Department of Pesticide Regulation
Environmental Monitoring Branch
1001 I Street
Sacramento, California 95812
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Study 224. A Preliminary Assessment of Pyrethroid Contamination of Surface Waters and Bed Sediments in High Pyrethroid-Use Regions of California

I. INTRODUCTION

Synthetic pyrethroid insecticides are applied to a variety of crops in California throughout the year. In 2002, over 200,000 pounds of pyrethroid insecticide active ingredients were applied to agricultural fields throughout the state. The six primary pyrethroids used in California agriculture, in order of decreasing amount applied in 2002, are permethrin, esfenvalerate, cyfluthrin, lambda-cyhalothrin, bifenthrin and cypermethrin (DPR 2004a). Use of pyrethroid insecticides is expected to increase as they are utilized more commonly as replacements for organophosphate insecticides.

Due to the aquatic toxicity of the pyrethroid insecticides (Table 1), offsite movement of these compounds into surface water is of concern. Recent monitoring studies conducted in California have shown pyrethroid contamination of both surface water/suspended sediment and stream bed sediment (Weston, *et.al*., 2004; Gill and Spurlock, 2004; Bacey *et al.*, 2003; Walters, *et al.*, 2002; Kelley and Starner, in preparation). Considering their high and potentially increasing use in California, reliable information regarding the environmental fate of these compounds is increasingly important.

The pyrethroid insecticides are extremely hydrophobic, with high adsorption coefficients and very low water solubility (Table 1). Due to their adsorption properties, determining the impact of pyrethroids on aquatic systems requires monitoring of suspended and bed sediments in addition to surface water (Weston *et al*., 2004).

In an effort to begin to assess the extent of pyrethroid contamination of surface waters in California, the current survey study is designed to measure the concentrations of the six primary pyrethroid insecticides in bed sediment and surface water/suspended sediment from water bodies in four regions of relatively high pyrethroid use.

Through analysis of data from the California Department of Pesticide Regulation's (DPR) Pesticide Use Reporting (PUR) database (1998-2002 data), several regions of the state were identified which historically have at least one period per year of relatively high use of pyrethroid insecticides lasting two or more months (DPR 2004a; see footnote). Agricultural use of pyrethroid insecticides is highest in the Salinas Valley region (Monterey County), the Imperial Valley region (Imperial County), the Feather River region in the Sacramento Valley, and several areas of the San Joaquin Valley (Figures 1 through 4, Table 2).

The four regions represent a variety of climates, pyrethroid use patterns, soil types and agricultural practices, factors which impact the potential for offsite movement of pyrethroids.

Salinas Valley/Salinas River Region, Monterey County

Over 33,000 pounds of pyrethroid insecticide active ingredients were applied to agricultural crops in Monterey County in 2002 (Figure 1). Of this amount, most was applied within an approximately 350 square mile area in the Salinas Valley region. This region extends approximately 50 miles along the

Note: All citations regarding pesticide use data are from DPR 2004a.

Salinas River from Monterey Bay north of the town of Marina southeast to about King City. Much of this area is cultivated year-round, with associated intensive use of pyrethroid insecticides and other pesticides. In 2002, over 1,000 pounds of pyrethroids were applied each month in eight of 12 months throughout the region.

The primary high-use period in the Salinas Valley region is from May through September; 70% of all pyrethroid use in the region occurs during this period (Figure 5). During this period in 2002, over 19,500 pounds of pyrethroid active ingredients were applied, with over 65% of this amount applied to lettuce. Use on lettuce, spinach, celery, artichokes and broccoli accounted for 90% of the amount applied in this period. Pyrethroid use in the region was similar during 1998-2001.

The peak period of organophosphate insecticide use in the region occurs during this same period, with over 95,000 pounds of diazinon and over 27,000 pounds of chlorpyrifos applied during May through September of 2002.

The Salinas River flows into the Monterey Bay National Marine Sanctuary, and the lower river is a primary migration corridor for endangered steelhead trout (*Onchorhynchus mykiss*)(Anderson, *et al.* 2003, Busby *et al.*, 1997).

Imperial Valley/Alamo River Region, Imperial County

Pyrethroid insecticides are applied throughout the year in Imperial County, with over 21,000 pounds applied in 2002 (Figure 2). Throughout the county, more than 1,000 pounds of the six primary pyrethroid insecticide active ingredients were applied each month in eight out of twelve months during 2002.

Virtually all agricultural use of pyrethroid insecticides in Imperial County takes place within an approximately 650 square mile area within the Imperial Valley. From the southeastern shoreline of the Salton Sea, the high use region extends east approximately 20 miles and south to the US/Mexico border. In 2002, over 20,000 pounds of the six pyrethroids were applied to agricultural crops within this 650 square mile area.

Two distinct periods of relatively high pyrethroid use occur within the Imperial Valley region (Figure 6). The highest use occurs during the fall (October and November), with applications made primarily to vegetable crops. A second period of high use occurs in late winter (February and March), when applications are made primarily to alfalfa. These two high use periods together account for over 60% of the annual pyrethroid use in the region.

In 2002, over 5,000 pounds of pyrethroid active ingredients were applied in the Imperial Valley region in February and March. Nearly 80% of this amount was applied to alfalfa, with less than 10% applied to lettuce.

During October and November of 2002, over 7,500 pounds of the pyrethroids were applied in the Imperial Valley region. Less than 10% of this amount was applied to alfalfa. Approximately 50% was applied to lettuce; applications to lettuce, onions, sugarbeets, broccoli and cauliflower together accounted for 80% of this use. Over the five year period from 1998 through 2002, the average amount of pyrethroids applied during October and November was over 9000 pounds.

The peak period of organophosphate application in the region also occurs in the fall. During the period from September through November of 2002, over 40,000 pounds of diazinon and over 55,000 pounds of chlorpyrifos were applied in the Imperial Valley.

Agricultural drain water from the Imperial Valley drains into the Salton Sea, providing over 70% of the freshwater input to the Sea. The Salton Sea supports a National Wildlife Refuge and is a critical stop on the Pacific Flyway for migrating birds (California Regional Water Quality Control Board, Colorado River Basin, 2003).

Sacramento Valley/Feather River Region

Pyrethroid insecticides are applied throughout the Sacramento Valley (Butte, Colusa, Glenn, Sacramento, Solano, Sutter, Tehama, Yolo and Yuba counties) on a variety of crops, including fruits, nuts, vegetables and rice. Over 29,000 pounds were applied in 2002 throughout the entire valley (Figure 3). The primary pyrethroid use period occurs during May through August (Figure 7), with applications to peaches and rice accounting for about 80 percent of the total use during that period. The primary use period for chlorpyrifos, an organophosphate insecticide, also occurs during this period in the Sacramento Valley.

There are several areas within the Sacramento Valley where pyrethroids are applied in significant amounts. However, the Feather River region, a 350 square mile region from the town of Biggs west approximately 10 miles and south about 35 miles (Figure 3), is the region of heaviest pyrethroid use in the Sacramento Valley. This region includes portions of Sutter, Yuba and Butte counties. From May to August of 2002, approximately 6700 pounds of pyrethroid active ingredients were applied to agricultural crops (primarily peaches) in this region; the use during this period accounts for 80% of the annual pyrethroid use in the region. Similar amounts were applied in the region in 1998 - 2001.

Within the Feather River region, much of the area west of the Feather River drain to the Sutter Bypass, which makes up about 80% of the Sutter National Wildlife Refuge, and into the Sacramento River. Areas east of the river drain into the Feather River (Dileanis *et al.* 2003). The Feather River is a tributary of the Sacramento River, which drains to the Sacramento Delta and to San Francisco Bay.

Northern San Joaquin Valley/San Joaquin River Region

Pyrethroid insecticides are applied throughout the eight county region of the San Joaquin Valley (Fresno, Kern, Kings, Madera, Merced, San Joaquin, Stanislaus and Tulare counties) on a variety of crops, including nuts, fruits, corn, cotton and alfalfa. Over 110,000 pounds of pyrethroid active ingredients were applied in 2002 throughout the entire valley.

While pyrethroid use is significant throughout the entire valley, the greatest potential for movement of pyrethroids to surface water bodies occurs in several localized (geographically distinct) areas of high use within the northern San Joaquin Valley (Figure 4). These areas include parts of San Joaquin, Stanislaus and Merced counties, and western Madera and northwestern Fresno counties.

In this region, the primary pyrethroid use period generally occurs between May and August (Figure 8). In 2002, over 13,000 pounds of pyrethroids were applied in the northern San Joaquin Valley from May through August, with applications to almonds and pistachios accounting for over 50 percent of the total use during the period (2002 PUR).

The area encompasses the San Joaquin River, which drains to the delta and San Francisco Bay, and several major tributaries of the San Joaquin, including the Stanislaus, Tuolumne and Merced Rivers.

II. OBJECTIVE

The primary objective of the study is to provide a preliminary assessment of the extent of pyrethroid insecticide contamination of bed sediment and surface water in several agricultural regions of California with high pyrethroid use. The secondary objective is to acquire additional surface water monitoring data for organophosphate insecticides in those same regions. Ongoing collection of diazinon and chlorpyrifos surface water data is needed because these organophosphate insecticides have been placed into re-evaluation by DPR (DPR 2003a and 2004b).

Results will be used to aid in the development of priorities for future monitoring and/or mitigation efforts.

III. PERSONNEL

The study will be conducted by staff from the Environmental Monitoring Branch, Surface Water Protection Program, under the general direction of Kean S. Goh, PhD., Agricultural Program Supervisor IV. Key personnel are listed below:

Project Leader:	Keith Starner
Field Coordinator:	Kevin Kelley
Senior Scientist:	Frank Spurlock
Laboratory Liaison:	Carissa Ganapathy
Chemists:	California Department of Food and Agriculture, Center for Analytical Chemistry Staff Chemists

Questions concerning this monitoring project should be directed to Keith Starner at kstarner@cdpr.ca.gov or (916) 324-4167.

IV. STUDY PLAN

Each of the four regions (Salinas River Valley, Imperial Valley, Sacramento Valley/Feather River, and Northern San Joaquin Valley) will be sampled three times during the 12-month study.

Where possible, sampling within a given region will be timed such that two of the sampling events within that region take place during or immediately following periods of historically high pyrethroid use within that region. Additionally, one of the sampling events will be timed such that sampling takes place during a period of relatively low historical pyrethroid use within the region.

Within each region, four sites will be sampled at each sampling interval. Locations of individual sites within a region will be determined based on the historical pyrethroid use patterns, proximity to the water bodies of interest, and previous pyrethroid monitoring data, where available. Where possible, sampling sites will be selected at locations where water velocities are low and where suspended sediment will tend to settle out of the water column. Such sites can loosely be described as settling areas or "receiving" sites (Kozlowski, Watson et. al., 2003). Such sampling sites will be selected where possible in order to increase the likelihood of sampling recently deposited sediment. At each sampling site, bed sediment will be collected for pyrethroid analysis, along with whole water (water plus suspended sediment) for both pyrethroid and organophosphate analysis.

Site selection will follow the general guidelines in Standard Operating Procedure (SOP) FSWA002.00 (Bennett, 1997) where applicable.

Sampling will commence in July 2004 and continue through June 2005.

V. SAMPLING METHODS

At each sampling site, duplicate sediment samples will be collected into separate pint Mason jars for pyrethroid analysis. An additional sediment sample will be collected for total organic carbon (TOC) analysis. Only the top 1 cm of the sediment column will be collected. Where possible, a trowel will be used to collect the samples by gently scraping the top layer of the sediment column. If necessary, a core tube will be used, retaining only the top 1 cm from each core and taking care to minimize disturbance of the top sediment layer during the sampling process. Where possible, sediment samples will be collected from a subregion of the selected sampling site most likely to result in collection of recently deposited sediment. Factors to be considered when determining this subregion for sampling include identifying locations of obvious deposition, noting stream flow rate, and observing obstructions to flow rate and alterations in flow direction that may have resulted in recent sediment deposition.

Sediment samples will be transported on wet ice and transferred as soon as practicable to frozen storage at 0^{o} C until extraction for chemical analysis.

At each sampling site, two surface water grab samples, one for pyrethroid analysis and one for organophosphate analysis, will be collected directly into separate 1-liter amber glass bottles. An additional surface water grab sample will be collected for total suspended solids (TSS) analysis. Grab samples will be collected as close to center channel as possible using a grab pole consisting of a glass bottle at the end of an extendable pole, or other sampling equipment designed to collect a sample directly into a 1-liter glass bottle. Samples will not be transferred from the original sample bottles until analysis at the lab. Amber bottles will be sealed with Teflon-lined lids and samples will be transported and stored on wet ice or refrigerated at 4^{o}C until extraction for chemical analysis.

Dissolved oxygen, pH, specific conductivity, and water temperature will be measured *in situ* at each site during each sampling period. Flow data will be collected using a digital flow meter.

VI. CHEMICAL ANALYSIS

Chemical analysis will be performed by the California Department of Food and Agriculture's Center for Analytical Chemistry. Analytical method titles and reporting limits for this study are given in Table 3. Details of the chemical analysis methods will be provided in the final report. Quality control will be conducted in accordance with Standard Operating Procedure QAQC001.00 (Segawa, 1995). Ten percent of the total number of analyses will consist of field blanks and blind spikes, to be submitted to the laboratory with field samples.

All sediment samples and whole water samples collected for pyrethroid analysis will be analyzed for bifenthrin, cyfluthrin, cypermethrin, esfenvalerate, lambda-cyhalothrin, and permethrin. For pyrethroid water analyses, the whole sample, including any suspended sediment, will be extracted in the sample bottle (*in toto*) and the pyrethroid residues will be reported on a whole sample basis (water plus suspended sediment). Replicate whole water samples will be collected at each sampling event and analyzed to determine suspended sediment concentration. Whole water samples collected for organophosphate insecticide analysis will be analyzed for a suite of organophosphates, listed in Table 3.

VII. DATA ANALYSIS

Concentrations of pesticides in water will be reported as micrograms per liter (µg/L) or nanograms per liter (ng/L). Concentrations of pesticides in sediment will be reported as micrograms per kilogram (µg/kg).

Resulting data will be analyzed and reported as appropriate, potentially including the following:

Comparison of analytical concentrations to concurrent use data, toxicity data, and Water Quality Criteria (CDFG 2000);

Spatial analysis of the resulting data using Geographic Information System (GIS) software in order to identify correlations between observed pesticide concentrations and region-specific geographical features such as climate, soil type, cropping patterns and agricultural practices;

Estimation of dissolved and sorbed pyrethroid concentration in whole water pyrethroid samples (Spurlock, 2003).

VIII. TIMETABLE

Field Sampling:	July 2004 through June 2005
Chemical Analysis:	July 2004 through September 2005
Final Report:	April 2006

An interim memo containing summary analytical results may be issued approximately midway through the study.

IX. BUDGET

Primary Analysis		Cost (@ $300/sample)		
Pyrethroid screen, sediment	2 reps x 4 sites x 12 months =	96 samples	=	28,800
Pyrethroid screen, water	1 rep x 4 sites x 12 months =	48 samples	=	14,400
Organophosphate screen (incl. diazinon)	1 rep x 4 sites x 12 months =	48 samples	=	14,400
Quality Control				
Blind spikes		10 samples	=	3,000
Field blanks		10 samples	=	3,000
Total				**63,600**

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Table 1. Pyrethroid Physical and Toxicological Characteristics

Pesticide	**K_{OC}**	**Solubility** (mg/l)	**Half-life Soil** (days)	**Hydrolytic** (pH 7) **Half-life** (days)	**Toxicity** [A] **LC_{50}** *Daphnia Magna* (ppb)
Bifenthrin	237,000	$1.4e^{-5}$	96-425	Stable	1.6
Cyfluthrin	124,000	$2.3e^{-3}$	12-34	183	0.16
Cypermethrin	310,000	$4.0e^{-3}$	28-55	274	1.25
Esfenvalerate	215,000 [A]	$6.0e^{-3}$	39-94	Stable	0.24
lambda-cyhalothrin	326,000	$5.0e^{-3}$	43	Stable	0.23
Permethrin	277,000	$5.5e^{-3}$	40-197	Stable	0.075

Source: Laskowski, 2002 except as indicted.
A. DPR 2003b.

Table 2. Pyrethroid use in high use counties, by active ingredient (ai), 2002.

County	**Pounds ai applied, 2002**						**Total by County**
	Bifenthrin	**Cyfluthrin**	**Cypermethrin**	**Esfenvalerate**	**Lambda-cyhalothrin**	**Permethrin**	
Butte	36	6	0	1030	589	1451	**3112**
Fresno	1067	3541	1083	4165	1626	16918	**28400**
Imperial	272	4025	1395	2675	2242	10833	**21442**
Madera	172	355	6	798	342	12126	**13799**
Merced	1417	840	33	1368	868	5362	**9888**
Monterey	726	0	1352	3027	2596	25411	**33112**
San Joaquin	301	127	0	2956	441	5728	**9553**
Stanislaus	1293	48	159	2892	731	5450	**10573**
Sutter	719	0	0	1503	905	3595	**6722**
Yuba	12	0	0	641	244	1201	**2098**
Total by ai	**6015**	**8942**	**4028**	**21055**	**10584**	**88075**	**138699**

Source: DPR 2004a.

Table 3: California Department of Food and Agriculture, Center for Analytical Chemistry Analytical Method Details.

Organophosphate Pesticides in Surface Water by GC Method: GC/FPD [1]

Compound	***Method Detection Limit (μg/L)***	***Reporting Limit (μg/L)***
Azinphos methyl	0.0099	0.05
Chlorpyrifos	see note 1	$\leq$ 0.014 [1]
Diazinon	0.011	0.04
DDVP (dichlorvos)	0.0098	0.05
Dimethoate	0.0079	0.04
Disulfoton	0.0093	0.04
Ethoprop	0.0098	0.05
Fenamiphos	0.0125	0.05
Fonofos	0.008	0.04
Malathion	0.0117	0.04
Methidathion	0.0111	0.05
Methyl Parathion	0.008	0.03
Thimet (Phorate)	0.0083	0.05
Profenofos	0.0114	0.05
Tribufos	0.0142	0.05

1. Analytical limits for chlorpyrifos not yet available pending testing of new analytical method with matrices similar to expected environmental samples (actual agricultural drain water and, when available, storm run off water).

Pyrethroid Pesticides in Surface Water; Method: GC/MSD [2]

Compound	***Method Detection Limit (ng/L)***	***Reporting Limit (ng/L)***
Bifenthrin	2.16	5.0
Cyfluthrin	55.5	80.0
Cypermethrin	56.6	80.0
Esfenvalerate	22.5	50.0
Lambda-Cyhalothrin	7.76	20.0
Permethrin	16.9	50.0

Pyrethroid Pesticides in Sediment; Method: GC/EC, confirmation by GC/MSD

Compound	***Method Detection Limit (μg/g)***	***Reporting Limit (μg/g)***
Bifenthrin	0.007	0.01
Cyfluthrin	0.008	0.01
Cypermethrin	0.008	0.01
Esfenvalerate	0.008	0.01
Lambda-Cyhalothrin	0.009	0.01
Permethrin	0.006	0.01

2. Method development to lower the analytical limits for pyrethroids in water and sediment is scheduled to begin in fall 2004.

Figure 1. Pyrethroid Use, Monterey County, 2002.

Note: Figures 1-4 depict combined agricultural use data for six pyrethroid active ingredients (permethrin, esfenvalerate, cyfluthrin, lambda-cyhalothrin, bifenthrin and cypermethrin), 2002.

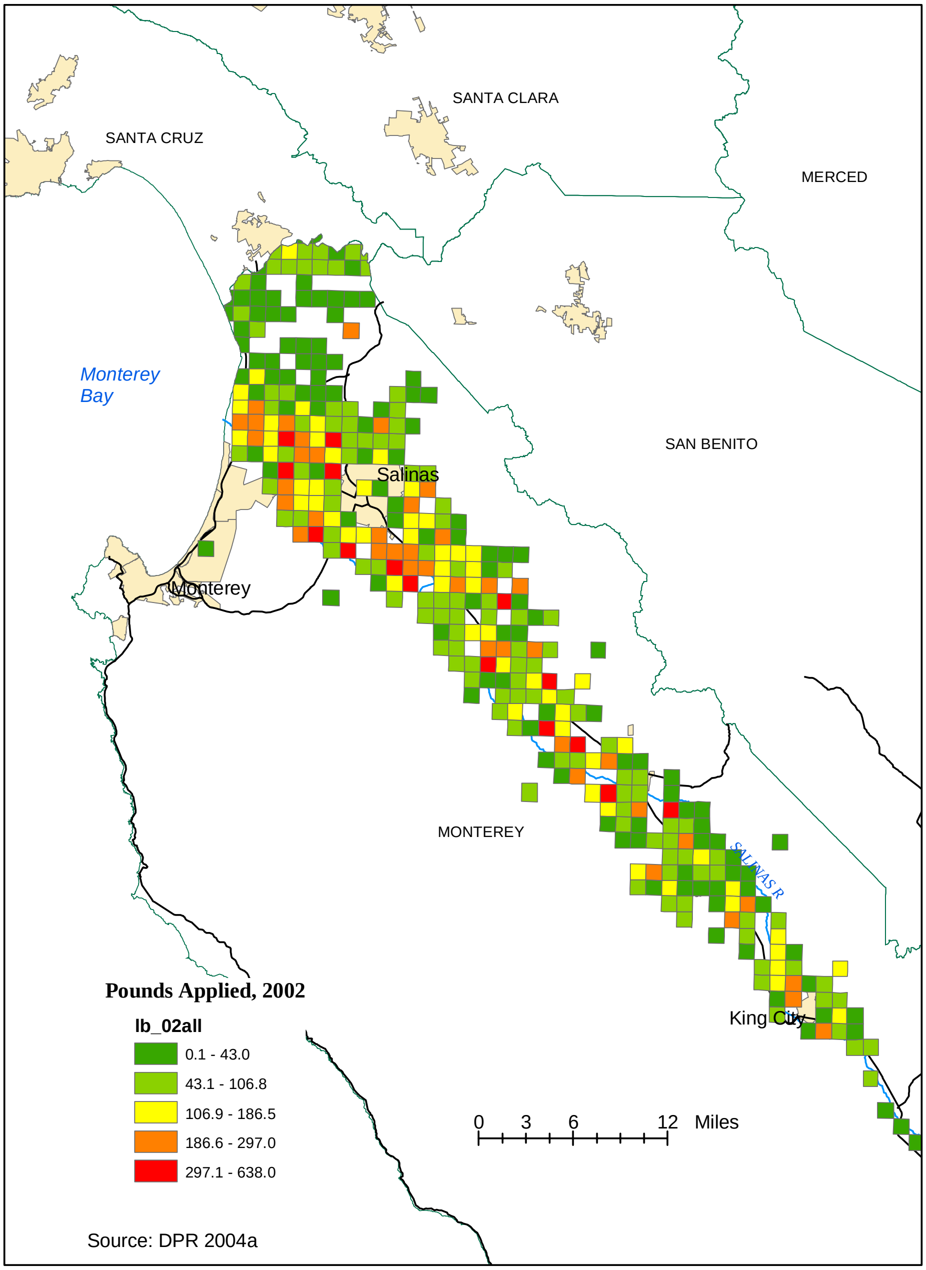


Figure 2. Pyrethroid Use, Imperial Valley, 2002.

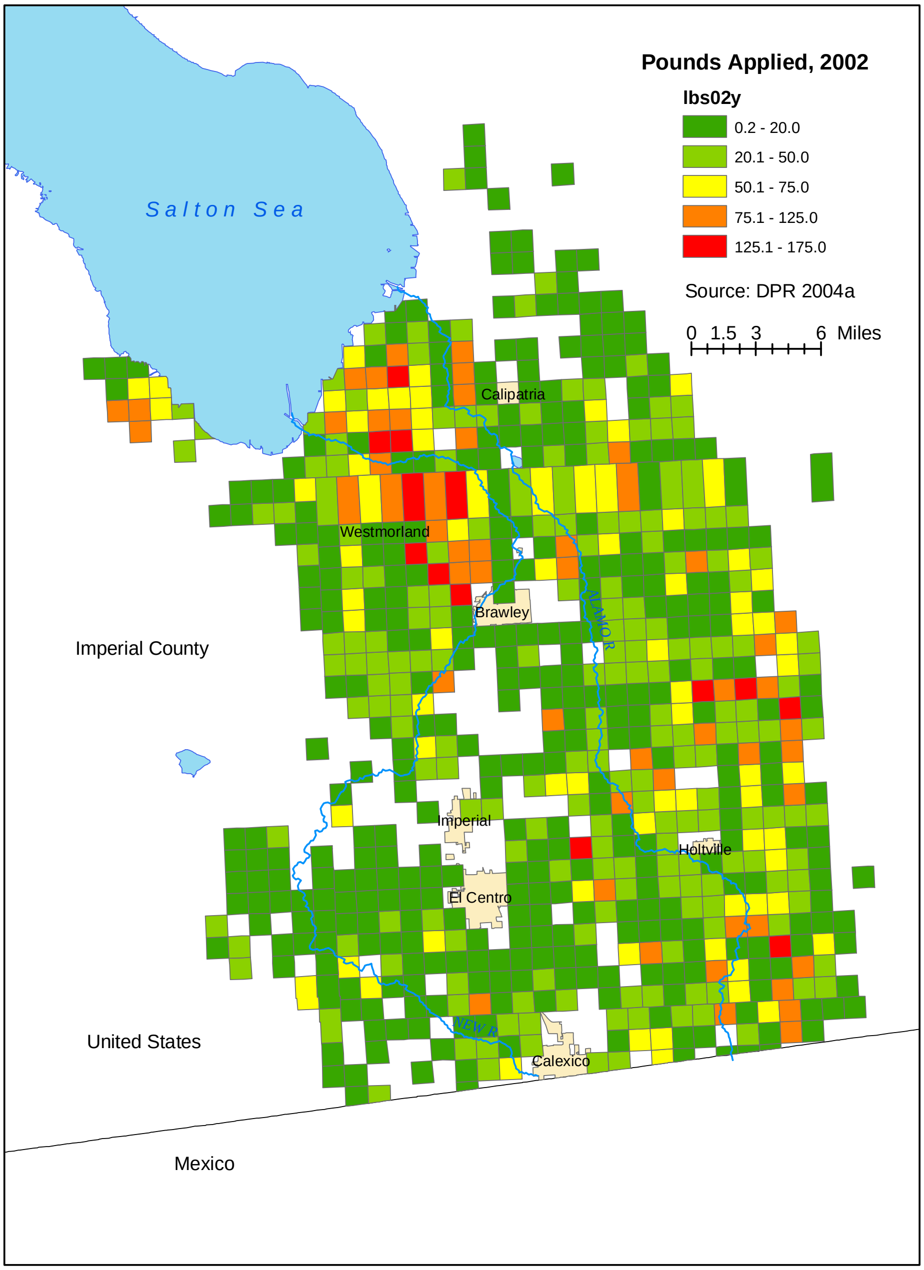


Figure 3. Pyrethroid Use, Sacramento Valley, 2002. Feather River Region outlined in black.

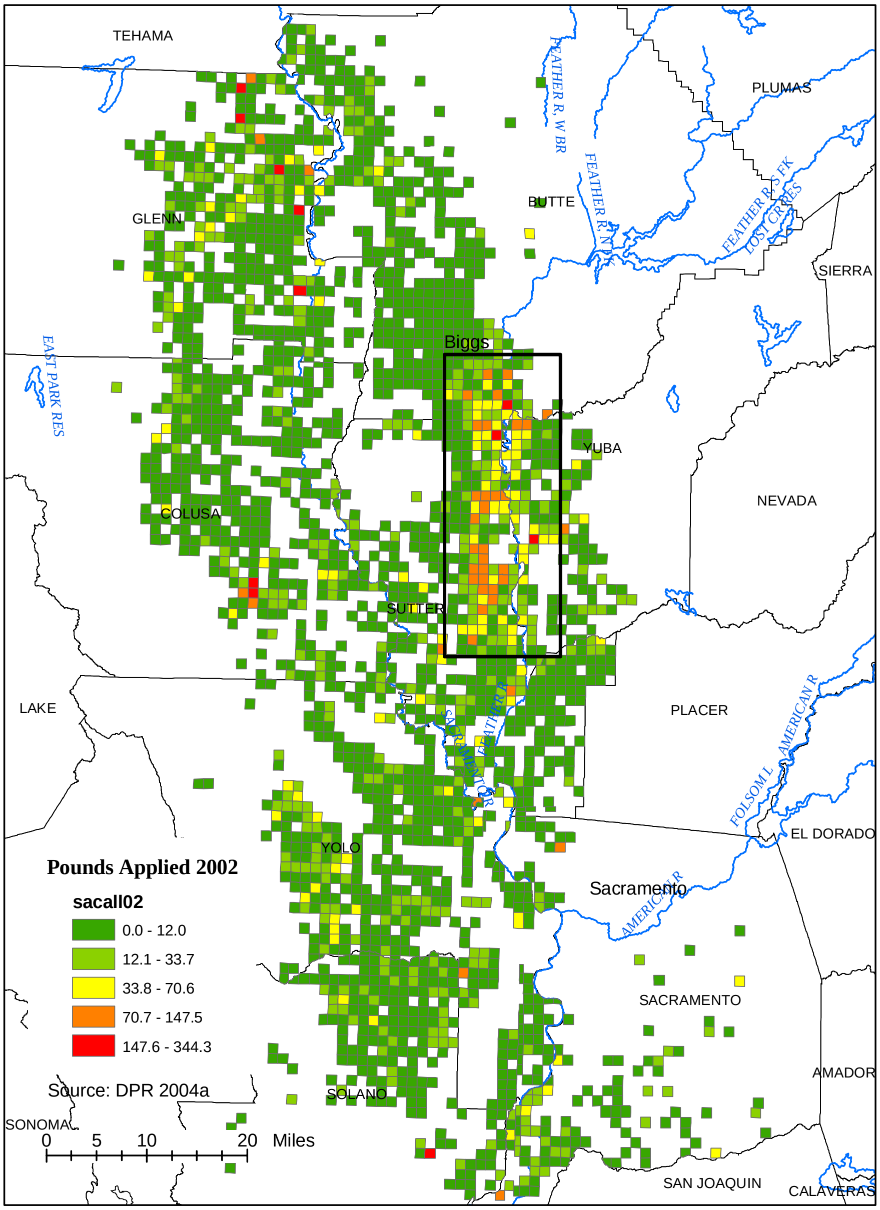


Figure 4. Pyrethroid Use, Northern San Joaquin Valley, 2002.

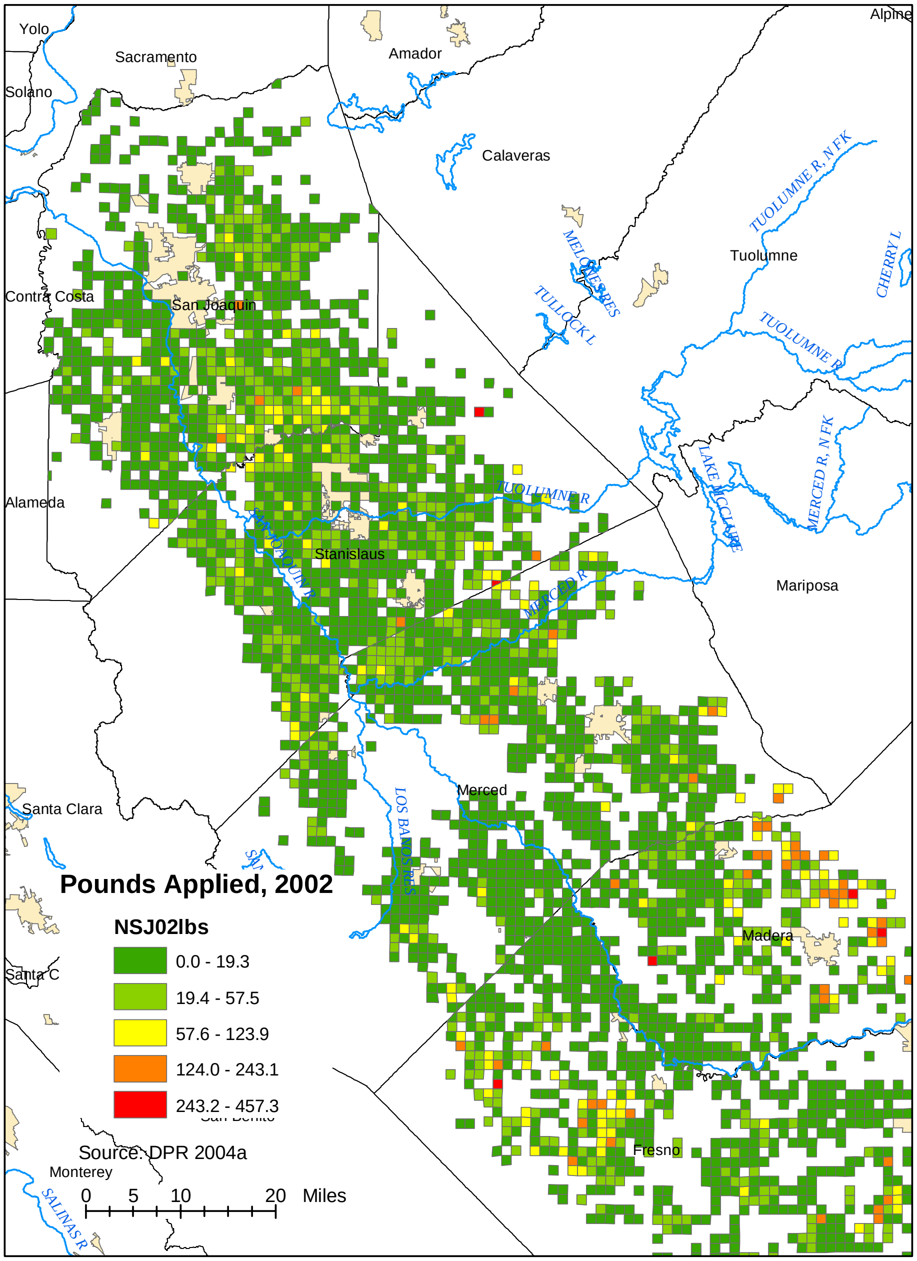
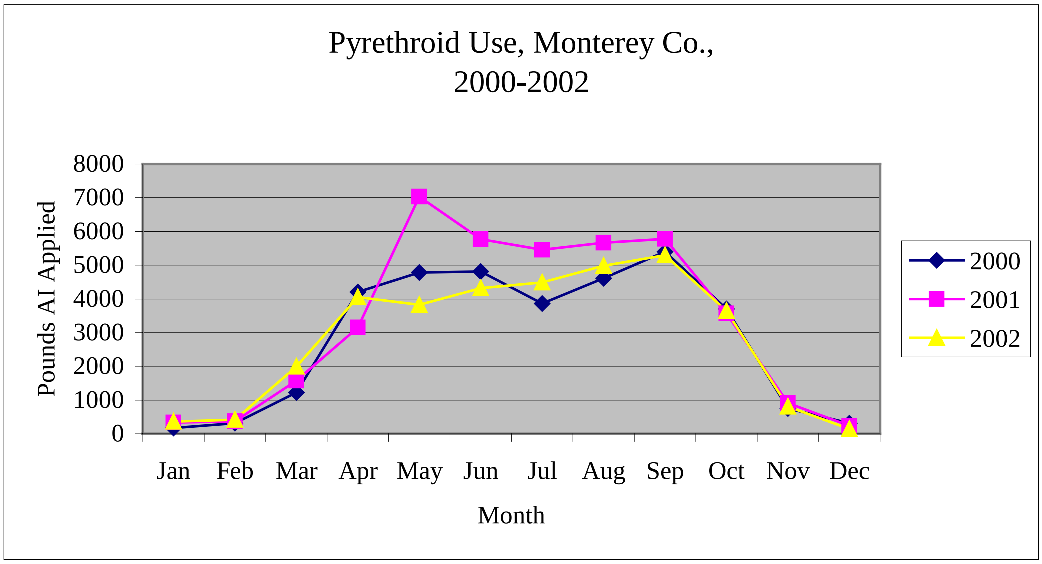


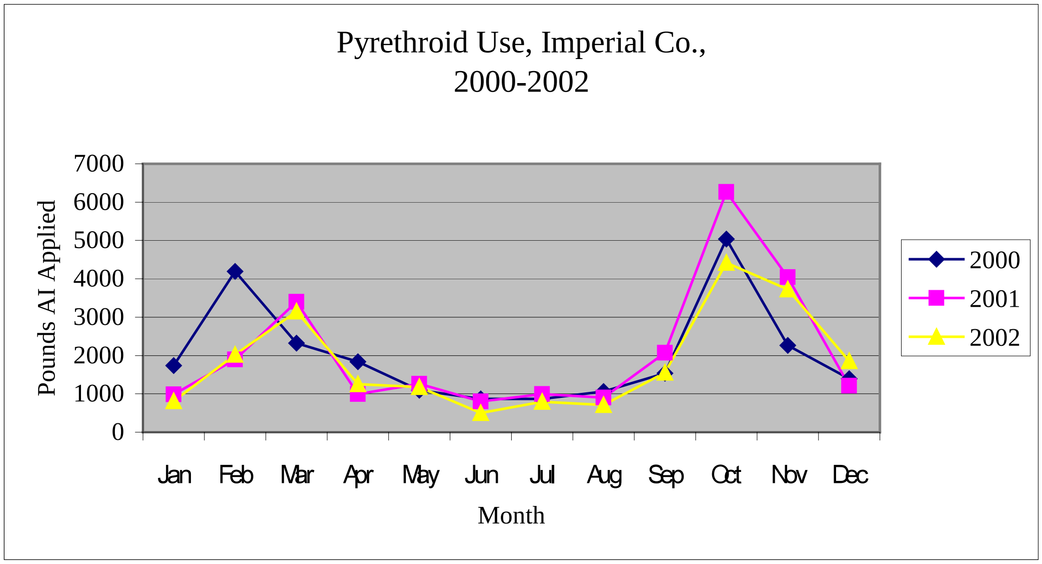
Figure 5. Monterey County Monthly Pyrethroid Use Data, 2000-2002.

All monthly use data are combined pounds of active ingredient for the six primary pyrethroid insecticides: permethrin, esfenvalerate, cyfluthrin, lambda-cyhalothrin, bifenthrin and cypermethrin. (DPR 2004a).



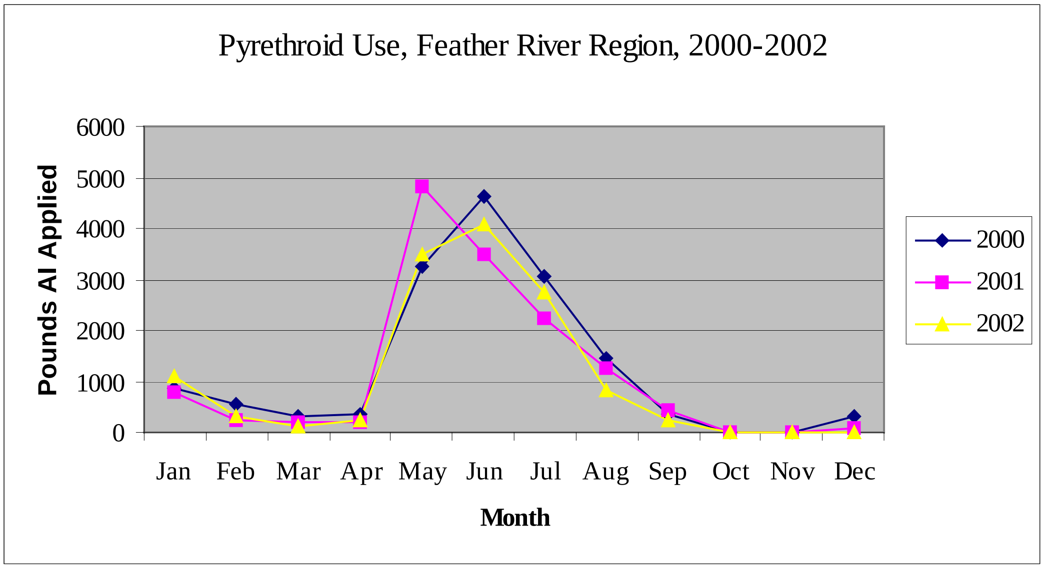
Monterey County monthly use data represents a use area of approximately 350 miles.

Figure 6. Imperial County Monthly Pyrethroid Use Data, 2000-2002.



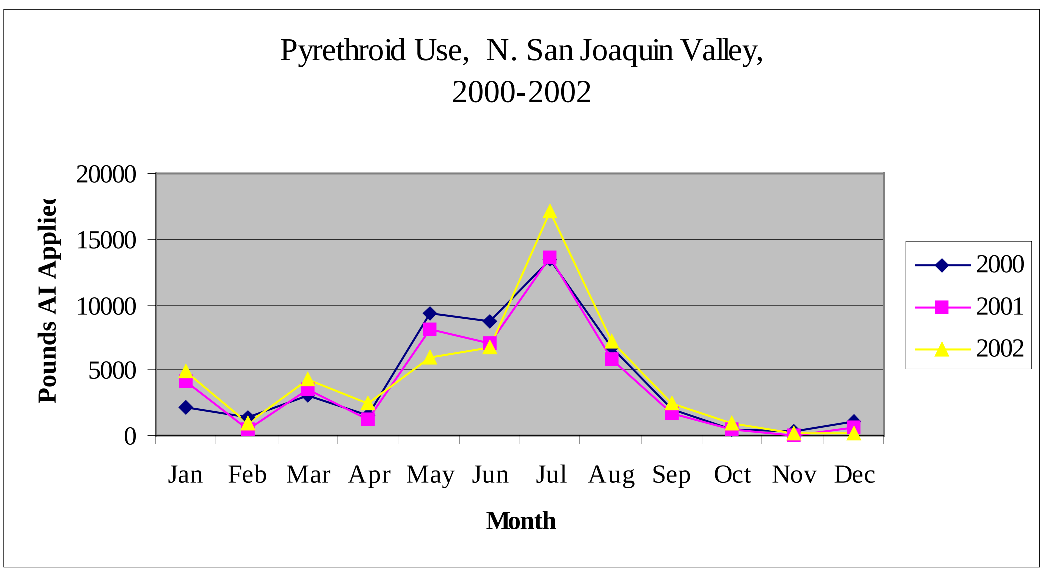
Imperial County monthly use data represents a use area of approximately 650 miles.

Figure 7. Feather River Region Monthly Pyrethroid Use Data, 2000-2002.



Monthly use data shown here represents all use in Sutter, Yuba and Butte counties. The Feather River Region is a subset of this area, with similar application timing (see Figure 3).

Figure 8. Northern San Joaquin Valley Monthly Pyrethroid Use Data, 2000-2002.



Northern San Joaquin Valley monthly use data represents a use area of approximately 6000 square miles.