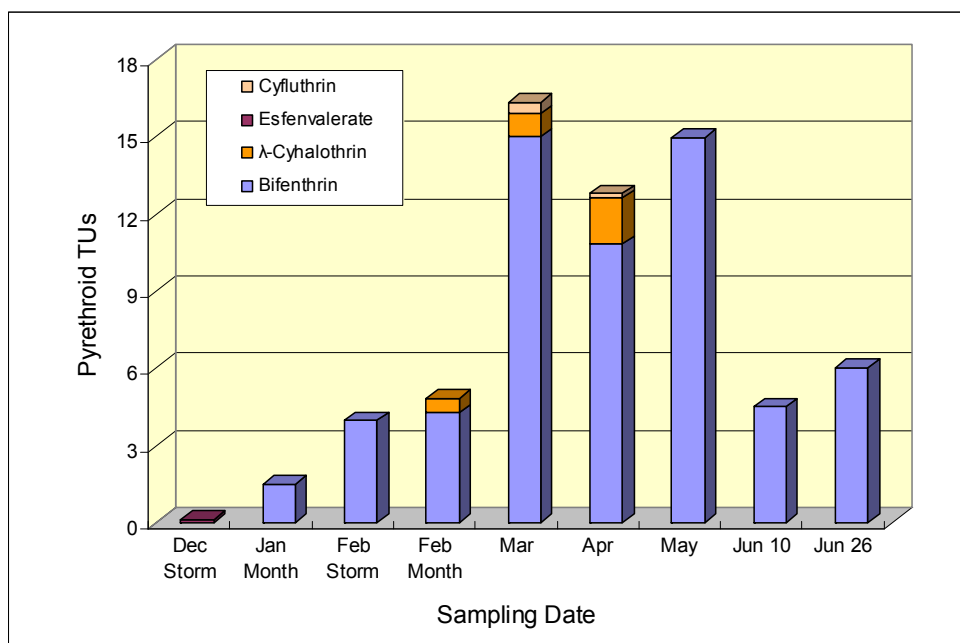


Figure 3. Toxicity units (TUs) of pyrethroids from sediment samples taken at Del Puerto Creek. TUs for trace detections were calculated using the MDL values.

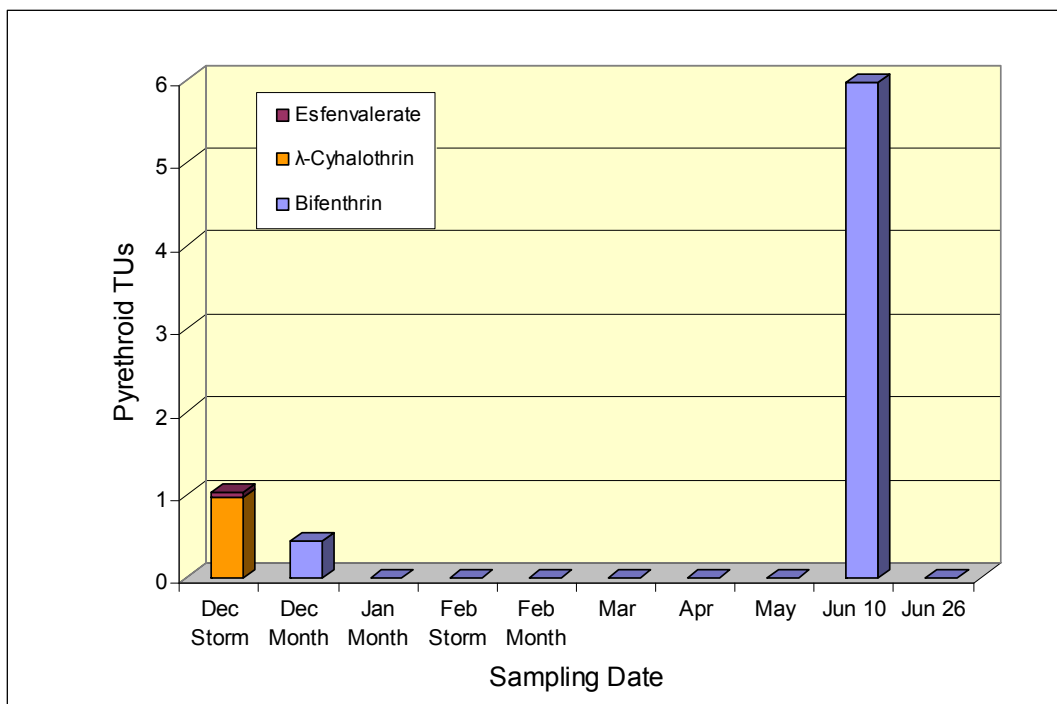


Figure 4. Toxicity units (TUs) of pyrethroids from sediment samples collected at Orestimba Creek. TUs for trace detections were calculated using the MDL values.

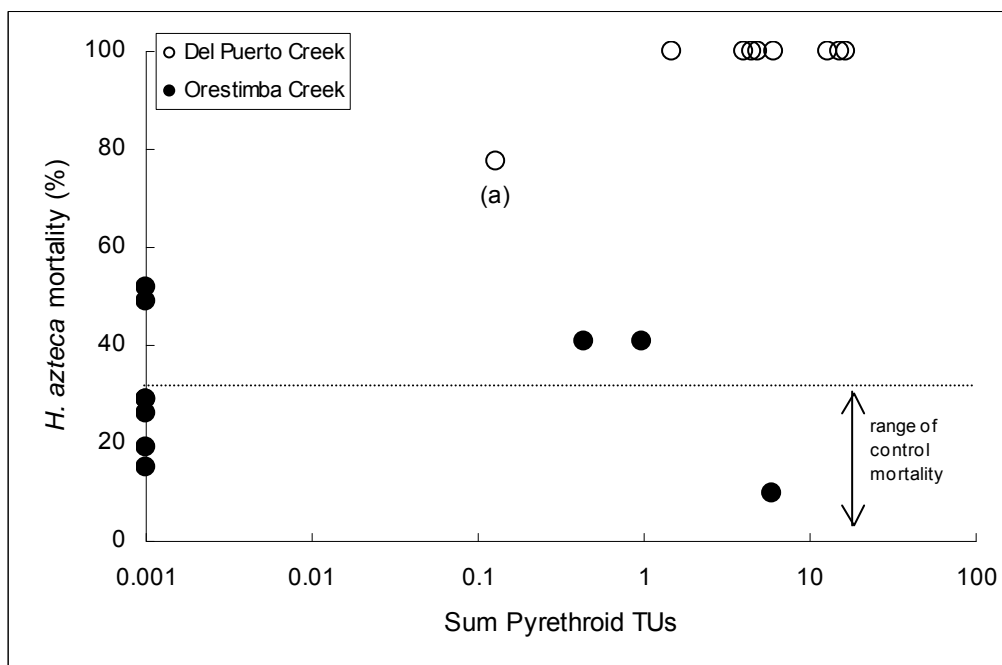


Figure 5. Relationship between the sum of pyrethroid toxicity units (TUs) in sediments and the toxicity to *H. azteca*. Non-detects were arbitrarily given a value of 0.001 TU and trace detections were calculated using MDL values.

Appendix 1. QC Data for Water Samples

Table A1. Holding times of the water samples for chemical analysis collected from Del Puerto and Orestimba Creeks. Red highlighted cells indicate exceedances.

Sample No.	Sampling Date	Date Received at Lab	Preservation Date	Sampling to Preservation (Days)	Extraction Date	Sampling to Extraction (Days)	Analysis Date	Sampling to Analysis (Days)
Chemistry – OP ¹								
101	18 Dec 07	19 Dec 07	None	NA ²	22 Dec 07	4	15 Jan 08	28
105	18 Dec 07	19 Dec 07	None	NA	22 Dec 07	4	15 Jan 08	28
110	26 Dec 07	27 Dec 07	None	NA	27 Dec 07	1	15 Jan 08	20
114	5 Jan 08	7 Jan 08	None	NA	8 Jan 08	3	15 Jan 08	10
119	5 Jan 08	7 Jan 08	None	NA	8 Jan 08	3	15 Jan 08	10
124	5 Jan 08	7 Jan 08	None	NA	8 Jan 08	3	15 Jan 08	10
125	14 Jan 08	14 Jan 08	14 Jan 08	0	19 Jan 08	5	13 Feb 08	30
129	14 Jan 08	14 Jan 08	14 Jan 08	0	19 Jan 08	5	13 Feb 08	30
133	20 Feb 08	20 Feb 08	20 Feb 08	0	22 Feb 08	2	7 Mar 08	16
137	20 Feb 08	20 Feb 08	20 Feb 08	0	22 Feb 08	2	7 Mar 08	16
141	28 Feb 08	28 Feb 08	None	NA	2 Mar 08	3	7 Mar 08	8
145	28 Feb 08	28 Feb 08	None	NA	2 Mar 08	3	7 Mar 08	8
151	18 Mar 08	18 Mar 08	None	NA	22 Mar 08	4	2 Apr 08	15
156	18 Mar 08	18 Mar 08	None	NA	22 Mar 08	4	2 Apr 08	15
160	18 Mar 08	18 Mar 08	None	NA	22 Mar 08	4	2 Apr 08	15
161	8 Apr 08	8 Apr 08	None	NA	10 Apr 08	2	21 Apr 08	13
166	8 Apr 08	8 Apr 08	None	NA	10 Apr 08	2	21 Apr 08	13
171	19 May 08	20 May 08	None	NA	22 May 08	3	6 June 08	18
177	19 May 08	20 May 08	None	NA	22 May 08	3	6 June 08	18
201	19 May 08	20 May 08	None	NA	22 May 08	3	6 June 08	18
204	19 May 08	20 May 08	None	NA	22 May 08	3	6 June 08	18
185	10 June 08	11 June 08	None	NA	11 June 08	2	2 July 08	22
190	10 June 08	11 June 08	None	NA	11 June 08	2	2 July 08	22
181	26 June 08	27 June 08	None	NA	1 July 08	5	1 Aug 08	36
211	26 June 08	27 June 08	None	NA	1 July 08	5	1 Aug 08	36
Chemistry – pyrethroid ³								
102	18 Dec 07	19 Dec 07	None	NA	22 Dec 07	4	17 Jan 08	30
106	18 Dec 07	19 Dec 07	None	NA	22 Dec 07	4	17 Jan 08	30
111	26 Dec 07	27 Dec 07	None	NA	27 Dec 07	1	17 Jan 08	22
115	5 Jan 08	7 Jan 08	None	NA	8 Jan 08	3	17 Jan 08	12
120	5 Jan 08	7 Jan 08	None	NA	8 Jan 08	3	17 Jan 08	12
126	14 Jan 08	14 Jan 08	14 Jan 08	0	19 Jan 08	5	6 Feb 08	23
130	14 Jan 08	14 Jan 08	14 Jan 08	0	19 Jan 08	5	6 Feb 08	23
134	20 Feb 08	20 Feb 08	22 Feb 08	2	22 Feb 08	2	19 Mar 08	28
138	20 Feb 08	20 Feb 08	22 Feb 08	2	22 Feb 08	2	19 Mar 08	28
142	28 Feb 08	28 Feb 08	29 Feb 08	1	2 Mar 08	3	19 Mar 08	20
146	28 Feb 08	28 Feb 08	29 Feb 08	1	2 Mar 08	3	19 Mar 08	20
149	28 Feb 08	28 Feb 08	29 Feb 08	1	2 Mar 08	3	19 Mar 08	20
152	18 Mar 08	18 Mar 08	None	NA	20 Mar 08	2	26 Mar 08	8
157	18 Mar 08	18 Mar 08	None	NA	20 Mar 08	2	26 Mar 08	8

Table A1, continued.

Sample No.	Sampling Date	Date Received at Lab	Preservation Date	Sampling to Preservation (Days)	Extraction Date	Sampling to Extraction (Days)	Analysis Date	Sampling to Analysis (Days)
162	8 Apr 08	8 Apr 08	None	NA	10 Apr 08	2	19 May08	41
167	8 Apr 08	8 Apr 08	None	NA	10 Apr 08	2	19 May08	41
170	8 Apr 08	8 Apr 08	None	NA	10 Apr 08	2	19 May08	41
174	19 May 08	20 May 08	None	NA	23 May 08	4	12 June 08	24
178	19 May 08	20 May 08	None	NA	23 May 08	4	12 June 08	24
202	19 May 08	20 May 08	None	NA	23 May 08	4	12 June 08	24
205	19 May 08	20 May 08	None	NA	23 May 08	4	12 June 08	24
186	10 June 08	11 June 08	None	NA	12 June 08	2	27 June 08	17
191	10 June 08	11 June 08	None	NA	12 June 08	2	27 June 08	17
182	26 June 08	27 June 08	None	NA	30 June 08	4	5 Aug 08	40
212	26 June 08	27 June 08	None	NA	30 June 08	4	5 Aug 08	40

¹OPs can be held 7 days prior to extraction without chemical preservation. Analysis within 40 days.

²Not applicable, chemical preservation was not needed prior to extraction.

³Pyrethroids can be held up to 4 days prior to extraction without chemical preservation. Analysis within 40 days.

Table A2. Percent recovery and RPD¹ of surrogate samples in water and in QC samples. Surrogates were triphenyl phosphate for OPs and dibromooctafluorobiphenyl for pyrethroids. Acceptable range of surrogate recovery was 50 – 150%. Yellow highlighted consecutive rows indicate samples that were also analyzed for RPD among the surrogates. Red highlighted cells indicate exceedances.

OP Water Sample No.	Percent Recovery of Triphenyl Phosphate (OPs)	Percent RPD among LCS/LCSD	Pyrethroid Water Sample No.	Percent Recovery of Dibromo-octafluorobiphenyl (Pyrethroids)	Percent RPD among LCS/LCSD
101	90.0		102	90.4	
105	81.5		106	98.6	
110	72.5		111	105.0	
MB ²	81.5		MB	95.5	
LCS	127	43.1	LCS	95.6	3.9
LCSD	82.0		LCSD	99.4	
114	78.0		115	95.6	
119	83.5		120	85.5	
124	83.0		MB	87	
MB	70.5		LCS	82.6	4.0
LCS	126.0	21.1	LCSD	86	
LCSD	102		126	80.4	
125	108		130	97.4	
129	94.8		MB	87.2	
MB	98.2		LCS	79.7	5.6
			LCSD	84.3	

Table A2, continued.

OP Water Sample No.	Percent Recovery of Triphenyl Phosphate (OPs)	Percent RPD among LCS/LCSD	Pyrethroid Water Sample No.	Percent Recovery of Dibromo-octafluorobiphenyl (pyrethroids)	Percent RPD among LCS/LCSD
LCS	95.7	2.3			
LCSD	93.5		134	80.6	
133	96.0		138	75.5	
137	97.6		MB	82.2	
MB	107		LCS	78.2	4.7
LCS	99.2	2.8	LCSD	74.6	
LCSD	102		146	82.1	
141	108		142	90.8	
145	98		149	88.9	
MB	104		MB	78.6	
LCS	98.2	1.8	LCS	75.1	10.0
LCSD	100		LCSD	83	
151	79.8		152	94.0	
156	108		157	118	
160	110		MB	89.6	
MB	118		LCS	87.6	16.2
LCS	106	17.2	LCSD	103	
LCSD	126		162	108	
161	72.2		167	110	
166	86.0		170	117	
MB	104		MB	109	
LCS	77.8	4.2	LCS	113	3.6
LCSD	74.6		LCSD	109	
171	92.8		174	68.9	
177	94.8		205	77.5	
201	93.1		202	64.8	
204	87.4		178	74.5	
MB	83.7		MB	73.8	
LCS	105	17.7	LCS	80.3	4.7
LCSD	87.9		LCSD	84.2	
185	103		186	93.9	
190	109		191	86.8	
MB	97.1		MB	74.3	
LCS	99.4	9.2	LCS	76.9	2.9
LCSD	109		LCSD	74.7	
181	110		182	97.7	
211	89.1		212	96.5	
MB	96.5		MB	87.4	
LCS	107	16.7	LCS	90.2	6.4
LCSD	90.5		LCSD	84.6	

¹Relative percent differences; acceptable RPDs are $\leq 25\%$ ²MB, method blank; LCS, laboratory control sample; LCSD, LCS duplicate

Table A3. Percent recoveries of spiked OP QC water samples. RPD¹ were calculated between the LCS and LCSD and are highlighted in yellow below the LCSD. Acceptable range for RPD were $\leq 25\%$. Exceedances or areas of QC concern are bolded and highlighted in red

QC Sample	Sampling Date	Azinphos methyl	Chlorpyrifos	Diazinon	Dimethoate	Disulfoton	Malathion	Methidathion	Methyl Parathion	Phorate	Phosmet
----- Percent Recovery or Percent RPD of QC Water Samples -----											
LCS	18/26 Dec 07	107	108	99.4	108	106	99.8	110	96.7	102	128
LCSD	18/26 Dec 07	101	94.3	97.9	108	90.7	110	108	101	82.5	110
Percent RPD		5.8	13.5	1.5	0	15.6	9.7	1.8	4.4	21.1	15.1
LCS	5 Jan 08	127	106	96.4	116	96.1	103	102	99.1	106	99.5
LCSD	5 Jan 08	108	93.3	85.9	79.9	96.1	105	104	93.4	94.9	114
Percent RPD		16.2	12.7	11.5	36.9	0	1.9	1.9	5.9	11.1	13.6
LCS	14 Jan 08	124	89.9	91.7	94.0	100	87.5	85.6	90.4	87	75.6
LCSD	14 Jan 08	120	101.0	94.0	86.4	88.9	101.0	91.0	86.5	89	99.3
Percent RPD		3.3	11.6	2.5	8.4	11.8	14.3	6.1	4.4	3.0	27.1
LCS	20 Feb 08	87.1	104	107	201	99.8	106	105	99.6	91.4	57.3
LCSD	20 Feb 08	106	93.9	105	179	86.9	97.1	102	93.4	94.0	44.3
Percent RPD		19.6	10.2	1.9	11.6	13.8	8.8	2.9	6.4	2.8	25.6
LCS	28 Feb 08	115	94.4	107	94.9	99.0	95.8	103	94.1	99.6	65.2
LCSD	28 Feb 08	137	105	112	90.0	101	107	109	98.3	96.8	59.2
Percent RPD		17.5	10.6	4.6	5.3	2.0	11.0	5.7	4.4	2.9	9.6
LCS	18 Mar 08	94.9	106	98.0	75.5	90.7	112	71.0	91.4	95.1	53.8
LCSD	18 Mar 08	110	118	121	89.7	105	78.4	89.5	123	111	84.0
Percent RPD		14.7	10.7	21.0	17.2	14.6	35.3	23.1	29.5	15.4	43.8
LCS	8 Apr 08	101	73.7	90.3	98.1	97.6	112	72.9	94.8	106	75.6
LCSD	8 Apr 08	100	122	82.6	98.5	112	70.4	89.4	112	114	73.9
Percent RPD		1.0	49.4	8.9	0.4	13.7	45.6	20.3	16.6	7.3	2.3
LCS	19 May 08	90.1	105	87.0	99.1	87.4	86.8	91.8	79.0	90.0	116
LCSD	19 May 08	82.6	112	104	92.6	89.5	84.6	77.0	81.0	70.2	100
Percent RPD		8.7	6.5	17.8	6.8	2.4	2.6	17.5	2.5	24.7	14.8
LCS	10 June 08	96.6	97.9	99.8	93.0	80.8	87.7	85.4	94.1	90.8	90.9
LCSD	10 June 08	108	97.6	103	109	101	96.8	99.2	100	90.1	116
Percent RPD		11.1	0.3	3.2	15.8	22.2	9.9	15.0	6.1	0.8	24.3
LCS	26 June 08	95.6	102	93.1	93.8	92.6	98.7	94.1	95.3	94.4	95.3
LCSD	26 June 08	82.7	93.6	109	81.3	79.8	89.5	87.9	82.0	83.5	98.1
Percent RPD		14.5	8.6	15.7	14.3	14.8	9.8	6.8	15.0	12.3	2.9

¹RPD, relative percent differences; LCS, laboratory control sample; LCSD, LCS duplicate

Table A4. Percent recoveries of spiked pyrethroid QC water samples. RPD¹ were calculated between the LCS and LCSD and are highlighted in yellow below the LCSD. Acceptable range for RPD were $\leq 25\%$. Exceedances or areas of QC concern are bolded and highlighted in red.

QC Sample	Sampling Date	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	λ -Cyhalothrin	Permethrin	Permethrin, cis	Permethrin, trans
----- Percent Recovery or Percent RPD of QC Water Samples -----											
LCS	18/26 Dec 07	82.9	84.6	75.5	85.9	102	86.6	95.8	76.8	NR	NR
LCSD	18/26 Dec 07	81	85.2	73	83	95.7	84.4	91.3	73.9	NR	NR
Percent RPD		2.3	0.7	3.4	3.4	6.4	2.6	4.8	3.8		
LCS	5 Jan 08	75.4	86.7	75.8	83.6	77.8	77.2	89.3	74.4	NR	NR
LCSD	5 Jan 08	70.7	84.6	72.1	85.8	87.6	87.8	83.3	75.9	NR	NR
Percent RPD		6.4	2.5	5.0	2.6	11.9	12.8	7.0	2.0		
LCS	14 Jan 08	68	100.0	83.4	76.4	77	93.7	77.3	101.0	NR	NR
LCSD	14 Jan 08	57	81.3	65.6	NR	58.1	79.3	61.6	77.6	NR	NR
Percent RPD		17.6	20.6	23.9		28.1	16.6	22.6	26.2		
LCS	20 Feb 08	74.1	96.2	79.1	70.7	89.4	82.8	90.8	90.5	NR	NR
LCSD	20 Feb 08	74.2	91.1	74.7	85.5	78.8	82.0	85.2	82.9	NR	NR
Percent RPD		0.1	5.4	5.7	19.0	12.6	1.0	6.4	8.8		
LCS	28 Feb 08	79.1	76.6	80.9	74.7	97.0	82.4	99.0	72.8	NR	NR
LCSD	28 Feb 08	58.4	76.5	71.5	60.8	80.0	81.2	79.3	63.3	NR	NR
Percent RPD		30.1	0.1	12.3	20.5	19.2	1.5	22.1	14.0		
LCS	18 Mar 08	94.2	104	122	70.0	103	99.8	81.1	NR	90.7	100
LCSD	18 Mar 08	88.9	85.3	101	84.4	65.2	124	65.5	NR	72.0	74.8
Percent RPD		5.8	19.8	18.8	18.7	44.9	21.6	21.3		23.0	28.8
LCS	8 Apr 08	95.3	116	118	86.0	102	117	105	NR	105	118
LCSD	8 Apr 08	92.9	118	117	102	107	125	113	NR	107	127
Percent RPD		2.6	1.7	0.9	17.0	4.8	6.6	7.3		1.9	7.3
LCS	19 May 08	70.0	95.7	81.8	67.1	84.0	78.2	88.9	NR	82.9	73.6
LCSD	19 May 08	90.0	112	106	84.5	99.0	103	103	NR	111	96.0
Percent RPD		25.0	15.7	25.8	23.0	16.4	27.4	14.7		29.0	26.4
LCS	10 June 08	91.8	91.0	94.6	101	96.3	90.4	92.5	NR	98.7	97.6
LCSD	10 June 08	85.0	97.3	93.1	103	100	93.8	83.6	NR	84.8	104
Percent RPD		7.7	6.7	1.6	2.0	3.8	3.7	10.1		15.1	6.3
LCS	26 June 08	129	102	89.5	70.7	103	93.9	91.8	NR	103	135
LCSD	26 June 08	80.7	100	72.8	75.6	94.7	108	102	NR	108	122
Percent RPD		46.1	2.0	20.6	6.7	8.4	14.0	10.5		4.7	10.1

¹RPD, relative percent differences; LCS, laboratory control sample; LCSD, LCS duplicate; NR, not run

Table A5. QC for field duplicate samples (FD) and field blanks (FB).

Sampling Date	5 Jan 08		28 Feb 08		18 Mar 08	8 April 08
Sample Type	Field sample	FD	Field sample	FD	FB	FB
Pyrethroids						
Bifenthrin	FD only collected for pyrethroids at this date		nd	nd	FB only collected for OP analysis	nd
Cyfluthrin			nd	nd		nd
Cypermethrin			nd	nd		nd
Deltamethrin			nd	nd		nd
Esfenvalerate			nd	nd		nd
Fenpropathrin			nd	nd		nd
λ-Cyhalothrin			nd	nd		nd
Permethrin			nd	nd		nd
OPs						
Azinphos methyl	nd	nd	FD only collected for OPs at this date		nd	FB only collected for pyrethroid analysis
Chlorpyrifos	nd	nd			nd	
Diazinon	nd	nd			trace*	
Dimethoate	nd	nd			nd	
Disulfoton	nd	nd			nd	
Malathion	nd	nd			nd	
Methidathion	nd	nd			nd	
Parathion, Methyl	nd	nd			nd	
Phorate	nd	nd			nd	
Phosmet	nd	nd			nd	

*above the RL but below the MDL

Table A6. Holding times water samples collect for TSS (total suspended solids) analysis. Exceedances or areas of QC concern are bolded and highlighted in red.

Sample id	Sampling Date	Analysis Date	Sampling to Analysis (Days)
103	18 Dec 07	27 Dec 07	9
107	18 Dec 07	27 Dec 07	9
109	18 Dec 07	27 Dec 07	9
112	26 Dec 07	27 Dec 07	1
117	5 Jan 08	7 Jan 08	2
121	5 Jan 08	7 Jan 08	2
121	5 Jan 08	7 Jan 08	2
123	5 Jan 08	7 Jan 08	2
116	14 Jan 08	16 Jan 08	2
127	14 Jan 08	16 Jan 08	2
131	14 Jan 08	16 Jan 08	2
135	20 Feb 08	21 Feb 08	1
139	20 Feb 08	21 Feb 08	1
143	28 Feb 08	29 Feb 08	1
147	28 Feb 08	29 Feb 08	1
150	28 Feb 08	29 Feb 08	1
153	18 Mar 08	21 Mar 08	3
155	18 Mar 08	21 Mar 08	3
158	18 Mar 08	21 Mar 08	3
163	8 Apr 08	11 Apr 08	3
165	8 Apr 08	11 Apr 08	3
168	8 Apr 08	11 Apr 08	3
175	19 May 08	17 July 08	59
179	19 May 08	17 July 08	59
189	10 June 08	17 Jul 08	37
192	10 June 08	17 Jul 08	37
193	10 June 08	17 Jul 08	37
183	26 June 08	17 Jul 08	21
184	26 June 08	17 Jul 08	21
213	26 June 08	17 Jul 08	21

Table A7. RPD¹ of TSS samples

Analysis Date	Sample Sediment Weight (mg L ⁻¹)	FD ² Sediment Weight (mg L ⁻¹)	RPD
27 Dec 07	12.39	7.42	50.2%
7 Jan 08	1766.45	1703.38	3.6%
16 Jan 08	116	30.45	1.9%
29 Feb 08	4.81	5.89	20.2%
21 Mar 08	11.64	12.35	5.9%
11 Apr 08	7.22	6.39	12.2%
17 Jul 08	98.1	104.4	6.3%
17 Jul 08	84.3	88.5	4.9%

¹Relative percent differences.

²Field duplicate

Appendix 2. QC Data for Sediment Samples

Table A8. Holding times for sediment samples for chemical analysis collected from Del Puerto and Orestimba Creeks.

Sediment Sample No.	Sampling Date	Date Received at CDFG Lab	Date Extracted	Sampling to Extraction (days)	Date analyzed	Sampling to Analysis (days) ¹
302	18 Dec 07	7 Jan 08	12 Mar 08	85	7 Apr 08	111
306	18 Dec 07	7 Jan 08	12 Mar 08	85	7 Apr 08	111
310	26 Dec 07	7 Jan 08	12 Mar 08	77	7 Apr 08	103
314	14 Jan 08	28 Feb 08	12 Mar 08	58	7 Apr 08	84
319	14 Jan 08	28 Feb 08	12 Mar 08	58	7 Apr 08	84
321	20 Feb 08	28 Feb 08	12 Mar 08	21	7 Apr 08	47
325	20 Feb 08	28 Feb 08	12 Mar 08	21	7 Apr 08	47
329	28 Feb 08	28 Feb 08	12 Mar 08	13	7 Apr 08	39
333	28 Feb 08	28 Feb 08	12 Mar 08	13	7 Apr 08	39
338	18 Mar 08	8 Apr 08	17 Apr 08	30	19 May 08	62
341	18 Mar 08	8 Apr 08	17 Apr 08	30	19 May 08	62
344	18 Mar 08	8 Apr 08	17 Apr 08	30	19 May 08	62
349	8 Apr 08	8 Apr 08	17 Apr 08	9	19 May 08	41
353	8 Apr 08	8 Apr 08	17 Apr 08	9	19 May 08	41
357	19 May 08	11 June 08	1 July 08	43	5 Aug 08	78
361	19 May 08	11 June 08	1 July 08	43	5 Aug 08	78
365	10 June 08	11 June 08	1 July 08	21	5 Aug 08	56
369	10 June 08	11 June 08	1 July 08	21	5 Aug 08	56
375	26 June 08	26 June 08	1 July 08	5	5 Aug 08	40
380	26 June 08	26 June 08	1 July 08	5	5 Aug 08	40

¹Sediments for chemical analysis can be held up to 6 months at -20°C

Table A9. Holding times for the sediment samples for TOC (total organic carbon) and grain size analysis.

Sampling Date ¹	TOC Analysis		Grain Size Analysis	
	Analysis Date	Sampling to Analysis Time (Months)	Analysis Date	Sampling to Analysis Time (Months)
18 Dec 07	11 June 08	5.87	25 June 09	7.3
26 Dec 07	12 June 08	5.63	25 June 09	7.0
14 Jan 08	12 June 08	5.00	25 June 09	6.4
20 Feb 08	12 June 08	3.77	25 June 09	5.2
28 Feb 08	12 June 08	3.50	25 June 09	4.9
18 Mar 08	12 June 08	2.87	25 June 09	4.3
8 Apr 08	13 June 08	2.20	25 June 09	3.6
19 May 08	13 June 08	0.83	25 June 09	2.2
10 June 08	13 June 08	0.10	25 June 09	1.5
26 June 08	3 Nov 08	4.3	25 June 09	1.0

¹Sediment samples were collected for TOC and grain size on the same date, and samples were collected from both Orestimba and Del Puerto Creeks.

Table A10. Percent recovery and RPD¹ of surrogate samples in sediment and in QC samples. Acceptable range of surrogate recovery was 50 – 150%. Highlighted consecutive rows (of the same color) indicate samples that were also analyzed for RPD among the surrogates or duplicates. Acceptable range for RPD were $\leq 25\%$.

Surrogate			Dibutylchloroendate		Dibromooctafluorobiphenyl	
Sediment Sample Number ²	Sampling Date	Analysis Date	Percent Recovery	Percent RPD	Percent Recovery	Percent RPD
302	18 Dec 07	7 Apr 08	115.0	12.0	92.7	1.3
302 LD	18 Dec 07	7 Apr 08	102.0		93.9	
306	18 Dec 07	7 Apr 08	95.3		88.5	
310	26 Dec 07	7 Apr 08	102.0		89.6	
314	14 Jan 08	7 Apr 08	89.8		87.0	
319	14 Jan 08	7 Apr 08	96.0		103.0	
321	20 Feb 08	7 Apr 08	106.0		98.7	
325	20 Feb 08	7 Apr 08	106.0		102.0	
329	28 Feb 08	7 Apr 08	114.0		92.2	
333	28 Feb 08	7 Apr 08	99.8		95.5	
MB	NA	7 Apr 08	103.0		100.0	
LCS	NA	7 Apr 08	106.0		72.3	
MS	NA	7 Apr 08	102.0	4.6	99.5	3.5
MSD	NA	7 Apr 08	97.4		96.1	
338	18 Mar 08	19 May 08	91.3		97.8	
341	18 Mar 08	19 May 08	88.0		97.4	
349	8 Apr 08	19 May 08	78.5	7.5	89.9	2.2
349 LD	8 Apr 08	19 May 08	84.6		91.9	
353	8 Apr 08	19 May 08	90.2		92.8	
344	18 Mar 08	19 May 08	87.3		87.4	
MB	NA	19 May 08	90.1		87.9	
LCS	NA	19 May 08	81.2	1.7	71.2	1.0
LCSD	NA	19 May 08	82.6		70.5	
MS	NA	19 May 08	86.8	4.3	68.8	15.1
MSD	NA	19 May 08	90.6		80.0	
357	19 May 08	5 Aug 08	103		89.2	
361	19 May 08	5 Aug 08	92.6	3.1	94.4	0.8
361 LD	19 May 08	5 Aug 08	95.5		95.2	
365	10 June 08	5 Aug 08	97.5		100	
369	10 June 08	5 Aug 08	73.1		71.3	
375	26 June 08	5 Aug 08	96.6		95.2	
380	26 June 08	5 Aug 08	98.3		96.3	
MB	NA	5 Aug 08	86.5		80.9	

Table A10 continued.

Surrogate			Dibutylchloroendate		Dibromooctafluorobiphenyl	
Sediment Sample Number	Sampling Date	Analysis Date	Percent Recovery	Percent RPD	Percent Recovery	Percent RPD
LCS	NA	5 Aug 08	82.4	7.3	86.8	2.4
LCSD	NA	5 Aug 08	76.6		88.9	
MS	NA	5 Aug 08	102	10.3	97.8	0
MDS	NA	5 Aug 08	92		97.8	

¹Relative percent differences

²MB, method blank; LCS, laboratory control sample; LCSD, LCS duplicate; MS, matrix spike; MSD, MS duplicate, LD, lab duplicate

Table A11. Percent recoveries of spiked QC sediment samples. RPD¹ were calculated between the laboratory control spikes and laboratory control spike duplicates or between the matrix spikes and matrix spike duplicates and are highlighted in yellow in the row below the respective calculation. Acceptable range for RPD were $\leq 25\%$. Exceedances or areas of QC concern are bolded and highlighted in red.

		----- Percent Recovery or Percent RPD of QC Sediment Samples -----								
QC	Analysis Date	Bifenthrin	Cyfluthrin	Cypermethrin	Deltamethrin	Esfenvalerate	Fenpropathrin	Permethrin cis	Permethrin trans	λ -Cyhalothrin
LCS ²	7 Apr 08	72.4	100.0	92.5	72.6	94.0	94.9	74.1	87.1	95.9
MS	7 Apr 08	116.0	105.0	107.0	99.7	106.0	99.5	92.6	102.0	97.8
MSD	7 Apr 08	117.0	106.0	104.0	89.6	104.0	101.0	90.1	84.6	100.0
Percent RPD		0.9	0.9	2.8	10.7	1.9	1.5	2.7	18.6	2.2
LCS	19 May 08	79.0	77.1	81.5	61.6*	78.3	80.0	83.8	80.7	74.0
LCSD	19 May 08	77.3	76.7	78.1	56.7*	75.8	78.2	80.9	95.8	73.0
Percent RPD		2.2	0.5	4.3	8.3	3.2	2.3	3.5	17.1	1.4
MS	19 May 08	91.8	99.8	101	79.3	99.6	93.0	78.7	91.1	104
MSD	19 May 08	103	86.5	102	75.6	94.9	91.2	83.5	83.7	104
Percent RPD		11.5	14.3	1.0	4.8	4.8	2.0	5.9	8.5	0.0
LCS	5 Aug 08	93.1	74.9	67.7*	60.2*	84.7	81.1	89.7	85.3	75.1
LCSD	5 Aug 08	98.9	71.5	71.4	68.1*	82.9	76.2	94.0	91.6	74.5
Percent RPD		6.0	4.6	5.3	12.3	2.1	6.2	4.7	7.1	0.8
MS	5 Aug 08	95.9	106	119	96.9	124	99.6	103	104	125
MSD	5 Aug 08	94.3	100	112	116	126	97.4	104	102	120
Percent RPD		1.7	5.8	6.1	17.9	1.6	2.2	1.0	1.9	4.1

¹Relative percent differences

²LCS, laboratory control sample; LCSD, LCS duplicate; MS, matrix spike; MSD, MS duplicate; RPD, relative percent difference

*LCS/LCSD is outside of control limits (low recovery)

Table A12. QC results of laboratory and field sediment duplicates (for chemical analysis).

QC duplicate type	Lab		Lab		Lab		Field	
Sampling date	18 Dec 07		8 Apr 08		19 May 08		18 Mar 08	
Sample/aliquot No.	302		349		361		338	341
	Aliquot 1	Aliquot 2	Aliquot 1	Aliquot 2	Aliquot 1	Aliquot 2	Aliquot 1	Aliquot 1
	Concentration (ng g ⁻¹ dry weight)							
Bifenthrin	nd	nd	nd	nd	74.4*	52.5*	nd	nd
Cyfluthrin, total	nd	nd	nd	nd	nd	nd	nd	nd
Cypermethrin, total	nd	nd	nd	nd	nd	nd	nd	nd
Deltamethrin	nd	nd	nd	nd	nd	nd	nd	nd
Esfenvalerate	trace‡	trace‡	nd	nd	nd	nd	nd	nd
Fenpropathrin	nd	nd	nd	nd	nd	nd	nd	nd
Permethrin, cis	nd	nd	nd	nd	nd	nd	nd	nd
Permethrin, trans	nd	nd	nd	nd	nd	nd	nd	nd
λ-Cyhalothrin, total	10	nd	nd	nd	nd	nd	nd	nd

* RPD for the two values, 34.5%

‡below the RL but above the MDL.

Table A13. QC for field and laboratory duplicates (TOC, grain size)

QC Type	-----Laboratory Duplicate -----					Field Duplicate	
-----TOC Concentration (ppm) -----							
Sample No.	315	342	350	354	362	339	341
Aliquot 1	8,700	8,430	12,546	9,650	10286	7,819	7,685
Aliquot 2	9,482	7,685	12,900	11,164	9,482		
RPD ¹	8.6%	9.2%	2.8%	14.5%	8.1%	1.7%	
-----Grain Size Analysis -----							
	% Pebble	% Granule	% Sand	% Silt	% Clay		
373	0.48	0.46	43.96	36.96	18.14		
373 Lab Duplicate	0.4	0.58	43.3	37.27	18.45		
RPD	18.2%	23.1%	1.5%	0.8%	1.7%		
379	0.4	0.47	18.06	53.06	28.01		
379 Lab Duplicate	0.33	0.39	19.34	52.41	27.53		
RPD	19.2%	18.6%	6.84%	1.2%	1.7%		
343	0.95	1.06	11.78	56.46	29.75		
348 Field Duplicate	0	0.06	7.98	60.74	31.22		
RPD	200%	178.6%	38.5%	7.3%	4.8%		

¹Relative percent difference (acceptable, $\leq 25\%$)

Appendix 3. UCD ATL QC Data

Table A14. Holding times of water samples for toxicity testing at UCD ATL.

Sample Type ¹		Sampling Date	Date received at UCD ATL	Sampling to Reception (days)	Date TOX test run	Sampling to TOX Test (days)
1001	P	18 Dec 07	19 Dec 07	1	19 Dec 07	1
1002	FD	18 Dec 07	19 Dec 07	1	19 Dec 07	1
1003	P	18 Dec 07	19 Dec 07	1	19 Dec 07	1
1004	FD	18 Dec 07	19 Dec 07	1	19 Dec 07	1
1005	FB	18 Dec 07	19 Dec 07	1	19 Dec 07	1
1006	P	26 Dec 07	27 Dec 07	1	27 Dec 07	1
1008	P	5 Jan 08	5 Jan 08	0	5 Jan 08	0
1009	P	5 Jan 08	5 Jan 08	0	5 Jan 08	0
1010	P	14 Jan 08	14 Jan 08	0	14 Jan 08	0
1011	P	14 Jan 08	14 Jan 08	0	14 Jan 08	0
1013	P	20 Feb 08	20 Feb 08	0	20 Feb 08	0
1015	P	20 Feb 08	20 Feb 08	0	20 Feb 08	0
1017	P	28 Feb 08	28 Feb 08	0	28 Feb 08	0
1019	P	28 Feb 08	28 Feb 08	0	28 Feb 08	0
1021	P	18 Mar 08	18 Mar 08	0	19 Mar 08	1
1023	P	18 Mar 08	18 Mar 08	0	19 Mar 08	1
1025	P	8 Apr 08	8 Apr 08	0	9 Apr 08	1
1027	P	8 Apr 08	8 Apr 08	0	9 Apr 08	1
1029	P	19 May 08	19 May 08	0	20 May 08	1
1031	P	19 May 08	19 May 08	0	20 May 08	1
1034	P	10 June 08	10 June 08	0	11 June 08	1
1036	P	10 June 08	10 June 08	0	11 June 08	1
1038	P	26 June 08	26 June 08	0	27 June 08	1
1040	P	26 June 08	26 June 08	0	27 June 08	1

¹P, primary sample; FD, field duplicate; FB, field blank

Table A15. Holding times of sediment samples for toxicity testing at UCD ATL.

Sample Type ¹		Sampling Date	Date received at UCD ATL	Sampling to Reception (days)	Date TOX test run	Sampling to TOX Test (days)
501	P	18 Dec 07	19 Dec 07	1	19 Dec 07	1
502	FD	18 Dec 07	19 Dec 07	1	19 Dec 07	1
503	FB	18 Dec 07	19 Dec 07	1	19 Dec 07	1
504	P	18 Dec 07	19 Dec 07	1	19 Dec 07	1
505	FD	18 Dec 07	19 Dec 07	1	19 Dec 07	1
506	P	26 Dec 07	27 Dec 07	1	27 Dec 07	1
507	P	14 Jan 08	14 Jan 08	0	17 Jan 08	3
508	P	20 Feb 08	20 Feb 08	0	22 Feb 08	2
509	P	20 Feb 08	20 Feb 08	0	22 Feb 08	2
510	P	28 Feb 08	28 Feb 08	0	1 Mar 08	2
511	P	28 Feb 08	28 Feb 08	0	1 Mar 08	2
512	P	18 Mar 08	18 Mar 08	0	19 Mar 08	1
513	P	18 Mar 08	18 Mar 08	0	19 Mar 08	1
515	P	8 Apr 08	8 Apr 08	0	10 Apr 08	2
516	P	8 Apr 08	8 Apr 08	0	10 Apr 08	2
517	P	19 May 08	19 May 08	0	21 May 08	2
518	P	19 May 08	19 May 08	0	21 May 08	2
519	P	10 June 08	10 June 08	0	12 June 08	2
520	P	10 June 08	10 June 08	0	12 June 08	2
521	P	26 June 08	26 June 08	0	28 June 08	2
522	P	26 June 08	26 June 08	0	28 June 08	2

¹P, primary sample; FD, field duplicate; FB, field blank

Table A16. QC for UCD ATL toxicity studies.

Source	Sample type	% Survival ¹
Water Samples (<i>C. dubia</i> toxicity)		
Lab	Control water	100 a
Del Puerto Creek	Field blank water	100 a
Orestimba Creek	Water primary sample	100 a
Orestimba Creek	Field duplicate water sample	100 a
Del Puerto Creek	Water primary sample	100 a
Del Puerto Creek	Field duplicate water sample	100 a
Sediment Samples (for <i>H. azteca</i> toxicity)		
Lab	Control sediment	85 a
Del Puerto Creek	Field blank sediment	75 ab
Orestimba Creek	Sediment primary sample	70 ab
Orestimba Creek	Field duplicate sediment sample	48 bc
Del Puerto Creek	Sediment primary sample	15 d
Del Puerto Creek	Field duplicate sediment sample	30 cd

¹Numbers with the same letter are not significantly different from each other.

Appendix 4. Field Measurements and Non-chemical Laboratory Analysis Results.

Table A17. Field measurements at the time of sampling Orestimba and Del Puerto Creeks.

Sampling Date	Astronomical Time	Event Type		pH	DO ¹ (mg L ⁻¹)	EC (μS cm ⁻¹)	Water Temp (°C)	Salinity (ppt)
		Storm	Monthly					
Orestimba Creek								
18 Dec 07	1040	X		7.39	6.23	835	7.8	0.4
26 Dec 07	0945		X	8.06	11.06	726	5.6	0.4
5 Jan 08	1010	X		7.50	9.39	325.5	9	0.2
14 Jan 08	1010		X	7.85	10.7	759	8.6	0.4
20 Feb 08	1115	X		7.57	9.4	793	11.8	0.4
28 Feb 08	1015		X	8.08	10.7	613	11.8	0.3
18 Mar 08	1010		X	7.80	8.03	1048	11.3	0.5
8 Apr 08	1030		X	8.16	9.6	1054	12.85	0.53
19 May 08	1100		X	7.75	6.43	1129	21.5	0.56
10 June 08	1000		X	8.07	8.09	1142	20.57	0.57
26 June 08	1000		X	7.94	7.43	1018	20.3	0.5
mean				7.83	8.82	858.4	12.8	0.43
Del Puerto Creek								
18 Dec 07	1235	X		7.09	3.56	1346	13.2	0.7
5 Jan 08	1047	X		8.23	10.67	683	8.2	0.3
14 Jan 08	1125		X	7.72	5.2	1598	8.6	0.8
20 Feb 08	1245	X		8.12	9.88	795	13.2	0.4
28 Feb 08	1140		X	8.73	11.95	843	14.7	0.4
18 Mar 08	1125		X	8.62	10.09	1235	13.1	0.6
8 Apr 08	1145		X	8.86	12.7	1575	15.44	0.8
19 May 08	1230		X	8.79	10.41	669	24.79	0.32
10 June 08	1235		X	7.86	5.35	1553	21.59	0.78
26 June 08	1240		X	8.04	6.84	1204	20.87	0.6
mean				8.21	8.67	1150	15.4	0.57

¹DO, dissolved oxygen; EC, electrical conductivity; Temp, temperature

Table A18. Total suspended sediments (TSS) in water samples collected at Orestimba and Del Puerto Creeks.

Site ¹	Sample Timing	Sediment Weight (mg/L)
ORC	Dec Storm	11.38
ORC	Dec Monthly	21.18
ORC	Jan Storm	1766.45
ORC	Jan Monthly	30.45
ORC	Feb Storm	27.44
ORC	Feb Monthly	31.82
ORC	Mar Monthly	11.64
ORC	Apr Monthly	7.22
ORC	May Monthly	19.01
ORC	June 10	98.10
ORC	June 26	84.26
DPC	Dec Storm	12.39
DPC	Jan Storm	148.08
DPC	Jan Monthly	287.06
DPC	Feb Storm	56.45
DPC	Feb Monthly	4.81
DPC	Mar Monthly	62.7
DPC	Apr Monthly	30.42
DPC	May Monthly	20.27
DPC	June 10	24.55
DPC	June 26	8.89

¹ORC, Orestimba Creek; DPC, Del Puerto Creek

Table A19. Grain size of sediment samples collected at Orestimba and Del Puerto Creeks.

Site ¹	Sample Timing	Pebble (%)	Granule (%)	Sand (%)	Silt (%)	Clay (%)
ORC	Dec Storm	0.66	0.94	23.32	51.14	23.94
ORC	Dec Monthly	0.42	0.1	18.35	55.07	26.06
ORC	Jan Monthly	0	0.04	41.92	40.94	17.1
ORC	Feb Storm	0.47	0.06	74.65	18.35	6.47
ORC	Feb Monthly	0.28	0.1	84.36	10.72	4.54
ORC	Mar Monthly	0.4	0.58	43.3	37.27	18.45
ORC	Apr Monthly	0.93	0.81	37.17	40.99	20.1
ORC	May Monthly	0.74	0.51	32.84	43.85	22.06
ORC	June 10	1.79	2.43	42	35.19	18.59
ORC	June 26	1.04	0.87	25.74	45.08	27.27
DPC	Dec Storm	3.02	1.25	66.98	19.26	9.49
DPC	Jan Monthly	0	0	35.36	44.13	20.51
DPC	Feb Storm	0	0	18.05	50.89	31.06
DPC	Feb Monthly	0.41	0.11	13.56	55.35	30.57
DPC	Mar Monthly	0.95	1.06	11.78	56.46	29.75
DPC	Apr Monthly	1.76	0.33	23.35	49.8	24.76
DPC	May Monthly	0	1.06	17.35	50.53	31.06
DPC	June 10	0.66	0.11	33.17	42.49	23.57
DPC	June 26	0.33	0.39	19.34	52.41	27.53

¹ORC, Orestimba Creek; DPC, Del Puerto Creek