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STUDY 270 (2013-14): URBAN PESTICIDE MONITORING IN SOUTHERN CALIFORNIA DURING FISCAL YEAR 2013-2014

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1.0 INTRODUCTION

Urban runoff is an important source of pesticide loading into surrounding waterways, justifying monitoring efforts to characterize pesticide composition in surface waters receiving urban inputs. In California, the Department of Pesticide Regulation (CDPR) receives pesticide use reports for urban applications by licensed applicators. Yearly, applicators generally report over 12 million pounds active ingredient (a.i.) of urban pesticide use in California (CDPR, 2009a). Reported use is categorized into agricultural and non-agricultural use. Agricultural use includes both production and non-production agricultural (i.e. golf courses, rights-of way, parks, watershed) applications. Non-agricultural use includes applications by a licensed pesticide applicator for residential, industrial, institutional, structural, or vector control purposes (CDPR, 2010a). However, urban pesticide use by individual homeowners is not reported, so that total use is greater than reported use. It has been estimated that urban pesticide use accounts for over 70% of the total pesticide use in California (UP3 Project, 2007). Appendix 1 shows the 2009 reported use of selected pesticides for non-agricultural purposes within Orange County, CA (CDPR 2010b). There were a total of 70,386 lbs of selected a.i. used for non-agricultural use in 2009, with pyrethroids making up 63% of total usage.

With this high volume of urban pesticide use there is a potential for pesticide runoff into urban creeks and rivers via storm drains. Numerous urban creeks are listed on the 2006 Federal Clean Water Act Section 303(d) list due to the historical presence of organophosphorus (OP) pesticides (Cal/EPA, 2009), partially attributable to their presence in urban runoff. While urban uses of OPs have been sharply curtailed due to Federal regulatory actions, recent monitoring has continued to identify the presence of OPs in some samples (Oki and Haver, 2009). Additionally, recent monitoring has shown that urban waterways are frequently contaminated with pyrethroids, OPs, and fipronil. Many of the detected pesticides are at concentrations that exceed the acute toxicity to sensitive aquatic organisms (Oki and Haver, 2009; Weston *et al.*, 2005; Weston *et al.*, 2009). In 2008 CDPR initiated a statewide urban monitoring project to more fully characterize the presence of pesticides in urban waterways (CDPR, 2009b). During the 2008-2009 monitoring events, CDPR detected carbaryl, diuron, simazine, triclopyr, dicamba, 2,4-D, and MCPA in addition to those mentioned above.

Study 270, which is a continuation of monitoring efforts of Studies 249 and 265, will provide data used to evaluate urban pesticide water quality trends. New surface water regulations were implemented in California in July, 2013. Long term monitoring at selected urban sites will help determine the effectiveness of these regulations on pesticide presence in urban waterways (CDRP, 2009c). This project will continue to monitor storm drains and urban waterways at selected monitoring sites from CDPR's 2008 study as well as at monitoring stations established by the University of California (Oki and Haver, 2009). This long-term monitoring may be used to track the performance of local mitigation measures or public outreach programs. Modifications from the FY 12-13 sampling plan is presented in section 4.2.

2.0 OBJECTIVE

The overall goal of this project is to assess urban pesticide use and water and sediment quality in drainages and receiving waters within two typical southern California urbanized areas during stormwater runoff and dry season conditions. Specific objectives include:

1) Determine presence and concentrations of selected pesticides in urban runoff under dry season and stormwater conditions;
2) Evaluate the magnitude of measured concentrations relative to water quality or aquatic toxicity benchmarks;
3) Collect data that will be used to evaluate the effectiveness of surface water regulations through long term (multiple year) monitoring at selected sampling locations;
4) Observe the mitigation effects of a small constructed wetland on pesticide concentrations in receiving waters;
5) Observe the mitigation effects of a small water treatment facility receiving dry season flow;
6) Monitor deposition of pyrethroids bound to sediments within watershed.

3.0 PERSONNEL

The study will be conducted by staff from the CDPR's Environmental Monitoring Branch under the general direction of Kean Goh, Program Manager. Key personnel are listed below:

Project Leader: Robert Budd, Ph.D.
Field Coordinator: Xin Deng, Ph.D.
Senior Scientist: Frank Spurlock, Ph.D.
Laboratory Liaison: Sue Peoples
Analytical Chemistry: Center for Analytical Chemistry, Department of Food and Agriculture (CDFA)
Collaborator: Darren Haver, Ph.D., University of California at Davis, Center Director/Water Resources and Water Quality Advisor, South Coast Research and Extension Center, 7601 Irvine Blvd., Irvine, CA, 92618, Phone: (949) 653-1814, email: dlhaver@ucdavis.edu

Please direct questions regarding this study to Robert Budd, Environmental Scientist, at (916) 445-2505 or rbudd@cdpr.ca.gov.

4.0 STUDY PLAN

4.1 Monitoring Sites

Water quality monitoring will be conducted at 10 sites within Orange County, California (Table 1). Details of site descriptions are provided in Appendix 2. There are eight sampling locations within the Salt Creek watershed (Figure 1) and two within the Wood Creek watershed (Figure 2).

Automated sampling equipment has been installed at two sites within Salt Creek and two within Wood Creek by the University of California (Oki and Haver, 2009); we will evaluate these sites for potential long-term monitoring in collaboration with the University of California.

Surrounding drainage areas at both watersheds consist of single family dwellings, multiple family dwellings, light commercial buildings, parks, schools, and a golf course.

Table 1. Summary of urban pesticide monitoring locations in California.

Area	Stormdrain Outfall	Receiving Water	Total Sites
Salt Creek	5	3	8
Wood Creek	1	1	2
Total	6	4	10

4.2 Sampling

Water sampling. Samples will be collected during three dry season and two storm sampling events. Dry season sampling will occur between August - September, 2013 and May-June, 2014. We will conduct storm sampling with the first major storm (rain) event of the 2013-2014 season (average highest precipitation is December – March) and with a major storm in the winter or early spring of 2014 (Table 2).

CDPR staff will collect water samples for chemical analysis and for determining total suspended solids (TSS) and total organic carbon (TOC). During creek sampling, CDPR will collect samples from the center channel using an extendable pole directly into 1-L amber glass bottles. When collecting water samples from storm drains, samples will be collected by hand directly into 1-L bottles. Water samples may also be collected by automated samplers set up by the University of California (Oki and Haver, 2009). All bottles will be sealed with Teflon® lined lids following CDPR SOP FSWA002.00 (Bennett, 1997). Samples will be stored and transported on wet ice or refrigerated at 4°C until analyzed.

Within the Salt Creek watershed, intensive sampling will occur at SC3. This site has been monitored for several years with consistently high detection frequencies of pesticides. This site is automated with sampler and water quality sensors, allowing for continuous flow and water quality parameters to be recorded. This site will serve as a signal of pesticides in urban runoff and will be developed as a long term monitoring station. Downstream sites (SC5 and SC7) are considered receiving waters of several urban inputs and will serve to evaluate pesticide concentrations in the watershed as well as downstream transport.

Samples at these three sites will be monitored for chlorothalonil, fipronil and metabolites, imidacloprid, dinitroanilines, phenoxy and triazine herbicides, and organophosphate and pyrethroid insecticides. Sediment samples will also be collected for pyrethroid analysis at these sites during all events, dependent on available sediment deposition. The other sites within Salt Creek will follow the same sampling schedule with a modified list of analytes (Table 2). SC7A is located adjacent to SC7. During the dry season water is pumped from an intake at SC7 through a small ozone water treatment facility and returning through an outlet at SC7a.

Within the Wood Creek watershed, samples will be collected at the inlet (WC1) and outlet (WC2) of a small (~0.18 acres) constructed wetland designed to mitigate pollutants receiving urban runoff. Wetland efficacy of pesticide removal will be evaluated through comparisons between outlet and inlet concentrations. Water samples will be collected during five events, with sediment collected during two dry season events.

Table 2. Sampling schedule for urban pesticide monitoring in Southern California.

Event	Date	CT	FP+ Met	FP+ OP	IM	DN	PX	PY-6	TR	PY-Sed	Tox	Total
Dry	Aug, 2013	3	7	3	10	3	10	10	3	5	5	59
Rain	Oct-Nov, 2013	3	6	3	9	3	9	9	3	2	5	52
Rain	Feb-Mar, 2014	3	6	3	9	3	9	9	3	2	5	52
Dry	May, 2014	-	6	3	-	-		9	-	2	-	20
Dry	June, 2014	3	7	3	10	3	10	10	3	5	5	59
	Total	12	32	15	38	12	38	47	12	16	20	242

CT=chlorothalonil, FP+Met=fipronil+metabolites, OP=organophosphates, IM=imidacloprid, DN=dinitroanaline, PX=phenoxy, PY=pyrethroid, TR=triazine,Sed=sediment,Tox=toxicity

Sediment sampling. Where applicable, sediment samples will be collected in 1 quart glass Mason Jars using passive sediment collection samplers (Budd, 2009) and analyzed for pyrethroids. Otherwise, enough sediment will be collected using stainless steel scoops from the top of the bed layer, biasing for fine sediments where possible.

Toxicity sampling. Water samples will be collected at a subset of sampling sites for toxicity analysis during four events of FY 13-14. Grab samples will be collected in 1 L amber I-Chem certified 200 bottles and transported to the Aquatic Health Program at the University of California. Toxicity testing will measure percent survival of the amphipod *Hyalella azteca* in water (96-hr).

Sample Transport. CDPR staff will transport samples following the procedures outlined in CDPR SOP QAQC004.01 (Jones, 1999). A chain-of-custody record will be completed and accompany each sample.

Modifications from FY 12-13. The current sampling plan is an extension of sampling conducted during fiscal years 2010-2013. The details of the previous sampling is described in the document titled Study 270: Urban pesticide monitoring in southern California, available at: http://www.cdpr.ca.gov/docs/emon/pubs/protocol/study270protocol.pdf. The sampling and analysis schedule is similar to that for FY 12-13, with a few notable modifications (Table 3). Modifications were based on previous monitoring data and a newly developed model designed to assist in prioritizing pesticides for monitoring surface waters in California (Luo et al., 2013). The model is based on current use patterns and aquatic toxicity benchmark data. The product is a relative prioritization score. Appendix 4 is an abbreviated list of pesticides from the most current prioritization of urban pesticides in Orange County, California for the years 2009 – 2011 (Budd et al., 2013). Scores below 6 are considered low priority due to their very low use and toxicity and are not shown. The list provides guidance to EM staff on pesticides to focus attention, however the decision to monitor for a pesticide is influenced by additional factors. For instance, metals (i.e. copper) have confounding factors such as variable background concentrations. Physiochemical properties play a significant role in chemical behavior. Pesticides such as diquat and paraquat dibromide are extremely hydrophobic, typically bound in a non-bioavailable form, so they are considered low priority for monitoring in surface waters. Laboratory analysis of chiral compounds typically report the total or partial mixtures and not individual isomers, even though they might have products on the market refined for the most active isomer (i.e. beta-cyfluthrin, gamma-cyhalothrin, S-cypermethrin). Therefore, even though a specific isomer is not being monitored, their presence in surface waters would be indicated by the associated mixture.

Table 3. Modifications from sampling plan for fiscal year 2012-2013

Change from FY 12-13	Justification
Adding dinitroanaline analytical suite to SC3, SC5, SC7	Prodiamine [16], oryzalin [9], and trifluralin [8] identified as high priority pesticides[a]
Reduce collection of OP to SC3, SC5, SC7	Low detection rates, limited use
Increase toxicity testing to 4 events at SC3, SC5, SC7, WC1, WC2	Toxicity data will allow for a more complete evaluation of water quality and mitigation effects
Third dry event added	Additional data point to evaluate effectiveness of surface water regulations (Objective 3)
One sampling event at new site in San Diego region (NSDS), location TBD	Scarcity of pesticide monitoring data in surface waters from area

[a] Budd et. al, 2013

4.3 Field Measurements

Physiochemical properties of water column will be determined using a YSI 6920 V2-2 multiparmeter Sonde according to the methods describe by Doo and He (2008). At each site, water parameters measured *in situ* will include pH, temperature, conductivity, turbidity, salinity, total dissolved solids, and dissolved oxygen.

Stormdrain discharge or stream flow rates will be measured to characterize the flow regime and to estimate the total loading of target pesticides. Flow will be calculated using a Global portable velocity flow probe (Goehring, 2008), or estimated utilizing a float or fill-bucket method.

4.4 Quality Assurance/Quality Control

Quality Assurance/Quality Control (QA/QC) will be conducted in accordance with Standard Operating Procedure QAQC001.00 (Segawa, 1995). Ten percent of the total number of samples will be submitted as field blanks, blind spikes, or field duplicates. In addition, QA/QC procedures developed by US EPA (2002) and for the Surface Water Ambient Monitoring Program (SWAMP) by California's State Water Resources Control Board (SWRCB) (Puckett, 2002) will be consulted where applicable.

5.0 LABORATORY ANALYSIS

The Center for Analytical Chemistry, California Department of Food and Agriculture, Sacramento, CA (CDFA) will conduct the pesticide analysis in water samples for the study. They will analyze seven different analyte groups which will include up to 27 chemical compounds for analysis (Table 4, Appendix 3).

Sediment samples will be sent to the California Department of Fish and Wildlife, Sacramento, CA (CDFW) for pesticide analysis. Sediment samples will be analyzed for pyrethroids pesticides (Table 4).

CDPR will analyze TSS in the water samples and will analyze TOC in both water samples and sediment samples. TSS samples will be analyzed following US EPA method 160.2 (US EPA, 1971) and as described in Kelley and Starner in CDPR Study Memo 219 (2004). TOC will be analyzed with a TOC-V CSH/CNS analyzer (Shimadzu Corporation, Kyoto, Japan).

Table 4. Chemical analysis of pesticides in the Southern California urban monitoring study.

Analyte Group	Media	Analytical Method	Method Detection Limit ($\mu g\ L^{-1}$)	Reporting Limit ($\mu g\ L^{-1}$)
Chlorothalonil	Water[a]	LC-MS/MS	0.0348	0.05
Fipronil & Degradates	Water	GC-MSD (SIM)	0.003 – 0.005	0.05
Imidacloprid	Water	GC-MS	0.01	0.05
Organophosphorus Insecticides	Water	GC-FPD	0.008 – 0.0142	0.05
	Water	GC-MS	0.0012 – 0.0079	0.01
Pendimethalin	Water	LC-MS/MS	0.012	0.05
Phenoxy Herbicides	Water	GC-MS	0.064	0.1
Pyrethroid Insecticides	Water	GC-ECD	1.09 – 7.68 (ng L^{-1})	5 – 15 (ng L^{-1})
	Sediment	GC-ECD	-	0.02 – 0.2 (ng g^{-1})

[a]Water samples analyzed by California Department of Food and Agriculture, sediment samples analyzed at the California Department of Fish and Game laboratory.

6.0 DATA ANALYSIS

All data generated by this project will be entered to a central database that holds all data including weather and field information, field measurements, and laboratory analytical data. We will use various nonparametric and parametric statistical methods to analyze the data. The data collected from this project may be used to develop or calibrate an urban pesticide runoff model.

TIMELINE

Field Sampling:	July 2013 – June 2014
Chemical Analysis:	July 2013 – October 2014
Data Entry:	March 2015 – June 2015

8.0 LABORATORY BUDGET

The estimated total cost forchemical analyses is $138,390.

Site Location	Analytical Suite	# Sites	Storm Samples	Dry Season Samples	Cost/ Sample	Cost
SC3, SC5, SC7	Chlorothalonil	3	2	2	660	7920
	Fipronil + OP (short)	3	2	3	840	15120
	Imidacloprid	3	2	2	600	7200
	Dinitroanalines	3	2	2	960	11520
	Phenoxy Herbicides	3	2	2	690	8280
	Pyrethroids-6	3	2	3	600	10800
	Triazines	3	2	2	450	7200
SC1, SC2, SC4, SC6, WC1, WC2	Fipronil + Metab.	6	2	3	600	14400
	Imidacloprid	6	2	2	600	9600
	Phenoxy Herbicides	6	2	2	690	11040
	Pyrethroids-6	6	2	3	600	14400
SC7a	Fipronil + Metab	1	-	2	600	1680
	Imidacloprid	1	-	2	600	1200
	Phenoxy Herbicides	1	-	2	690	1380
	Pyrethroids-6	1	-	2	600	1200
NSDS	Fipronil + OP (Short)	1	-	1	840	840
	Imidacloprid	1	-	1	600	600
	Phenoxy Herbicides	1	-	1	690	690
	Pyrethroids-6	1	-	1	600	600
					Total	**$136,590**

OP = organophosphate, Metab.=Metabolites

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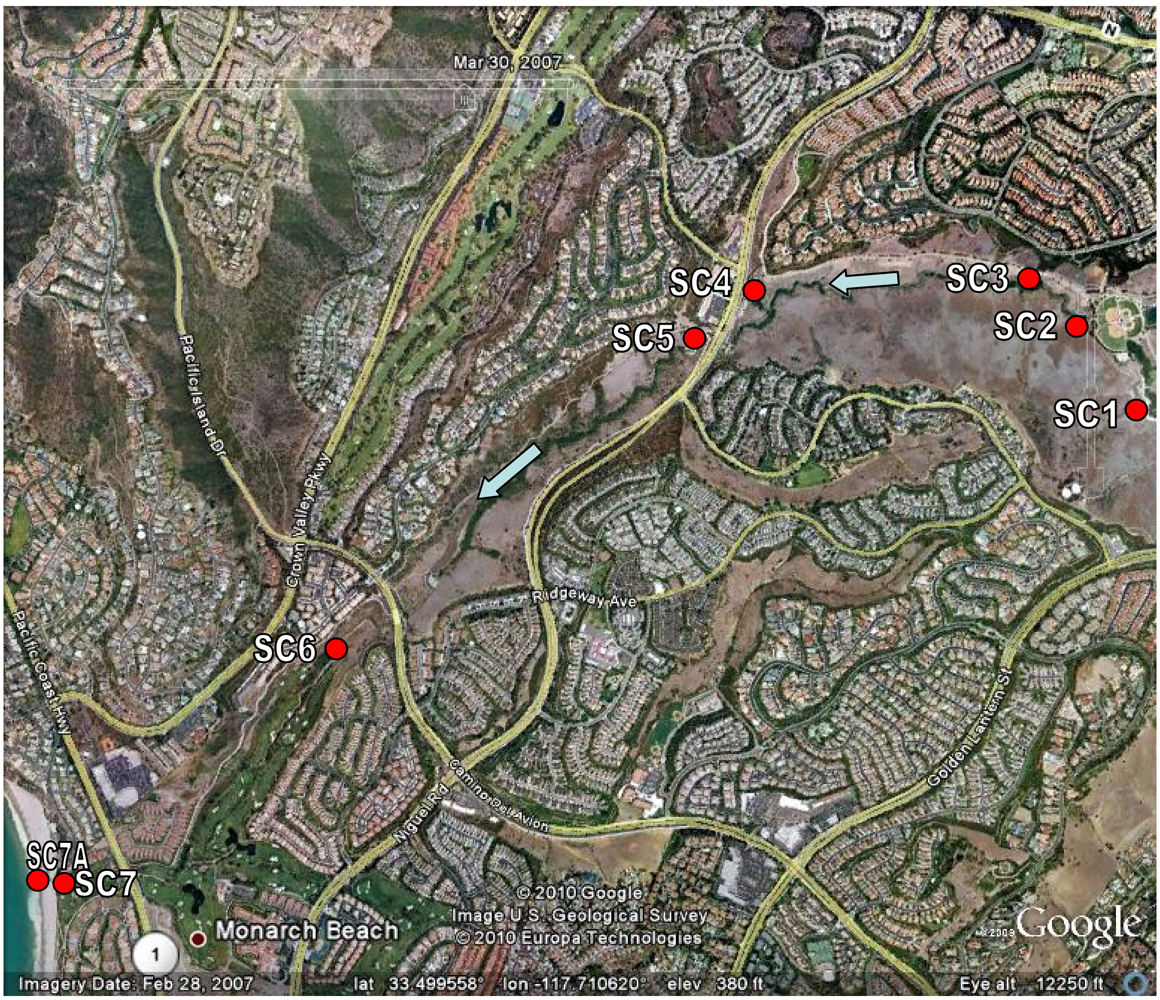

Figure 1. Sampling locations within Salt Creek watershed, Orange County, CA

Figure 2. Sampling locations within Wood Creek watershed, Orange County, CA

Appendix 1: Non-agricultural pesticide usage (lbs) in Orange County, CA

Analyte	2009 Use	2005-2009 Total	2005-2009 Average
Carbamates			
Carbaryl	49	559	112
Chloronitrils			
Chlorothalonil	14,726	69,829	13,966
Dinitoanalines			
Pendamethalin	1,779	7,185	1,437
Fipronil + Degradates			
Fipronil	3,968	38,842	7,768
Neonicotinoids			
Imidacloprid	1,000	41,283	8,257
Organophosphates			
Chlorpyrifos	263	6,312	1,262
Diazinon	15	565	113
Dimethoate	18	77	15
Malathion	1,134	8,755	1,751
Total Organophosphates		15,709	
Phenoxy			
2,4-D	7	179	36
Dicamba	132	727	145
Triclopyr	2,891	26,679	5,336
Total Phenoxy		27,585	
Pyrethroids			
Bifenthrin	3,489	39,729	7,946
Cyfluthrin	1,508	7,663	1,533
Cypermethrin	2,673	16,386	3,277
Deltamethrin	317	2,219	444
Esfenvalerate	28	108	22
Fenpropathrin		0	0
λ-Cyhalothrin	490	5,255	1,051
Permethrin	35,801	203,636	40,727
Resmethrin	98	346	69
Total Pyrethroids		275,341	
Triazines			
Bromacil	597	2,980	596
Diuron	4,979	22,028	4,406
Hexazinone	920	2,036	407
Simazine	91	6,304	1,261
Total Triazines		33,348	

Appendix 2. Detailed sampling site information

Watershed	Site ID	Northing	Easting	Site type
Salt Creek	SC-1	33 30 32.92	117 41 26.53	Stormdrain
Salt Creek	SC-2	33 30 40.57	117 41 40.67	Stormdrain
Salt Creek	SC-3	33 30 43.02	117 41 49.55	Stormdrain
Salt Creek	SC-4	33 30 31.00	117 42 26.34	Stormdrain
Salt Creek	SC-5	33 30 20.23	117 42 30.87	Receiving water
Salt Creek	SC-6	33 29 31.91	117 43 02.68	Stormdrain
Salt Creek	SC-7	33 28 53.97	117 43 26.55	Receiving water
Salt Creek	SC-7A	33 28 54.12	117 43 27.37	Receiving water
Wood Creek	WC-1	33 34.56.56	117 44 43.02	Stormdrain
Wood Creek	WC-2	33 34 53.70	117 44 44.65	Receiving water

Appendix 3. Active ingredients within analytical chemical suites

Chloronitriles	**Neonicotinoids**	**Pyrethroids**
Chlorothalonil	Imidacloprid	Bifenthrin
		Cyfluthrin
Dinitroanalines	**Organophosphates**	Cypermethrin
Benfluralin	Chlorpyrifos	Fenvalerate/esfenvalerate
Ethalfluralin	Diazinon	λ-cyhalothrin/epimer
Oryzalin	Malathion	cis-Permethrin
Oxyfluorfen		trans-Permethrin
Pendimethalin		
Prodiamine		
Trifluralin		
Fipronil + Metabolites	**Phenoxy Suite**	**Triazine Herbicides**
Desulfinyl fipronil	2,4-D	Bromacil
Desulfinyl fipronil amide	Dicamba	Diuron
Fipronil	MCPA	Hexazinone
Fipronil amide	Triclopyr	Prometon
Fipronil sulfide		Norflurazon
Fipronil sulfone		Simazine

Appendix 4. Priority model pesticides based on urban usage in Orange County, California (2009-2011).

Pesticide	Use [Score]	Benchmark [Score]	Final score
Permethrin	41,842 [5]	0.01 [7]	35
Bifenthrin	12,394 [5]	0.075 [6]	30
lambda-Cyhalothrin	2,006 [4]	0.0035 [7]	28
Cyfluthrin	2,138 [4]	0.0125 [6]	24
beta-Cyfluthrin	2,010 [4]	0.034 [6]	24
Chlorothalonil	15,749 [5]	1.8 [4]	20
Fipronil	4,790 [4]	0.11 [5]	20
Copper	2,393 [4]	2.05 [4]	16
Prodiamine	2,078 [4]	3 [4]	16
Cypermethrin	1,742 [3]	0.195 [5]	15
Malathion	939 [3]	0.3 [5]	15
Diquat dibromide	637 [3]	0.75 [5]	15
Mancozeb	7,164 [4]	47 [3]	12
Triclopyr, butoxyethyl ester	3,822 [4]	70 [3]	12
Pendimethalin	1,583 [3]	5.2 [4]	12
Diuron	1,324 [3]	2.4 [4]	12
Chlorfenapyr	960 [3]	2.915 [4]	12
Deltamethrin	517 [2]	0.055 [6]	12
Chlorpyrifos	83 [2]	0.05 [6]	12
2,4-D, 2-ethylhexyl ester	1,908 [3]	66 [3]	9
Oryzalin	1,672 [3]	15.4 [3]	9
Imidacloprid	1,571 [3]	35 [3]	9
Iprodione	1,543 [3]	50 [3]	9
Propiconazole	968 [3]	21 [3]	9
PCNB	643 [3]	50 [3]	9
Dichlobenil	616 [3]	30 [3]	9
Thiophanate-methyl	3,404 [4]	930 [2]	8
Oxadiazon	540 [2]	5.2 [4]	8
Pyraclostrobin	160 [2]	1.5 [4]	8
Bromacil	106 [2]	6.8 [4]	8
Trifluralin	87 [2]	7.52 [4]	8
gamma-Cyhalothrin	0 [1]	0.00024 [8]	8
Diflubenzuron	0 [1]	0.0014 [7]	7
(s)-Cypermethrin	0 [1]	0.0018 [7]	7
Acephate	1,066 [3]	550 [2]	6
2,4-D	572 [2]	13.1 [3]	6
Dithiopyr	559 [2]	20 [3]	6
Hydroprene	278 [2]	65 [3]	6
MCPA, 2-ethyl hexyl ester	220 [2]	20 [3]	6
Azoxystrobin	201 [2]	49 [3]	6
MCPP-p, dimethylamine salt	198 [2]	14 [3]	6
Pyriproxyfen	181 [2]	56 [3]	6
Fludioxonil	176 [2]	70 [3]	6
Pyrethrins	160 [2]	12.5 [3]	6
Dicamba	138 [2]	61 [3]	6
Imazapyr, isopropylamine salt	103 [2]	24 [3]	6
Thiamethoxam	88 [2]	17.5 [3]	6

Pesticide	Use [Score]	Benchmark [Score]	Final score
Esfenvalerate	47 [2]	0.025 [6]	6
DDVP	7 [1]	0.035 [6]	6
Abamectin	6 [1]	0.05 [6]	6
Chlorsulfuron	5 [1]	0.055 [6]	6
Tralomethrin	1 [1]	0.0195 [6]	6

Yellow = in current monitoring plan, green = previously monitored, blue= monitored as part of isomer mixture.