Department of Pesticide Regulation

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MEMORANDUM

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Winston H. Hickox
Secretary, California Environmental Protection Agency

TO: Patricia Dunn
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FROM: DeeAn Jones
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DATE: October 3, 2000

SUBJECT: PRELIMINARY RESULTS OF ACUTE AND CHRONIC TOXICITY TESTING OF SURFACE WATER MONITORED IN THE SAN JOAQUIN RIVER WATERSHED, WINTER 1999-2000.

SCOPE OF THIS MEMORANDUM

This memorandum provides results of water sampling conducted on the San Joaquin River (SJR) by the Department of Pesticide Regulation (DPR) for the Dormant Spray Water Quality Program. Information presented is from December 6, 1999 to March 3, 2000 and includes results from chemical analyses conducted by the California Department of Food and Agriculture (CDFA) and bioassays conducted by the California Department of Fish and Game (DFG). This memorandum summarizes the fourth of a five-year study begun in 1996, designed to monitor dormant spray insecticides (chlorpyrifos, diazinon, and methidathion) in the SJR watershed. In an effort to obtain more information about pesticide residues in surface water, samples were analyzed for additional insecticides as well as selected herbicides. This memorandum does not include an in-depth interpretation of the data, which will be provided in the final report.

BACKGROUND

The SJR flows west from the Sierra Nevada Mountain Range, then heads north through the San Joaquin Valley and terminates in the Sacramento-San Joaquin Delta. The river extends approximately 134 miles from Friant Dam to Stevinson where flows are intermittent, and from Stevinson to Vernalis (about 60 miles) where flows are perennial (Figure 1). The river basin, including tributary watersheds, drains approximately 15,880 square miles (Central Valley Regional Water Quality Control Board, 1994). Runoff from rainfall occurring in the San Joaquin Valley and Sierra Nevada foothills during the rainy season, October to March, creates short term increases in river discharge. With little significant rain from June to September, river discharge during the summer is composed of dam releases of snow-melt water which is subsequently used for agricultural, urban, recreational and wildlife purposes. The dormant spray season for nut and stone fruit trees coincides with the rainy season. During this period dormant spray insecticides, mainly chlorpyrifos, diazinon and methidathion, are applied along with weed oil to control peach twig borer, San Jose scale, European red mite and brown mite pests. Rainfall and subsequent surface runoff from agricultural areas provides a mechanism for off-site movement of pesticides to the SJR. From 1988 to 1990, the Central Valley Regional Water Quality Control Board (CVRWQCB) conducted an aquatic toxicity survey in the San Joaquin Valley. Surface water samples collected from certain reaches of the San Joaquin River watershed during this survey were acutely toxic to the water flea, *Ceriodaphnia dubia* (Foe and Connor, 1991). The cause of toxicity was not determined but was attributed to pesticides in general. Further study was conducted in the Valley during the winter of 1991-92, and the resultant toxicity was attributed to the presence of chlorpyrifos and diazinon (Foe and Sheipline, 1993; Foe, 1995; Kuivila and Foe, 1995). The toxicity found in these studies was in violation of the CVRWQCB's narrative water quality objective (Foe, 1995) which states that, "All waters shall be maintained free of toxic substances in concentrations that produce detrimental physiological responses in human, plant, animal, or aquatic life" (CVRWQCB, 1994).

DPR monitored the San Joaquin River watershed during the winters of 1991-92 and 1992-93, and reported the detection of chlorpyrifos, diazinon, and methidathion in 10, 72, and 18 percent of the 108 water samples collected, respectively. Of these positive samples, 2, 13, and 1 percent respectively, exceeded the LC_{50} for *C. dubia* indicating potential acute toxicity to this organism (Ross, *et al.*, 1997). In addition, in a 1993 CVRWQCB study, diazinon concentrations in the San Joaquin River at Vernalis ranged from 0.148 to 1.07 µg/L on 12 consecutive days, and the authors concluded that chronic toxicity due to diazinon might be problematic at this site (Kuivila and Foe, 1995). Diazinon was also detected at levels acutely toxic to *C. dubia* in Orestimba Creek, a tributary to the San Joaquin River, during the 1992-93 dormant spray period (Domagalski, 1995). Consequently, methods designed to reduce the mass of dormant spray insecticides leaving target areas have been under investigation by DPR and growers (Ross, *et al.*, 1997; Ando, 1996; Anonymous, 1996; Biermann, 1996).

A U.S. Geological Survey (USGS) report of an investigation of pesticides in storm runoff from agricultural areas at a site along the Tuolumne River and from urban drains in Modesto, California found six different pesticides in the predominantly agricultural samples and 15 in the mainly urban runoff samples (Kratzer, 1998). Diazinon and chlorpyrifos were the insecticides detected in agricultural runoff in 100 and 88 percent of the 8 samples, respectively. Diazinon, carbaryl, chlorpyrifos, and malathion were each detected in 100% of the 10 urban samples collected. The herbicides simazine, napropamide and dacthal were detected in 100% of the 8 samples collected from the agricultural runoff site and metolachlor was detected in 88% of the samples. Simazine, dacthal and trifluralin were detected in all the samples collected in the urban runoff, and metolachlor, EPTC, benfluralin, pendimethalin, prometon, napropamide, and propanil were detected in 10 to 90% of the samples.

During the winter of 1996-97, DPR conducted toxicity monitoring at two sites in the San Joaquin River Watershed (Bennett *et al*., 1998). The first half of winter was unusually wet with flooding followed by unseasonably dry weather during the second half of winter. Water samples from Orestimba Creek contained residues of diazinon, carbofuran, and dimethoate in 20, 13, and 7 percent of the 15 samples collected, respectively. The maximum diazinon, carbofuran, and dimethoate concentrations detected were 0.092, 0.238, and 0.082 ug/L, respectively. Three (12%) of the 24 water samples from the San Joaquin River near Vernalis contained diazinon residues, with a maximum concentration of 0.070 ug/L. *Ceriodaphnia dubia* survival ranged from 40 to 100 percent for acute toxicity tests from Orestimba Creek. Only one of the samples collected on January 29, 1997 was significantly different from the control. However, there were no pesticides detected in this sample. Chronic toxicity was not detected in the eight weekly sets of water samples collected from the San Joaquin River near Vernalis.

During the winter of 1997-98, river discharge remained high from January through the end of sampling in March compared to historical levels for these months (Anderson *et al.*, 1995). It was a wet season (Department of Water Resources, 1998) much like the winter before. Chlorpyrifos and methyl parathion were each detected once at 0.093 and 0.190 ug/L, respectively, in the same sample collected from Orestimba Creek on January 26, 1998. Diazinon was detected in three of 18 samples (17%) collected at the site with concentrations ranging from 0.059 to 0.139 ug/L. Diazinon was detected on January 12 and then again in the samples collected on February 2 and 4. Four of the nine herbicides analyzed for were detected in Orestimba Creek. Bromacil was detected in three of the 18 samples (17%) with concentrations ranging from 0.066 to 0.115 ug/L. Cyanazine was detected in one of 18 samples (6%) at 0.25 ug/L. Diuron was detected in six of 18 samples (33%) at concentrations ranging from 0.078 to 0.388 ug/L and simazine was detected in five of 18 samples (28%) at concentrations ranging from 0.063 to 0.711 ug/L. Three of the samples collected on December 1, 1997, January 12 and 19, 1998 each contained residues of three different herbicides.

For acute toxicity tests conducted on Orestimba Creek samples, four of 18 samples had significantly reduced survival compared to the control. The four samples were collected on January 21, and February 4, 11 and 23. Pesticides analyzed in this study were all below detection limits in the January 21 and February 11 and 23 samples. The only sample with reduced survival that correlated with a detection was collected on February 4, with diazinon, diuron and simazine detections.

During the same study period, at the San Joaquin River, diazinon and methidathion were detected. Diazinon was detected in 10 of 30 samples (33%) collected at Vernalis (Table 7).

These detections came in two groupings. The first detection occurred on January 7 and was at the detection limit. There was no detection January 9. On January 12, 14 and 16 diazinon was again detected at concentrations ranging from 0.063 to 0.102 ug/L. The second group of detections began on January 30 and continued through February 11 with concentrations ranging from 0.042 to 0.093 ug/L. Methidathion was detected in three samples (10%) collected on January 7, 12 and February 2. The three detections ranged from 0.053 to 0.112 ug/L and coincided with diazinon detections. The herbicides bromacil, cyanazine, diuron and simazine were also detected in the SJR. Bromacil was detected in four (13%) and cyanazine in two of 30 samples (7%). Diuron was detected in all samples at concentrations ranging from 0.056 to 2.95 ug/L. Simazine was detected in 16 samples (53%) with levels ranging from 0.050 to 0.470 ug/L.

There was no *C. dubia* survival in the chronic toxicity test sample collected on February 2 with renewal water collected February 4 and 6. The sample collected on February 2 had detectible levels of diazinon, methidathion, diuron and simazine and there were no detections in the February 4 and 6 samples. There was no significant toxicity found in the remaining samples.

During the study last winter, 1998-99, rainfall was significantly lower than the previous year during the same period. During this study there were no detections of organophosphate or carbamate insecticides in the 20 samples collected at the Orestimba Creek site. However, several herbicides were detected. Diuron was detected in all 20 samples at concentrations ranging from 0.061 to 1.7 µg/L. Bromacil was detected three times (15%) at concentrations ranging from 0.080 to 0.089 µg/L and simazine four times ranging from 0.066 to 0.10 µg/L. Cyanazine and prometryn were each detected once (5%) at 0.37 and 0.076 µg/L, respectively. Five samples (25%) contained more than one herbicide residue. Five herbicides were detected in 1 sample that was collected one day after a rain event on February 22, 1999. There was no significant mortality detected in any acute toxicity sample collected at Orestimba Creek.

Diazinon was detected in 3 of the 30 samples (10%) collected from the SJR at Vernalis. The detections occurred on January 20, 1999 at 0.15 µg/L, January 21 at 0.090 µg/L and on February 10 at 0.053 µg/L. Each detection occurred 1 to 2 days after a rain event. Similar to the Orestimba Creek samples, there were no other organophosphate or carbamate insecticides detected. Diuron was detected in every sample collected at Vernalis ranging in concentration from 0.10 to 1.9 µg/L. Bromacil was detected in one of 30 samples (3.3%) at a concentration of 0.059 µg/L. Cyanazine came in two pulses, and was detected in a total of six samples (20%) ranging in concentrations from 0.097 to 0.42 µg/L. Prometryn was detected in two samples (7%) ranging from 0.053 to 0.071 µg/L and simazine was detected seven times (23%) with concentrations ranging from 0.059 to 0.12 µg/L. Thirteen samples (43%) had more than one triazine detection and three of them (10%) had three detections in one sample. There was no significant mortality in any chronic toxicity sample collected at the SJR.

This study is part of a 5-year effort to monitor the occurrence of toxicity to *C. dubia* in water collected from the SJR watershed. Monitoring was conducted specifically during winter months for organophosphate and carbamate insecticides that are historically applied to dormant nut and stone fruit orchards (Table 1). Orestimba Creek, a small tributary of the SJR, was monitored for acute toxicity since small tributaries historically carry higher insecticide concentrations than the mainstem of the SJR. Orestimba Creek contains runoff from the coastal range and agricultural areas in the valley floor. The SJR at Vernalis, a site downstream from agricultural and urban areas, yet above tidal influence from the delta, was examined for chronic toxicity. Additionally, in order to gain more information about pesticide residues in state surface waters, samples were analyzed for selected herbicides (Table 1). A companion study was also conducted to monitor pesticide levels and toxicity in the Sacramento River (Nordmark, 1998). Long-term monitoring of acute and chronic toxicity will help scientists at DPR to evaluate the effectiveness of programs designed to decrease the runoff of dormant spray insecticides.

MATERIALS AND METHODS

Site Description

The same sites were selected as in the three previous studies. One site each was selected for acute and chronic toxicity monitoring. Acute toxicity was monitored in Orestimba Creek, a western tributary to the SJR, where runoff in the winter is composed of drainage from the Coastal Range and from agricultural areas in the watershed (Figure 1). Samples for chronic toxicity were collected from the SJR near Vernalis, where discharges from the River's major agricultural tributaries, including the Merced, Tuolumne, and Stanislaus Rivers, are received. Discharge records for both monitoring sites were available from collocated gauging stations.

Sample Collection

Background samples were collected during the week of December 6, 1999, prior to the onset of the dormant spray season. Dormant-season sampling began on January 3, 2000, and continued through March 3, 2000, when dormant-spray applications ceased.

Chemical analyses were performed on each water sample that was collected for acute or chronic toxicity tests. Pesticides included in our analyses were chosen based on historical use during the dormant spray season in the watershed (DPR, 1995), previous detections in the watershed, and to standardize analyses between the Sacramento and San Joaquin River studies. For this study, organophosphate and carbamate insecticides and soil applied herbicides (triazine herbicides, diuron and bromacil) were analyzed in three separate screen analyses with diazinon being analyzed in a fourth (Table 1).

Acute toxicity tests were performed twice per week, with samples collected on Monday and Wednesday. One chronic toxicity test was conducted weekly using water samples collected on Monday, Wednesday and Friday. Water collected on Monday was used to begin the chronic toxicity tests. Water collected on Wednesday and Friday was used to renew chronic test water.

Water samples were collected near center channel at both Orestimba Creek at River Road and the SJR near Vernalis. Each sample was taken using a depth-integrated sampler (D-77), equipped with a Teflon bottle and nozzle. When flow was too low to use the D-77 sampler at Orestimba Creek, a Teflon bottle attached to a pole was used to collect the sample. At both sites, sub-samples were composited in a larger 38-liter stainless steel container until 12 liters were collected. This composited sample was stored on wet ice until it was delivered to the DPR West Sacramento field office later that day.

Samples were transported to the field office and split using a Geotech® 10-port splitter, into nine 1-liter amber glass bottles with Teflon® lined caps. For both sites, two 1-liter samples were submitted to DFG for toxicity testing. Four 1-liter samples were submitted for chemical analyses: one each for the organophosphate, carbamate, diazinon and herbicide analyses. Two 1-liter backups were stored at West Sacramento and one-liter was used for acidification purposes (see below).

Samples designated for organophosphate and carbamate chemical analysis were preserved by acidification with 3N hydrochloric acid to a pH of 3.0 to 3.5. Most organophosphate and carbamate pesticides are sufficiently preserved at this pH (Ross *et al.*, 1996). Diazinon, however, rapidly degrades under acidic conditions and therefore was analyzed from a separate unacidified sample. Herbicide samples are stable enough without acidification so were not acidified. Samples were stored in a 4^{o} C refrigerator until transported to the appropriate laboratory (on wet ice) for analysis. All primary samples were delivered to the testing laboratory the same day they were collected except when there was a Monday holiday, then samples were delivered early on Tuesdays.

Environmental Measurements

Temperature, dissolved oxygen (DO), pH and electrical conductivity (EC) were measured *in situ* at each sampling site. DO was measured with an YSI (Yellow Springs Instruments®) DO meter (model 58). Water pH was measured with a Sentron® pH meter (model 1001). Water temperature and EC were measured with an Orion® conductivity meter (model 142). Additionally, alkalinity, hardness, and ammonia were measured by the DFG aquatic toxicity lab (ATL) upon delivery of their samples. Totals of alkalinity and hardness were measured with a Hach®titration kit. Total ammonia was measured with an Orion® multi-parameter meter (model 290A) fitted with an Orion®ammonia ion selective electrode (model 95-12).

Daily rainfall and discharge data were also gathered for the study area. Daily rainfall measurements were obtained courtesy of the Modesto Irrigation District office in Modesto, CA about 15 miles east of the Vernalis site (Modesto Irrigation District, 2000) and Yancey Lumber and Hardware in Patterson, CA, 8 miles north west of the Orestimba Creek site (Figure 1) (Quiroba, 2000). Discharge data were collected at collocated USGS gauging stations at both the SJR at Vernalis and Orestimba Creek at River Road. Rainfall and discharge information will be used to follow annual changes in pesticide concentrations with respect to fluctuations in flow and will also be useful for modeling efforts, should they be undertaken.

Pesticide Analysis and Toxicity Tests

Pesticide analyses were performed by the CDFA Center for Analytical Chemistry. The organophosphate insecticides were analyzed using gas chromatography (GC) equipped with a flame photometric detector. The carbamate insecticides were analyzed using high performance liquid chromatography (HPLC), post column-derivatization and a fluorescence detector. The herbicides (triazine herbicides, diuron and bromacil) were analyzed by both HPLC equipped with a UV detector and by GC equipped with a nitrogen phosphorus detector. The pesticides and reporting limits are listed in Table 1. Detailed analytical methods will be provided in the final report.

Quality control (QC) for the chemistry portion of this study was conducted in accordance with Standard Operating Procedure QAQC001.00 (DPR, 1996) and consisted of a continuing QC program, plus the submission of five rinse blanks of the splitting equipment and 26 blind spikes submitted for the Sacramento and San Joaquin studies. Blind spike and continuing QC results for each of the analytical screens are presented in Tables 2 though 6. Study 184 and 185 refer to the Sacramento and San Joaquin River studies, respectively. There were no detections of any pesticides in any of the five rinse blank samples (not in tables). The 26 blind spikes were submitted for both studies along with the regular field samples. The blind spikes contained 34 chemical analytes (Table 2). More detailed quality control data, including method development, the establishment of control limits, spike recoveries and analysis of QC will be included in the final report.

<u>Toxicity Tests</u>

For Orestimba Creek, one sample per collection event was delivered to DFG s ATL for acute toxicity testing. Acute tests were performed in undiluted sample water using 96-hour, static-renewal bioassays with the cladoceran *C. dubia* in accordance with current U. S. Environmental Protection Agency procedures (U.S. EPA, 1993). From the SJR, one sample per collection event was delivered to DFG s ATL for chronic toxicity testing. Chronic tests were performed using a 7-day bioassay with *C. dubia* in accordance with current U.S. EPA (1994) procedures. Test organisms used in chronic testing were placed in sample water on day one of the testing, with test water replenished on days three and five with new water collected from the site on Wednesdays and Fridays. All bioassays were commenced and renewal water used within 36 hours of sample collection. Data were reported as the percent survival for both acute and chronic tests, and the average number of offspring per female adult (fecundity) for the chronic tests.

RESULTS

Environmental Measurements

Orestimba Creek

Temperature measurements at the Orestimba Creek site ranged between 6.2 and 12.6 ^{o}C (Figure 2). Dissolved oxygen ranged from 8.78 to 12.24 mg/L (Figure 2), with percent saturation ranging from 80 to 99%. pH ranged between 7.3 and 8.4 and EC measurements ranged from 234 to 753 µS/cm. Alkalinity ranged from 80 to 176 mg/L and hardness was between 96 and 254 mg/L. All ammonia concentrations were below the detection limit of 50 µg/L.

Compared to last year's dormant-spray seasons, this season was much wetter. During the weekly monitoring period from January 3 to March 3, 2000, rainfall reported at Patterson, California totaled 8.52 inches (Quiroba, 2000) (Figure 3). During last year's study 2.82 inches of rain was recorded at the same site. Daily mean discharge at the USGS gauging station along Orestimba Creek at River Road ranged from 2.7 to 986 cfs (cubic feet per second). These data are provisional and subject to change.

San Joaquin River near Vernalis

Water temperature measurements at the SJR site ranged from 8.3 to 14.1^{o} C (Figure 4). Dissolved oxygen ranged from 8.00 to 11.16 mg/L (Figure 4), with percent saturation ranging from 75 to 96%. pH ranged from 6.5 to 7.9 and EC measurements were between 216 and 1063 µS/cm. Alkalinity ranged from 46 to 150 mg/L and hardness from 44 to 214 mg/L. All ammonia concentrations were below the detection limit of 50 µg/L.

During the weekly monitoring period from January 3 to March 3, 2000, discharge at the SJR site ranged from 1581 to 15,514 cfs (Figure 5). Rainfall data collected by the Modesto Irrigation District totaled 10.28 inches during the same period (MID, 2000). Last year's rainfall totaled 5.20 inches during the same three month period at the same site. These data are provisional and subject to change.

Pesticide Concentrations and Toxicity Data

Pesticide Concentrations

During this study there were no detections of carbamate insecticides in the 20 samples collected at the Orestimba Creek site (Table 7). However, a few organophosphates were detected. Diazinon was detected in 4 of the 20 samples (20%). The detections occurred on January 12, January 19, January 24, and January 26, 2000 at 0.161, 0.043, 0.069, and 0.051 µg/L, respectively. All of these detections occurred during or one day after a rain event. Methidathion was detected twice (10%) on January 26 at 1.74 µg/L and again on February 23, 2000, at 0.063 µg/L. Dimethoate was also detected twice (10%) on January 19, and February 23, 2000, at 0.213 and 0.117 µg/L, respectively. Several herbicides were also detected at this site. Diuron was detected in 16 of 20 samples (80%) at concentrations ranging from 0.059 to 1.99 µg/L (Figure 3). Prometryn was detected five times (25%) ranging from 0.128 to 3.127 µg/L and simazine 11 times (55%) ranging from 0.060 to 2.892 µg/L. Bromacil and prometon were each detected once (5%) at 0.082 and 0.078 µg/L, respectively. Fourteen samples (70%) contained more than one herbicide residue. Four herbicides were detected in two samples that were collected on January 24 and 26, after heavy rain events on January 24 and 25, 2000. There was no significant mortality detected in any acute toxicity sample collected at Orestimba Creek.

Diazinon was detected in 3 of the 30 samples (10%) collected from the SJR at Vernalis (Table 7). The detections occurred on January 21, 2000 at 0.100 µg/L, January 24 at 0.045 µg/L and on January 26 at 0.051 µg/L (Figure 5). Fonofos was also detected once, on January 24, at 0.065 µg/L. Each detection occurred within 1 day after a rain event. There were no other organophosphate or carbamate insecticides detected. Diuron was detected in every sample collected at Vernalis during the dormant spray season ranging in concentration from 0.115 to 3.72 µg/L (Figure 5). Simazine was detected in 15 of the 30 samples (50%) ranging in concentration from 0.050 to 0.299 µg/L. Bromacil was detected in three of 30 samples (10%) at concentrations from 0.056 to 0.074 µg/L. Prometryn was detected in three samples (10%) ranging from 0.064 to 0.203 µg/L and cyanazine was detected once (3.3%) at 0.350 µg/L. Fifteen samples (50%) had more than one triazine detection and four of them (13.3%) had three or four detections in one sample. There was no significant mortality in any chronic toxicity sample collected at the SJR.

If you have any questions, please feel free to call me at 916-324-4110.

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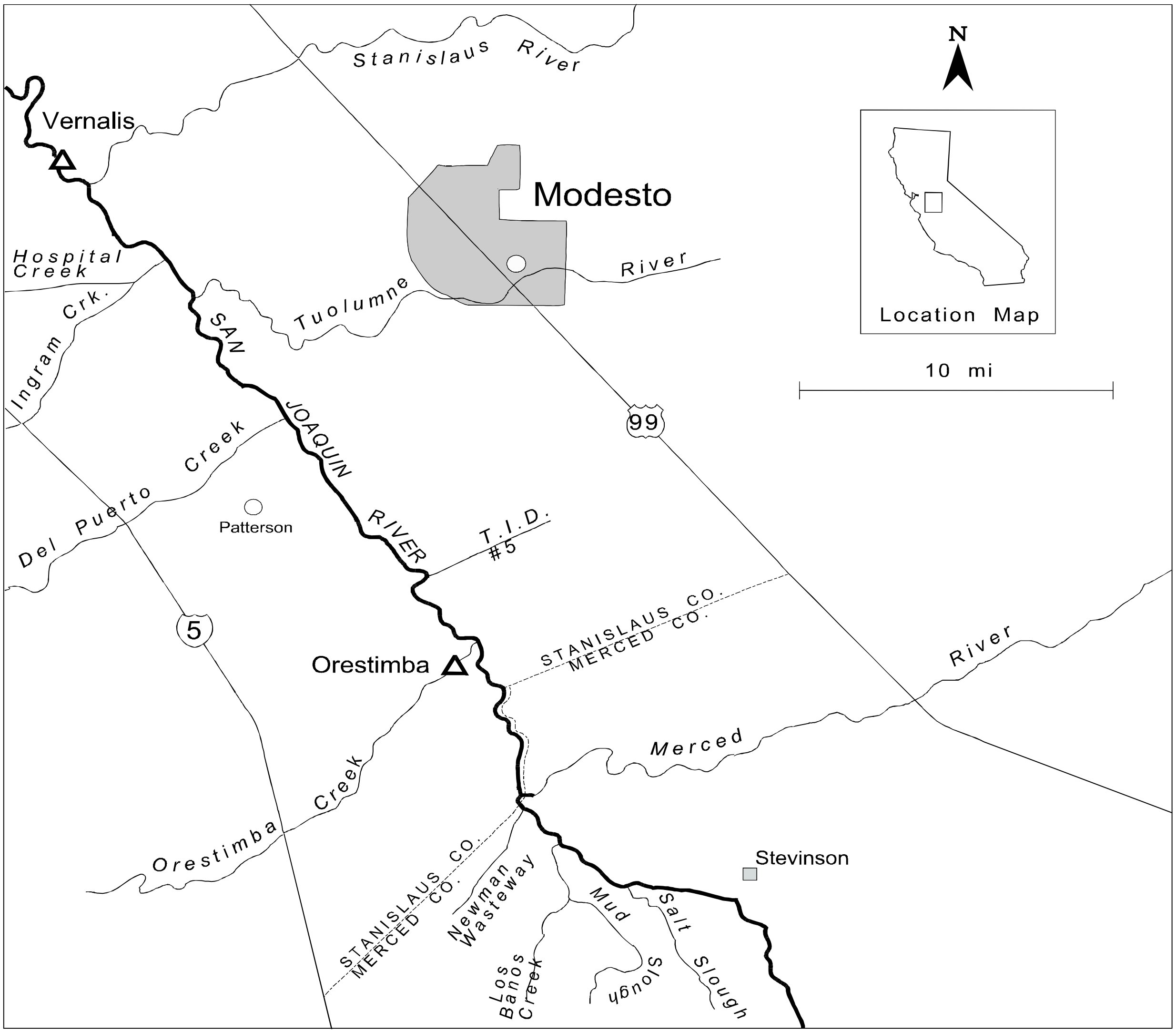

Figure 1. Location of toxicity sampling sites (△) and rainfall stations (○) in the San Joaquin River Watershed: Winter 1999-2000.

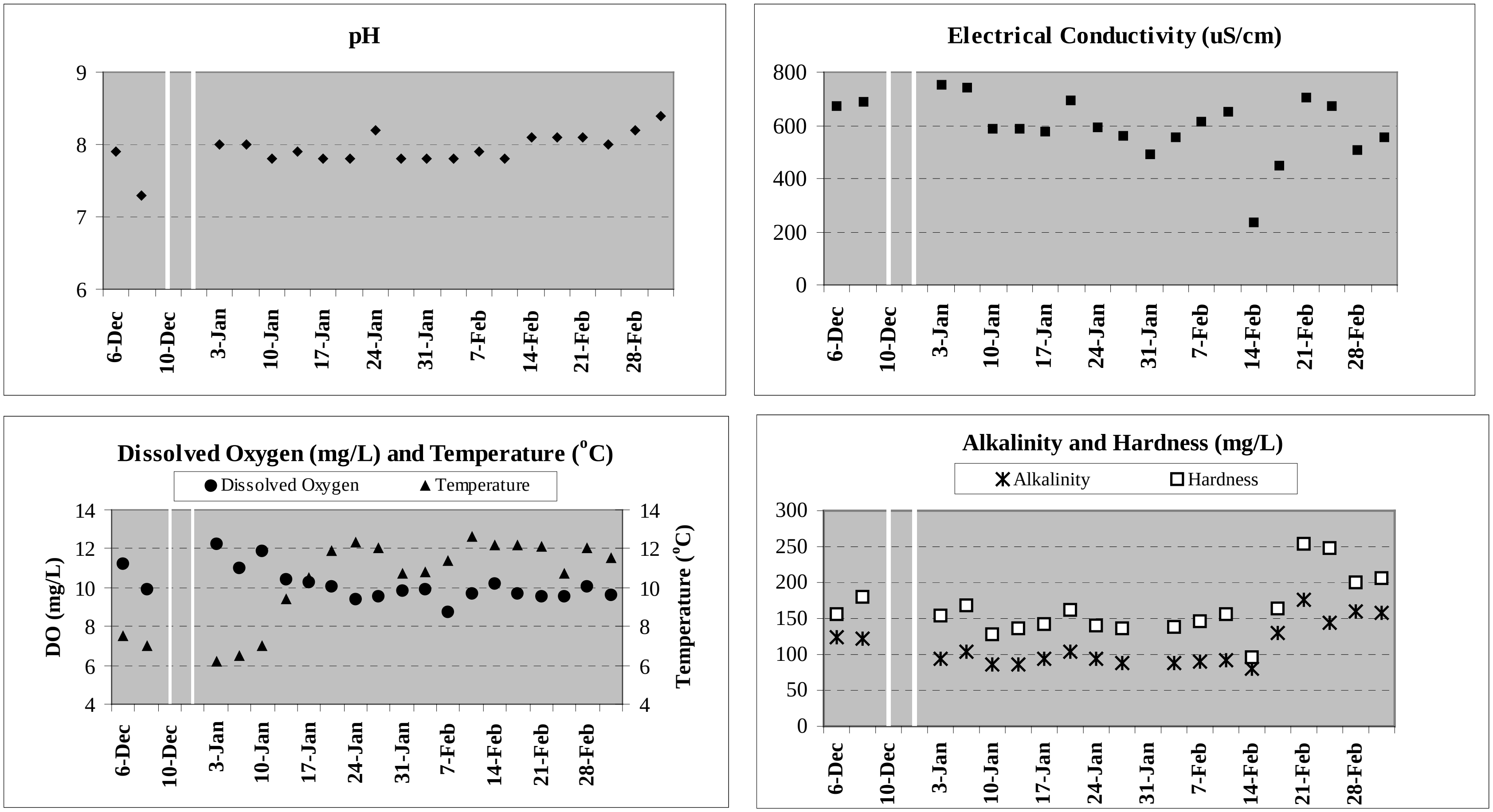

Figure 2. Environmental measurements for Orestimba Creek at River Road. Data Collected from December 6 through 8, 1999 and January 3 through March 1, 2000. Data was collected twice each week during the study period. Double bar denotes a break in sampling between background and dormant season samples.

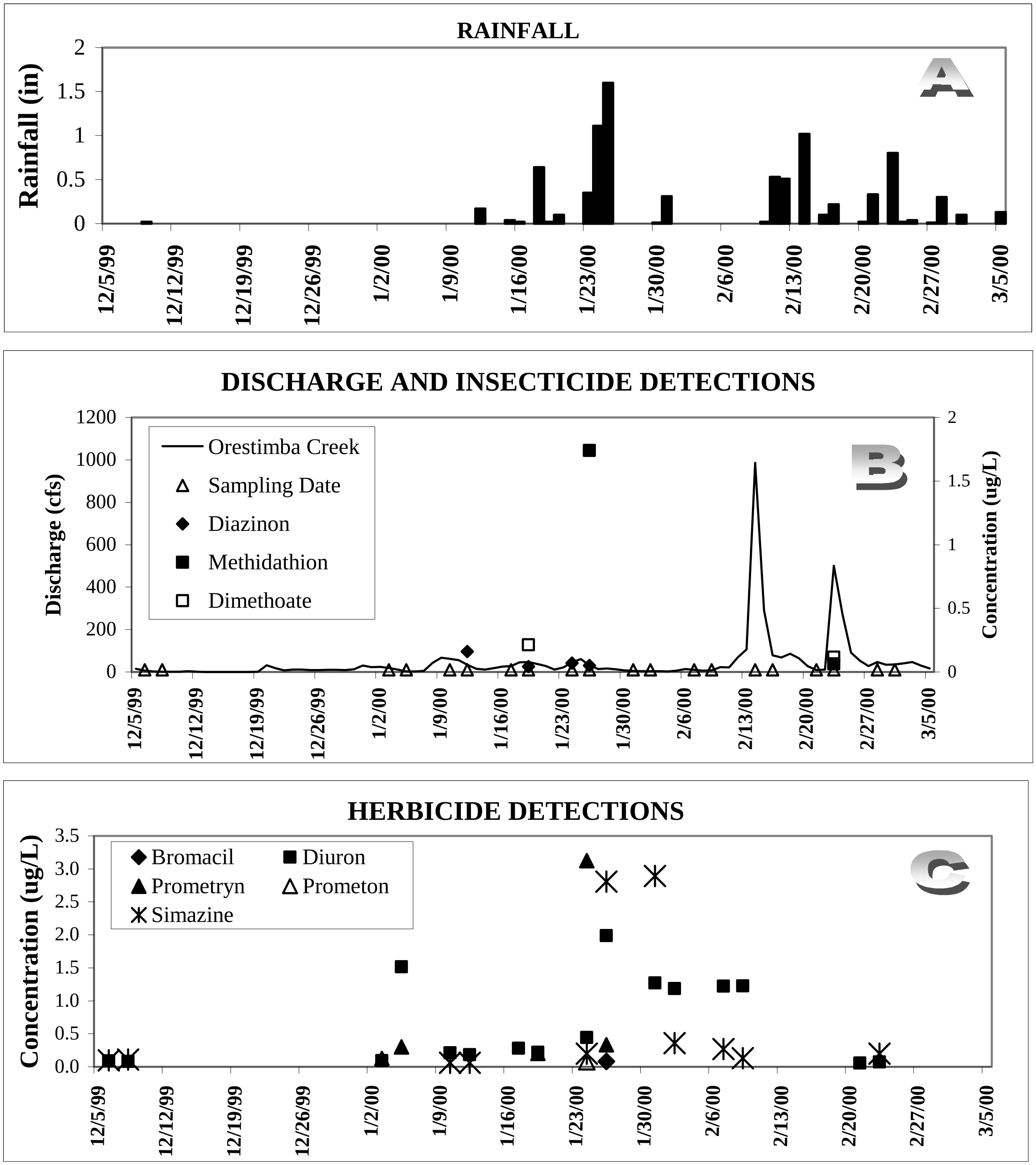

Figure 3. (A) Rainfall measured at Patterson; (B) Discharge and insecticides were measured in Orestimba Creek at River Road; (C) Detections of herbicides, winter 1999-2000.

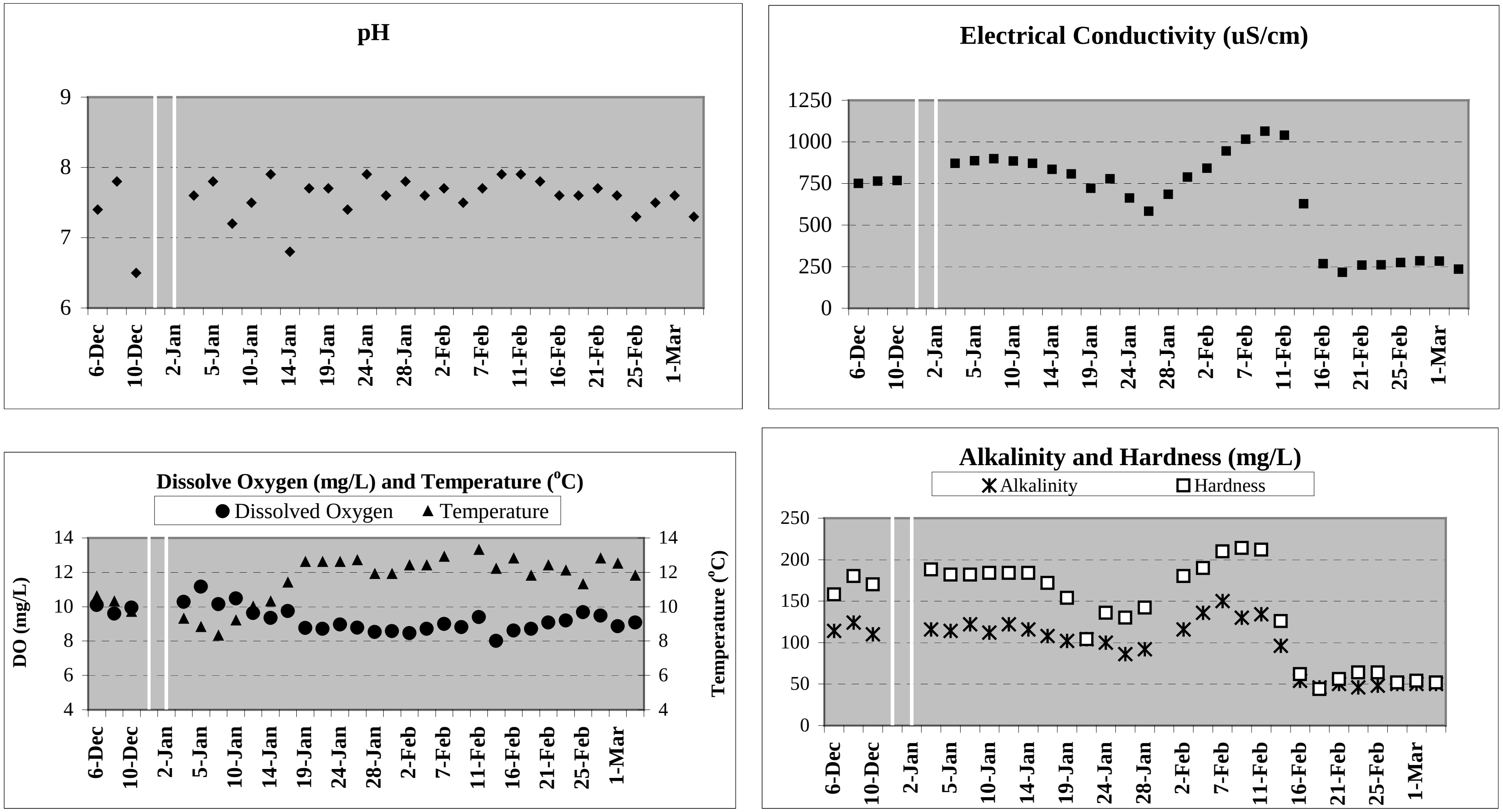

Figure 4. Environmental measurements for the San Joaquin River at Vernalis, CA. Data Collected from December 6 through 10, 1999 and January 3 through March 3, 2000. Data was collected three times each week during the study period. Double bar denotes a break in sampling between background and dormant season samples.

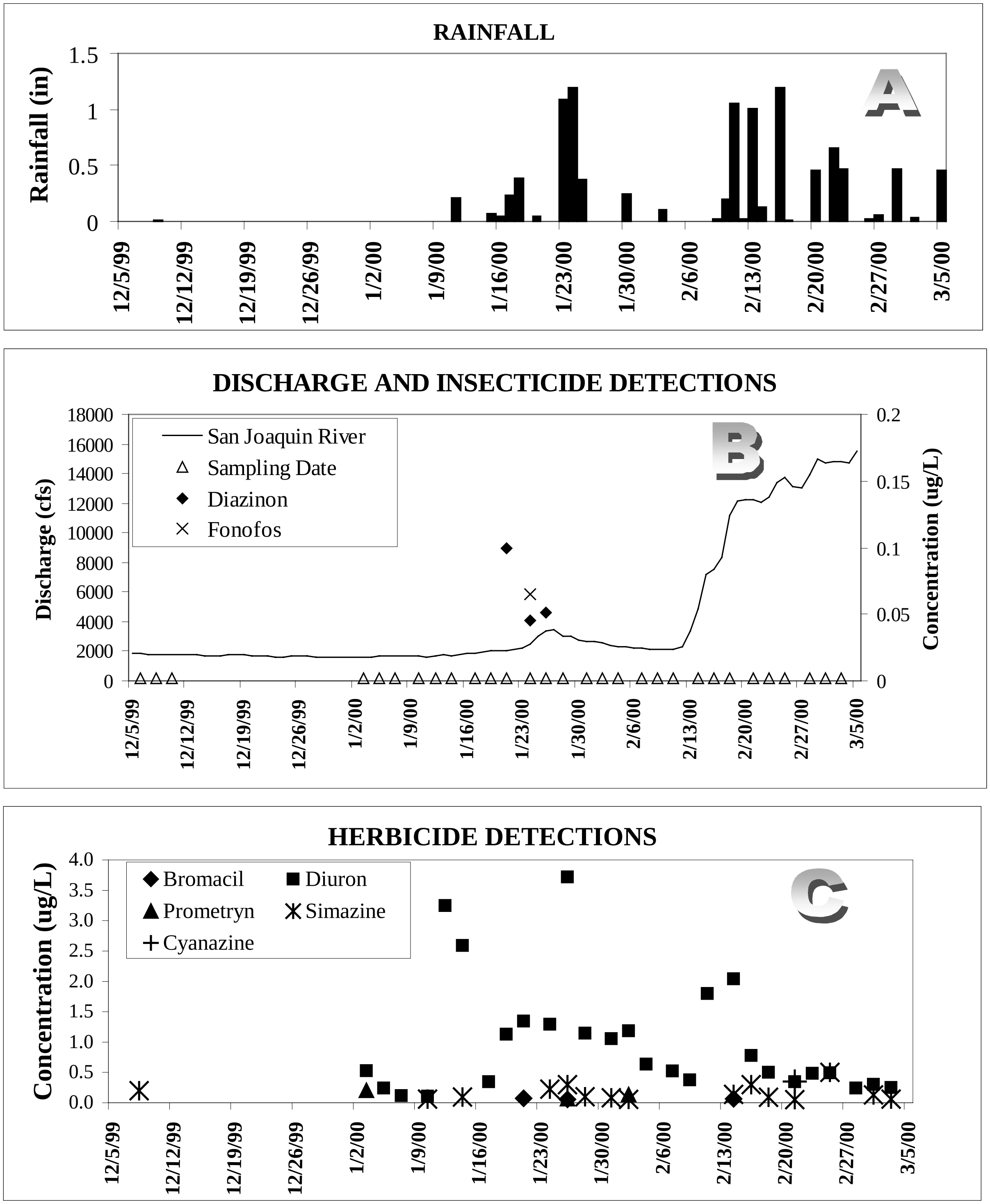

Figure 5. (A) Rainfall measured at Modesto; (B) Discharge and insecticides were measured in the San Joaquin River near Vernalis; (C) Detections of herbicides, winter 1999-2000.

Table 1. California Department of Food and Agriculture, Center for Analytical Chemistry organophosphate and carbamate insecticide and multiple herbicide screens for the San Joaquin River toxicity monitoring study.

Organophosphate Pesticides in Surface Water by GC **Method: GC/FPD**		**N-Methyl Carbamate in Surface Water by HPLC** **Method: HPLC/Post Column-fluorescence**		**Herbicides in Surface Water by HPLC** **Method: HPLC/Post Column-fluorescence**	
Compound	**Reporting Limit (µg/L)**	**Compound**	**Reporting Limit (µg/L)**	**Compound**	**Reporting Limit (µg/L)**
Chlorpyrifos	0.04	Carbaryl	0.05	Atrazine	0.05
Diazinon[1]	0.04	Carbofuran	0.05	Bromacil	0.05
Dimethoate (Cygon)	0.05			Diuron	0.05
Fonofos	0.05			Cyanazine	0.2
Malathion	0.05			Hexazinone	0.2
Methidathion	0.05			Metribuzin	0.2
Methyl parathion	0.05			Prometon	0.05
Phosmet	0.05			Prometryn	0.05
				Simazine	0.05

[1] Diazinon is analyzed from a separate, unpreserved, split sample. Other OP and CB chemical samples are preserved with 3N HCl to a pH of 3-3.5 to retard analyte degradation. See text.

Table 2. Blind Spike Recoveries for the Sacramento and San Joaquin River Studies.

Extraction Date	Study Number[a]	Sample Number	Screen	Pesticide	Spike Level	Recovery	Percent Recovery	Exceed CL[b]
1/11/00	184	107	OP	Fonofos	0.2	0.152	76.0	LCL
				Phosmet	0.4	0.435	109	
1/13/00	185	169	OP	Dimethoate	0.2	0.225	112.5	
				Methidathion	0.1	0.11	110.0	
1/18/00	184	136	DI	Diazinon	0.1	0.0911	91.1	
1/20/00	184	170	TR	Prometryn	0.3	0.262	87.3	
				Hexazinone	0.5	0.545	109	
1/20/00	184	169	CB	Carbaryl	0.1	0.107	107	UCL
				Carbofuran	0.2	0.192	96	
1/21/00	183	556	DI	Diazinon	0.2	0.194	97.0	
1/25/00	185	525	TR	Bromacil	0.2	0.178	89	
				Prometon	0.25***	0.253	101	
1/28/00	185	170	TR	Atrazine	0.2	0.194	97.0	
2/1/00	185	171	CB	Carbaryl	0.2	0.209	104.5	UCL
2/3/00	185	294	TR	Simazine	0.2	0.192	96	
2/3/00	185	292	OP	Malathion	0.2	0.182	91.0	
				Chlorpyrifos	0.3	0.266	89	
2/7/00	184	362	TR	Diuron	0.3	0.297	99	
				Cyanazine	0.5	0.489	98	
2/8/00	184	361	OP	Methyl Parathion	0.2	0.201	101	
2/15/00	184	319	DI	Diazinon	0.2	0.192	96.0	
2/19/00	185	293	DI	Diazinon	0.1	0.1	100.0	
2/21/00	184	476	TR	Prometon	0.4	0.435	108.8	
2/22/00	184	475	OP	Phosmet	0.3	0.34	113.3	
2/24/00	184	477	DI	Diazinon	0.2	0.171	85.5	
2/25/00	185	295	TR	Metribuzine	0.5	0.442	88.4	
2/28/00	184	364	OP	Chlorpyrifos	0.3	0.25	83.3	
2/28/00	185	332	TR	Bromacil	0.3	0.294	98.0	
				Atrazine	0.3	0.325	108.3	
2/28/00	184	363	OP	Chlorpyrifos	0.3	0.256	85.3	
3/1/00	185	334	CB	Carbaryl	0.25	0.239	95.6	
3/2/00	185	333	DI	Diazinon	0.2	0.164	82.0	
3/3/00	185	336	OP	Dimethoate	0.2	0.251	125.5	UCL
3/3/00	185	335	CB	Carbofuran	0.35	0.343	98.0	

[a] 184 refers to the study number for the Sacramento River, 185 refers to the SJR.

[b] CL=Control Limit; Upper CL (UCL), Lower CL (LCL). CLs for these pestcides are listed in Tables 3 through 6.

*** Prometon was accidentally spiked at 0.25ppb but was supposed to be 0.5ppb

Table 3. Continuing Quality Control- Organophosphate Screen

Extraction Date	Sample Numbers	Percent Recovery Chlorpyrifos	Diazinon	Dimethoate	Fonofos	Malathion	Methidathion	Methyl Parathion	Phosmet
12/7/99	**184**- 1, 7, 13 **185**- 1, 7	87.5	82.5	103	80.0	96.0	100	93.0	83.0
12/10/99	**184**- 19, 25, 31 **185**- 13, 19	101	96.3	111	93.0	104	114	107	100
12/14/99	**184**- 37 **185**- 25	88.8	85.0	105	86.0	97.0	103	100	91.6
1/13/00	**184**- 108, 114, 120 **185**- 79, 85, 169	93.8	90.0	86.0	86.0	103	90.0	107	88.4
1/19/00	**184**- 126, 132, 137, 143, 149. **185**- 91, 97, 103	92.5	88.8	82.0	83.0	108	90.0	108	95.8
1/20/00	**184**- 155, 161, 167 **185**- 109, 115	100	96.3	111	93.0	101	120	103	104
1/25/00	**184**- 175, 181, 185, 191, 197 **185**- 121, 127, 131, 137	88.8	88.8	102	85.0	92.0	97.0	90.0	91.2
1/27/00	**184**- 203, 209, 215 **185**- 140, 143, 149	91.3	87.5	106	86.0	106	120	112	116
2/1/00	**184**- 221, 227, 231, 237, 243 **185**- 155, 161, 167	91.3	78.8	99.0	73.0	99.0	109	96.0	95.4
2/3/00	**184**- 249, 255, 261 **185**- 176, 182, 292	91.3	91.3	108	89.0	97.0	104	98.0	97.8
2/8/00	**184**- 267, 273, 279, 285, 361 **185**- 188, 189, 194, 198, 204	88.8	92.5	98.0	86.0	98.0	108	101	106
2/10/00	**184**- 291, 297, 303 **185**- 210, 216	95	93.8	108	85.0	105	111	114	107
2/15/00	**184**- 309, 315, 320, 326 **185**- 222, 228, 234	101	98.8	108	96.0	104	112	108	101
2/17/00	**184**- 338, 344, 350 **185**- 240, 246	98.8	93.8	113	94.0	110	112	116	103
2/22/00	**184**- 365, 368, 374, 380, 475 **185**- 251, 258, 262, 267	98.8	96.3	115	95.0	103	117	112	92.8
2/24/00	**184**- 386, 392, 398 **185**- 274, 280	85.0	81.3	105	78.0	92.0	101	93.0	105
2/29/00	**184**- 363, 364, 404, 411, 417, 423 **185**- 286, 296, 302	97.5	97.5	103	95.0	105	113	108	95.8
3/1/00	**184**- 429, 435, 441 **185**- 308, 314	95.0	93.8	110	89.0	105	112	110	97.2
3/7/00	**184**- 447, 453, 457, 478, 484 **185**- 320, 326, 336	105	104	109	104	112	118	112	102
3/9/00	**184**- 463, 469, 490	96.3	91.3	114	90.0	105	111	103	115
3/14/00	**184**- 496, 524	97.5	92.5	86.0	93.0	105	111	103	109
Average Recovery		94.9	92.1	103.5	88.9	102.8	108.7	105.2	101.2
Standard Deviation		5.03	5.95	9.88	7.18	5.46	9.06	7.40	7.70
CV		5.30	6.46	9.54	8.07	5.31	8.34	7.03	7.60
Upper Control Limit		116	122	116	102	114	124	116	118
Upper Warning Limit		110	113	110	100	109	116	110	113
Lower Warning Limit		83	78	86	94	87	83	85	95
Lower Control Limit		76	69	80	92	81	75	79	90

*Highlighted cells are percent recoveries exceeding control limits

Table 4. Continuing Quality Control-Carbamate Screen

Extraction Date	Sample Numbers	Percent Recovery Carbofuran	Carbaryl
1/20/00	**184**- 156, 162, 168, 169 **185**- 110, 116	87.5	96.0
1/26/00	**184**- 176, 182, 186, 192, 198 **185**- 122, 128, 132, 138	82.8	93.4
1/27/00	**184**- 204, 210, 216 **185**- 144, 150	92.2	95.7
2/1/00	**184**- 222, 228, 232, 238, 244 **185**- 156, 162, 168, 171	73.8	90.8
2/3/00	**184**- 250, 256, 262 **185**- 177, 183	81.2	97.0
2/8/00	**184**- 268, 274, 280, 286 **185**- 195, 199, 205	90.5	99.2
2/22/00	**184**- 357, 369, 375, 381 **185**- 253, 259, 263, 269	74.2	93.0
2/10/00	**184**- 292, 298, 304 **185**- 211, 217	98.8	100
2/15/00	**184**- 310, 316, 321, 327, 333 **185**- 223, 229, 235	89.4	98.2
2/17/00	**184**- 339, 345, 351 **185**- 241, 247	88.8	95.1
2/24/00	**184**- 387, 393, 399 **185**- 275, 281	75.0	90.5
2/29/00	**184**- 405, 412, 418, 424 **185**- 287, 297, 303	91.0	99.5
3/2/00	**184**- 430, 436, 442 **185**- 309, 315, 334	80.1	94.2
3/7/00	**184**- 448, 454, 458, 479, 485 **185**- 191, 321, 327, 335	86.2	98.4
3/9/00	**184**- 464, 470, 491	76.5	93.3
3/13/00	**184**- 497, 525	83.4	95.0
Average Recovery		84.5	95.6
Standard Deviation		7.33	2.98
CV		8.68	3.11
Upper Control Limit		99.8	99.5
Upper Warning Limit		95.7	96.0
Lower Warning Limit		79.2	82.2
Lower Control Limit		75.0	78.7

*Highlighted cells are percent recoveries exceeding control limits

Table 5. Continuing Quality Control-Diazinon Analysis

Extraction Date	Sample Numbers	Percent Recovery Diazinon
12/7/99	**184**- 3, 9, 15 **185**- 3, 9	86.5
12/9/99	**184**- 21, 27, 33 **185**- 15, 22	91.3
12/14/99	**184**- 39 **185**- 27	98.8
1/4/00	**184**- 45, 51, 57 **185**- 33, 39	112
1/18/00	**184**- 128, 133, 136, 139, 145, 151 **185**- 93, 99, 105	89.1
1/21/00	**184**- 157, 163, 171 **185**- 111, 117	91.3
1/25/00	**184**- 177, 183, 187, 193, 199 **185**- 123, 129, 133, 139	113
1/27/00	**184**- 205, 211, 217 **185**- 145, 151	106
2/3/00	**184**- 251, 257, 263 **185**- 178, 184	92.5
2/8/00	**184**- 269, 275, 281, 287 **185**- 190, 196, 200, 206	78.8
2/10/00	**184**- 293, 299, 305 **185**- 212, 218	97.5
2/15/00	**184**- 311, 317, 319, 322, 328, 334 **185**- 224, 230, 236	85.0
2/17/00	**184**- 340, 346, 352 **185**- 242, 248, 293 189- 804	92.5
2/22/00	**184**- 358, 370, 376, 382 **185**- 254, 260, 264, 270	96.3
2/24/00	**184**- 388, 394, 400, 477 **185**- 276, 282	90.0
2/29/00	**184**- 407, 413, 419, 425 **185**- 288, 298, 304	102
3/2/00	**184**- 431, 437, 443 **185**- 310, 316, 333	80.0
3/9/00	**184**- 449, 455, 459, 465, 471, 480, 486, 492 **185**- 322, 328	103
3/14/00	**184**- 498, 526	91.3
Average Recovery		93.8
Standard Deviation		9.3
CV		9.90
Upper Control Limit		109
Upper Warning Limit		103
Lower Warning Limit		77.6
Lower Control Limit		71.4

Table 6. Continuing Quality Control- Triazine/Diuron/Bromacil Screen

Extraction Date	Sample Numbers	Percent Recovery Bromacil	Simazine	Atrazine	Diuron	Prometon	Prometryn	Hexazinone	Cyanazine	Metribuzin
12/09/99	**184**- 6, 12, 18 **185**- 6, 11	99.8	107.7	73.7	75.4	88.7	85.0	95.9	100.0	86.1
12/13/99	**184**- 24, 30, 136, 42 **185**- 18, 24, 30	88.6	93.6	78.3	72.6	83.2	80.1	96.9	91.0	88.0
1/11/00	**184**- 94, 100, 106, 84, 88 **185**- 72, 78, 60, 66	88.1	108.2	93.4	90.7	89.2	87.5	93.9	92.1	90.0
1/13/00	**184**- 113, 119, 125 **185**- 84, 90	106.3	114.4	113.3	96.6	94.2	100.4	102.3	93.6	87.7
1/18/00	**184**- 131, 134, 142, 148 154 **185**- 96, 102, 108	84.0	79.0	97.5	93.8	94.8	99.8	98.8	93.7	103.2
1/20/00	**184**- 160, 166, 170, 174 **185**- 114, 120	91.5	95.2	88.2	87.4	94.8	99.8	98.8	93.7	103.2
1/21/00	**184**- 180, 184 **185**- 126, 130, 525	80.8	79.9	79.1	85.8	95.8	104.1	117.9	93.5	83.6
1/26/00	**184**- 19, 196, 202 **185**- 136, 142	100.5	103.9	92.6	102.1	87.1	102.6	102.5	114.3	102.1
1/27/00	**184**- 208, 214, 220 **185**- 148, 159	82.9	81.3	79.8	92.0	97.7	100.1	107.9	95.7	86.1
2/1/00	**184**- 226, 230, 236, 242, 248 **185**- 160, 166, 170, 175	94.0	107.3	88.4	104.8	102.5	108.6	109.4	96.3	92.0
2/3/00	**184**- 254, 260, 266 **185**- 181, 187, 295	86.8	87.1	82.2	96.9	74.3	98.1	104.0	96.4	92.8
2/8/00	**184**- 272, 278, 284, 290, 362 **185**- 193, 197, 203, 209	89.1	104.4	97.5	110.3	96.8	108.0	94.0	86.8	85.5
2/10/00	**184**- 296, 302, 308 **185**- 215, 221	88.4	101.3	81.3	89.3	94.5	108.4	110.1	103.9	93.6
2/15/00	**184**- 314, 318, 325, 331, 337 **185**- 227, 233, 239	90.9	93.2	80.7	88.4	93.7	94.6	100.0	91.3	82.7
2/17/00	**184**- 343, 349, 355 **185**- 245, 252	91.0	93.2	106.6	100.6	88.1	82.4	100.7	93.5	87.1
2/22/00	**184**- 367, 373, 379, 385, 476 **185**- 257, 268, 273	96.6	89.6	117.4	90.3	96.1	93.9	103.4	112.5	99.5
2/24/00	**184**- 391, 397, 403 **185**- 279, 285	93.6	105.8	96.3	89.1	106.2	99.3	95.3	93.2	86.4
2/29/00	**184**- 410, 416, 422, 428 **185**- 291, 295, 301, 307, 332	93.1	94.8	99.2	90.3	96.6	93.9	119.2	117.2	101.1
3/2/00	**184**- 434, 440, 446 **185**- 313, 319	82.1	89.8	82.8	89.2	105.7	100.8	96.3	92.0	94.0
3/7/00	**184**- 452, 456, 462, 483, 489 **185**- 325, 329	84.3	98.1	117.6	105.9	89.6	88.8	93.1	88.9	79.8
3/9/00	**184**- 468, 474, 495	106.7	116.8	109.4	111.0	108.1	92.7	99.6	91.9	89.9
3/14/00	**184**- 501, 527	91.1	93.4	85.4	94.7	91.1	78.7	93.2	89.8	88.9
Average Recovery		91.1	96.8	94.4	95.5	94.8	97.1	102	96.5	91.5
Standard Deviation		7.26	10.9	12.8	7.78	7.61	8.19	7.63	8.57	7.14
CV		7.97	11.2	13.5	8.15	8.03	8.43	7.48	8.88	7.80
Upper Control Limit		115	126	121	117	111	115	123	121	105
Upper Warning Limit		109	118	114	108	104	108	115	114	101
Lower Warning Limit		86.5	86.4	85.0	74.6	75.9	79.1	84.5	87.4	84.5
Lower Control Limit		80.9	78.5	77.7	66.2	68.9	71.9	76.8	80.7	80.4

*Highlighted cells are percent recoveries exceeding control limits

Table 7. Concentrations of pesticides (ppb) detected in samples from Orestimba Creek and the San Joaquin River, winter 1999-2000

ORESTIMBA CREEK at RIVER ROAD										**SAN JOAQUIN RIVER near VERNALIS**								
Sampling Date	Diazinon	Methidathion	Dimethoate	Bromacil	Diuron	Prometryn	Prometon	Simazine	Acute Toxicity (% survival[1])	Diazinon	Fonofos	Bromacil	Diuron	Prometryn	Simazine	Cyanazine	Chronic Toxicity (% survival[1])	Chronic Toxicity (offspring/female[1])
12/6/99	nd[2]	nd	nd	nd	0.0890	nd	nd	0.0990	100/100	nd	nd	nd	nd	nd	nd	nd		
12/8/99	nd	nd	nd	nd	0.0800	nd	nd	0.1100	100/90	nd	nd	nd	nd	nd	0.2000	nd		
12/10/99										nd	nd	nd	nd	nd	nd	nd	100/100	40.2/17.7
1/3/00	nd	nd	nd	nd	0.0955	0.1280	nd	nd	100/100	nd	nd	nd	0.5293	0.2030	nd	nd		
1/5/00	nd	nd	nd	nd	1.5164	0.3020	nd	nd	100/85[3]	nd	nd	nd	0.2432	nd	nd	nd		
1/7/00										nd	nd	nd	0.1150	nd	nd	nd	90/90	32.6/22.9
1/10/00	nd	nd	nd	nd	0.2110	nd	nd	0.0600	95/95	nd	nd	nd	0.1090	nd	0.0620	nd		
1/12/00	0.1610	nd	nd	nd	0.1830	nd	nd	0.0600	100/100	nd	nd	nd	3.2470	nd	nd	nd		
1/14/00										nd	nd	nd	2.5920	nd	0.0950	nd	100/100	20.5/13.5
1/17/00	nd	nd	nd	nd	0.2820	nd	nd	nd	90/100	nd	nd	nd	0.3480	nd	nd	nd		
1/19/00	0.0430	nd	0.2130	nd	0.2190	0.2020	nd	nd	100/100	nd	nd	nd	1.1300	nd	nd	nd		
1/21/00										0.1000	nd	0.0740	1.3470	nd	nd	nd	80/80	29.6/30.7
1/24/00	0.0690	nd	nd	nd	0.4450	3.1270	0.0780	0.2000	100/95	0.0450	0.0650	nd	1.2910	nd	0.2270	nd		
1/26/00	0.0510	1.7400	nd	0.0820	1.9900	0.3360	nd	2.8090	85/100	0.0510	nd	0.0560	3.7200	0.0640	0.2990	nd		
1/28/00										nd	nd	nd	1.1480	nd	0.0980	nd	90/90	18.1/14.7
1/31/00	nd	nd	nd	nd	1.2720	nd	nd	2.8920	100/100	nd	nd	nd	1.0550	nd	0.0820	nd		
2/2/00	nd	nd	nd	nd	1.1870	nd	nd	0.3600	100/90	nd	nd	nd	1.1840	0.1320	0.0550	nd		
2/4/00										nd	nd	nd	0.6350	nd	nd	nd	100/100	25.0/15.3
2/7/00	nd	nd	nd	nd	1.2230	nd	nd	0.2690	100/95	nd	nd	nd	0.5240	nd	nd	nd		
2/9/00	nd	nd	nd	nd	1.2280	nd	nd	0.1310	100/100	nd	nd	nd	0.3760	nd	nd	nd		
2/11/00										nd	nd	nd	1.8000	nd	nd	nd	90/70[4]	37.3/23.6
2/14/00	nd	nd	nd	nd	nd	nd	nd	nd	90/100	nd	nd	0.0660	2.0400	nd	0.1390	nd		
2/16/00	nd	nd	nd	nd	nd	nd	nd	nd	85/100	nd	nd	nd	0.7780	nd	0.2970	nd		
2/18/00										nd	nd	nd	0.5030	nd	0.0890	nd	100/100	32.8/21.2
2/21/00	nd	nd	nd	nd	0.0590	nd	nd	nd	95/100	nd	nd	nd	0.3450	nd	0.0500	0.3500		
2/23/00	nd	0.0630	0.1170	nd	0.0740	nd	nd	0.2010	100/95	nd	nd	nd	0.4860	nd	nd	nd		
2/25/00										nd	nd	nd	0.4930	nd	0.0500	nd	80/90	35.6/28.6
2/28/00	nd	nd	nd	nd	nd	nd	nd	nd	100/100	nd	nd	nd	0.2440	nd	nd	nd		
3/1/00	nd	nd	nd	nd	nd	nd	nd	nd	100/100	nd	nd	nd	0.3010	nd	0.1290	nd		
3/3/00										nd	nd	nd	0.2520	nd	0.0590	nd	90/90	40.9/46.2

1. Two numbers are reported for toxicity tests. The first is the result from the sample and the second is the result from the corresponding control.
2. nd = none detected
3. Test not rerun although control survival was < 90%
4. Test not rerun although control survival was < 80%

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