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Study 290. Surface Water Monitoring for Pesticides in Agricultural Areas of California, 2014

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March 11, 2014

I. INTRODUCTION

Surface water monitoring for pesticides in agricultural areas of California is one of the California Department of Pesticide Regulation's (CDPR's) key environmental monitoring activities. This project was initiated in 2008 with a long term goal of collecting data to better assess potential impacts of pesticides in agricultural runoff on California aquatic environments. Project findings help guide CDPR in the development and implementation of regulatory and non-regulatory mitigation activities. In the past six years, the project has identified geographic areas with heavy pesticide uses via the Pesticide Use Report (PUR) database and selected sites adjacent to agricultural fields with high runoff potential for long term monitoring efforts. The Salinas, Santa Maria and Imperial valleys have previously been designated as high priority areas for long-term surface water monitoring due to high pesticide uses (Starner 2010, 2013). This study is a continuation of the agricultural monitoring project following the same monitoring strategies that were established in previous years in selecting monitoring sites and active ingredient (AI) candidates.

II. OBJECTIVE

The goal of the project is to provide data for a long-term assessment of surface water pesticide contamination in agricultural areas of California. Results will provide useful data on the environmental fate and transport of current-use pesticides under a variety of conditions for use in the development of management responses. Objectives of the project are as follows:

- 1) Identify runoff sampling sites in counties of high pesticide uses;
- 2) Annually prioritize monitoring AI candidates;
- 3) Measure chemical occurrences and concentrations of highly prioritized pesticides in runoff samples;
- 4) Characterize pesticide compositions in agricultural waterways;
- 5) Analyze chemistry data to evaluate potential impacts on aquatic environments.

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, Environmental Program Manager (Supervisor) I. Key personnel are listed below:

Project Leader:	Xin Deng, PhD
Field Coordinator:	Kevin Kelley
Laboratory Liaison:	Sue Peoples
Chemists:	California Department of Food and Agriculture, Center for Analytical Chemistry Staff Chemists

Questions concerning this monitoring project should be directed to Xin Deng PhD at (916) 445-2506 or by email at xdeng@cdpr.ca.gov.

IV. STUDY PLAN

According to the PUR database, over 600 pesticide AIs in a total amount of 135.6 million pounds were applied in agricultural areas of the state in 2011 (CDPR 2013). Those pesticide AIs possess a wide range of toxicity to aquatic organisms (US EPA 2013). In order to conduct the statewide monitoring effectively and better use limited resources, CDPR recently developed a pesticide Monitoring Prioritization Model (MPM) that automates the process of identifying potential monitoring candidates. The model develops a ranking of AIs based on their use amounts that were reported to the CDPR's PUR database (CDPR 2013) and their toxicity "Aquatic Life Benchmarks" that were developed by US EPA (US EPA 2013). Pesticide AIs that were selected as monitoring candidates for 2014 were identified using the MPM.

As a result of our statewide assessment using the MPM that was based on average use data from 2009-2011 and single-year use data from 2011, the top 10 statewide priority AIs were identified (Table 1). Of these, seven were selected for inclusion into this project: chlorpyrifos, malathion, permethrin, bifenthrin, pendimethalin, oxyfluorfen, and chlorothalonil. These AIs were also identified as monitoring candidates in the previous year (Starnier 2013), except for bifenthrin which replaced trifluralin on the top 10 list. Analytical methods are available for all seven AIs.

Three of the top 10 AIs from the assessment were not included in the current project because analytical methods are not yet available for paraquat dichloride and ziram, and for copper, environmental concentrations would not provide meaningful information on agricultural uses of copper due to confounding non-pesticide sources. In the future, additional factors will be assessed to determine if monitoring, and therefore the development of analytical methods, is warranted for these AIs. These factors may include chemical/physical properties, environmental fate, and detailed use patterns.

For the seven selected AIs, areas with periods of intensive use in the vicinity of surface water were identified through spatial/temporal analysis of PUR data (CDPR 2013). Chlorpyrifos, malathion and permethrin uses are high (>5000 lbs. active ingredient) in the Salinas and Santa Maria valleys throughout the irrigation season as well as in the Imperial Valley in the fall (Figure 1). DPR has previously designated these three geographic areas as high priority areas for long-term surface water monitoring, largely due to the high use of these AIs (Starnier 2010). This assessment supports that designation as well. For permethrin and bifenthrin analysis, the analytical method includes four additional pyrethroids: Lambda-cyhalothrin, cyfluthrin, cypermethrin and fenvalerate/esfenvalerate (Table 3).

Uses of oxyfluorfen and pendimethalin are high during the irrigation season in Salinas and Santa Maria valleys, and are high in the Imperial Valley and the Palo Verde area in Riverside County in the spring (Figure 1). Monitoring for these AIs will be conducted in these areas. The herbicide analytical method includes five additional dinitroanilines (i.e., oryzalin, ethalfluralin, trifluralin, benfluralin and proflumicafone) that will be analyzed together as well.

Chlorothalonil use is high in several areas of the Central Valley, as well as in Salinas, Santa Maria, and Imperial valleys. Monitoring will include two areas of the Central Valley (Figure 1), where use is high on tomatoes, as well as in the three high priority monitoring regions. CDPR started to monitor for chlorothalonil in agricultural areas in 2013.

For each of the regions selected above for inclusion in the project, an additional region-specific assessment was conducted using the pesticide MPM. The goal of these assessments was to identify AIs that have significant aquatic toxicity and high use within a specific geographic region, but for which use

was not high enough on a statewide basis to rank in the statewide analysis. The assessment was conducted using PUR data from 2009-2011 as well as the single-year data from 2011. The regional assessment for the Salinas Valley (Monterey County) resulted in the addition of diazinon, methomyl, pyraclostrobin and imidacloprid for monitoring in that area. Significant use of pendimethalin and malathion in Palo Verde concurrent with the high trifluralin use was also identified; those AIs will be included in the monitoring. Diacylhydrazine insecticides including methoxyfenozide and tebufenozide were previously detected at high frequencies in the Salinas Valley and will be included in the monitoring.

Monitoring in each area will be conducted for the appropriate AIs during the season or seasons of historically high pesticide use (CDPR 2013, Table 2). Sampling will commence in March 2014 and continue through October 2014.

V. SAMPLING METHODS

At each sampling site, surface water grab samples for chemical analysis will be collected into 1-liter amber glass bottles. Grab samples will be collected using either a grab pole consisting of a glass bottle at the end of an extendable pole. Glass bottles will be sealed with Teflon-lined lids and samples will be transported and stored on wet ice or refrigerated at 4°C until extraction for chemical analysis. Appropriate CDPR QA/QC Standard Operating Procedures will be followed.

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 analytes, method detection limits, and reporting limits for this study are given in Table 2. 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).

VII. DATA ANALYSIS

Concentrations of pesticides in water will be reported as micrograms per liter ($\mu\text{g/L}$) / parts per billion (ppb) or nanograms per liter (ng/L) / parts per trillion (ppt). Resulting data will be analyzed and reported as appropriate, potentially including the following:

Comparison of pesticide concentrations to aquatic toxicity benchmarks, water quality limits and other toxicity data (CCVRWQCB 2012, US EPA 2012); spatial analysis of data in order to identify correlations between observed pesticide concentrations and region-specific pesticide use and geographical features; assessment of multiple years of data to characterize patterns and trends in detection frequencies; assessment of results to determine potential additional monitoring in regions with similar pesticide use patterns.

VIII. TIMETABLE

Field Sampling:	March 2014 through October 2014
Chemical Analysis:	March 2014 through December 2014
Draft Report:	June 2015

IX. BUDGET

Analysis	Samples	Cost/sample	Cost Estimate
Organophosphate	85	600	51000
Diazinon	30	510	15300
Chlorothalonil	35	660	23100
Pyrethroids	30	960	28800
Dinitroanilines	30	960	28800
Methomyl	18	480	8640
Imidacloprid	40	720	28800
Strobilurins	45	840	37800
Diacylhydrazines	17	720	12240
Subtotal Analysis			234480

Continuing QC	Samples	Cost/sample	Cost Estimate
Organophosphate	9	600	5400
Diazinon	4	510	2040
Chlorothalonil	4	660	2640
Pyrethroids	3	960	2880
Dinitroanilines	3	960	2880
Methomyl	2	480	960
Imidacloprid	4	720	2880
Strobilurins	4	840	3360
Diacylhydrazines	2	720	1440
Subtotal QC			24480

Total			258960
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X. REFERENCES

CCVRWQCB (California Central Valley Regional Water Quality Control Board) 2012. Criteria reports. Accessed January 10 2013.

http://www.swrcb.ca.gov/rwqcb5/water_issues/tmdl/central_valley_projects/central_valley_pesticides/criteria_method/index.shtml

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<http://calpip.cdpr.ca.gov/>

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Segawa, R. 1995. Chemistry Laboratory Quality Control. Environmental Hazards Assessment Program QAQC001.00. Department of Pesticide Regulation, Sacramento, CA.

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<http://www.cdpr.ca.gov/docs/emon/pubs/protocol/study282protocol.pdf>

US EPA 2013. Aquatic Life Benchmark Table.

http://www.epa.gov/oppefed1/ecorisk_ders/aquatic_life_benchmark.htm

Table 1. Top 10 Pesticides Identified by Monitoring Prioritization Model Based on Statewide Yearly-Average Use Data from 2009-2011

Chemical Name	Use in pound	Use score	Benchmark (µg/L)	Toxicity score	Final score
CHLORPYRIFOS	1271377.5	5	0.05	6	30
PERMETHRIN	110277.6	3	0.01	7	21
COPPER	4938598.8	5	2.05	4	20
PENDIMETHALIN	1802207.6	5	5.2	4	20
PARAQUAT DICHLORIDE	831544.0	4	0.396	5	20
OXYFLUORFEN	611999.4	4	0.29	5	20
MALATHION	500470.3	4	0.3	5	20
BIFENTHRIN	85113.5	3	0.075	6	18
CHLOROTHALONIL	844393.7	4	1.8	4	16
ZIRAM	746473.6	4	9.7	4	16

Table 2. Monitoring Plan, 2014

Region	Season	Analytical Screen	Events
Salinas	spring through fall	Organophosphate	6
		Diazinon	6
		Chlorothalonil	3
		Pyrethroids	3
		Strobilurins	3
		Dinitroanilines	2
		Methomyl	3
		Imidacloprid	3
Santa Maria	spring through fall	Diacylhydrazines	3
		Organophosphate	3
		Chlorothalonil	1
		Pyrethroids	1
		Dinitroanilines	1
		Imidacloprid	2
Imperial	spring	Organophosphate	1
		Chlorothalonil	1
		Dinitroanilines	1
Imperial	fall	Organophosphate	1
		Diazinon	1
		Pyrethroids	1
		Imidacloprid	1
Palo Verde	spring	Organophosphate	1
		Dinitroanilines	1
Los Banos/SJ Delta	fall	Chlorothalonil	1

Table 3. Department of Food and Agriculture, Center for Analytical Chemistry analytical method details

Organophosphate (OP) Insecticides in Surface Water by GC/FPD (Short)

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Chlorpyrifos	0.0008	0.01
Diazinon	0.0012	0.01
Dimethoate	0.0079	0.04
Malathion	0.0117	0.02
Methidathion	0.0111	0.05

Dinitroaniline (DN) Herbicides/Oxyfluorfen in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Oryzalin	0.01	0.05
Ethalfuralin	0.01	0.05
Trifluralin	0.01	0.05
Benfluralin	0.01	0.05
Prodiamine	0.01	0.05
Pendamethalin	0.01	0.05
Oxyfluorfen	0.01	0.05

Chlorothalonil in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Chlorothalonil	0.111	0.05

Diacylhydrazine Insecticides in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Methoxyfenozide	0.00641	0.05
Tebufenozide	0.00573	0.05

Pyrethroid Insecticides (PY) in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Bifenthrin	0.00176	0.005
Lambda-cyhalothrin	0.00115	0.015
Permethrin (cis)	0.00352	0.015
Permethrin (trans)	0.00352	0.015
Cyfluthrin	0.0173	0.015
Cypermethrin	0.00175	0.015
Fenvalerate/esfenvalerate	0.00175	0.015

Imidacloprid (IMD) in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Imidacloprid	0.0101	0.05

Methomyl in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Methomyl	0.0265	0.05

Strobilurin Fungicides in Surface Water

<i>Chemical</i>	<i>Method Detection Limit (µg/L)</i>	<i>Reporting Limit (µg/L)</i>
Azoxystrobin	0.0225	0.05
Kresoxim-methyl	0.0190	0.05
Pyraclastrobin	0.0207	0.05
Trifloxystrobin	0.0172	0.05

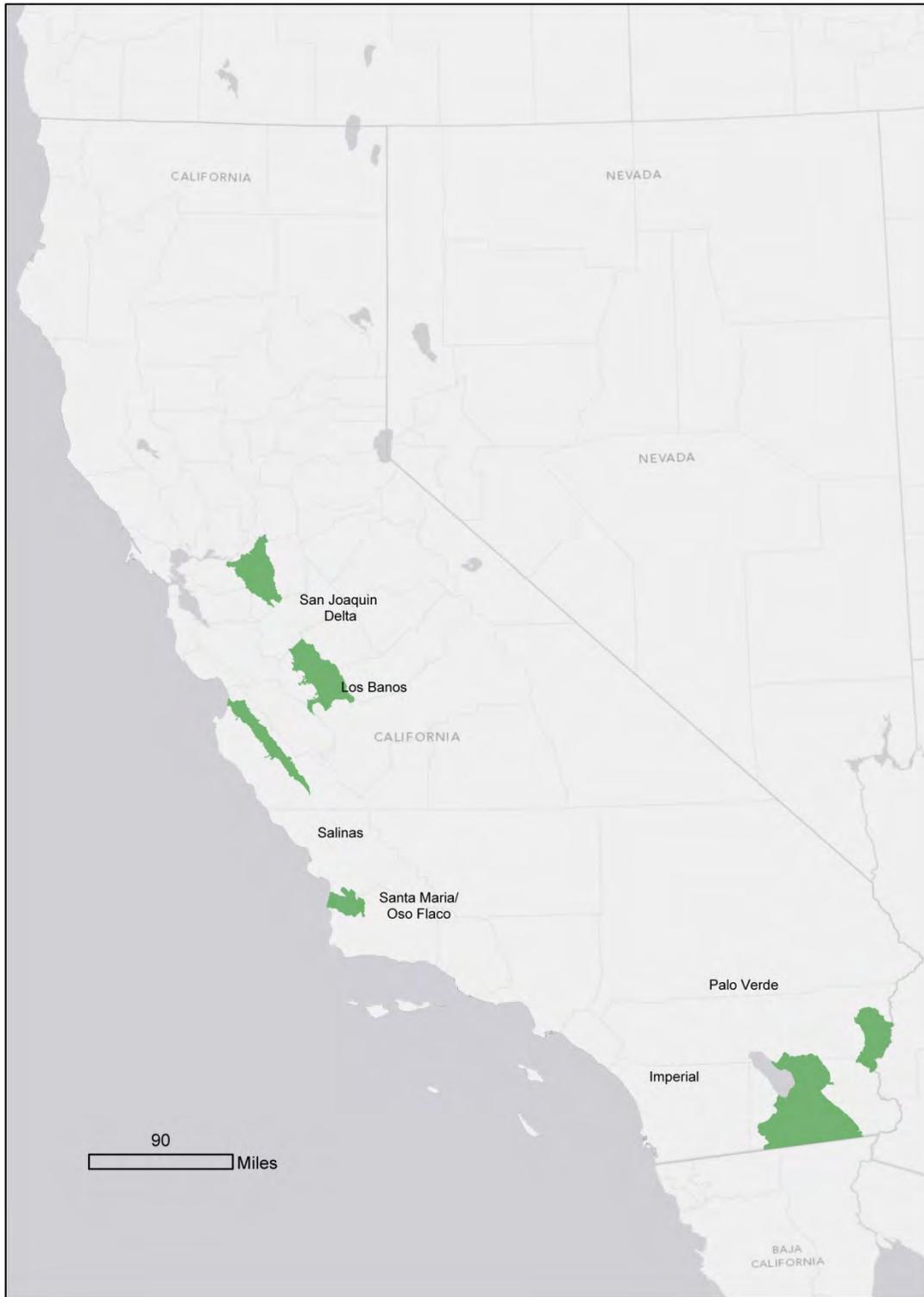


Figure 1. California Agricultural Monitoring Regions, 2014.

IX. BUDGET (revised, Study 290)

Analysis	Samples	Cost/sample	Cost Estimate
Organophosphate	83	600	49800
Diazinon	48	510	24480
Chlorothalonil	24	660	15840
Pyrethroids	38	960	36480
Dinitroanilines	35	840	29400
Methomyl	18	480	8640
Imidacloprid	60	720	43200
Strobilurins	29	840	24360
Diacylhydrazines	19	720	13680
Subtotal Analysis			245880

QC	Samples	Cost/sample	Cost Estimate
Organophosphate	8	600	4800
Diazinon	7	510	3570
Chlorothalonil	4	660	2640
Pyrethroids	4	960	3840
Dinitroanilines	3	840	2520
Methomyl	2	480	960
Imidacloprid	6	720	4320
Imidacloprid	3	840	2520
Diacylhydrazines	2	720	1440
Subtotal QC			26610

Total **272490**

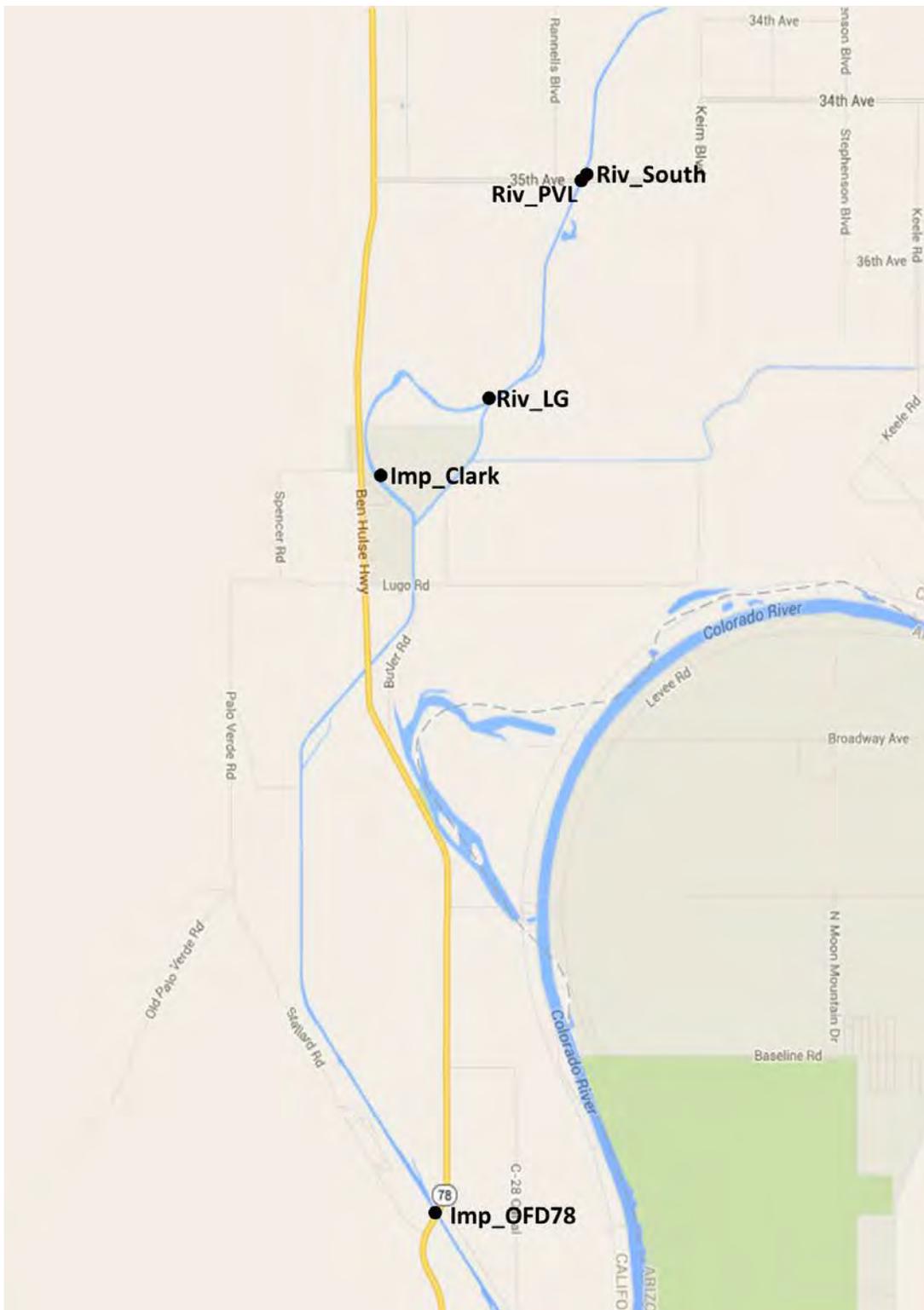
* Lab blind spiked samples are not included.

Appendix II. Sampling Site Information for Study 290 in 2014

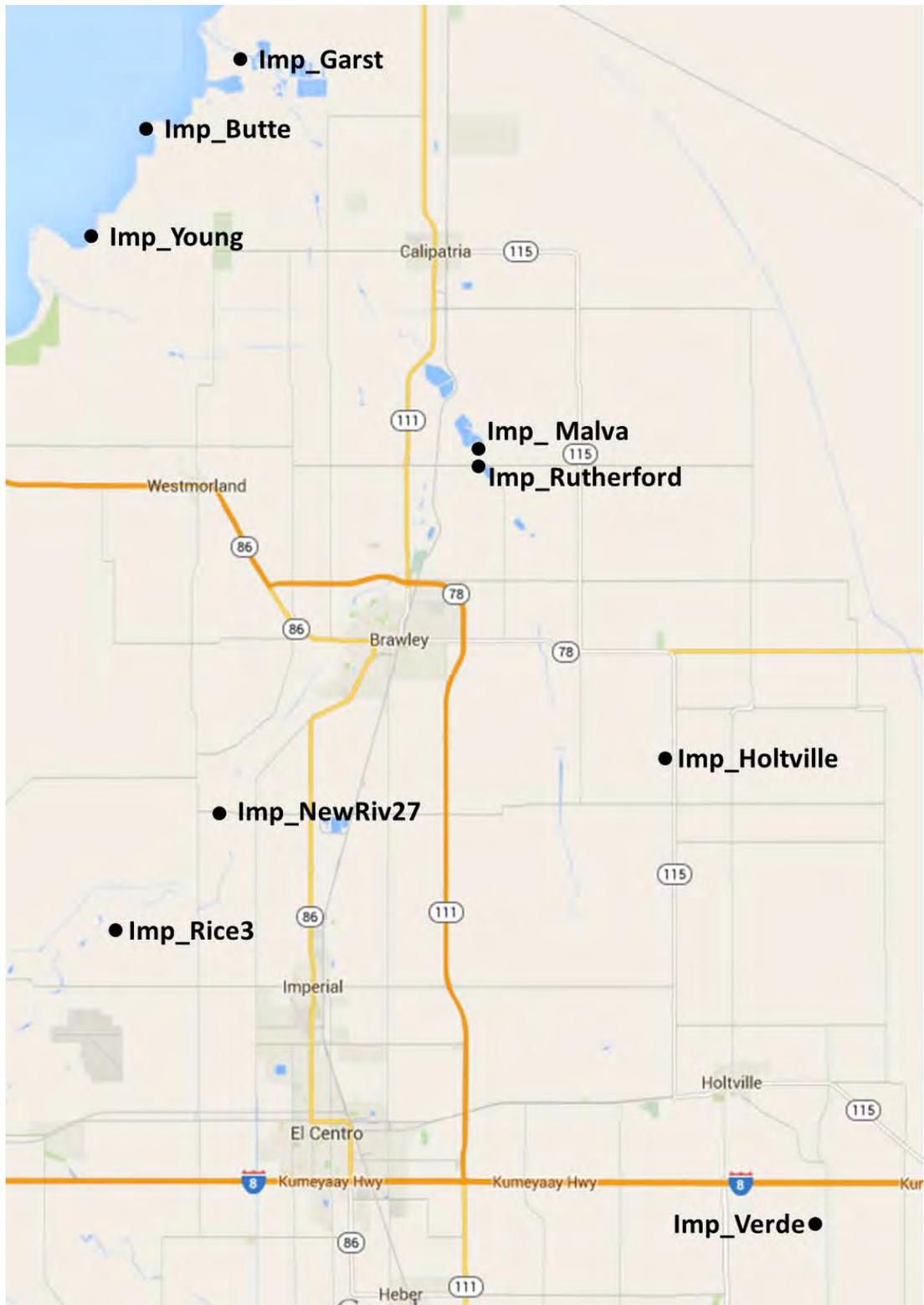
Site ID	County	Watershed	Latitude	Longitude	Site Type	Site Location
Imp_Garst	Imperial	Alamo River	33.199	-115.59696	Receiving Water	Alamo River at Garst Road
Imp_Holtville	Imperial	Alamo River	32.9309	-115.40611	Receiving Water	Holtville Main Drain at HWY115
Imp_Malva	Imperial	Alamo River	33.0518	-115.48862	Ag Drain	Malva Drain nr. Park Avenue
Imp_Young	Imperial	New River	33.1328	-115.66646	Ag Drain	Vail Drain nr Young Road
Imp_Verde	Imperial	Alamo River	32.7555	-115.33697	Ag Drain	Verde Drain at Bonds Corner Road
Imp_Clark	Imperial	Palo Verde Drain	33.428	-114.73	Receiving Water	Palo Verde Outfall Drain (PVOD2) - Colorado River Region - SWAMP station code 715CPVOD2
Imp_Rutherford	Imperial	Alamo River	33.0447	-115.48829	Receiving Water	Alamo River at Rutherford Rd (upstream of Imperial State Wildlife Area)
Imp_Butte	Imperial	Salton Sea	33.1753	-115.63722	Receiving Water	Salton Sea at Obsidian Butte
Imp_Rice3	Imperial	New River	32.8691	-115.651	Ag Drain	Rice Drain III at Weinert Road
Imp_NewRiv27	Imperial	New River	32.9136	-115.60646	Receiving Water	New River at HWY S27/Keystone Road
Imp_OFD78	Imperial	Palo Verde Drain	33.3613	-114.72299	Ag Drain	Outfall Drain at HWY78
Sal_Rec3	Monterey	Salinas River	36.6592	-121.61567	Receiving Water	Reclamation Ditch site 3
Sal_SanJon	Monterey	Tembladero Slough	36.7049	-121.70506	Receiving Water	Rec Ditch at San Jon Road
Sal_Davis	Monterey	Salinas River	36.647	-121.70219	Receiving Water	Salinas River at Davis Road
Sal_Monte	Monterey	Salinas River	36.7319	-121.7824	Receiving Water	Salinas River at Del Monte Road
Sal_Potrero	Monterey	Old Salinas River	36.79056	-121.79064	Receiving Water	Old Salinas River at Potrero Rd
Sal_Dunes	Monterey	Old Salinas River	36.7719	-121.78971	Receiving Water	Old Salinas R. at Monterey Dunes Way

Appendix II. (Continued)

Site ID	County	Watershed	Latitude	Longitude	Site Type	Site Location
Sal_Molera	Monterey	Tembladero Slough	36.7721	-121.78763	Receiving Water	Tembladero Sl. at Molera Road
Sal_Haro	Monterey	Tembladero Slough	36.7596	-121.75433	Receiving Water	Tembladero Slough at Haro Street
Sal_Quail	Monterey	Salinas River	36.6092	-121.56269	Receiving Water	Quail Creek at HWY 101, btwn Spence and Potter Roads (trib. to Salinas R.)
Sal_Hartnell	Monterey	Salinas River	36.6435	-121.57836	Receiving Water	Alisal Creek at Hartnell Rd
Sal_Chualar	Monterey	Salinas River	36.5584	-121.52964	Ag Drain	Chualar Creek at Chualar River Rd., ca. 1.2 mi. from HWY 101 (trib. to Salinas R.)
Sal_Blanco	Monterey	Salinas River	36.6987	-121.73517	Ag Drain	Blanco Drain at Cooper Rd, ca 0.2 mi. S of Nashua Rd, drains to Salinas River
Riv_LG	Riverside	Palo Verde Drain	33.436	-114.7162	Receiving Water	Palo Verde Lagoon (LG1) - Colorado River Region - SWAMP station code 715CPVLG1
Riv_PVL	Riverside	Palo Verde Drain	33.4559	-114.70551	Receiving Water	Palo Verde Lagoon @ 35 th Avenue
Riv_South	Riverside	Palo Verde Drain	33.4562	-114.70501	Receiving Water	South End Drain @Palo Verde Lagoon
SM_OFC	San Luis Obispo	Oso Flaco Creek	35.0164	-120.58755	Ag Drain	Oso Flaco Creek @ OFL Road
SM_Solomon	Santa Barbara	Orcutt Creek	34.9414	-120.5742	Receiving Water	Solomon Creek @ HWY 1
SM_Orcutt	Santa Barbara	Orcutt Creek	34.9576	-120.63244	Receiving Water	Orcutt Creek @ Main Street
SM_Main	Santa Barbara	Santa Maria River	34.95474	-120.48501	Ag Drain	Main Ditch @ Main Street
SM_Simas	Santa Barbara	Orcutt Creek	34.9423	-120.5563	Ag Drain	Green Valley Creek @ Simas Road



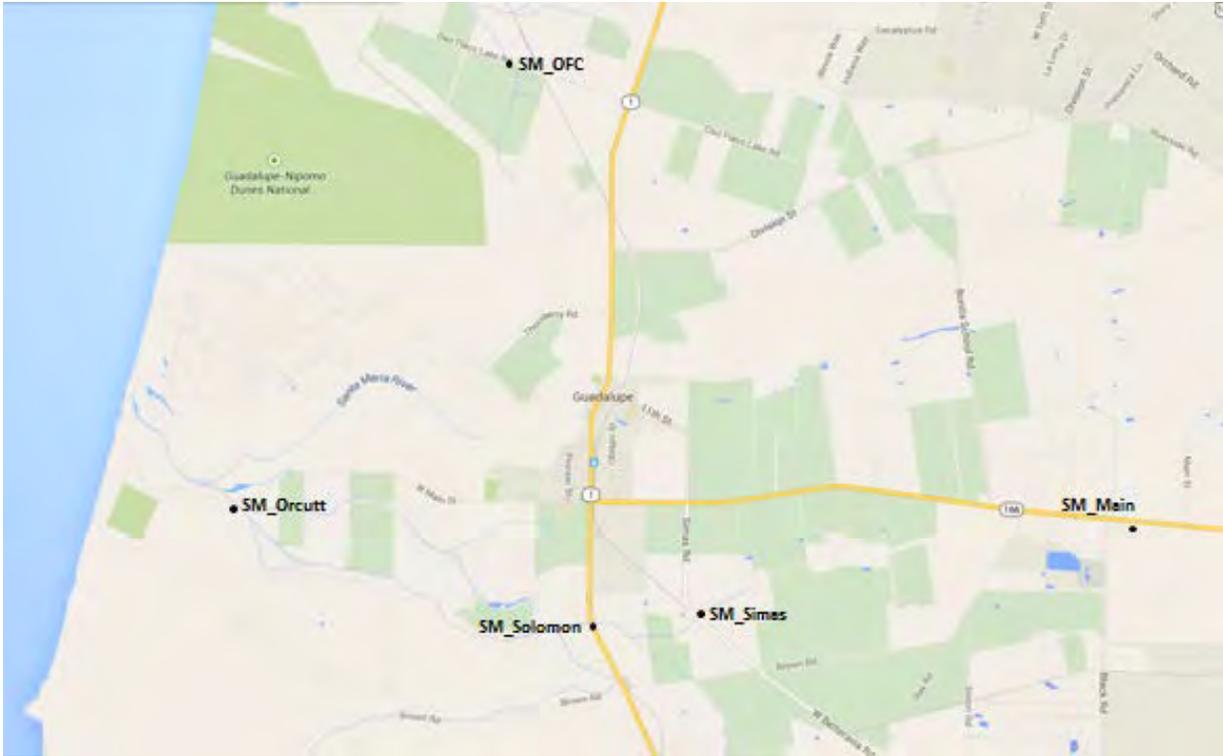
Map 1. Monitoring Sites in Palo Verde Outfall Drain in Imperial and Riverside Counties.



Map 2. Monitoring Sites in Alamo River and New River in Imperial County.



Map 3. Monitoring Sites in Salinas River and Tembladero Slough in Monterey County.



Map 4. Monitoring Sites in Orcutt Creek and Oso Flaco Creek in Santa Barbara and San Luis Obispo Counties.

Appendix III. Water Quality Data for Study 290 in 2014

Site ID	Sample Date	Site Type	Event Type	Water Flow	Temp (C°)	pH (unit)	DO (mg/L)	Conductivity (mS/cm)	Salinity (ppt)
Imp_Garst	3/18/2014	Receiving Water	Nonstorm	Flowing	19.2	7.76	4.1	2.819	1.5
Imp_Young	3/18/2014	Ag Drain	Nonstorm	Flowing	23.5	7.14	7.03	2.553	1.3
Imp_Butte	3/18/2014	Receiving Water	Nonstorm	Ponded	22.7	8.41	5.91	65.8	44.6
Imp_Rice3	3/18/2014	Ag Drain	Nonstorm	Flowing	14.7	7.54	3.3	2.732	1.4
Imp_Verde	3/18/2014	Ag Drain	Nonstorm	Flowing	16.5	7.69	4.66	3.342	1.8
Imp_Holtville	3/18/2014	Receiving Water	Nonstorm	Flowing	16.4	7.98	5.05	2.914	1.5
Imp_Malva	3/18/2014	Ag Drain	Nonstorm	Flowing	17.7	8.16	5.33	2.595	1.3
Imp_Rutherford	3/18/2014	Receiving Water	Nonstorm	Ponded	18.5	7.83	4.65	2.920	1.5
Imp_NewRiv27	3/18/2014	Receiving Water	Nonstorm	Flowing	18.4	7.64	8.0	4.621	2.5
Riv_PVL	3/19/2014	Receiving Water	Nonstorm	Ponded	18.1	7.69	6.71	2.007	1.0
Riv_LG	3/19/2014	Receiving Water	Nonstorm	Ponded	19.3	7.52	7.16	1.989	1.0
Imp_Clark	3/19/2014	Receiving Water	Nonstorm	Flowing	19.0	7.5	7.53	1.991	1.0
Imp_OFD78	3/19/2014	Receiving Water	Nonstorm	Ponded	18.8	7.61	6.62	2.093	1.1
Riv_South	3/19/2014	Ag Drain	Nonstorm	Ponded	18.7	7.75	6.73	2.515	1.3
Sal_Quail	4/15/2014	Ag Drain	Nonstorm	Flowing	18.6	8.7	6.68	1.121	0.6
Sal_Hartnell	4/15/2014	Receiving Water	Nonstorm	Flowing	24.3	7.98	6.25	0.873	0.4
Sal_Rec3	4/15/2014	Receiving Water	Nonstorm	Flowing	21.0	7.13	7.98	1.146	0.6

Site ID	Sample Date	Site Type	Event Type	Water Flow	Temp (C°)	pH (unit)	DO (mg/L)	Conductivity (mS/cm)	Salinity (ppt)
Sal_Haro	4/15/2014	Receiving Water	Nonstorm	Flowing	22.2	8.09	9.66	2.643	1.4
Sal_Molera	4/15/2014	Receiving Water	Nonstorm	Flowing	20.1	8.53	6.65	5.28	2.9
Sal_Chualar	4/16/2014	Ag Drain	Nonstorm	Flowing	10.1	8.59	10.27	1.201	0.6
Sal_Davis	4/16/2014	Receiving Water	Nonstorm	Flowing	17.3	8.12	8.31	2.057	1.1
Sal_Blanco	4/16/2014	Ag Drain	Nonstorm	Flowing	15.8	8.27	15.61	3.046	1.6
Sal_Monte	4/16/2014	Receiving Water	Nonstorm	Ponded	18.2	9.29	11.59	2.400	1.4
Sal_Dunes	4/16/2014	Receiving Water	Nonstorm	Flowing	17.7	8.97	10.64	5.38	2.9
Sal_Quail	5/13/2014	Ag Drain	Nonstorm	Flowing	22.8	8.11	8.07	1.268	0.6
Sal_Chualar	5/13/2014	Ag Drain	Nonstorm	Flowing	27.5	8.91	11.35	1.313	0.7
Sal_Hartnell	5/13/2014	Receiving Water	Nonstorm	Flowing	20.1	7.94	5.49	0.994	0.5
Sal_Rec3	5/13/2014	Receiving Water	Nonstorm	Flowing	26.7	8.93	6.52	1.005	0.5
Sal_Haro	5/13/2014	Receiving Water	Nonstorm	Flowing	24.3	8.57	8.6	2.415	1.2
Sal_Monte	5/13/2014	Receiving Water	Nonstorm	Ponded	21.3	7.83	9.63	1.261	0.6
SM_Simas	5/14/2014	Ag Drain	Nonstorm	Flowing	22.0	8.13	9.32	3.502	1.8
SM_Solomon	5/14/2014	Receiving Water	Nonstorm	Flowing	26.4	7.52	10.32	3.091	1.6
SM_Orcutt	5/14/2014	Receiving Water	Nonstorm	Flowing	23.5	7.99	8.84	2.762	1.4
SM_Main	5/14/2014	Ag Drain	Nonstorm	Flowing	29.4	8.28	13.25	1.481	0.7
SM_OFC	5/14/2014	Ag Drain	Nonstorm	Flowing	19.8	7.51	5.11	2.872	1.5

Site ID	Sample Date	Site Type	Event Type	Water Flow	Temp (C°)	pH (unit)	DO (mg/L)	Conductivity (mS/cm)	Salinity (ppt)
Sal_Monte	6/17/2014	Receiving Water	Nonstorm	Ponded	MV*	MV	MV	MV	MV
Sal_Quail	6/17/2014	Ag Drain	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Chualar	6/17/2014	Ag Drain	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Hartnell	6/17/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Rec3	6/17/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_SanJon	6/17/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Davis	6/18/2014	Receiving Water	Nonstorm	Ponded	MV	MV	MV	MV	MV
Sal_Potrero	6/18/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Molera	6/18/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Dunes	6/18/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Haro	6/18/2014	Receiving Water	Nonstorm	Flowing	MV	MV	MV	MV	MV
Sal_Hartnell	7/15/2014	Receiving Water	Nonstorm	Flowing	21.6	8.03	5.92	0.950	0.5
Sal_Rec3	7/15/2014	Receiving Water	Nonstorm	Flowing	24.6	8.61	15.3	1.0127	0.5
Sal_Haro	7/15/2014	Receiving Water	Nonstorm	Flowing	24.4	8.72	7.39	2.070	1.05
Sal_Quail	7/15/2014	Ag Drain	Nonstorm	Flowing	20.0	8.35	4.64	0.851	0.40
Sal_Chualar	7/15/2014	Ag Drain	Nonstorm	Flowing	26.7	9.44	16.99	1.859	0.90
SM_Solomon	7/16/2014	Receiving Water	Nonstorm	Flowing	21.2	7.8	12.22	3.1282	1.64

Site ID	Sample Date	Site Type	Event Type	Water Flow	Temp (C°)	pH (unit)	DO (mg/L)	Conductivity (mS/cm)	Salinity (ppt)
SM_Orcutt	7/16/2014	Receiving Water	Nonstorm	Flowing	21.1	7.72	7.35	2.739	1.42
SM_Main	7/16/2014	Ag Drain	Nonstorm	Flowing	22.5	8.38	7.79	1.607	0.80
SM_OFC	7/16/2014	Ag Drain	Nonstorm	Flowing	17.7	7.21	2.26	2.420	1.25
Sal_Quail	8/19/2014	Ag Drain	Nonstorm	Flowing	20.8	8.13	8.74	1.1287	0.56
Sal_Chualar	8/19/2014	Ag Drain	Nonstorm	Flowing	20.9	8.15	8.75	1.1276	0.56
Sal_Hartnell	8/19/2014	Receiving Water	Nonstorm	Flowing	20.5	7.85	7.35	0.9218	0.45
Sal_Rec3	8/19/2014	Receiving Water	Nonstorm	Flowing	20.0	8.15	11.52	0.996	0.49
Sal_Haro	8/19/2014	Receiving Water	Nonstorm	Flowing	21.1	7.91	9.75	2.698	1.40
SM_Orcutt	8/20/2014	Receiving Water	Nonstorm	Flowing	20.4	7.65	6.30	3.463	1.81
SM_Main	8/20/2014	Ag Drain	Nonstorm	Flowing	27.4	8.71	11.47	1.286	0.64
SM_Solomon	8/20/2014	Receiving Water	Nonstorm	Flowing	23.9	7.40	13.6	4580	2.46
SM_OFC	8/20/2014	Ag Drain	Nonstorm	Flowing	17.8	7.22	MV	2.784	1.48
Sal_Quail	9/16/2014	Ag Drain	Nonstorm	Flowing	16.7	7.81	6.72	1.454	0.73
Sal_Chualar	9/16/2014	Ag Drain	Nonstorm	Flowing	24.1	9.46	19.39	0.93	0.46
Sal_Hartnell	9/16/2014	Receiving Water	Nonstorm	Flowing	19.8	7.91	4.82	1.383	0.70
Sal_Rec3	9/16/2014	Receiving Water	Nonstorm	Flowing	20.4	8.71	2.82	0.706	0.35
Sal_Haro	9/16/2014	Receiving Water	Nonstorm	Flowing	18.5	8.05	7.04	2.577	1.33

Site ID	Sample Date	Site Type	Event Type	Water Flow	Temp (C°)	pH (unit)	DO (mg/L)	Conductivity (mS/cm)	Salinity (ppt)
SM_OFC	9/17/2014	Ag Drain	Nonstorm	Flowing	20.1	7.61	2.94	2.383	1.23
SM_Solomon	9/17/2014	Receiving Water	Nonstorm	Flowing	23.2	8.08	16.95	3.822	2.02
SM_Orcutt	9/17/2014	Receiving Water	Nonstorm	Flowing	20.0	7.85	6.51	3.194	1.67
SM_Main	9/17/2014	Ag Drain	Nonstorm	Flowing	25.8	8.8	16.22	1.339	0.67
Imp_NewRiv27	10/15/2014	Receiving Water	Nonstorm	Flowing	22.3	7.63	5.66	5.543	3.0
Imp_Rice3	10/15/2014	Ag Drain	Nonstorm	Flowing	21.8	8.05	8.1	4.076	2.17
Imp_Verde	10/15/2014	Ag Drain	Nonstorm	Flowing	22.9	8.11	7.85	4.081	2.16
Imp_Holtville	10/15/2014	Receiving Water	Nonstorm	Flowing	21.8	8.16	8.18	3.642	1.92
Imp_Malva	10/15/2014	Ag Drain	Nonstorm	Flowing	21.3	8.32	9.35	2.594	1.34
Imp_Rutherford	10/15/2014	Receiving Water	Nonstorm	Flowing	23.0	8.03	7.89	3.038	1.58
Imp_Young	10/15/2014	Ag Drain	Nonstorm	Flowing	24.2	7.75	3.46	2.453	1.26
Imp_Butte	10/15/2014	Receiving Water	Nonstorm	Ponded	30.7	8.43	12.22	65.01	43.9
Imp_Garst	10/15/2014	Receiving Water	Nonstorm	Flowing	23.3	8.05	7.11	3.171	1.66

*MV = missing value.

Appendix IV. Water Monitoring Data for Study 290 in 2014

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group*	Result (µl/L)**	RL (µl/L)	MDL (µl/L)
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Oxyfluorfen	OXY	0.092	0.050	0.0230
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Butte	Receiving Water	3/18/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Chlorpyrifos	OP	0.584	0.010	0.0102
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Dimethoate	OP	4.24	0.040	0.0120
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Malathion	OP	0.802	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Pendimethalin	DN	1.79	0.050	0.0190
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Garst	Receiving Water	3/18/14	Nonstorm	Trifluralin	DN	0.339	0.050	0.0150
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Chlorothalonil	CT	0.105	0.050	0.0348
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Chlorpyrifos	OP	0.904	0.010	0.0102
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Dimethoate	OP	2.97	0.040	0.0120
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Malathion	OP	3.71	0.020	0.0094
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Oxyfluorfen	OXY	0.083	0.050	0.0230
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Pendimethalin	DN	3.18	0.050	0.0190
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Holtville	Receiving Water	3/18/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Chlorpyrifos	OP	0.794	0.010	0.0102
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Dimethoate	OP	0.068	0.040	0.0120
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Oxyfluorfen	OXY	0.132	0.050	0.0230
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Pendimethalin	DN	2.26	0.050	0.0190
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Malva	Ag Drain	3/18/14	Nonstorm	Trifluralin	DN	1.03	0.050	0.0150
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Chlorpyrifos	OP	0.390	0.010	0.0102
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Dimethoate	OP	3.06	0.040	0.0120
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Malathion	OP	1.01	0.020	0.0094
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Pendimethalin	DN	1.42	0.050	0.0190
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_NewRiv27	Receiving Water	3/18/14	Nonstorm	Trifluralin	DN	0.178	0.050	0.0150
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Chlorpyrifos	OP	0.644	0.010	0.0102
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Dimethoate	OP	16.4	0.040	0.0120
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Malathion	OP	0.071	0.020	0.0094
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Pendimethalin	DN	4.31	0.050	0.0190
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Rice3	Ag Drain	3/18/14	Nonstorm	Trifluralin	DN	0.052	0.050	0.0150
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Chlorpyrifos	OP	0.689	0.010	0.0102

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Dimethoate	OP	4.79	0.040	0.0120
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Malathion	OP	0.423	0.020	0.0094
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Oxyfluorfen	OXY	0.058	0.050	0.0230
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Pendimethalin	DN	1.99	0.050	0.0190
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Rutherford	Receiving Water	3/18/14	Nonstorm	Trifluralin	DN	0.338	0.050	0.0150
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Chlorpyrifos	OP	1.75	0.010	0.0102
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Dimethoate	OP	1.29	0.040	0.0120
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Pendimethalin	DN	2.14	0.050	0.0190

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Verde	Ag Drain	3/18/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Imp_Young	Ag Drain	3/18/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Young	Ag Drain	3/18/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Imp_Young	Ag Drain	3/18/14	Nonstorm	Chlorpyrifos	OP	0.374	0.010	0.0102
Imp_Young	Ag Drain	3/18/14	Nonstorm	Dimethoate	OP	0.451	0.040	0.0120
Imp_Young	Ag Drain	3/18/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Young	Ag Drain	3/18/14	Nonstorm	Malathion	OP	0.123	0.020	0.0094
Imp_Young	Ag Drain	3/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Young	Ag Drain	3/18/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Young	Ag Drain	3/18/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
Imp_Young	Ag Drain	3/18/14	Nonstorm	Pendimethalin	DN	0.911	0.050	0.0190
Imp_Young	Ag Drain	3/18/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Young	Ag Drain	3/18/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Dimethoate	OP	0.142	0.040	0.0120
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Pendimethalin	DN	Trace	0.050	0.0190
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_Clark	Receiving Water	3/19/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Dimethoate	OP	0.239	0.040	0.0120
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Pendimethalin	DN	Trace	0.050	0.0190
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Imp_OFD78	Ag Drain	3/19/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Riv_LG	Receiving Water	3/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Riv_LG	Receiving Water	3/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Riv_LG	Receiving Water	3/19/14	Nonstorm	Dimethoate	OP	0.155	0.040	0.0120
Riv_LG	Receiving Water	3/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Riv_LG	Receiving Water	3/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Riv_LG	Receiving Water	3/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Riv_LG	Receiving Water	3/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Riv_LG	Receiving Water	3/19/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Riv_LG	Receiving Water	3/19/14	Nonstorm	Pendimethalin	DN	0.057	0.050	0.0190
Riv_LG	Receiving Water	3/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Riv_LG	Receiving Water	3/19/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Dimethoate	OP	0.139	0.040	0.0120
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Pendimethalin	DN	0.065	0.050	0.0190
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Riv_PVL	Receiving Water	3/19/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Riv_South	Ag Drain	3/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Riv_South	Ag Drain	3/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Riv_South	Ag Drain	3/19/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Riv_South	Ag Drain	3/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Riv_South	Ag Drain	3/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Riv_South	Ag Drain	3/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Riv_South	Ag Drain	3/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Riv_South	Ag Drain	3/19/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Riv_South	Ag Drain	3/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Riv_South	Ag Drain	3/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Riv_South	Ag Drain	3/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Bifenthrin	PY	0.0182	0.001	0.0009
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Methoxyfenozide	DA	0.214	0.050	0.0064
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Haro	Receiving Water	4/15/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Bifenthrin	PY	0.00229	0.001	0.0009
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Methoxyfenozide	DA	0.163	0.050	0.0064
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Permethrin	PY	0.00933	0.002	0.0011
Sal_Hartnell	Receiving Water	4/15/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Bifenthrin	PY	0.00352	0.001	0.0009
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Molera	Receiving Water	4/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Bifenthrin	PY	0.0255	0.001	0.0009
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Methoxyfenozide	DA	Trace	0.050	0.0064
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Permethrin	PY	0.1940	0.002	0.0011
Sal_Quail	Ag Drain	4/15/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Bifenthrin	PY	0.00207	0.001	0.0009
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Malathion	OP	0.454	0.020	0.0094
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Methoxyfenozide	DA	0.095	0.050	0.0064
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Permethrin	PY	0.00375	0.002	0.0011
Sal_Rec3	Receiving Water	4/15/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Blanco	Ag Drain	4/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Diazinon	OP	0.047	0.010	0.0109
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Methoxyfenozide	DA	Trace	0.050	0.0064

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Chualar	Ag Drain	4/16/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Davis	Receiving Water	4/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Bifenthrin	PY	0.00294	0.001	0.0009
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Dunes	Receiving Water	4/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Monte	Receiving Water	4/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Azoxystrobin	STR	0.060	0.050	0.0225
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Benfluralin	DN	Trace	0.050	0.0150
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Bensulide	IMD/BEN	3.64	0.040	0.0198
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Chlorpyrifos	OP	0.043	0.010	0.0102
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	2.19	0.050	0.0394
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Chualar	Ag Drain	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Bensulide	IMD/BEN	Trace	0.040	0.0198
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Diazinon	OP	0.026	0.010	0.0109
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	0.371	0.050	0.0394
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Oxyfluorfen	OXY	0.054	0.050	0.0230
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Haro	Receiving Water	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Azoxystrobin	STR	0.198	0.050	0.0225
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Bensulide	IMD/BEN	2.13	0.040	0.0198
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	1.41	0.050	0.0394

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Pyraclostrobin	STR	0.062	0.050	0.0207
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Hartnell	Receiving Water	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Monte	Receiving Water	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	0.455	0.050	0.0394
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Malathion	OP	ND	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Oxyfluorfen	OXY	ND	0.050	0.0230
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Pyraclostrobin	STR	0.072	0.050	0.0207
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Quail	Ag Drain	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Azoxystrobin	STR	0.144	0.050	0.0225
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Bensulide	IMD/BEN	0.109	0.040	0.0198
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Imidacloprid	IMD/BEN	1.00	0.050	0.0394
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Malathion	OP	0.02	0.020	0.0094
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Rec3	Receiving Water	5/13/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Main	Ag Drain	5/14/14	Nonstorm	Azoxystrobin	STR	ND	0.050	0.0225
SM_Main	Ag Drain	5/14/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Main	Ag Drain	5/14/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Main	Ag Drain	5/14/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Main	Ag Drain	5/14/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Main	Ag Drain	5/14/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Main	Ag Drain	5/14/14	Nonstorm	Imidacloprid	IMD/BEN	0.340	0.050	0.0394
SM_Main	Ag Drain	5/14/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Main	Ag Drain	5/14/14	Nonstorm	Malathion	OP	0.056	0.020	0.0094
SM_Main	Ag Drain	5/14/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Main	Ag Drain	5/14/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Main	Ag Drain	5/14/14	Nonstorm	Oxyfluorfen	OXY	0.060	0.050	0.0230

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µ/L)	RL (µ/L)	MDL (µ/L)
SM_Main	Ag Drain	5/14/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Main	Ag Drain	5/14/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Main	Ag Drain	5/14/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Main	Ag Drain	5/14/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Main	Ag Drain	5/14/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
SM_OFC	Ag Drain	5/14/14	Nonstorm	Azoxystrobin	STR	0.745	0.050	0.0225
SM_OFC	Ag Drain	5/14/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_OFC	Ag Drain	5/14/14	Nonstorm	Bensulide	IMD/BEN	0.078	0.040	0.0198
SM_OFC	Ag Drain	5/14/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_OFC	Ag Drain	5/14/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_OFC	Ag Drain	5/14/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_OFC	Ag Drain	5/14/14	Nonstorm	Imidacloprid	IMD/BEN	1.12	0.050	0.0394
SM_OFC	Ag Drain	5/14/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_OFC	Ag Drain	5/14/14	Nonstorm	Malathion	OP	0.287	0.020	0.0094
SM_OFC	Ag Drain	5/14/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_OFC	Ag Drain	5/14/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_OFC	Ag Drain	5/14/14	Nonstorm	Oxyfluorfen	OXY	0.295	0.050	0.0230
SM_OFC	Ag Drain	5/14/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_OFC	Ag Drain	5/14/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_OFC	Ag Drain	5/14/14	Nonstorm	Pyraclostrobin	STR	0.222	0.050	0.0207
SM_OFC	Ag Drain	5/14/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_OFC	Ag Drain	5/14/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Imidacloprid	IMD/BEN	0.621	0.050	0.0394
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Oxyfluorfen	OXY	Trace	0.050	0.0230
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Orcutt	Receiving Water	5/14/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Simas	Ag Drain	5/14/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
SM_Simas	Ag Drain	5/14/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Simas	Ag Drain	5/14/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Simas	Ag Drain	5/14/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Simas	Ag Drain	5/14/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Simas	Ag Drain	5/14/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Simas	Ag Drain	5/14/14	Nonstorm	Imidacloprid	IMD/BEN	0.464	0.050	0.0394
SM_Simas	Ag Drain	5/14/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Simas	Ag Drain	5/14/14	Nonstorm	Malathion	OP	0.023	0.020	0.0094
SM_Simas	Ag Drain	5/14/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Simas	Ag Drain	5/14/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Simas	Ag Drain	5/14/14	Nonstorm	Oxyfluorfen	OXY	0.164	0.050	0.0230
SM_Simas	Ag Drain	5/14/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Simas	Ag Drain	5/14/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Simas	Ag Drain	5/14/14	Nonstorm	Pyraclostrobin	STR	Trace	0.050	0.0207
SM_Simas	Ag Drain	5/14/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Simas	Ag Drain	5/14/14	Nonstorm	Trifluralin	DN	0.050	0.050	0.0150
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Azoxystrobin	STR	0.078	0.050	0.0225

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Imidacloprid	IMD/BEN	1.83	0.050	0.0394
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Malathion	OP	0.056	0.020	0.0094
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Oxyfluorfen	OXY	0.137	0.050	0.0230
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Pyraclostrobin	STR	Trace	0.050	0.0207
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Solomon	Receiving Water	5/14/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Bensulide	IMD/BEN	0.486	0.040	0.0198
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Diazinon	OP	0.014	0.010	0.0109

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Dimethoate	OP	0.325	0.040	0.0120
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.314	0.050	0.0394
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Methoxyfenozide	DA	Trace	0.050	0.0064
Sal_Chualar	Ag Drain	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Bensulide	IMD/BEN	0.444	0.040	0.0198
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.862	0.050	0.0394
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Methoxyfenozide	DA	0.076	0.050	0.0064
Sal_Hartnell	Receiving Water	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Methoxyfenozide	DA	0.160	0.050	0.0064
Sal_Monte	Receiving Water	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.367	0.050	0.0394
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Methoxyfenozide	DA	Trace	0.050	0.0064
Sal_Quail	Ag Drain	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Bensulide	IMD/BEN	0.875	0.040	0.0198
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.745	0.050	0.0394

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Methoxyfenozide	DA	0.116	0.050	0.0064
Sal_Rec3	Receiving Water	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Bensulide	IMD/BEN	0.046	0.040	0.0198
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.208	0.050	0.0394
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Methoxyfenozide	DA	1.01	0.050	0.0064
Sal_SanJon	Receiving Water	6/17/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Methoxyfenozide	DA	Trace	0.050	0.0064
Sal_Davis	Receiving Water	6/18/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Methoxyfenozide	DA	0.084	0.050	0.0064
Sal_Dunes	Receiving Water	6/18/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Imidacloprid	IMD/BEN	0.082	0.050	0.0394
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Methoxyfenozide	DA	0.232	0.050	0.0064
Sal_Haro	Receiving Water	6/18/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Diazinon	OP	0.010	0.010	0.0109
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Imidacloprid	IMD/BEN	0.063	0.050	0.0394
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Methoxyfenozide	DA	0.261	0.050	0.0064
Sal_Molera	Receiving Water	6/18/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Methoxyfenozide	DA	0.135	0.050	0.0064

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Potrero	Receiving Water	6/18/14	Nonstorm	Tebufenozide	DA	ND	0.050	0.0057
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Diazinon	OP	0.011	0.010	0.0109
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Azoxystrobin	STR	0.259	0.050	0.0225
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Bensulide	IMD/BEN	7.73	0.040	0.0198
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Chlorpyrifos	OP	0.013	0.010	0.0102
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Dimethoate	OP	2.08	0.040	0.0120
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.482	0.050	0.0394
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Methomyl	ME	0.096	0.050	0.0110
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Pyraclostrobin	STR	0.088	0.050	0.0207

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	7/15/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Bifenthrin	PY	0.0178	0.001	0.0009
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.111	0.050	0.0394
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Methomyl	ME	0.354	0.050	0.0110
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	7/15/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Azoxystrobin	STR	0.212	0.050	0.0225
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Bensulide	IMD/BEN	0.590	0.040	0.0198
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Bifenthrin	PY	0.00389	0.001	0.0009
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Dimethoate	OP	0.071	0.040	0.0120
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.823	0.050	0.0394
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Malathion	OP	0.201	0.020	0.0094
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Methomyl	ME	0.852	0.050	0.0110
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Permethrin	PY	0.0280	0.002	0.0011
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Pyraclostrobin	STR	0.111	0.050	0.0207

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Hartnell	Receiving Water	7/15/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Azoxystrobin	STR	0.077	0.050	0.0225
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Bensulide	IMD/BEN	9.68	0.040	0.0198
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Bifenthrin	PY	0.00260	0.001	0.0009
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.528	0.050	0.0394
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Malathion	OP	0.073	0.020	0.0094
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Methomyl	ME	0.304	0.050	0.0110
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Permethrin	PY	0.0189	0.002	0.0011
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Quail	Ag Drain	7/15/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Azoxystrobin	STR	0.073	0.050	0.0225
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Bensulide	IMD/BEN	0.160	0.040	0.0198
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Bifenthrin	PY	0.00117	0.001	0.0009
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.903	0.050	0.0394
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Lambda Cyhalothrin	PY	0.00229	0.002	0.0017
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Methomyl	ME	0.214	0.050	0.0110
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Rec3	Receiving Water	7/15/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Main	Ag Drain	7/16/14	Nonstorm	Azoxystrobin	STR	0.053	0.050	0.0225
SM_Main	Ag Drain	7/16/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Main	Ag Drain	7/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_Main	Ag Drain	7/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Main	Ag Drain	7/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_Main	Ag Drain	7/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Main	Ag Drain	7/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Main	Ag Drain	7/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_Main	Ag Drain	7/16/14	Nonstorm	Imidacloprid	IMD/BEN	0.989	0.050	0.0394
SM_Main	Ag Drain	7/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Main	Ag Drain	7/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Main	Ag Drain	7/16/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
SM_Main	Ag Drain	7/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Main	Ag Drain	7/16/14	Nonstorm	Methomyl	ME	0.064	0.050	0.0110
SM_Main	Ag Drain	7/16/14	Nonstorm	Permethrin	PY	0.0341	0.002	0.0011
SM_Main	Ag Drain	7/16/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Main	Ag Drain	7/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_OFC	Ag Drain	7/16/14	Nonstorm	Azoxystrobin	STR	0.078	0.050	0.0225

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_OFC	Ag Drain	7/16/14	Nonstorm	Bensulide	IMD/BEN	0.052	0.040	0.0198
SM_OFC	Ag Drain	7/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_OFC	Ag Drain	7/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_OFC	Ag Drain	7/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_OFC	Ag Drain	7/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_OFC	Ag Drain	7/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_OFC	Ag Drain	7/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_OFC	Ag Drain	7/16/14	Nonstorm	Imidacloprid	IMD/BEN	0.343	0.050	0.0394
SM_OFC	Ag Drain	7/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_OFC	Ag Drain	7/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_OFC	Ag Drain	7/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
SM_OFC	Ag Drain	7/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_OFC	Ag Drain	7/16/14	Nonstorm	Methomyl	ME	ND	0.050	0.0110
SM_OFC	Ag Drain	7/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_OFC	Ag Drain	7/16/14	Nonstorm	Pyraclostrobin	STR	0.086	0.050	0.0207
SM_OFC	Ag Drain	7/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Bensulide	IMD/BEN	Trace	0.040	0.0198
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Imidacloprid	IMD/BEN	0.578	0.050	0.0394
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Malathion	OP	0.975	0.020	0.0094
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Methomyl	ME	0.055	0.050	0.0110
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Orcutt	Receiving Water	7/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Azoxystrobin	STR	0.082	0.050	0.0225
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Bensulide	IMD/BEN	0.440	0.040	0.0198
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Imidacloprid	IMD/BEN	1.34	0.050	0.0394
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Malathion	OP	1.52	0.020	0.0094
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Methomyl	ME	ND	0.050	0.0110
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Pyraclostrobin	STR	Trace	0.050	0.0207
SM_Solomon	Receiving Water	7/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Bensulide	IMD/BEN	12.9	0.040	0.0198
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Chlorpyrifos	OP	0.043	0.010	0.0102
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Dimethoate	OP	0.767	0.040	0.0120
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Imidacloprid	IMD/BEN	0.793	0.050	0.0394
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Chualar	Ag Drain	8/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Diazinon	OP	0.076	0.010	0.0109
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Imidacloprid	IMD/BEN	0.149	0.050	0.0394
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Haro	Receiving Water	8/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Bensulide	IMD/BEN	10.0	0.040	0.0198
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Diazinon	OP	0.021	0.010	0.0109
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Imidacloprid	IMD/BEN	1.80	0.050	0.0394
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Malathion	OP	0.054	0.020	0.0094
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Hartnell	Receiving Water	8/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Bensulide	IMD/BEN	2.13	0.040	0.0198
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Imidacloprid	IMD/BEN	0.742	0.050	0.0394
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Quail	Ag Drain	8/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Bensulide	IMD/BEN	1.38	0.040	0.0198
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Diazinon	OP	0.015	0.010	0.0109
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Imidacloprid	IMD/BEN	1.05	0.050	0.0394

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
Sal_Rec3	Receiving Water	8/19/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Main	Ag Drain	8/20/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Main	Ag Drain	8/20/14	Nonstorm	Bensulide	IMD/BEN	0.355	0.040	0.0198
SM_Main	Ag Drain	8/20/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Main	Ag Drain	8/20/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Main	Ag Drain	8/20/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Main	Ag Drain	8/20/14	Nonstorm	Imidacloprid	IMD/BEN	0.234	0.050	0.0394
SM_Main	Ag Drain	8/20/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
SM_Main	Ag Drain	8/20/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Main	Ag Drain	8/20/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Main	Ag Drain	8/20/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Main	Ag Drain	8/20/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Main	Ag Drain	8/20/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_OFC	Ag Drain	8/20/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_OFC	Ag Drain	8/20/14	Nonstorm	Bensulide	IMD/BEN	Trace	0.040	0.0198
SM_OFC	Ag Drain	8/20/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_OFC	Ag Drain	8/20/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_OFC	Ag Drain	8/20/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_OFC	Ag Drain	8/20/14	Nonstorm	Imidacloprid	IMD/BEN	0.679	0.050	0.0394
SM_OFC	Ag Drain	8/20/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
SM_OFC	Ag Drain	8/20/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_OFC	Ag Drain	8/20/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_OFC	Ag Drain	8/20/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_OFC	Ag Drain	8/20/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_OFC	Ag Drain	8/20/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Dimethoate	OP	0.129	0.040	0.0120
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Imidacloprid	IMD/BEN	1.03	0.050	0.0394
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Malathion	OP	0.033	0.020	0.0094
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Orcutt	Receiving Water	8/20/14	Nonstorm	Trifluralin	DN	ND	0.050	0.0150
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Benfluralin	DN	ND	0.050	0.0150
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Bensulide	IMD/BEN	trace	0.040	0.0198
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Dimethoate	OP	0.287	0.040	0.0120
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Ethalfuralin	DN	ND	0.050	0.0170
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Imidacloprid	IMD/BEN	3.98	0.050	0.0394
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Oryzalin	DN	ND	0.050	0.0210
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Pendimethalin	DN	ND	0.050	0.0190
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Prodiamine	DN	ND	0.050	0.0200
SM_Solomon	Receiving Water	8/20/14	Nonstorm	Trifluralin	DN	Trace	0.050	0.0150
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Azoxystrobin	STR	0.065	0.050	0.0225
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Bensulide	IMD/BEN	4.13	0.040	0.0198
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Bifenthrin	PY	0.0114	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Diazinon	OP	0.037	0.010	0.0109
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Imidacloprid	IMD/BEN	1.74	0.050	0.0394
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Malathion	OP	Trace	0.020	0.0094
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Methomyl	ME	1.07	0.050	0.0110
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Permethrin	PY	0.0171	0.002	0.0011
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Chualar	Ag Drain	9/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Bifenthrin	PY	0.00322	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Dimethoate	OP	0.098	0.040	0.0120
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Imidacloprid	IMD/BEN	0.172	0.050	0.0394
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Malathion	OP	0.136	0.020	0.0094
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Methomyl	ME	0.114	0.050	0.0110
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
Sal_Haro	Receiving Water	9/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Azoxystrobin	STR	0.238	0.050	0.0225
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Bensulide	IMD/BEN	0.305	0.040	0.0198
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Bifenthrin	PY	0.00207	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Diazinon	OP	0.022	0.010	0.0109
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Dimethoate	OP	0.551	0.040	0.0120
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Imidacloprid	IMD/BEN	1.33	0.050	0.0394
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Malathion	OP	0.024	0.020	0.0094
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Methomyl	ME	0.687	0.050	0.0110
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Permethrin	PY	0.0053	0.002	0.0011
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Pyraclostrobin	STR	Trace	0.050	0.0207
Sal_Hartnell	Receiving Water	9/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Azoxystrobin	STR	0.063	0.050	0.0225
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Bensulide	IMD/BEN	1.40	0.040	0.0198
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Bifenthrin	PY	0.00574	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Imidacloprid	IMD/BEN	0.924	0.050	0.0394
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Lambda Cyhalothrin	PY	0.00424	0.002	0.0017
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Malathion	OP	0.145	0.020	0.0094
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Methomyl	ME	6.51	0.050	0.0110
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Permethrin	PY	0.0717	0.002	0.0011
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Pyraclostrobin	STR	0.198	0.050	0.0207
Sal_Quail	Ag Drain	9/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Azoxystrobin	STR	0.100	0.050	0.0225
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Bensulide	IMD/BEN	0.269	0.040	0.0198
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Bifenthrin	PY	0.00152	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Chlorothalonil	CT	ND	0.050	0.0348
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Imidacloprid	IMD/BEN	2.01	0.050	0.0394
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Lambda Cyhalothrin	PY	0.00525	0.002	0.0017
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Methomyl	ME	1.56	0.050	0.0110
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Pyraclostrobin	STR	0.129	0.050	0.0207
Sal_Rec3	Receiving Water	9/16/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Main	Ag Drain	9/17/14	Nonstorm	Azoxystrobin	STR	ND	0.050	0.0225
SM_Main	Ag Drain	9/17/14	Nonstorm	Bensulide	IMD/BEN	0.438	0.040	0.0198
SM_Main	Ag Drain	9/17/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Main	Ag Drain	9/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Main	Ag Drain	9/17/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_Main	Ag Drain	9/17/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Main	Ag Drain	9/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Main	Ag Drain	9/17/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_Main	Ag Drain	9/17/14	Nonstorm	Imidacloprid	IMD/BEN	1.39	0.050	0.0394
SM_Main	Ag Drain	9/17/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Main	Ag Drain	9/17/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Main	Ag Drain	9/17/14	Nonstorm	Malathion	OP	0.03	0.020	0.0094
SM_Main	Ag Drain	9/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Main	Ag Drain	9/17/14	Nonstorm	Methomyl	ME	ND	0.050	0.0110
SM_Main	Ag Drain	9/17/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_Main	Ag Drain	9/17/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Main	Ag Drain	9/17/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_OFC	Ag Drain	9/17/14	Nonstorm	Azoxystrobin	STR	0.075	0.050	0.0225
SM_OFC	Ag Drain	9/17/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_OFC	Ag Drain	9/17/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_OFC	Ag Drain	9/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_OFC	Ag Drain	9/17/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_OFC	Ag Drain	9/17/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_OFC	Ag Drain	9/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_OFC	Ag Drain	9/17/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_OFC	Ag Drain	9/17/14	Nonstorm	Imidacloprid	IMD/BEN	0.577	0.050	0.0394
SM_OFC	Ag Drain	9/17/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_OFC	Ag Drain	9/17/14	Nonstorm	Lambda Cyhalothrin	PY	0.00945	0.002	0.0017
SM_OFC	Ag Drain	9/17/14	Nonstorm	Malathion	OP	0.227	0.020	0.0094
SM_OFC	Ag Drain	9/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_OFC	Ag Drain	9/17/14	Nonstorm	Methomyl	ME	ND	0.050	0.0110
SM_OFC	Ag Drain	9/17/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_OFC	Ag Drain	9/17/14	Nonstorm	Pyraclostrobin	STR	0.095	0.050	0.0207
SM_OFC	Ag Drain	9/17/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Azoxystrobin	STR	Trace	0.050	0.0225
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	0.00880	0.005	0.0017
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Imidacloprid	IMD/BEN	1.54	0.050	0.0394
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Malathion	OP	0.036	0.020	0.0094
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Methomyl	ME	0.110	0.050	0.0110
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Orcutt	Receiving Water	9/17/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Azoxystrobin	STR	ND	0.050	0.0225
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Imidacloprid	IMD/BEN	9.14	0.050	0.0394

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Kresoxim-methyl	STR	ND	0.050	0.0190
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Malathion	OP	0.123	0.020	0.0094
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Methomyl	ME	ND	0.050	0.0110
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Pyraclostrobin	STR	ND	0.050	0.0207
SM_Solomon	Receiving Water	9/17/14	Nonstorm	Trifloxystrobin	STR	ND	0.050	0.0172
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.199	0.040	0.0198
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Butte	Receiving Water	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.688	0.040	0.0198
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Chlorpyrifos	OP	0.222	0.010	0.0102
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.352	0.050	0.0394
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Garst	Receiving Water	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Bensulide	IMD/BEN	4.78	0.040	0.0198
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Bifenthrin	PY	0.00168	0.001	0.0009
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Chlorpyrifos	OP	0.085	0.010	0.0102
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.182	0.050	0.0394
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Holtville	Receiving Water	10/15/14	Nonstorm	Permethrin	PY	0.00233	0.002	0.0011
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Bensulide	IMD/BEN	ND	0.040	0.0198
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Chlorpyrifos	OP	1.220	0.010	0.0102
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	ND	0.050	0.0394
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Malva	Ag Drain	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.051	0.040	0.0198
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Chlorpyrifos	OP	0.035	0.010	0.0102
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.065	0.050	0.0394
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	0.00221	0.002	0.0017
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_NewRiv27	Receiving Water	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.148	0.040	0.0198
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Chlorpyrifos	OP	0.049	0.010	0.0102

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	0.00716	0.005	0.0017
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.210	0.050	0.0394
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	0.00208	0.002	0.0017
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Rice3	Ag Drain	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.776	0.040	0.0198
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Chlorpyrifos	OP	0.124	0.010	0.0102
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.411	0.050	0.0394

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Rutherford	Receiving Water	10/15/14	Nonstorm	Permethrin	PY	ND	0.002	0.0011
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Bensulide	IMD/BEN	3.73	0.040	0.0198
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Chlorpyrifos	OP	ND	0.010	0.0102
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	0.00543	0.005	0.0017
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.099	0.050	0.0394
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	0.00339	0.002	0.0017
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Verde	Ag Drain	10/15/14	Nonstorm	Permethrin	PY	0.00357	0.002	0.0011
Imp_Young	Ag Drain	10/15/14	Nonstorm	Bensulide	IMD/BEN	0.740	0.040	0.0198
Imp_Young	Ag Drain	10/15/14	Nonstorm	Bifenthrin	PY	ND	0.001	0.0009

Site ID	Site Type	Sample Date	Event Type	Analyte Name	Analyte Group	Result (µl/L)	RL (µl/L)	MDL (µl/L)
Imp_Young	Ag Drain	10/15/14	Nonstorm	Chlorpyrifos	OP	0.028	0.010	0.0102
Imp_Young	Ag Drain	10/15/14	Nonstorm	Cyfluthrin	PY	ND	0.002	0.0146
Imp_Young	Ag Drain	10/15/14	Nonstorm	Cypermethrin	PY	ND	0.005	0.0015
Imp_Young	Ag Drain	10/15/14	Nonstorm	Diazinon	OP	ND	0.010	0.0109
Imp_Young	Ag Drain	10/15/14	Nonstorm	Dimethoate	OP	ND	0.040	0.0120
Imp_Young	Ag Drain	10/15/14	Nonstorm	Fenvalerate/Esfenvalerate	PY	ND	0.005	0.0017
Imp_Young	Ag Drain	10/15/14	Nonstorm	Imidacloprid	IMD/BEN	0.903	0.050	0.0394
Imp_Young	Ag Drain	10/15/14	Nonstorm	Lambda Cyhalothrin	PY	ND	0.002	0.0017
Imp_Young	Ag Drain	10/15/14	Nonstorm	Malathion	OP	ND	0.020	0.0094
Imp_Young	Ag Drain	10/15/14	Nonstorm	Methidathion	OP	ND	0.050	0.0114
Imp_Young	Ag Drain	10/15/14	Nonstorm	Permethrin	PY	0.0024	0.002	0.0011

* DA = Diacylhydrazines; DN = Dinitroanilines; DZ = Diazinon; IMD/BEN = Imidacloprid/Bensulide; ME = Methomyl; OP = Organophosphates; OXY = Oxyfluorfen; PY = Pyrethroids; STR = Strobilurins.

**ND = not detected; Trace = MDL < concentration < RL.

Appendix V. Aquatic Toxicity Data for Study 290 on September and October, 2014*

Site ID	Site Type	Sample Date	Hyalella Survival		Hyalella Growth		Chironomus Survival		Chironomus Growth	
			%	SD**	mg/ind	SD	%	SD	mg/ind	SD
Sal_Chualar	Ag Drain	9/16/14	0	0	NA	NA	73	4	0.301	0.052
Sal_Hartnell	Receiving Water	9/16/14	38	8	0.067	0.02	0	0	NA	NA
Sal_Quail	Ag Drain	9/16/14	0	0	NA	NA	2	4	NA	NA
Sal-Rec3	Receiving Water	9/16/14	30	10	0.058	0.01	4	8	NA	NA
Sal_Haro	Receiving Water	9/16/14	59	9	0.065	0.02	83	12	1.860	0.799
SM_Main	Ag Drain	9/17/14	94	5	0.111	0.01	92	10	2.095	1.671
SM_Solomon	Receiving Water	9/17/14	98	4	0.078	0.01	0	0	NA	NA
SM-Orcutt	Receiving Water	9/17/14	50	20	0.051	0.03	48	14	0.121	0.100
SM_OFc	Ag Drain	9/17/14	0	0	NA	NA	42	34	0.223	0.146
Lab QA			82	8	0.099	0.01	100	0	2.399	1.486

Site ID	Site Type	Sample Date	Hyalella Survival		Hyalella Growth		Chironomus Survival		Chironomus Growth	
			%	SD**	mg/ind	SD	%	SD	mg/ind	SD
Imp_Garst	Receiving Water	10/15/14	0	0	NA	NA	69	10	2.729	1.237
Imp_Rugherford	Receiving Water	10/15/14	0	0	NA	NA	79	16	2.008	1.571
Imp_Holtville	Receiving Water	10/15/14	0	0	NA	NA	82	3	1.143	0.787
Imp_Malva	Ag Drain	10/15/14	0	0	NA	NA	0	0	NA	NA
Imp_NewRiv27	Receiving Water	10/15/14	78	13	0.106	0.01	96	8	1.475	1.017
Imp_Rice3	Ag Drain	10/15/14	40	24	0.138	0.04	88	16	1.404	0.358
Imp_Young	Ag Drain	10/15/14	96	9	0.119	0.00	86	7	1.529	1.141
Imp_Verde	Ag Drain	10/15/14	22	4	0.160	0.05	96	5	0.803	0.609
Lab QA			96	5	0.096	0.01	96	5	0.923	0.279

*Toxicity tests were conducted for 10 days for *Hyalella azteca* and *Chironomus dilutus*. The endpoints were percent survivals and average individual growth rate (mg/ind); Highlighted cells indicate significant differences from controls.

**SD = standard deviation.

Appendix VI. Analytical Methods used for Study 290 in 2014

Determination of organophosphate pesticides in surface water using gas chromatography with mass selective detection (MSD) (CDFA)

Determination of methoxyfenozide and tebufenozide in surface water by ultra performance liquid chromatography coupled to tandem mass spectrometry (CDFA)

Determination of N-methylcarbamate pesticides in surface water using high performance liquid chromatography and post-column derivatization (CDFA)

Determination of bensulide and imidacloprid in surface water (CDFA)

Determination of pyrethroids in sediment water using triple quadrupole GC/MS/MS (CDFA)

Determination of ethalfluralin, trifluralin, benfluralin, prodiamine, pendimethalin, oxyfluorfen, and oryzalin in surface water (CDFA)

Determination of chlorothalonil in ground and surface water (CDFA)

Determination of azoxystrobin, kresoxim methyl, pyraclostrobin and trifloxystrobin in surface water by ultra performance liquid chromatography coupled to tandem mass spectrometry (CDFA)

Title: Determination of Chlorothalonil in Ground and Surface Water

1. Scope:

This section method (SM) provides stepwise procedure for chlorothalonil analysis in ground and surface water. It is followed by all authorized EA personnel.

2. Principle:

The chlorothalonil is extracted from the acidified ground water and surface water samples with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated on a rotary evaporator and then a solvent exchange is performed with methanol. The extract is concentrated to a final volume of 1 mL and then vialled into 2 autosampler vials for analysis on an Ultra Performance Liquid Chromatography (UPLC) coupled to a negative atmosphere pressure chemical ionization triple quadrupole mass spectrometry (APCI-LC/MS/MS).

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

4. Interferences:

There were no matrix interferences for chlorothalonil at the time of method development.

5. Apparatus and Equipment:

- 5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)
- 5.2 Nitrogen Evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)
- 5.3 Balance (Mettler PC 4400 or equivalent)
- 5.4 Vortex-vibrating mixer
- 5.5 UPLC equipped with a triple quadrupole mass spectrometry and APCI ion source.

6. Reagents and Supplies:

- 6.1 Chlorothalonil CAS#1897-45-6
- 6.2 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.3 Sulfuric Acid, Conc. ACS Grade
- 6.4 Water, MS grade, Burdick & Jackson or equivalent
- 6.5 Methanol, MS grade, Burdick & Jackson or equivalent
- 6.6 Separatory funnel, 2 L
- 6.7 Boiling flask, 500 mL
- 6.8 Sodium Sulfate, ACS grade
- 6.9 Funnels, long stem, 60°, 10 mm diameter
- 6.10 Graduated conical tubes with glass stopper, 15 mL
- 6.11 Glass wool, Pyrex® fiber glass slivers 8 microns
- 6.12 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.13 Recommended analytical column:
Waters Acquity BEH 1.7µm, 2.1 x 50 mm

7. Standards Preparation:

- 7.1 An individual stock standard of 1.0 mg/mL was obtained from the CDFA/CAC Standards Repository. The standard was diluted to 10 µg/mL with methanol for identification purposes.

The following concentrations: 1, 0.5, 0.25, 0.1, 0.05, 0.025, µg/mL were prepared in methanol for LC instrument calibration.

- 7.2 Keep all standards in the designated refrigerator for storage.
- 7.3 The expiration date of each standard is six months from the preparation date.

8. Sample Preservation and Storage:

Store all samples waiting for extraction in a separate refrigerator (4±3°C).

9. Test Sample Preparation:

- 9.1 Background Preparation

The Department of Pesticide Regulation (DPR) provided the ground water and surface water for background to be used in method validation and QC.

9.2 Preparation of blank and spike

Matrix blank: Weigh out 1000 g of background water and follow the test sample extraction procedure.

Matrix spike: Weigh out 1000 g of background water. Spike a client requested amount of fungicide into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.3 Test Sample Extraction

- 9.3.1 Record the weight of water samples to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a funnel.
- 9.3.2 Add 2.5 mL of sulfuric acid to each separatory funnel and mix well.
- 9.3.3 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.
- 9.3.4 After phases have separated, drain the lower methylene chloride layer through 25 ± 4 g of anhydrous sodium sulfate and glass wool into a 500 mL boiling flask.
- 9.3.5 Repeat steps 9.3.3 & 9.3.4 two more times using 80 ± 5 mL of methylene chloride for 1 minute each time. Combine the extracts in the same boiling flask.
- 9.3.6 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.3.7 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 – 20 inch Hg vacuum. Add 2-4 mL of methanol and rotoevaporate to 1-2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.3.8 Rinse flask 3 more times with 2 - 4 mL of methanol and transfer each rinse to the same test tube.

9.3.9 Evaporate the sample extract to a volume slightly less than 1 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with methanol, mix well and transfer to 2 autosampler vials with inserts.

10. Instrument Calibration:

- 10.1 The calibration standard curve consists of a minimum of three levels. The lowest level must be at or below the corresponding reporting limits.
- 10.2 The calibration curve for the LCMS instrument was obtained using Linear fit.

11. Analysis:

11.1 UPLC-MS/MS

11.1.1 UPLC Instrument: Waters Acquity Ultra Performance LC
Column: Waters Acquity BEH 1.7 μ m, 2.1 x 50 mm
Column Temperature: 60 °C
Mobile Phase: Gradient
Solvent 1: Water
Solvent 2: Methanol
Gradient:

<u>Time(min)</u>	<u>Flow rate</u>	<u>Solvent 1</u>	<u>Solvent 2</u>
0	0.50	90.0	10.0
1.0	0.50	90.0	10.0
1.5	0.50	5.0	95.0
3.5	0.50	5.0	95.0
3.55	0.50	90.0	10.0
5.0	0.50	90.0	10.0

Injection Volume:2.0 μ L

11.1.2 Mass Spectrometry and Operating Parameters

Model: Waters Xevo Triple Quadrupole
Ion ProbeType: Atmospheric Pressure Chemical Ionization (APCI)
Ion Mode: APCI-
APCI Probe Temp: 500 °C
Source Temp: 150 °C

Compound	Retention Time (min)	Precursor ion	Product Ion	Dwell (s)	Cone(V)	Collision Energy/-ev
Chlorothalonil	1.90	244.95	174.95	0.061	46.0	28.0
		244.95	181.91	0.061	46.0	30.0

Quantitation ions are in bold.

Note: The column conditions, temperature, mobile phase, etc. may slightly shift retention time.

12. Quality Control:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 well water samples and 7 surface water samples are spiked at 0.1ppb and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

$$MDL = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicates used to determine the MDL, t=3.143.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is chosen in a range 1-5 times the MDL, as per client agreement. The reporting limit for Chlorothalonil in well water and surface water is 0.05 ppb.

12.3 Method Validation

The method validation consisted of five sample sets. Each set included five levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the analytes are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation. The upper and lower warning and control limits are set at ± 2 and 3 standard deviations of the % recovery, respectively, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 per cent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. **Calculations:**

Quantitation is based on an external standard (ESTD) calculation using either the peak area or height. The LCMS software used a linear curve fit, with all levels weighted 1/x.

$$\text{ppb} = \frac{(\text{sample peak area or ht}) \times (\text{std conc}) \times (\text{std vol. injected}) \times (\text{final vol of sample})(1000 \mu\text{L/mL})}{(\text{std.peak area or ht}) \times (\text{sample vol injected}) \times (\text{sample wt (g)})}$$

14. **Reporting Procedure:**

Sample results are reported out according to the client's analytical laboratory specification sheets.

15. **Discussion and References:**

15.1 Upon infusion of chlorothalonil, we found the principal ion to be 245 ion rather than the anticipated molecular ion at 264. This is consistent with substitution of the chlorine by hydroxyl within the source.

15.2 Acid is not necessary for the extraction of chlorothalonil but was added with the intent of including its metabolites at a later date.

- 15.3 A storage stability study was done with this project for well water only. The storage stability study consisted of a 1.0 ppb spike level and 2 replicates over a 28 day period. Fourteen liters of background well water were spiked and then transferred to fourteen one liter amber bottles. These spiked samples were stored in the refrigerator until analyzed at 0, 2, 5, 7, 14, 21 and 28 days. Along with the storage spikes a blank and method control spike were also extracted. This storage study showed no degradation for the chlorothalonil within the 28 days. Results for the storage studies are shown in Appendix 3.
- 15.4 We have observed gradual losses in sensitivity and peak tailing caused by the sample matrix. We recommend cleaning the cones when this occurs.
- 15.5 References:
- 15.51 Wakefield, Mike (Principal MS Applications Specialist); UPLC-MS/MS conditions for Chlorothalonil, Waters Corporation
- 15.52 Hsu, J. and White, J.; *Determination of Azoxystrobin, Azoxystrobin Acid, Azoxystrobin Z-metabolite, Dicloran, Iprodione, Isoiprodione, Vinclozalin and 3,5-Dichloroaniline in Well Water*, 2010, Environmental Analysis Section Method, Center for Analytical Chemistry, CDFA

Appendix 1

The Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Results:	Well Water	Surface Water
Spk\Analyte	Chlorothalonil	Chlorothalonil
	<u>Spike Level: 0.1 ppb</u>	<u>Spike Level: 0.1 ppb</u>
Spike 1	0.0919	0.0898
Spike 2	0.127	0.0959
Spike 3	0.0958	0.102
Spike 4	0.106	0.0797
Spike 5	0.104	0.115
Spike 6	0.107	0.101
Spike 7	0.106	0.0928
SD	0.0112	0.0111
MDL	0.0351	0.0348
RL	0.05	0.05

Appendix 2

Results: Well Water

Analyte	Spike ppb	Recovery					%	%
		(%) Set 1	(%) set 2	(%) set 3	(%) set 4	(%) set 5		
Chlorothalonil	0.1	113	100	95.8	111	85.4	Mean:	93.2
	0.2	98.3	110	79.7	115	84.0	SD:	12.4
	0.5	73.2	116	78.3	91.8	79.1	UCL:	130.4
	1.0	93.3	91.6	87.7	89.0	82.9	UWL:	118.0
	2.0	83.0	101	85.8	99.5	86.2	LWL:	68.5
							LCL:	56.1

Results: Surface Water

Analyte	Spike ppb	Recovery					%	%
		(%) Set 1	(%) set 2	(%) set 3	(%) set 4	(%) set 5		
Chlorothalonil	0.1	95.2	79.7	76.2	94.3	100	Mean:	93.3
	0.2	103	89.8	81.8	102	110	SD:	9.9
	0.5	101	92.4	74.9	102	97.6	UCL:	123.0
	1.0	90.8	92.7	81.8	104	90.7	UWL:	113.1
	2.0	106	91.8	79.3	104	90.5	LWL:	73.4
							LCL:	63.5

Appendix 3 Storage Stability Study

Spike Level: 1.0ppb

Chlorothalonil Results:

Storage Day	EMON Lab#	Sample	1st injection result ppb	2nd injection result ppb	Average ppb	% Recovery
Day 0	2010-1618	Blank	ND	ND	ND	N/A
	2010-1619	SPK 1	0.785	0.676	0.731	73.1%
	2010-1620	Spk 2	0.765	0.721	0.743	74.3%
Day 2	2010-1621	Blank	ND	ND	ND	N/A
	2010-1622	QC spk	0.887	0.844	0.866	86.6%
	2010-1623	SPK 1	1.04	1.07	1.055	106%
	2010-1624	Spk 2	1.02	0.931	0.976	97.6%
Day 5	2010-1625	Blank	ND	ND	ND	N/A
	2010-1626	QC spk	0.783	0.718	0.751	75.1%
	2010-1627	SPK 1	0.810	0.764	0.787	78.7%
	2010-1628	Spk 2	0.882	0.718	0.800	80.0%
Day 7	2010-1629	Blank	ND	ND	ND	N/A
	2010-1630	QC spk	0.902	0.907	0.905	90.5%
	2010-1631	SPK 1	0.889	0.842	0.866	86.6%
	2010-1632	Spk 2	0.903	0.848	0.876	87.6%
Day 14	2010-1633	Blank	ND	ND	ND	N/A
	2010-1634	QC spk	1.21	1.23	1.22	122%
	2010-1635	SPK 1	0.984	0.849	0.917	91.7%
	2010-1636	Spk 2	0.968	0.878	0.923	92.3%

Appendix 3 Storage Stability Study continued:

Day 21	2010-1637	Blank	ND	ND	ND	N/A
	2010-1638	QC spk	0.977	0.959	0.968	96.8%
	2010-1639	SPK 1	0.766	0.739	0.753	75.3%
	2010-1640	Spk 2	0.834	0.787	0.811	81.1%
Day 28	2010-1641	Blank	ND	ND	ND	N/A
	2010-1642	QC spk	0.953	0.899	0.926	92.6%
	2010-1643	SPK 1	0.887	0.875	0.881	88.1%
	2010-1644	Spk 2	0.994	0.914	0.954	95.4%

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Elaine Wong
Environmental Program Manager I

Date

Title: Determination of Pyrethroids in Sediment Water Using Triple Quadrupole GC/MS/MS

1. Scope:

This section method (SM) documents a selective pyrethroid analysis in sediment water and is followed by all authorized EMON personnel. This method uses the triple quadrupole to improve sensitivity and enables the lowering of the reporting limit over the previous method which used the ECD and MSD.

2. Principle:

The SM describes the method for determination of resmethrin, bifenthrin, fenpropathrin, lambda cyhalothrin epimer, lambda cyhalothrin, permethin cis, permethrin trans, cyfluthrin, cypermethrin, fenvalerate/ esfenvalerate and deltamethrin in sediment water. The pyrethroids are extracted from the sediment water using liquid-liquid extraction with hexane. The extracts are concentrated and then cleaned up with florisil before being analyzed with a gas chromatography equipped with triple quadrupole detector. The reporting limit is 10 ppt for resmethrin, 2 ppt for bifenthrin and 5 ppt for all the rest of the compounds.

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Hexane is a flammable and toxic solvent; it should be handled with care in a ventilated area.

4. Interferences:

There were no interferences at the time of validation for the background water provided.

5. Apparatus and Equipment:

5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)

5.2 Nitrogen evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)

5.3 Balance, (Mettler PC 4400 or equivalent)

5.4 Vortex-vibrating mixer

5.5 Gas Chromatograph equipped with a triple quadrupole

6. Reagents and Supplies:

- | | | |
|------|---|-----------------|
| 6.1 | Bifenthrin | CAS#42576-02-3 |
| 6.2 | Fenpropathrin | CAS#39515-41-8 |
| 6.3 | Lambda cyhalothrin epimer | CAS# unknown |
| 6.4 | Lambda cyhalothrin | CAS#91465-08-06 |
| 6.5 | Permethrin cis | CAS#54774-45-7 |
| 6.6 | Permethrin trans | CAS#51877-74-8 |
| 6.7 | Cyfluthrin | CAS#68369-37-5 |
| 6.8 | Cypermethrin | CAS#52315-07-8 |
| 6.9 | Fenvalerate | CAS#51630-58-1 |
| 6.10 | Deltamethrin | CAS#52918-63-5 |
| 6.11 | Resmethrin | CAS#10453-86-8 |
| 6.12 | Hexanes, nanograde or equivalent pesticide grade | |
| 6.13 | Diethylether, nanograde or equivalent pesticide grade | |
| 6.14 | Separatory funnel, 2 L | |
| 6.15 | Boiling flask, 500 mL | |
| 6.16 | Sodium Sulfate, ACS grade | |
| 6.17 | Funnels, short stem, 60°, 10 mm diameter | |
| 6.18 | Glass wool, Pyrex® fiberglass slivers 8 microns | |
| 6.19 | Beaker, 1 L | |
| 6.20 | Florisil SPE cartridge, 2 grams with 20 mL reservoir | |
| 6.21 | Volumetric Pipette, 1 mL | |
| 6.22 | Test tube, 50 mL | |
| 6.23 | Test tube, 15 mL | |
| 6.24 | Disposable Pasteur pipettes, and other laboratory ware as needed | |
| 6.25 | Recommended analytical columns:
Varian –VF-5ms arylene stabilized phase equivalent to 5% phenyl, 95% dimethylpolysiloxane fused silica column, 30 m x 0.25 mm id x 0.25 um film thickness. | |

7. Standards Preparation:

- 7.1 The individual pyrethroid stock standards of 1.0 mg/mL were obtained from the CDFA/CAC Standards Repository. The standards were diluted to 10 µg/mL with hexanes for identification purposes.

A combination standard of 10 µg/mL was prepared from individual mg/mL standards with acetone to be used for fortification. Another 10 µg/mL combination standard was prepared in hexanes and was diluted to the following

concentrations: 0.005, 0.01, 0.025, 0.05, 0.1, 0.2, 0.5 µg/mL in hexanes for instrument calibration. The calibration standards are added to blank matrix extracts to correct for matrix background response enhancement.

7.2 Keep all standards in the designated refrigerator for storage.

7.3 The expiration date of each standard is six months from the preparation date.

8. Sample Preservation and Storage:

Store all samples waiting for extraction in a separate refrigerator (32-40 °F)

9. Test Sample Preparation:

9.1 Background Preparation

The Department of Pesticide Regulation (DPR) provided the sediment water for background to be used in method validation and QC. The sediment water was prepared by adding 5 g of soil to approximately a liter of American river water.

9.2 Spike

Take a liter of background sediment water from refrigerator and allow it to come to room temperature. Fortify at a level requested by client. After fortification mix well and process same as samples.

9.3 Test Sample Extraction

9.3.1 Remove water samples from refrigerator and allow samples to come to room temperature before weighing them. Record weight.

9.3.2 Transfer the water sample to a 2 L separatory funnel leaving as much of the sediment as possible in the sample bottle.

9.3.3 Add 60 mL of hexanes to the sample bottle and manually shake for 30 seconds.

9.3.4 Transfer hexane and sediment into the separatory funnel and shake for 2 min., venting frequently.

- 9.3.5 Allow the layers to separate, drain the lower aqueous layer into a 1L beaker. Pour the hexane layer through a funnel containing a plug of glasswool and approximately 40 g sodium sulfate into a 500 mL boiling flask.
- 9.3.6 Transfer the water from the beaker into the separatory funnel and repeat steps 9.3.3 – 9.3.6 two more times shaking for 1 min. Combine the extracts in the same boiling flask. Record sample bottle weight.
- 9.3.7 Rotary evaporate to ~ 5 mL under vacuum at approximately 20-24 inch Hg in a water bath at 42-45° C.
- 9.3.8 Transfer the extract to a 15 mL test tube. Rinse flask 3 times with approximately 2 mL of hexane and transfer each rinsate to the same test tube.
- 9.3.9 Place the test tube on a nitrogen evaporator under a gentle stream of nitrogen with water bath set at 40-45° C and concentrate to ~ 2 mL final volume.

Cleanup

- 9.3.10 Condition a 2 g florisil SPE cartridge with 10 mL of 15% diethylether in hexane followed by 20 mL hexane. Do not allow cartridges to go to dryness.
- 9.3.11 Carefully load the sample extract onto the conditioned florisil SPE cartridge. Rinse the tube that previously contained the extract twice with 2 mL hexane. Add rinses to florisil cartridge.
- 9.3.12 Elute the pesticides from the cartridge with 30 mL of 15% diethylether in hexane and collect in a 50 mL tube.
- 9.3.13 Evaporate the sample eluants to dryness under a gentle stream of nitrogen in a 40-45° C water bath.
- 9.3.14 Pipet 1mL of hexane into the test tube and vortex well. Vial extract into 2 autosampler vials with inserts.

10. Instrument Calibration:

- 10.1 The calibration standards are added to blank matrix extracts to correct for matrix background response enhancement.
- 10.2 The calibration standard curve consists of a minimum of three levels. The recommended concentrations levels of standards are 0.001, 0.005, 0.01, 0.025, 0.05, 0.1, 0.2, or 0.5 µg/mL. Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995.

11. Analysis:

11.1 Injection Scheme

The instrument may need to be conditioned with a matrix blank or old sample before running the following sequence of Standard Curve, Hexane, Matrix Blank, Matrix Spike, Test Samples (maximum of 10 – 12) and Standard Curve.

11.2 GC-Triple Quadrupole Instrumentation

11.2.1 Gas Chromatograph: Varian CP-3800

Column: Varian Factor Four VF-5ms 30M x 0.25mm x 0.25µm.

Temperature Program: initial column temperature 80 °C, hold 1 min., ramp at 40 °C/min. to 180 °C hold for 0 min., ramp at 5 °C/min. to 305 °C hold for 0.5 min..

Injector Temperature: 250°C

Injection Volume: 1 µL

Carrier Gas: Helium 1mL/min.

Triple Quadrupole: Varian Triple Quad 320-MS

Ionization: Positive Electron Impact

Transfer Line: 300°C

Source Temp: 200°C

Collision Gas: Argon @ 1.8 mTorr

Compound	Retention Time (min.)	Precursor Ion	Product Ion	Collision Energy/-ev
Resmethrin 1	16.3	171	128,143	12
Resmethrin 2	16.5	171	128,143	12
Bifenthrin	17.3	181	166	15
Fenpropathrin	17.6	265	210	15
λ Cyhalothrin epimer	18.8	208	181	10
λ Cyhalothrin	19.2	208	181	10
Permethrin cis	20.7	183	168	23
Permethrin trans	20.9	183	168	23
Cyfluthrin 1,	21.7	226	206	15
Cyfluthrin 2	21.9	226	206	15
Cyfluthrin 3	22.0	226	206	15
Cyfluthrin 4	22.1	226	206	15
Cypermethrin 1,	22.3	181	152	20
Cypermethrin 2	22.5	181	152	20
Cypermethrin 3	22.6	181	152	20
Cypermethrin 4	22.7	181	152	20
Fenvalerate	24.2	167	125	15
Esfenvalerate	24.4	167	125	15
Deltamethrin	25.5	253	174	10

12. Quality Control:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 sediment water samples are spiked at 5 ppt except resmethrin, which was spiked at 10 ppt and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicates used to determine the MDL, t=3.143. The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The reporting limit for resmethrin is 10 ppt, bifenthrin is 2 ppt and for all other compounds is 5ppt.

12.3 Method Validation

The method validation consisted of three sample sets. Each set included three levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the pyrethroids are tabulated in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation for each analyte. The upper and lower control limits are set at ± 3 standard deviations of the % recovery, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 per cent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results exceed the calibration curve.

13. Calculations:

Lambda cyhalothrin/epimer, cyfluthrin, cypermethrin and fenvalerate are expressed as the sum of their isomers. Therefore, the total residues should be calculated using the sum of their peak responses.

Quantitation is based on external standard (ESTD) calculation using either the peak area or height. The MSD uses linear regression fit, with all levels weighted equally. Alternatively, at chemist discretion, concentrations may be calculated using the response factor for the standard whose value is closest to the level in the sample.

$$\text{ppt} = \frac{(\text{sample peak area or ht}) \times (\text{std conc}) \times (\text{std vol. Injected}) \times (\text{final vol of sample})(1000)(1000)}{(\text{std.peak area or ht}) \times (\text{sample vol injected}) \times (\text{sample wt (g)})}$$

14. Reporting Procedure:

Sample results are reported in accordance with the client's analytical laboratory specification sheets.

15. Discussion:

15.1 This method was developed to lower the reporting limit for the pyrethroids by using triple quadrupole mass spectrometry. The only change from the previous method EMON-SM-05-003 is the instrumentation. Since the extraction procedure is the same as the previous method a reduced number of spikes were analyzed for validation.

15.2 Negative chemical ionization (NCI) in selected ion monitor mode was also tried for the pyrethroids and showed some promise for all compounds except resmethrin which provided no signal. Method detection limits and validation resembled those found in EI mode. Future samples that have high background noise will be analyzed by both techniques since in chemical ionization mode the background noise has a different chemical origin and might offer some improvement. In the case of the background matrix provided for the QC there was little benefit observed by running samples under CI mode.

15.3 The sample matrix may require that the injector liner be changed more frequently and the column trimmed to maintain sensitivity. The ion volume and the source may also need to be cleaned more frequently.

15.4 This method was adapted from the methods listed in the references below.

16. References:

16.1 J. White, *Analysis of Pyrethroids in Sediment Water* Emon-SM-05-003, 2006, California Department of Food and Agriculture, Center for Analytical Chemistry, Environmental Analysis Section, 3292 Meadowview Road, Sacramento, California 95832

- 16.2 J. You, D.P. Weston, M. J. Lydy, *A Sonication Extraction Method for the Analysis of Prethroid, Organophosphate, and Organochlorine Pesticides from Sediment by Gas Chromatography with Electron-Capture Detection*, Archives Environmental Contamination and Toxicology 47, 141-147 (2004)
- 16.3 J. You, M. J. Lydy, *Evaluation of Desulfuration Methods for Pyrethroid, Organophosphate, and Organochloride Pesticides in Sediment with High Sulfur Content*, Archives Environmental Contamination and Toxicology 47, 148 -153 (2004)
- 16.4 J. White, H. Feng, Determination of Pyrethroids in Sediment Water, EMON-SM-52-7.1, 2004, California Department of Food and Agriculture, Center for Analytical Chemistry, Environmental Monitoring Laboratory, 3292 Meadowview Road, Sacramento, California 95832

Appendix 1

The determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Spike level is 5 ppt for all compounds except Resmethrin, which is 10 ppt

	Bifenthrin	Fenopropathrin	λ cyhalothrin Epimer/ λ cyhalothrin	Permethrin cis	Permethrin trans	Cyfluthrin
	ppt	ppt	ppt	ppt	ppt	ppt
blk sed	n/d	n/d	n/d	n/d	n/d	n/d
spk1	5.74	5.51	5.87	5.72	4.10	5.68
spk2	4.98	4.57	5.23	6.04	4.06	5.23
spk 3	5.57	5.00	5.46	5.54	4.39	5.46
spk 4	5.36	4.71	5.71	5.40	3.85	5.53
spk 5	5.46	5.58	6.76	6.01	4.75	6.32
spk 6	5.02	4.42	5.93	5.29	4.24	5.90
spk 7	4.86	5.07	5.39	5.36	3.92	5.04
Std dev	0.29	0.42	0.55	0.34	0.33	0.47
MDL	0.91	1.32	1.74	1.05	1.05	1.46
RL	2 ppt	5 ppt	5 ppt	5 ppt	5 ppt	5 ppt

	Cypermethrin	Fenvalerate/ Esfenvalerate	Deltamethrin	Resmethrin
	ppt	ppt	ppt	ppt
blk sed	n/d	n/d	n/d	n/d
spk1	6.27	5.11	4.86	9.22
spk2	4.99	4.44	5.29	8.68
spk 3	6.31	5.92	4.50	10.70
spk 4	5.02	4.75	4.93	10.20
spk 5	5.71	5.41	6.29	10.19
spk 6	5.45	4.99	5.14	12.79
spk 7	5.65	4.82	5.36	9.56
Std dev	0.49	0.53	0.56	1.33
MDL	1.54	1.66	1.77	4.18
RL	5 ppt	5 ppt	5 ppt	10 ppt

Appendix 2

Method Validation Data and Control Limits

Analyte	Spike ppt	Recovery % set 1	set 2	set 3		
Bifenthrin	5	88.6	93.0	75.8	Mean:	80.2
	10	79.9	83.3	75.7	SD:	6.81
	25	72.8	75.6	76.8	UCL:	101
					LCL:	59.7
Fenpropathrin	5	91.0	91.6	108	Mean:	91.0
	10	83.8	86.9	76.1	SD:	9.39
	25	88.8	102	90.8	UCL:	119
					LCL:	62.8
λ cyhalothrin /epimer	5	97.2	101	76.2	Mean:	87.4
	10	84.4	92	76.2	SD:	8.59
	25	90.4	85.2	83.6	UCL:	113
					LCL:	61.6
Permethrin cis	5	98.2	132	71.2	Mean:	93.9
	10	95.7	85.0	93.5	SD:	16.8
	25	82.4	88.4	98.8	UCL:	144
					LCL:	43.6
Permethrin trans	5	91.8	129	78.4	Mean:	96.2
	10	108	87.4	92.6	SD:	14.5
	25	92.8	91.6	94.0	UCL:	140
					LCL:	52.7
Cyfluthrin	5	105	120	103	Mean:	102
	10	101	103	89.4	SD:	8.43
	25	95.2	103	96.8	UCL:	127
					LCL:	76.5
Cypermethrin	5	102	113	74.6	Mean:	95.8
	10	101	96.1	101	SD:	10.7
	25	90.0	96.4	88.4	UCL:	128
					LCL:	63.5
Fenvalerate / Esf	5	96.8	122	93.8	Mean:	95.2
	10	90.7	100	89.9	SD:	11.0
	25	86.8	90.0	86.4	UCL:	128
					LCL:	62.2

Appendix 2 continued

Method Validation Data and Control Limits

Analyte	Spike ppt	Recovery % set 1	set 2	set 3		
Deltamethrin	5	110	104	95.6	Mean:	96.4
	10	93.5	99.0	64.5	SD:	13.4
	25	95.2	109	96.4	UCL:	137
					LCL:	56.2
Resmethrin	15	85.3	74.0	69.3	Mean:	74.5
	25	80.7	63.7	67.0	SD:	8.4
	50	79.8	65.7	84.8	UCL:	99.7
					LCL:	49.3

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Date

Title: Determination of Bensulide and Imidacloprid in Surface Water

1. Scope:

This section method (SM) documents Bensulide and Imidacloprid pesticide Residue analysis in surface water. It is to be followed by all authorized section personnel.

2. Principle:

The surface water sample is extracted with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated to almost dryness on a rotary evaporator and diluted to a final volume of 1.0 mL with methanol. The extract is then analyzed by an Ultra Performance Liquid Chromatography (UPLC) coupled to a triple quadrupole using electrospray ionization in positive ion mode.

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

3.3 All solvents should be handled with care in a ventilated area.

4. Interferences:

There is no known interference for this analysis.

5. Apparatus and Equipment:

5.1 Rotary evaporator (Büchi/Brinkman or equivalent)

5.2 Nitrogen evaporator (Meyer N-EVAP Organomation Model # 112 or equivalent)

5.3 Vortex-vibrating mixer

5.4 Balance (Mettler PC 4400) or equivalent

5.5 Liquid Chromatograph equipped with an ion trap mass spectrometer

6. Reagents and Supplies

- 6.1 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.2 Methanol, nanograde or equivalent pesticide grade
- 6.3 Anhydrous Sodium Sulfate, granular
- 6.4 Bensulide CAS# 741-58-2
- 6.5 Imidacloprid CAS# 138261-41-3
- 6.6 Conical tube with glass stopper, 15-mL graduated, 0.1 mL subdivision
- 6.7 Separatory funnel, 2 L
- 6.8 Boiling flask, 500 mL
- 6.9 Funnel, long stem, 10 mm diameter
- 6.10 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.11 Recommended analytical columns: Waters Symmetry HSS T3 1.8 μ m 2.1x100 mm column

7. Standards Preparation:

- 7.1 The individual bensulide and Imidacloprid stock standards of 1.0mg/mL were obtained from the CDFA/CAC Environmental Analysis Standards Repository. The standards were diluted to 10 μ g/mL with methanol for identification purposes. A combination standard of 10 μ g/mL was prepared from the individual mg/mL standards in methanol. The combination 10 μ g/mL standard was used to dilute the following concentrations: 0.025, 0.05, 0.1, 0.25, 0.5, 1.0 μ g/mL in methanol.
- 7.2 Store standards according to manufacturing requirement. Keep all standards in designated refrigerator for storage.
- 7.3 The expiration date of working standard is six months from the preparation date of the stock standard

8. Sample Preservation and Storage:

All water samples and sample extracts shall be stored in the refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Sample Preparation

- 9.1.1 Remove samples from refrigerator and allow samples to come to room temperature before extraction.

9.1.2 Preparation of matrix blank and matrix spike:

The Department of Pesticide Regulations (DPR) provided the background water for matrix blank and spikes.

- 9.1.2.1 Matrix blank: Weigh out approximate 1000 g of background water and follow the test sample extraction procedure.
- 9.1.2.2 Matrix spike: Weigh out approximate 1000 g of background water. Spike a client requested amount of bensulide/imidacloprid into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.2 Test Sample Extraction

- 9.2.1 Record the weight of the whole bottle water sample to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.
- 9.2.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.
- 9.2.3 After phases have separated, drain lower methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glasswool, into a 500 mL boiling flask.
- 9.2.4 Repeat steps 9.2.2 & 9.2.3 two more times using 80 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.
- 9.2.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.2.6 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 - 20 inch Hg vacuum. Add 2 - 4 mL of methanol and rotoevaporate to 1 - 2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.2.7 Rinse flask 3 more times with 2 - 4 mL of methanol and transfer each rinse to the same test tube.

9.2.8 Evaporate the extract to a volume slightly less than 1 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with methanol, mix well and transfer into two autosampler vials.

9.2.9 Submit extract for LC-MS analysis.

10. Instrument Calibration:

10.1 A calibration standard curve consists of minimum of three levels. Standard concentrations of 0.025, 0.05, 0.1, 0.25, 0.5 or 1.0 $\eta\text{g}/\mu\text{L}$ are recommended. Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995.

11. Analysis:

11.1 UPLC-MS/MS

11.1.1 UPLC instrument: Waters Acquity Ultra Performance LC
Column: Waters Acquity HSS T3 1.8 μm 2.1x100 mm
Column Temperature: 50°C
Mobile Phase: Gradient
Solvent 1: Water + 4% acetic acid
Solvent 2: Methanol + 4% acetic acid
Gradient:

<u>Time (min)</u>	<u>Flow rate</u>	<u>Solvent 1</u>	<u>Solvent 2</u>
0	0.50	90.0	10.0
0.5	0.50	90.0	10.0
3.5	0.50	10.0	90.0
4.5	0.50	10.0	90.0
5.0	0.50	90.0	10.0
6.0	0.50	90.0	10.0

Injection Volume: 1.0 μL

11.1.2 Mass Spectrometry and Operating Parameters

Model: Waters Xevo Triple Quadrupole
Ion ProbeType: Electrospray Ionization (ES)
Ion Mode: ESI (+)
Desolvation Temp: 500 °C
Source Temp: 150 °C

Compound	Retention Time (min)	Precursor ion	Product Ion	Dwell (s)	Cone(V)	Collision Energy/-ev
Imidacloprid	2.51	256.08	175.02	0.025	24.0	16.0
			209.1	0.025	24.0	16.0
Bensulide	3.94	398.16	158.01	0.061	14.0	34.0
			314	0.061	46.0	30.0

Quantitation ions are in bold.

12. Quality Control:

12.1 Method Detection Limits (MDL)

The method detection limit refers to the lowest concentration of analyte that a method can detect reliably. To determine the MDL, 7 replicate water samples are spiked at 0.10 ppb. The standard deviation from the spiked sample recoveries are used to calculate the MDL for the analyte using the following equation:

$$MDL = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicate used to determine the MDL, t=3.143.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting limit (RL):

The reporting limit (RL) refers to the level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. Per client agreement, the RL is chosen in a range 1-5 times the MDL. The reporting limit for Bensulide is 0.04ppb and Imidacloprid is 0.05ppb

12.3 Method Validation

The method validation for bensulide and Imidachloprid consisted of three sample sets. Each set included five levels of fortification and a method blank. All spikes

and method blanks were processed through the entire analytical method. Spikes levels and recoveries for bensulide and Imidacloprid are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation. The upper and lower control limits are set at ± 3 standard deviation of the % recovery, shown in Appendix 2. The control chart range generated from this validation data was narrower than that of the previous method for Bensulide. It was decided that the control charts would be used but the upper and lower control limits would be set with the limits from the previous methods Bensulide 56.7 – 130.6 and Imidacloprid 77.2-121.9. The new data for Bensulide fit within these limits and the data for Imidacloprid was almost the same as the old control limits.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 per cent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. Calculations:

Quantitation is based on external standard (ESTD) calculation using either the peak area or height. The software uses a linear or quadratic curve fit, with all levels weighted equally. Alternatively, at chemist discretion, results may be calculated using the response factor for the standard whose value is closest to the level in the sample.

$$\text{ppb} = \frac{(\text{sample peak ht. or area}) (\text{std. conc.}) (\text{std. vol. injected}) (\text{sample final vol., (mL)}) (1000 \mu\text{L/mL})}{(\text{std. peak ht. or area}) (\text{sample vol. injected}) (\text{sample wt., g})}$$

14. Reporting Procedure:

Sample results are reported out according to the client's analytical laboratory specifications sheets.

15. Discussion:

This SOP combines the analysis of bensulide and Imidacloprid into a single method. In the past both compounds were extracted and analyzed separately.

16. References:

- 16.1. Lee, Paul; *Determination of Bensulide in Surface Water Using Liquid Chromatography Mass Spectrometry*, 2002, Environmental Monitoring method, Center for Analytical Chemistry, CDFA.
- 16.2. Hernandez, Jorge; *HPLC Determination of Imidacloprid in Surface and Well Water*, 2001, Environmental Monitoring method, Center for Analytical Chemistry, CDFA.

APPENDIX I

The determination of Method Detection Limit (MDL) data and Reporting Limit (RL) for Bensulide and Imidacloprid in surface water:

Spk\Analyte	Bensulide ppb	Imidacloprid ppb
0.1 ppb spk 1	0.105	0.112
0.1 ppb spk 2	0.107	0.094
0.1 ppb spk 3	0.190	0.100
0.1 ppb spk 4	0.105	0.093
0.1 ppb spk 5	0.106	0.080
0.1 ppb spk 6	0.097	0.095
0.1 ppb spk 7	0.097	0.073
SD	0.00629	0.00125
MDL	0.0198	0.0394
RL	0.04	0.05

APPENDIX II

Method Validation Data and Control Limit

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	%	%
Bensulide	0.1	109	112	92.2	Mean:	102
	0.2	113	104	106	SD:	7.43
	0.5	106	89.2	97.4	UCL:	124.4
	1.0	103	108	102	UWL:	117
	2.0	95.0	103	92.0	LWL:	87.3
					LCL:	79.8
Imidacloprid	0.1	103	97.0	105	Mean:	100
	0.2	108	104	103	SD:	7.78
	0.5	104	87.0	103	UCL:	123.5
	1.0	104	108	108	UWL:	115.7
	2.0	90.0	93.0	84.9	LWL:	84.6
					LCL:	76.8

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Title: Determination of Methoxyfenozide and Tebufenozide in Surface Water by Ultra Performance Liquid Chromatography Coupled to Tandem Mass Spectrometry

1. Scope:

This section method (SM) provides stepwise procedure for methoxyfenozide and tebufenozide analysis in surface water. It is followed by all authorized EA personnel.

2. Principle:

The methoxyfenozide and tebufenozide are extracted from the surface water sample with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated on a rotary evaporator and then a solvent exchange is performed with methanol. The extract is concentrated to a final volume of 1 mL and then vialled into an autosampler vial for analysis on Ultra Performance Liquid Chromatography (UPLC) coupled to a positive electrospray ionization triple quadrupole mass spectrometry (ES-LC/MS/MS).

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

4. Interferences:

There were no matrix interferences for methoxyfenozide and tebufenozide at the time of method development.

5. Apparatus and Equipment:

5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)

5.2 Nitrogen Evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)

5.3 Balance (Mettler PC 4400 or equivalent)

5.4 Vortex-vibrating mixer

5.5 UPLC equipped with a triple quadrupole mass spectrometry and ES ion source.

6. Reagents and Supplies:

- 6.1 Methoxyfenozide CAS#161050-58-4
- 6.2 Tebufenozide CAS#112410-23-8
- 6.3 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.4 Water, MS grade, Burdick & Jackson or equivalent
- 6.5 Methanol, MS grade, Burdick & Jackson or equivalent
- 6.6 Formic Acid, HPLC grade
- 6.7 Ammonium formate, reagent grade or equivalent
- 6.8 Separatory funnel, 2 L
- 6.9 Boiling flask, 500 mL
- 6.10 Sodium Sulfate, ACS grade
- 6.11 Funnels, long stem, 60°, 100 mm I.D.
- 6.12 Volumetric Pipette, 0.5 mL
- 6.13 Graduated conical tubes with glass stopper, 15 mL
- 6.14 Glass wool, Pyrex® fiber glass slivers 8 microns
- 6.15 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.16 Recommended analytical column:
Waters Acquity BEH C18 1.7µm, 2.1 x 100 mm
- 6.17 Aqueous Solution: For 500 mL, mix 470 ± 2mL water, 25 ± 0.5 mL methanol, 4.50 ± 0.25 mL 1 M ammonium formate and 0.5 ± 0.05 mL formic acid.
- 6.18 Organic Solution: For 500mL, mix 450 ± 2mL methanol and 45 ± 0.5 mL water with 4.50 ± 0.25 mL 1 M ammonium formate and 0.5 ± 0.05 mL formic acid.

7. Standards Preparation:

- 7.1 The individual stock standards of 1.0 mg/mL were obtained from the CDFA/CAC Standards Repository. The standards were diluted to 10 µg/mL with methanol for identification purposes.

A combination standard of 1 µg/mL was prepared from the individual 10 µg/mL standards with methanol. The standard was also used to dilute the following concentrations: 0.025, 0.05, 0.1, 0.25 and 0.5 µg/mL in methanol for instrument calibration.

- 7.2 Keep all standards in the designated refrigerator for storage.
- 7.3 The expiration date of each standard is six months from the preparation date.

8. Sample Preservation and Storage:

Store all samples waiting for extraction in a separate refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Background Preparation

The Department of Pesticide Regulations (DPR) provides the background water for matrix blank and spikes.

9.2 Preparation of blank and spike

Matrix blank: Weigh out 500 g of background water and follow the test sample extraction procedure.

Matrix spike: Weigh out 500 g of background water. Spike a client requested amount of insecticides into the background water, mix well and let it stand for one minute. Follow the test sample extraction procedure.

9.3 Test Sample Extraction

9.3.1 Remove samples from the refrigerator and allow them to reach ambient temperature.

9.3.2 Mix sample well before weighting aliquot. Weight 500 ± 0.1 g of water samples by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.

9.3.3 Shake with 80 ± 5 mL of methylene chloride for 1 minute. Vent frequently to relieve pressure.

9.3.4 After phases have separated, drain the lower methylene chloride layer through 25 ± 4 g of anhydrous sodium sulfate and glass wool into a 500 mL boiling flask.

9.3.5 Repeat steps 9.3.3 & 9.3.4 two more times using 60 ± 5 mL of methylene chloride and shake for 1 minute each time. Combine the extracts in the same boiling flask.

- 9.3.6 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.3.7 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 – 20 inch Hg vacuum. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.3.8 Rinse flask 3 more times with 2 - 4 mL of methylene chloride and transfer each rinse to the same test tube.
- 9.3.9 Evaporate the sample extract to dryness in a water bath at 40 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 0.5 mL with methanol, mix well and transfer to an autosampler vial. Submit extract for LC-MS analysis.

10. Instrument Calibration:

- 10.1 The calibration standard curve consists of a minimum of three levels. The lowest level must be at or below the corresponding reporting limit. The current working standard levels are 0.025, 0.05, 0.1, 0.25, and 0.5 µg/mL.
- 10.2 Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995, with all levels weighted 1/x.

11. Analysis:

11.1 Injection Scheme

The LC-MS needs to be conditioned with standard or a sample extract 2 to 5 runs before running the following sequence: A set of calibration standards, a matrix blank, a matrix spike, a set of up to 12 test samples, then a set of standards, etc.

11.2 UPLC-MS/MS

- 11.2.1 UPLC Instrument: Waters Acquity Ultra Performance LC
Column: Waters Acquity BEH C18 1.7µm, 2.1 x 100 mm
Column Temperature: 60 °C
Mobile Phase: Gradient

Solvent 1: Aqueous Solution
Solvent 2: Organic Solution
Gradient:

<u>Time(min)</u>	<u>Flow rate (mL/min)</u>	<u>Solvent 1</u>	<u>Solvent 2</u>
0	0.60	90.0	10.0
0.5	0.60	90.0	10.0
7.00	0.60	10.0	90.0
7.80	0.60	10.0	90.0
8.00	0.60	90.0	10.0
8.50	0.60	90.0	10.0

Injection Volume: 1.0 µL

11.2.2 Mass Spectrometry and Operating Parameters

Model: Waters Xevo Triple Quadrupole
Ion ProbeType: Electrospray Ionization (ESI)
Ion Mode: Positive
Source Temp: 150 °C

Compound	Retention Time (min)	Precursor ion	Product Ion	Dwell (s)	Cone(V)	Collision Energy/-ev
Methoxyfenozide	5.80	369.24	149.05	0.128	12.0	16.0
		369.24	313.13	0.128	12.0	6.00
Tebufenozide	6.33	353.25	133.05	0.128	14.0	16.0
		353.25	297.15	0.128	14.0	6.00

Quantitation ions are in bold.

12. Quality Control:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 surface water samples are spiked at 0.1ppb and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with $n-1$ degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the $n=7$ replicates used to determine the MDL, $t=3.143$.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is chosen in a range 1-5 times the MDL, as per client agreement. The reporting limit for methoxyfenozide and tebufenozide is 0.05 ppb.

12.3 Method Validation

The method validation consisted of five sample sets. Each set included five levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the analytes are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation for each analyte. The upper and lower warning and control limits are set at ± 2 and 3 standard deviations of the percent recovery, respectively, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 percent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. Calculations:

Quantitation is based on an external standard (ESTD) calculation using either the peak area or height. The triple quadrupole LCMS software used a linear curve fit, with all levels weighted 1/x. Alternatively, at the chemist's discretion, sample results may be calculated using the response factor for the standard.

$$\text{ppb} = \frac{(\text{sample peak area or ht}) \times (\text{std conc.}) \times (\text{std vol. injected}) \times (\text{final vol. of sample})(1000 \mu\text{L/mL})}{(\text{std peak area or ht}) \times (\text{sample vol. injected}) \times (\text{sample wt (g)})}$$

14. Reporting Procedure:

Sample results are reported out according to the client's analytical laboratory specification sheets.

15. Discussion and References:

15.1 A storage stability study was done with this project. The storage stability study consisted of a 1.0 ppb spike level and 3 replicates over a 28 day period. Twelve liters of background well water were spiked and then transferred to twelve one liter amber bottles. These spiked samples were stored in the refrigerator until analyzed on 0, 2, 4, 7, 15, 21 and 28 days. Along with the storage spikes, a blank and method control spike were also extracted. This storage study showed no significant degradation for these compounds within 28 days. Results for the storage study are shown in Appendix 3.

15.2 Solid phase extraction using an Oasis HLB 500mg cartridge was also tried. A 500 mL surface water sample was filtered through a glass fiber filter. The filtered sample was passed through a solid phase extraction HLB cartridge and methoxyfenozide and tebufenozide were eluted from the solid phase cartridge with acetonitrile. The extract was concentrated to just dryness with nitrogen in a heated water bath, and then adjusted to a 0.5 mL volume with methanol. Recoveries were good and in the 80-90% range. There were some concerns about filtering away the sediment that could be in more turbid samples and the possible loss of methoxyfenozide and tebufenozide that might occur during that process. It was decided to retain liquid /liquid extraction as the primary extraction process.

16. References:

- 16.1 Hall, Gregory; Engebretson, Jo; Hengel, Mathew J. and Shibamoto, Takayuki
“Analysis of Methoxyfenozide Residues in Fruits, Vegetables, and Mint by Liquid
Chromatography-Tandem Mass Spectrometry (LC-MS/MS), J. Agric. Food
Chem. 2004, 52, 672-676
- 16.2 “Crop Protection Handbook, 2010”, MeisterPro Executive Office 27722 Euclid
Ave., Willoughby, OH

Appendix 1

The Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Lab #	Spk\Analyte	Methoxyfenozide	Tebufenozide
2011-1806	blk	nd	nd
2011-1807	0.1ppb spk 1	0.086	0.088
2011-1808	0.1ppb spk 2	0.087	0.087
2011-1809	0.1ppb spk 3	0.086	0.087
2011-1810	0.1ppb spk 4	0.089	0.089
2011-1811	0.1ppb spk 5	0.089	0.088
2011-1812	0.1ppb spk 6	0.092	0.092
2011-1813	0.1ppb spk 7	0.088	0.0088
	SD	0.00204	0.001822
Reported	MDL	0.00641	0.00573
	RL	0.05	0.05

All concentrations are expressed in ppb.

Appendix 3

Storage study Summary for Methoxyfenozide & Tebufenozide in Surface Water

Analyte \ Recovery %		Day 0	Day 2	Day 4	Day 7	Day 14	Day 21	Day 28
Methoxyfenozide	blank	ND	ND	ND	ND	ND	ND	ND
	QC spike		81.5%	85.9%	82.4%	84.7%	87.5%	84.7%
	Spike 1	85.5%	81.5%	81.3%	91.4%	87.2%	91.9%	86.7%
	Spike 2	91.5%	87.7%	88.3%	85.2%	82.5%	89.6%	87.8%
	Spike 3	86.6%	82.9%	87.8%	86.2%	82.5%	88.0%	83.4%
Tebufenozide	blank	ND	ND	ND	ND	ND	ND	ND
	QC spike		80.5%	85.2%	81.6%	84.2%	88.6%	84.2%
	Spike 1	86.1%	81.8%	81.5%	90.0%	87.1%	91.1%	87.7%
	Spike 2	90.5%	88.4%	85.8%	86.4%	82.6%	87.4%	85.3%
	Spike 3	85.7%	82.5%	87.9%	86.0%	81.3%	87.7%	79.4%

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Title: Determination of N-methylcarbamate Pesticides in Surface Water using High Performance Liquid Chromatography and Post-column derivatization

1. Scope:

This section method (SM) documents the selected N-methylcarbamate pesticides analysis in surface water by all authorized section personnel.

2. Principle:

The surface water sample is extracted with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated to almost dryness then diluted to a final volume of 0.40 mL with methanol. The extract is then analyzed by HPLC. The analytes are derivatized with OPA (ortho-phthalaldehyde) in a post column reaction and detected with a fluorescence detector. The reporting limit for this method is 0.05 ppb for all compounds.

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

3.3 All solvents should be handled with care in a ventilated area.

4. Interferences:

There are matrix interferences that cause quantitative problems. Therefore the calibration standards will be made up in appropriate matrix.

5. Apparatus and Equipment:

- 5.1 Rotary evaporator (Büchi/Brinkman or equivalent)
- 5.2 Nitrogen evaporator (Meyer N-EVAP Organomation Model # 112 or equivalent)
- 5.3 Vortex-vibrating mixer
- 5.4 Balance (Mettler PC 4400) or equivalent

5.5 HPLC with post column derivatization system and fluorescence detector.

6. Reagents and Supplies

- 6.3 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.4 Methanol, nanograde or equivalent pesticide grade
- 6.5 Anhydrous Sodium Sulfate, granular
- 6.6 Aldicarb Sulfoxide CAS# 1646-87-3
- 6.7 Aldicarb Sulfone CAS# 1646-88-4
- 6.8 Oxamyl CAS# 23135-22-0
- 6.9 Methomyl CAS# 16752-775
- 6.10 3-OH-Carbofuran CAS# 16655-82-6
- 6.11 Aldicarb CAS# 116-06-3
- 6.12 Carbofuran CAS# 1563-66-2
- 6.13 Carbaryl CAS# 63-25-2
- 6.14 Methiocarb CAS# 2032-65-7
- 6.15 Hydrolysis reagent (Pickering Laboratories CB130 or equivalent)
- 6.16 O-phthalaldehyde (Pickering Laboratories 012 or equivalent)
- 6.17 O-phthalaldehyde diluent (Pickering Laboratories CB910 or equivalent)
- 6.18 2-mercaptoethanol
- 6.19 OPA Reagent- Dissolve 100mg O-Phthalaldehyde in 10mL methanol. Add this mixture to 950 mL O-Phthalaldehyde diluent and mix well. Add 1 mL 2-mercaptoethanol and pour solution into reagent reservoir.
- 6.20 Conical tube with glass stopper, 15-mL graduated, 0.1 mL subdivision
- 6.21 Separatory funnel, 250 mL
- 6.22 Boiling flask, 500 mL
- 6.23 Funnel, long stem, 10 mm diameter
- 6.24 Nitrogen Evaporator, Organomation
- 6.25 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.26 0.2 μ nylon filters (Acrodisc 28143-274 or equivalent)
- 6.27 Recommended analytical columns:
Carbamate analysis C18 4.6mm ID X 250 mm. (Pickering Laboratories 1846250 or equivalent)

7. Standards Preparation:

- 7.1 Dilute the 1 mg/mL Carbamate standards obtained from the CDFFA/CAC Environmental Analysis Standards Repository with methanol to make up a series of

mixed working standards (see 10.2). These standards shall be prepared to cover the linear range from 0.0125 $\eta\text{g}/\mu\text{L}$ to 0.5 $\eta\text{g}/\mu\text{L}$ for the carbamate screen.

7.2 Store standards according to manufacturing requirement. Keep all standards in designated refrigerator for storage.

7.3 The expiration date of each mixed working standard is six months from the preparation date or same as stock standards, if sooner.

8. Sample Preservation and Storage:

All water samples and sample extracts shall be stored in the refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Sample Preparation

9.1.1 Remove samples from refrigerator and allow samples to come to room temperature before extraction.

9.1.2 Preparation of matrix blank and matrix spike:

The Department of Pesticide Regulations (DPR) provides the background water for matrix blank and spikes.

9.1.2.1 Matrix blank: Weigh out 100 grams of background water and follow the test sample extraction procedure.

9.1.2.2 Matrix spike: Weigh out 100 grams of background water. Spike a client requested amount of carbamate pesticides into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.2 Test Sample Extraction

9.2.1 Shake each sample then weigh out 100 grams of sample and transfer to a separatory funnel.

9.2.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.

- 9.2.3 After phases have separated, drain lower methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glasswool, into a 500 mL boiling flask.
- 9.2.4 Repeat steps 9.2.2 & 9.2.3 two more times using 100 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.
- 9.2.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.2.6 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 - 20 inch Hg vacuum. Pass sample through 0.2 μ filter into a calibrated 15 mL graduated test tube.
- 9.2.7 Rinse flask 2-3 more times with 2 - 4 mL of methylene chloride and filter the rinse into the same test tube.
- 9.2.8 Evaporate the extract to a volume slightly less than 0.5 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Add in approx. 1 mL methanol. Evaporate the extract to less than 300 μ L. Transfer extract to a calibrated vial insert. Wash the tube with a few drops of Methanol and add to insert. Adjust the final volume of 0.4 mL with methanol.
- 9.2.9 Submit extract for HPLC analysis.

10. Instrument Calibration:

10.1 A calibration standard curve consists of minimum of three levels. Standard concentrations of 0.0125, 0.025, 0.05, 0.1, and 0.5 η g/ μ L are recommended. Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995.

10.2 Compositions of calibration mixed standards are as follows:

CB-A Mixed Standard

Aldicarb Sulfoxide
Aldicarb Sulfone
Methomyl
3-Hydroxycarbofuran
Aldicarb
Carbofuran
Carbaryl

CB-B Mixed Standard

Oxamyl
Methiocarb

11. Analysis:

11.1 Injection Scheme

Follow the sequence of calibration standards, QC samples, test samples (maximum of 10-12 samples) and final calibration standards.

11.2 HPLC Instrumentation

11.2.1 Analyze carbamate pesticides by HPLC equipped with post column reaction module and a fluorescence detector.

11.2.2 Recommended instrument HPLC gradient::

	A= 1% methanol in water	B= acetonitrile
Time (min)	% A	%B
0.00	98.0	2.0
1.00	98.0	2.0
16.00	30.0	70.0
18.00	30.0	70.0
22.00	100.0	0.0
25.00	100.0	0.0
25.10	98.0	2.0
30.00	98.0	2.0

11.2.3 Injection volume 25 µL.

12. Quality Control:

12.1 Each set of samples shall have a matrix blank and minimum of one matrix spike sample.

12.2 The matrix blank should be free of target compounds.

12.3 The recoveries of the matrix spike shall be within the control limits.

12.3.1 When spike recoveries fall outside the control limits, the chemist must investigate the cause. The entire extraction set of samples is re-analyzed. If the spike recoveries fall within the limit, then the results from the re-analyzed samples shall be reported.

12.3.2 If the spike recoveries still fall outside the control limits, the client will be notified. The backup samples will be re-extracted for analysis.

12.4 The retention time should be within ± 2 percent of that of the standard.

12.5 The sample must be diluted if results fall outside the linear range of the standard curve.

12.6 Bracketing standard curves should have a percent change less than 20 % for all compounds.

12.7 Method Detection Limits (MDL)

The method detection limit refers to the lowest concentration of analyte that a method can detect reliably. To determine the MDL, 7 replicate water samples are spiked at 0.05 ppb for OP screen and 7 replicate water samples are spiked at 10 ppt for low level diazinon and chlorpyrifos. The standard deviation from the spiked sample recoveries are used to calculate the MDL for each analyte using the follow equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicate used to determine the MDL, t=3.143.

12.8 Reporting limit (RL):

The reporting limit (RL) refers to the level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. Per client agreement, the RL is chosen in a range 1-5 times the MDL except in special cases. (See 15.5)

MDL data and the RL are tabulated in Appendix IA and IB.

12.9 Method Validation Recovery Data and Control Limits:

12.9.1 The method validation consisted of three sample sets. Each set included five levels of fortification (0.0125, 0.025, 0.05, 0.1, 1.0 ppb) and a method blank. All spikes and method blank samples were processed through the entire analytical method.

12.9.2 Upper and lower warning and control limits are set at ± 2 and ± 3 standard deviations of the average % recovery, respectively.

12.10 Estimated Measurement Uncertainty:

12.11 Trend Identification

12.11.1 All matrix spike recoveries for carbamate analysis will be put into control

Charts and monitored for trends. Three trend characteristics will be evaluated at least bi-yearly by the supervisor or designee.

2 of 3 points above or below 2/3 of the UCL or LCL.

7 continuous points above or below the center line (CL)

14 points alternating above and below the CL.

12.11.2 When results indicate an out of control situation the supervisor or designee will indicate this on the control chart and take appropriate corrective action, which may include monitoring the results more closely to initiating a formal corrective action with root cause investigation.

13. Calculations:

Quantitation is based on external standard (ESTD) calculation using either the peak area or height. The software uses a linear or quadratic curve fit, with all levels weighted equally. Alternatively, at chemist discretion, concentrations may be calculated using the response factor for the standard whose value is closest to the level in the sample.

$$\text{ppb} = \frac{(\text{sample peak ht. or area}) (\text{std. conc.}) (\text{std. vol. injected}) (\text{sample final vol., (mL)})(1000 \mu\text{L/mL})}{(\text{std. peak ht. or area}) (\text{sample vol. injected}) (\text{sample wt., g})}$$

14. Reporting Procedure:

14.1 Identification of Analyte

For responses within calibration range, compare the retention time of the peaks with the retention time of standards. For positive results retention times shall not vary from the standards more than 2 percent.

14.2 Sample results are reported out according to the client's analytical laboratory specifications.

15. References:

Muth, G.L., Erro, F. A Rapid Carbamate Multiresidue Procedure of Vegetable Crops Environmental Contamination & Toxicology, 1980, 24, 759-765

Keith, Lawrence H., Principles of Environmental Analysis, Anal Chem, 1983, 55, 2210-2218

APPENDIX IA

The determination of Method Detection Limit (MDL) data and Reporting Limit (RL)

	Aldicarb sulfoxide	Aldicarb sulfone/Oxamyl	Methomyl	3-OH Carbofuran	Aldicarb	Carbofuran	Carbaryl	Methiocarb
MDL#1	0.02433	0.06784	0.02939	0.0322165	0.024847	0.02892	0.03208	0.03113
MDL#2	0.02126	0.05778	0.02545	0.02704	0.02244	0.0267	0.02718	0.02444
MDL#3	0.0248	0.06608	0.02709	0.03169	0.02316	0.02691	0.02924	0.02875
MDL#4	0.02172	0.05155	0.02367	0.02685	0.02164	0.02417	0.0245	0.03718
MDL#5	0.01686	0.05218	0.02204	0.02594	0.01776	0.02235	0.02388	0.02301
MDL#6	0.02388	0.05906	0.029	0.03161	0.02579	0.02815	0.02841	0.02617
MDL#7	0.026	0.06423	0.03	0.035	0.0245	0.03114	0.03286	0.02866
SD	0.00307	0.00651	0.00306	0.00343	0.00268	0.00294	0.00344	0.00473
3.1416 xSD	0.01026	0.01882	0.00967	0.01133	0.00871	0.00964	0.01038	0.01578
MDL	0.011	0.020	0.010	0.011	0.010	0.010	0.011	0.016

All concentrations are expressed in ppb.

APPENDIX IIA

Method Validation Data and Control Limit for Carbamates Table 1

Level µg/L (ppb)	Aldicarb Sulfoxide	Percent recovery	Aldicarb Sulfone	Percent recovery	Methomyl	Percent recovery	3-OH- Carbofuran	Percent recovery
0.0125	0.0089	71.2	0.0114	91.2	0.0070	88.8	0.0144	115
	0.0093	74.4	0.0109	87.6	0.0140	84.4	0.0123	98.4
	0.0108	86.2	0.0115	91.8	0.0114	86.4	0.0124	99.2
0.025	0.0196	78.5	0.0211	84.2	0.0201	78.1	0.0214	85.7
	0.0216	86.6	0.0232	92.8	0.0268	90.4	0.0231	92.5
	0.0238	95.2	0.0274	110	0.0213	97.6	0.0303	121
0.05	0.0495	99.2	0.0470	94.0	0.0438	87.2	0.0541	108
	0.0467	93.6	0.0459	91.8	0.0404	87.4	0.0481	96.2
	0.0248	85.7	0.0439	87.7	0.0440	85.0	0.0440	87.9
0.10	0.0944	94.4	0.0978	97.8	0.0948	94.8	0.0954	95.4
	0.0904	90.4	0.1031	103	0.0928	92.8	0.1097	110
	0.0896	89.6	0.1033	103	0.0996	99.6	0.1102	110
1.00	0.8064	80.6	0.9223	92.2	0.8858	88.6	0.9238	92.4
	0.8259	82.6	0.9318	93.2	0.8752	87.5	0.9300	93.0
	0.8578	85.8	0.9842	98.4	0.9673	96.7	0.9859	98.6
SD		7.88		6.79		5.73		10.30
SD X 3		23.64		20.38		17.19		30.89

Table 2

Level µg/L (ppb)	Aldicarb	Percent recovery	Carbofuran	Percent recovery	Carbaryl	Percent recovery
0.0125	0.0119	95.2	0.0138	110	0.0132	106
	0.0108	86.4	0.0119	95.2	0.0119	95.6
	0.0107	85.8	0.0118	94.4	0.0119	95.2
0.025	0.0188	75.3	0.0208	83.0	0.0213	85.1
	0.0222	88.9	0.0234	93.5	0.0234	93.6
	0.0252	101	0.0284	114	0.0276	110
0.05	0.0416	83.2	0.0488	97.6	0.0480	96.0
	0.0418	83.6	0.0462	92.4	0.0454	90.8
	0.0397	79.4	0.0436	87.2	0.0435	86.9
0.10	0.0886	88.6	0.0956	95.6	0.0946	94.6
	0.0984	98.4	0.1018	102	0.1023	102
	0.1038	102	0.1063	106	0.1049	105
1.00	0.8776	87.8	0.9238	92.4	0.9178	91.8
	0.8309	83.1	0.9122	91.2	0.9267	92.7
	0.9291	92.9	0.9743	97.4	0.9711	97.1
SD		7.79		8.26		6.97
3 X SD		23.38		24.79		20.90

Table 3

Level µg/L (ppb)	Oxamyl	Percent recovery	Methiocarb	Percent recovery
0.0125	0.0116	92.8	0.0124	99.2
	0.0118	94.8	0.0124	99.2
	0.0103	82.4	0.0105	83.6
0.025	0.0242	96.9	0.0247	98.9
	0.0233	93.2	0.0237	94.7
	0.0248	99.2	0.0232	92.8
0.05	0.0462	92.4	0.0424	84.8
	0.0449	89.8	0.0412	82.4
	0.0458	91.5	0.0442	88.3
0.10	0.0940	94.0	0.0949	94.9
	0.0848	84.8	0.0992	99.2
	0.0964	96.4	0.1042	104
1.00	0.9099	91.0	0.9043	90.4
	0.8909	89.1	0.8887	88.9
	0.9159	91.6	0.9263	92.6
SD		4.37		6.50
3 X SD		13.12		19.50

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Title: Determination of Ethalfluralin, Trifluralin, Benfluralin, Prodiamine, Pendimethalin, Oxyfluorfen, and Oryzalin in Surface Water

1. Scope:

This section method (SM) provides stepwise procedure for selective Dinitroaniline compounds and Oxyfluorfen analysis in surface water. It is followed by all authorized EA personnel.

2. Principle:

The dinitroanilines and oxyfluorfen are extracted from surface water samples with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated on a rotary evaporator and then a solvent exchange is performed with acetone. The extract is concentrated to a final volume of 1 mL where 0.5 mL is removed and vialled for GCMS-SIM (Gas Chromatography with Mass Spectrometer operated in the Single Ion Monitoring mode) or GCMS/MS analysis. The remaining 0.5mL is evaporated to just dryness and then brought up to a final volume of 0.5mL with methanol for analysis of oryzalin on LCMS.

3. Safety:

- 3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.
- 3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

4. Interferences:

There were no matrix interferences that caused quantitative problems during method development and validation.

5. Apparatus and Equipment:

- 5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)
- 5.2 Nitrogen Evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)
- 5.3 Balance (Mettler PC 4400 or equivalent)
- 5.4 Vortex-vibrating mixer

- 5.5 Gas Chromatograph equipped with a mass selective detector (MSD)
- 5.6 Gas Chromatograph equipped with a triple stage quadropole detector (MS/MS)
- 5.7 Liquid Chromatograph equipped with an ion trap (LCMS)

6. Reagents and Supplies:

- 6.1 Ethalfluralin CAS#55283-68-6
- 6.2 Trifluralin CAS#1582-09-8
- 6.3 Benfluralin CAS#1861-40-1
- 6.4 Prodiamine CAS#29091-21-2
- 6.5 Pendimethalin CAS#40487-42-1
- 6.6 Oxyfluorfen CAS#42874-03-3
- 6.7 Oryzalin CAS#19044-88-3
- 6.8 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.9 Acetone, nanograde or equivalent pesticide grade
- 6.10 Water, MS grade, Burdick & Jackson or equivalent
- 6.11 Methanol, MS grade, Burdick & Jackson or equivalent
- 6.12 Formic Acid, HPLC grade
- 6.13 Ammonium formate, reagent grade or equivalent
- 6.10 Separatory funnel, 2 L
- 6.11 Boiling flask, 500 mL
- 6.12 Sodium Sulfate, ACS grade
- 6.13 Funnels, long stem, 60°, 10 mm diameter
- 6.14 Volumetric Pipette, 0.5 mL
- 6.15 Graduated conical tubes with glass stopper, 15 mL
- 6.16 Glass wool, Pyrex® fiber glass slivers 8 microns
- 6.17 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.18 Recommended analytical columns:

For MSD - 5% (Phenyl)-methylpolysiloxane (HP-5MS or equivalent) fused silica column, 30 m x 0.25 mm id x 0.25 µm film thickness.

For HPLC/MS – Waters SymmetryShieldRP₁₈ 5 µm, 3.9 x 150 mm cartridge
Guard column: Waters SymmetryShieldRP₁₈ 5 µm, 3.9 x 20 mm cartridge
Guard column holder: Waters Sentry guard holder universal.

7. Standards Preparation:

- 7.1 The individual dinitroaniline and oxyfluorfen stock standards of 1.0 mg/mL were obtained from the CDFCA/CAC Standards Repository. The standards were diluted

to 10 µg/mL with acetone for identification purposes. Oryzalin was prepared in methanol at a concentration of 10 µg/mL for infusion into the LCMS.

A combination standard of 10 µg/mL was prepared from the individual mg/mL standards with acetone. The standard was also used to dilute the following concentrations: 0.025, 0.05, 0.1, 0.2, 0.5, and 1 µg/mL in acetone for GC instrument calibration. The 10 µg/mL of oryzalin in methanol was diluted to the same concentrations as above for LC instrument calibration.

7.2 Keep all standards in the designated refrigerator for storage.

7.3 The expiration date of each standard is six months from the preparation date.

8. **Sample Preservation and Storage:**

Store all samples waiting for extraction in a separate refrigerator (0 - 5 °C).

9. **Test Sample Preparation:**

9.1 Background Preparation

The Department of Pesticide Regulation (DPR) provided the surface water for background to be used in method validation and QC.

9.2 Preparation of blank and spike

Matrix blank: Weigh out 1000 g of background water and follow the test sample extraction procedure.

Matrix spike: Weigh out 1000 g of background water. Spike a client requested amount of herbicides into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.3 Test Sample Extraction

9.3.1 Record the weight of water samples to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.

- 9.3.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.
- 9.3.3 After phases have separated, drain lower the methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glass wool, into a 500 mL boiling flask.
- 9.3.4 Repeat steps 9.3.1 & 9.3.2 two more times using 80 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.
- 9.3.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.3.6 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 – 20 inch Hg vacuum. Add 2-4 mL of acetone and rotoevaporate to 1-2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.3.7 Rinse flask 3 more times with 2 - 4 mL of acetone and transfer each rinse to the same test tube.
- 9.3.8 Evaporate the sample extract to a volume slightly less than 1 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with acetone, mix well and transfer 0.5mL to two autosampler vials with inserts. Submit extract for GCMS-Triple Stage quadrapole analysis.
- 9.3.9 The remaining 0.5 mL sample extract is placed back in the water bath and evaporated to just dryness. Pipet 0.5 mL of methanol into the test tube and vortex well. Transfer extract to an autoamplifier vial to analyze on LCMS for oryzalin.

10. **Instrument Calibration:**

- 10.1 The calibration standard curve consists of a minimum of three levels. The lowest level must be at or below the corresponding reporting limits.
- 10.2 The calibration curves for the GCMS and Triple Quad were obtained using quadratic fit. The LCMS calibration curves were obtained using linear regression.

11. Analysis:

11.1 HPLC-MS

11.1.1 HPLC Instrument: Waters model 2695 HPLC and auto-sampler with column heater and remote control through Thermo Finnigan Xcalibur system.

Column: Waters SymmetryShield RP₁₈ 5 µm, 3.9 x 150 mm column

Column Temperature: 40 °C

Mobile Phase: Gradient

Solvent 1: 3762 mL water, 200 mL methanol, 38 mL 1M ammonium formate and 4.0 mL formic acid.

Solvent 2: 3600 mL methanol, 360 mL water, 36 mL 1.0 M ammonium formate, 4 mL formic acid.

Gradient:

<u>Time(min)</u>	<u>Flow rate</u>	<u>Mobile Phase 1</u>	<u>Mobile Phase 2</u>
0	0.75	85.0	15.0
3.0	0.75	85.0	15.0
4.0	0.75	50.0	50.0
10.0	0.75	50.0	50.0
14.0	0.75	40.0	60.0
16.0	0.75	5.0	95.0
22.0	0.75	5.0	95.0
24.5	0.75	85.0	15.0
27.0	0.75	85.0	15.0

Injection Volume:20 µL

11.1.2 Liquid Chromatograph Mass spectrometer (LC-MS) and Operating Parameters

Model:	Finnigan Model DECA ion trap MS
Ion Source Type:	Atmospheric pressure Ionization (APCI)
Source Polarity:	Positive
APCI Vaporizer Temp:	450 °C
Capillary Temperature:	220 °C
Sheath Gas:	60
Auxiliary Gas:	10
Mode of operation:	MS/MS

Compound Name	Retention Time (min.)	Molecular Weight	Mass Range	Product Ions
Oryzalin	18.96	346.36	95-400	288, 305

Note: The column conditions, temperature, mobile phase, etc. may slightly shift retention time.

11.1.3 Operating parameter

Parent Mass(m/z)	Isolation Width (m/z)	Normalized Collision Energy (%)	Activation Q	Activation Time (msec.)
347	2.0	30.0	0.250	30.0

11.2 GC-Triple Quad Instrumentation:

11.2.1 Model: Varian Triple Quad 320-MS

Column: Varian Factor Four VF-5ms x 0.25mm x 0.25µm

Temperature Program: initial column temperature 80 °C, hold 1 min., ramp at 15 °C/min. to temperature of 180 °C and hold for 3 min. ramp at 15 °C/min. to final temperature of 300°C and hold for 3 min.;

Injector Temperature: 250 °C

Injection volume: 1 µL.

Compound	Retention Time (min)	Precursor ion	Product Ion	Collison Energy/-ev
Ethalfuralin	10.28	333	316	-10
Trifluralin	10.52	335	290	-15
Benfluralin	10.62	335	276	-15
Prodiamine	13.91	350	275	-10
Pendimethalin	14.86	281	252	-10
Oxyfluorfen	15.97	361	300	-15

11.3 GCMS Instrumentation:

11.3.1 Model: Agilent GCMS

Column: 5% (Phenyl)-methylpolysiloxane (HP-5MS or equivalent) fused silica column, 30 m x 0.25 mm id x 0.25 µm film thickness.

Temperature Program: initial column temperature 80 °C, hold 1 min., ramp at 15 °C/min. to temperature of 180 °C and hold for 3 min. ramp at 15 °C/min. to final temperature of 300°C and hold for 3 min.;

Injector Temperature: 250 °C
Transfer line Temperature: 280 °C

Compound	Retention Time (min.)	Selected ions	Starting time (min.)
Ethalfuralin	9.41	276 , 316, 333	6.00
Trifluralin	9.62	264, 306 , 335	9.52
Benfluralin	9.69	264, 292 , 335	9.52
Prodiamine	13.27	279, 321 , 333	12.50
Pendimethalin	14.23	252 , 253, 281	13.85
Oxyfluorfen	15.38	252 , 300, 361	14.85

Quantitation ions are in bold.

12. Quality Control:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 surface water samples are spiked at 0.05ppb and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicates used to determine the MDL, t=3.143.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is chosen in a range 1-5 times the MDL, as per client agreement. The reporting limit for the dinitroanilines and oxyfluorfen is 0.05 ppb.

12.3 Method Validation

The method validation consisted of four sample sets. Each set included five levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the selective dinitroaniline and oxyfluorfen are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation for each analyte. The upper and lower warning and control limits are set at ± 2 and 3 standard deviations of the % recovery, respectively, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 per cent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. Calculations:

Quantitation is based on an external standard (ESTD) calculation using either the peak area or height. The LCMS software used a linear curve fit, with all levels weighted equally. The software for the triple quadrupole uses a quadratic curve fit, with all levels weighted $1/nx$ and the GCMS uses quadratic curve fit, with all levels weighted equally. Alternatively, at the chemist's discretion, sample results may be calculated using the response factor for the standard.

$$\text{ppb} = \frac{(\text{sample peak area or ht}) \times (\text{std conc}) \times (\text{std vol. injected}) \times (\text{final vol of sample})(1000 \mu\text{L/mL})}{(\text{std. peak area or ht}) \times (\text{sample vol injected}) \times (\text{sample wt (g)})}$$

14. **Reporting Procedure:**

Sample results are reported out according to the client's analytical laboratory specification sheets.

15. **Discussion and References:**

- 15.1 The triple quadrupole will be used as the primary instrument for the analysis of the dinitroanilines and oxyfluorfen. The MSD will be used as a backup instrument. The LCMS is used for the analysis of oryzalin since it wasn't very sensitive on the GC.
- 15.2 A storage stability study was done with this project. The storage stability study consisted of a 5 ppb spike level and 3 replicates over a 28 day period. Fifteen bottles containing background water were spiked and stored in the refrigerator until analyzed on 0, 4, 7, 14, and 28 days. Along with the storage spikes a blank and method control spike were also extracted. This storage study showed no degradation for the dinitroaniline compounds or oxyfluorfen. The results are shown in Appendix 3.
- 15.3 We have observed gradual losses in sensitivity caused by the sample matrix. We recommend changing the injector liner and trimming the column when this occurs.
- 15.4 Solid phase extraction has been tried for sample preparation as part of our method development. The recoveries were low and inconsistent for some compounds.
- 15.5 GC-Triple Quad analysis of the samples produced a sample response and quantitation varied depending on matrix background in the samples. Therefore the calibration standards were added to a matrix blank extract to correct for matrix background interference. This is unnecessary for LCMS analysis.
- 15.6 References:
- 15.61 J.L. Kish, E.M. Thrumann, E.A. Scribner, and L.R. Zimmerman; *Methods of Analysis by the U.S. Geological Survey Organic Geochemistry Research Group—Determination of Selected Herbicides Metabolites and Their Degradation Products in Water Using Solid-Phase Extraction and Gas*

Chromatography/Mass, U.S. Geological Survey Kansas Water Science Center

15.62 Hsu, J. and Feng, H.; *Determination of Organophosphate Pesticides in the surface water using Gas Chromatography*, 2004, Environmental monitoring method, Center for Analytical Chemistry, CDFA

Appendix 1

The Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Results: Varian GC/TQMS

Spk\Analyte	Ethalfuralin	Trifluralin	Benfluralin	Prodiamine	Pendimethalin	Oxyfluorfen
0.05ppb spk 1	0.0366	0.0410	0.0384	0.0375	0.0358	0.0332
0.05ppb spk 2	0.0463	0.0402	0.0387	0.0384	0.0359	0.0369
0.05ppb spk 3	0.0416	0.0399	0.0451	0.0414	0.0377	0.0343
0.05ppb spk 4	0.05	0.0478	0.0465	0.0427	0.0464	0.0403
0.05ppb spk 5	0.0479	0.0398	0.0433	0.0391	0.0385	0.0375
0.05ppb spk 6	0.0461	0.0408	0.0437	0.0415	0.0394	0.0381
0.05ppb spk 7	0.0495	0.0512	0.0487	0.0493	0.0425	0.0425
SD	0.00479	0.00460	0.00382	0.00395	0.00382	0.00322
MDL	0.0150	0.0144	0.0120	0.0124	0.0120	0.0101
RL	0.05	0.05	0.05	0.05	0.05	0.05

Appendix 1: continued

Results: Agilent GC/MSD

Spk\Analyte	Ethalfuralin	Trifluralin	Benfluralin	Prodiamine	Pendimethalin	Oxyfluorfen
0.05ppb spk 1	0.044	0.040	0.038	0.051	0.045	0.052
0.05ppb spk 2	0.052	0.047	0.044	0.060	0.054	0.061
0.05ppb spk 3	0.048	0.044	0.041	0.057	0.051	0.059
0.05ppb spk 4	0.059	0.054	0.051	0.069	0.062	0.070
0.05ppb spk 5	0.047	0.044	0.041	0.054	0.048	0.052
0.05ppb spk 6	0.049	0.045	0.042	0.057	0.051	0.056
0.05ppb spk 7	0.056	0.052	0.049	0.066	0.059	0.068
SD	0.00528	0.0049	0.0047	0.0064	0.0059	0.0072
MDL	0.017	0.015	0.015	0.020	0.019	0.023
RL	0.05	0.05	0.05	0.05	0.05	0.05

Results: Finningan LCQ Deca

Spk\Analyte	Oryzalin
0.05ppb spk 1	0.057
0.05ppb spk 2	0.057
0.05ppb spk 3	0.057
0.05ppb spk 4	0.056
0.05ppb spk 5	0.055
0.05ppb spk 6	0.057
0.05ppb spk 7	0.053
SD	0.001528
MDL	0.021
RL	0.05

Appendix 2

Method Validation Data

Results: Varian GC/TQMS							
Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4	%	%
Ethalfluralin	0.15	110	97.7	101	97.9	Mean:	98.7
	0.3	108	107	86.2	96.4	SD:	6.4
	1	94.7	94	98.9	93.9	UCL:	117.9
	2	94.7	105	108	96.7	UWL:	111.5
	5	90.0	95.9	102	95.0	LWL:	85.9
						LCL:	79.5
Trifluralin	0.15	109	91.4	103	89.8	Mean:	97.4
	0.3	108	104	88.5	92.7	SD:	6.6
	1	96.5	92	97.6	95.2	UCL:	117.2
	2	96.8	106	106	91.5	UWL:	110.6
	5	92.6	89.9	102	95.6	LWL:	84.2
						LCL:	77.6
Benfluralin	0.15	103	86	101	87.7	Mean:	96.7
	0.3	107	104	83.5	92.6	SD:	7.0
	1	98.3	92.5	101	93.6	UCL:	117.7
	2	94.9	108	104	95.1	UWL:	110.7
	5	91	90.4	102	97.3	LWL:	82.7
						LCL:	75.7
Prodiamine	0.15	120	95.1	112	99.0	Mean:	101
	0.3	117	113	77.6	97.2	SD:	11.4
	1	102	93.7	113	90.2	UCL:	135.2
	2	92.6	108	115	92.5	UWL:	123.8
	5	90.1	93.9	100.9	91.0	LWL:	78.2
						LCL:	66.8

Results: **Varian GC/TQMS**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4	%	%
Pendimethlin	0.15	109	94.4	109	95.3	Mean:	98.0
	0.3	112	106	85.1	98.0	SD:	8.9
	1	101	91.6	105	90.2	UCL:	124.7
	2	94.2	99.6	115	92.2	UWL:	115.8
	5	86.6	88.3	99.3	88.5	LWL:	80.2
						LCL:	71.3
Oxyfluorfen	0.15	114	96.5	112.4	95.3	Mean:	100.4
	0.3	115	113	75.2	101	SD:	12.8
	1	105	90.7	107	91.3	UCL:	138.8
	2	97.6	109	128	91.9	UWL:	126.0
	5	84.1	89.8	106	85.7	LWL:	74.8
						LCL:	62.0

Results: **Agilent GC/MSD**

Analyte	Spike ppb	Recovery Set 1	(%) set 2	set 3	set 4	%	%
Ethalfuralin	0.15	94.3	84.9	95.9	91.2	Mean:	99.6
	0.3	104	114	105	90.6	SD:	9.6
	1	123	107	100	106	UCL:	128.4
	2	96.3	114	103	89.2	UWL:	118.8
	5	96.9	92.9	92.7	92.6	LWL:	80.4
						LCL:	70.8
Trifluralin	0.15	91.3	82	91.3	87.3	Mean:	97.1
	0.3	101	111	102	87.7	SD:	9.4
	1	119	104	96.5	102	UCL:	125.3
	2	94.6	112	102	88.0	UWL:	115.9
	5	95.8	92.4	91.4	92.0	LWL:	78.3
						LCL:	68.9

Results:		Agilent GC/MSD						
Benfluralin	0.15	90.0	80.0	89.3	84.7	Mean:	96.0	
	0.3	99.7	110	99.1	85.7	SD:	9.5	
	1	118	103	96.1	101	UCL:	124.5	
	2	94.0	111	101	87.5	UWL:	115.0	
	5	95.8	92.0	91.4	91.6	LWL:	77.0	
						LCL:	67.5	
Prodiamine	0.15	116	96.7	117	108	Mean:	112	
	0.3	121	135	121	102	SD:	11.0	
	1	130	116	113	115	UCL:	145.0	
	2	106	121	120	97.5	UWL:	134.0	
	5	105	103	99.2	98.8	LWL:	90.0	
						LCL:	79.0	
Pendimethlin	0.15	112	89.6	106	98.1	Mean:	108	
	0.3	117	126	120	95.5	SD:	10.6	
	1	123	111	108	111	UCL:	139.8	
	2	105	120	119	95.0	UWL:	129.2	
	5	106	100	97.6	97.9	LWL:	86.8	
						LCL:	76.2	
Oxyfluorfen	0.15	124	87.3	111	103	Mean:	113	
	0.3	125	131	134	102	SD:	12.1	
	1	123	120	115	118	UCL:	149.6	
	2	110	120	123	102	UWL:	137.2	
	5	114	105	99.9	100	LWL:	88.8	
						LCL:	76.7	

Results:		Finningan LCQ Deca						
Analyte	Spike ppb	Recovery (%)				%		
		Set 1	set 2	set 3	set 4			
Oryzlin	0.15	92.7	96.0	73.3	84.0	Mean:	83.6	
	0.3	86.0	91.7	100	77.7	SD:	9.3	
	1	87.2	91.2	77.9	68.0	UCL:	111.5	
	2	93.0	70.6	80.5	68.5	UWL:	102.2	
	5	90.4	81.8	79.6	81.0	LWL:	65.0	
						LCL:	55.7	

Appendix 3 Storage Stability Study

Analyte	Day 0		Day 4		Day 7		Day 14		Day 28		
	ppb	%R	ppb	%R	ppb	%R	ppb	%R	ppb	%R	
Ethalfuralin	blk	nd	nd		nd		nd		nd		
	spk	0.836	83.6%	0.875	87.5%	0.849	84.9%	0.796	79.6%	0.804	80.4%
	spk 1	0.865	86.5%	0.894	89.4%	0.877	87.7%	0.961	96.1%	1.00	100%
	spk 2	0.873	87.3%	0.857	85.7%	0.858	85.8%	1.03	103%	1.04	104%
	spk 3	0.831	83.1%	0.821	82.1%	0.895	89.5%	0.941	94.1%	0.872	87.2%
Trifluralin	blk	nd	nd		nd		nd		nd		
	spk	0.795	79.5%	0.851	85.1%	0.877	87.7%	0.818	81.8%	0.83	83.0%
	spk 1	0.825	82.5%	0.862	86.2%	0.828	82.8%	0.948	94.8%	0.964	96.4%
	spk 2	0.734	73.4%	0.838	83.8%	0.88	88.0%	1.06	106.0%	1.03	103%
	spk 3	0.797	79.7%	0.833	83.3%	0.913	91.3%	0.94	94.0%	0.832	83.2%
Benfluralin	blk	nd	nd		nd		nd		nd		
	spk	0.840	84.0%	0.827	82.7%	0.859	85.9%	0.806	80.6%	0.838	83.8%
	spk 1	0.875	87.5%	0.854	85.4%	0.858	85.8%	0.983	98.3%	0.962	96.2%
	spk 2	0.853	85.3%	0.874	87.4%	0.878	87.8%	1.03	103%	1.06	106%
	spk 3	0.856	85.6%	0.828	82.8%	0.879	87.9%	0.930	93.0%	0.885	88.5%
Prodiamine	blk	nd	nd		nd		nd		nd		
	spk	0.858	85.8%	0.852	85.2%	0.899	89.9%	0.832	83.2%	0.813	81.3%
	spk 1	0.906	90.6%	0.881	88.1%	0.834	83.4%	1.02	102%	0.97	97.0%
	spk 2	0.905	90.5%	0.910	91.0%	0.953	95.3%	1.09	109%	1.10	110%
	spk 3	0.899	89.9%	0.851	85.1%	0.908	90.8%	0.979	97.9%	0.907	90.7%
Pendimethlin	blk	nd	nd		nd		nd		nd		
	spk	0.82	82.0%	0.825	82.5%	0.881	88.1%	0.796	79.6%	0.802	80.2%
	spk 1	0.898	89.8%	0.836	83.6%	0.785	78.5%	0.948	94.8%	0.953	95.3%
	spk 2	0.900	90.0%	0.875	87.5%	0.871	87.1%	1.02	102%	1.04	104%
	spk 3	0.868	86.8%	0.783	78.3%	0.857	85.7%	0.906	90.6%	0.868	86.8%

Oxyfluorfen	blk	nd									
	spk	0.775	77.5%	0.824	82.4%	0.884	88.4%	0.819	81.9%	0.726	72.6%
	spk 1	0.889	88.9%	0.810	81.0%	0.788	78.8%	0.984	98.4%	0.977	97.7%
	spk 2	0.857	85.7%	0.849	84.9%	0.913	91.3%	0.991	99.1%	1.04	104%
	spk 3	0.838	83.8%	0.752	75.2%	0.869	86.9%	0.867	86.7%	0.837	83.7%
Oryzalin	blk	nd									
	spk	0.900	90.0%	0.963	96.3%	0.95	95.0%	0.960	96.0%	0.795	79.5%
	spk 1	0.963	96.3%	0.929	92.9%	0.937	93.7%	0.918	91.8%	0.881	88.1%
	spk 2	0.898	89.8%	0.824	82.4%	0.867	86.7%	1.02	102%	0.884	88.4%
	spk 3	0.999	99.9%	0.997	99.7%	0.803	80.3%	1.03	103%	0.870	87.0%

Written By:

Original signed by :

3/17/2009

Jean Hsu
Chemist

Date

Written By:

Original signed by :

3/17/2009

Jane White
Chemist

Date

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Original signed by :

3/17/2009

Steve Siegel
Section Supervisor

Date

Approved By:

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3/19/2009

Elaine Wong
Program Supervisor

Date

Determination of Organophosphate Pesticides in Surface water using Gas Chromatography with mass selective detection (MSD).

1. Scope:

This section method (SM) documents the selected organophosphate pesticides analysis in surface water by all authorized section personnel. This method is not applicable for Ethoprop, Azinphos-methyl and Profenofos.

2. Principle:

The surface water sample is extracted with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated to almost dryness on a rotary evaporator and diluted to a final volume of 1.0 mL with acetone. The extract is then analyzed by a gas chromatograph equipped with a mass selective detector (MSD).

3. Safety:

3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.

3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

3.3 All solvents should be handled with care in a ventilated area.

4. Interferences:

There are matrix interferences that cause quantitative problems. Therefore the calibration standards will be made up in appropriate matrix.

5. Apparatus and Equipment:

5.1 Rotary evaporator (Büchi/Brinkman or equivalent)

5.2 Nitrogen evaporator (Meyer N-EVAP Organomation Model # 112 or equivalent)

5.3 Vortex-vibrating mixer

- 5.4 Balance (Mettler SM-L) or equivalent
- 5.5 Gas Chromatograph equipped with a mass selective detector (MSD)

6. Reagents and Supplies

- 6.1 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.2 Acetone, nanograde or equivalent pesticide grade
- 6.3 Anhydrous Sodium Sulfate, granular
- 6.4 Diazinon CAS# 333-41-5
- 6.5 Disulfoton CAS# 298-04-4
- 6.6 Chlorpyrifos CAS# 2921-88-2
- 6.7 Malathion CAS# 121-75-5
- 6.8 Methidation CAS# 950-37-8
- 6.9 Fenamiphos CAS# 22224-92-6
- 6.10 Dichlorvos CAS# 62-73-7
- 6.11 Phorate CAS# 298-02-2
- 6.12 Fonofos CAS# 66767-39-3
- 6.13 Dimethoate CAS# 60-51-5
- 6.14 Parathion methyl CAS# 298-00-0
- 6.15 Tribufos (DEF) CAS# 78-48-8
- 6.16 Conical tube with glass stopper, 15-mL graduated, 0.1 mL subdivision
- 6.17 Separatory funnel, 2 L
- 6.18 Boiling flask, 500 mL
- 6.19 Funnel, long stem, 10 mm diameter
- 6.20 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.21 Recommended analytical columns:

For MSD - 1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane (Restek Rxi-5Sil MS or equivalent) fused silica column, 30 m x 0.25 mm x 0.25 μ m film thickness.

7. Standards Preparation:

- 7.1 Dilute the 1 mg/mL Organophosphate standards obtained from the CDFCA/CAC Environmental Analysis Standards Repository with acetone to make up a series of mixed working standards (see 10.2). These standards shall be prepared to cover the linear range from 0.025 η g/ μ L to 0.5 η g/ μ L for OP screen and 0.01 η g/ μ L to 0.5 η g/ μ L for low level diazinon and chlorpyrifos.
- 7.2 The calibration standards are added to matrix blank extracts (9.1.2.1) to correct for matrix background interference.

- 7.3 Store standards according to manufacturing requirement. Keep all standards in designated refrigerator for storage.
- 7.4 The expiration date of each mixed working standard is six months from the preparation date or same as stock standards, if sooner.
- 7.5 A portion of the new standard will be vialled and set aside in the refrigerator. This will be used when doing the intermediate check and the check for a new set of standards. The intermediate check will be performed before the standard is 3 months old and be documented along with the comparison for that set of standards. There should be <20% difference between the response of the new standard or the intermediate check standard and the response of the vialled standard.

8. Sample Preservation and Storage:

All water samples and sample extracts shall be stored in the refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Sample Preparation

9.1.1 Remove samples from refrigerator and allow samples to come to room temperature before extraction.

9.1.2 Preparation of matrix blank and matrix spike:

The Department of Pesticide Regulations (DPR) provides the background water for matrix blank and spikes.

9.1.2.1 Matrix blank: Weigh out approximate 1000 g of background water and follow the test sample extraction procedure.

9.1.2.2 Matrix spike: Weigh out approximate 1000 g of background water. Spike a client requested amount of organophosphate pesticides into the background water and let it stand for 1 minute. Follow the test sample extraction procedure.

9.2 Test Sample Extraction

- 9.2.1 Record the weight of the whole bottle water sample to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.
- 9.2.2 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.
- 9.2.3 After phases have separated, drain lower methylene chloride layer through 20 ± 4 g of anhydrous sodium sulfate and glass wool, into a 500 mL boiling flask.
- 9.2.4 Repeat steps 9.2.2 & 9.2.3 two more times using 80 ± 5 mL of methylene chloride each time. Combine the extracts in the same boiling flask.
- 9.2.5 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.2.6 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 - 20 inch Hg vacuum. Add 2 - 4 mL of acetone and rotoevaporate to 1 - 2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.2.7 Rinse flask 3 more times with 2 - 4 mL of acetone and transfer each rinse to the same test tube.
- 9.2.8 Evaporate the extract to a volume slightly less than 1 mL in a water bath at 38 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with acetone, mix well and transfer into two autosampler vials.
- 9.2.9 Submit extract for GC/MS analysis.

10. Instrument Calibration:

- 10.1 The calibration standards are added to a matrix blank extract to correct for matrix background.
- 10.2 A calibration standard curve consists of minimum of three levels. Standard concentrations of 0.01, 0.025, 0.05, 0.1, 0.25 and 0.5 $\text{ng}/\mu\text{L}$ are recommended. Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995.

11.1 Injection Scheme

Follow the sequence of Solvent, Calibration standards, Solvent, Matrix Blank, Matrix Spike, Test Samples (maximum of 10-12 samples) and Calibration standards. Injection of an old sample or matrix blank before the sequence analysis to condition the instrument is recommended.

11.2 GC Instrumentation

11.2.1 Recommended instrument (GC/MSD) parameters: Injector 250 °C; MSD transfer line heater 280 °C; oven temperature 80 °C, hold 2 min., ramp @ 20 °C/min. to 250 °C, hold 4 min.; injection volume 2 or 3 µL.

Ions Selected for SIM Acquisition:

Diazinon	137, 152, 179, 304 ,	Retention time: 11.9 min
Disulfoton	88 , 97, 142, 274,	Retention time: 12.2 min
Malathion	93, 125, 127, 173 ,	Retention time: 14.1 min
Chlorpyrifos	125, 197 , 258, 314,	Retention time: 11.2 min
Methidathion	58, 85, 93, 145 ,	Retention time: 9.88 min
Fenamiphos	154, 217, 288, 303 ,	Retention time: 9.26 min
DDVP	79, 109 , 185,	Retention time: 11.2 min
Phorate	75 , 97, 121, 260,	Retention time: 9.72 min
Dimethoate	87 , 93, 125, 126,	Retention time: 12.0 min
Fonofos	109 , 137, 246,	Retention time: 10.7 min
Me Parathion	63, 109, 125, 263 ,	Retention time: 9.94 min
DEF	169 , 202,	Retention time: 9.73 min

(Quantitation ions are in bold)

12. Quality Control:

12.1 Each set of samples shall have a matrix blank and minimum of one matrix spike sample.

12.2 The matrix blank should be free of target compounds.

12.3 The recoveries of the matrix spike shall be within the control limits.

12.3.1 When spike recoveries fall outside the control limits, the chemist must

investigate the cause. The entire extraction set of samples is re-analyzed. If the spike recoveries fall within the limit, then the results from the re-analyzed samples shall be reported.

12.3.2 If the spike recoveries still fall outside the control limits, the client will be notified. The backup samples will be re-extracted for analysis.

12.4 The retention time should be within ± 2 percent of that of the standard.

12.5 All calibration standards analyzed for a sample set will be used in the calibration curve. If the calibration curve does not meet the acceptance criteria the samples shall be re-run. If the calibration criteria are met the sample results will be reported. If the calibration criteria are still not met a method deviation will be prepared and approved by the supervisor or designee. The client will be notified of the deviation and a copy of the method deviation detailing what was changed and why it was changed will be included with the sample results and the data will be flagged to let the data user know of the deviation.

12.6 The sample must be diluted if results fall outside the linear range of the standard curve.

12.7 Bracketing standard curves should have a percent change less than 20%.

12.8 Method Detection Limits (MDL)

The method detection limit refers to the lowest concentration of analyte that a method can detect reliably. To determine the MDL, 7 replicate water samples are spiked at 0.05 ppb for OP screen and 7 replicate water samples are spiked at 10 ppt for low level diazinon and chlorpyrifos and 7 replicates were spikes at 0.02 ppb for malathion. The standard deviation from the spiked sample recoveries are used to calculate the MDL for each analyte using the follow equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicate used to determine the MDL, t=3.143.

12.9 Reporting limit (RL):

The reporting limit (RL) refers to the level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. Per client

agreement, the RL is chosen in a range 1-5 times the MDL except in special cases. (See 15.5)

MDL data and the RL are tabulated in Appendix IA and IB.

12.10 Method Validation Recovery Data and Control Limits:

12.10.1 The method validation consisted of five sample sets. Each set included seven levels of fortification (0.01, 0.025, 0.05, 0.10, 0.25, 0.5 ppb) and a method blank. All spikes and method blank samples were processed through the entire analytical method.

12.10.2 Upper and lower warning and control limits are set at ± 2 and ± 3 standard deviations of the average % recovery, respectively.

12.10.3 The method validation consisted of five sample sets. Each set included six levels of fortification and a method blank. All spikes and method blank samples were processed through the entire analytical method.

Method validation results and control limits are tabulated in Appendix IB.

12.11 Estimated Measurement Uncertainty:

Total uncertainty for this method is 17% at 95% confidence interval.

12.12 Trend Identification

12.12.1 All matrix spike recoveries for OP analysis will be put into control charts and monitored for trends. Three trend characteristics will be evaluated at least bi-yearly by the supervisor or designee.
2 of 3 points above or below 2/3 of the UCL or LCL.
7 continuous points above or below the center line (CL)
14 points alternating above and below the CL.

12.12.2 When results indicate an out of control situation the supervisor or designee will indicate this on the control chart and take appropriate corrective action, which may include monitoring the results more closely to initiating a formal corrective action with root cause investigation.

13. Calculations:

Quantitation is based on external standard (ESTD) calculation using either the peak area or height. The software uses a linear or quadratic curve fit, with all levels weighted equally. Alternatively, at chemist discretion, concentrations may be calculated using

the response factor for the standard whose value is closest to the level in the sample.

$$\text{ppb} = \frac{(\text{sample peak ht. or area}) (\text{std. conc.}) (\text{std. vol. injected}) (\text{sample final vol., (mL)})(1000)}{(\text{std. peak ht. or area}) (\text{sample vol. injected}) (\text{sample wt., g})}$$

14. Reporting Procedure:

14.1 Identification of Analyte

For responses within calibration range, compare the retention time of the peaks with the retention time of standards. For positive results retention times shall not vary from the standards more than 2 percent.

14.2 Sample results are reported out according to the client's analytical laboratory specifications.

15. Discussion and References:

- 15.1 Sample response and quantitation vary depending on matrix background in the samples. The calibration standards were added to a matrix blank extract to correct for matrix background interference.
- 15.2 Some of the late eluting compounds were observed to suffer gradual losses in sensitivity. We recommend changing the injector liner and trimming the column when this occurs.
- 15.3 The client requested a lower reporting limit for both diazinon and chlorpyrifos. We re-validated this method using GC/MSD as the analysis instrument to achieve the lower reporting limit for those two compounds.

16. References:

- 16.1 *EPA Method 507, Pesticides, Capillary Column*. EPA Test Method for Drinking Water and Raw Source Water, 1987.
- 16.2 Hsu, J. and Hernandez J. *Determination of Organophosphate Pesticides in Surface Water using Gas Chromatography*, 1997, Environmental Monitoring Method, Center for Analytical Chemistry, CDFA.

Appendix IA

Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Spike/analyte	Diazinon			Disulfoton			Chlorpyrifos		
			Avg.			Avg.			Avg.
0.05/ ppb Spk 1	0.04709	0.04664	0.04687	0.04203	0.04528	0.04366	0.04784	0.04804	0.04794
0.05/ ppb Spk 2	0.04901	0.04975	0.04938	0.03938	0.03474	0.03706	0.04991	0.05010	0.05001
0.05/ ppb Spk 3	0.04465	0.04871	0.04668	0.04050	0.03653	0.03852	0.04580	0.04566	0.04573
0.05/ ppb Spk 4	0.04851	0.05026	0.04939	0.04640	0.04365	0.04503	0.04775	0.04768	0.04772
0.05/ ppb Spk 5	0.04405	0.04447	0.04426	0.04774	0.04583	0.04679	0.04459	0.04420	0.04440
0.05/ ppb Spk 6	0.04154	0.04181	0.04168	0.04740	0.04446	0.04593	0.04222	0.04262	0.04242
0.05/ ppb Spk 7	0.03949	0.04188	0.04069	0.03821	0.03487	0.03654	0.04093	0.04070	0.04082
		SD	0.00348			0.00441			0.00326
MDL= 3.14 * SD		MDL	0.01093			0.01384			0.01024
		RL	0.01			0.04			0.01

Spike/analyte	Malathion			Methidathion			Fenamiphos		
			Avg.			Avg.			Avg.
0.05/ ppb Spk 1	0.04549	0.04553	0.04551	0.03980	0.04117	0.04049	0.04614	0.04229	0.04422
0.05/ ppb Spk 2	0.04877	0.04895	0.04886	0.04612	0.04541	0.04577	0.04490	0.04879	0.04685
0.05/ ppb Spk 3	0.04489	0.04101	0.04295	0.03971	0.03883	0.03927	0.04202	0.04175	0.04189
0.05/ ppb Spk 4	0.04693	0.04568	0.04631	0.04224	0.04092	0.04158	0.04880	0.04839	0.04860
0.05/ ppb Spk 5	0.04169	0.04129	0.04149	0.03380	0.03328	0.03354	0.04403	0.04333	0.04368
0.05/ ppb Spk 6	0.04208	0.04177	0.04193	0.03967	0.03922	0.03945	0.04305	0.04289	0.04297
0.05/ ppb Spk 7	0.04121	0.04039	0.04080	0.04004	0.03957	0.03981	0.04196	0.03691	0.03944
		SD	0.00298			0.00362			0.00305
MDL= 3.14 * SD		MDL	0.00935			0.01136			0.00957
		RL	0.02			0.05			0.05

Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Spike/analyte	Malathion		
			Avg.
0.02/ ppb Spk 1	0.02160	0.02590	0.02375
0.02/ ppb Spk 2	0.01830	0.02260	0.02045
0.02/ ppb Spk 3	0.01690	0.02170	0.01930
0.02/ ppb Spk 4	0.01850	0.02230	0.02040
0.02/ ppb Spk 5	0.01710	0.02340	0.02025
0.02/ ppb Spk 6	0.01410	0.01960	0.01685
0.02/ ppb Spk 7	0.01830	0.02220	0.02025
Standard deviation		SD	0.00203
MDL= 3.14 * SD		MDL	0.00638
Reporting limit		RL	0.02

Appendix IB

Spike/analyte	DDVP	Avg.	Phorate	Avg.	Fonofos	Avg.			
0.05/ ppb Spk 1	0.04130	0.04339	0.04235	0.04292	0.04329	0.04311	0.04369	0.04362	0.04366
0.05/ ppb Spk 2	0.04210	0.04447	0.04329	0.04396	0.04350	0.04373	0.04652	0.04794	0.04723
0.05/ ppb Spk 3	0.04034	0.04069	0.04052	0.04084	0.04006	0.04045	0.04155	0.04126	0.04141
0.05/ ppb Spk 4	0.03780	0.04184	0.03982	0.04263	0.04252	0.04258	0.04368	0.04409	0.04389
0.05/ ppb Spk 5	0.03835	0.03789	0.03812	0.04031	0.03962	0.03997	0.04167	0.04151	0.04159
0.05/ ppb Spk 6	0.03834	0.03724	0.03779	0.03725	0.03734	0.03730	0.03935	0.03893	0.03914
0.05/ ppb Spk 7	0.03534	0.03528	0.03531	0.03577	0.03555	0.03566	0.03822	0.03774	0.03798
		SD	0.00276			0.00305			0.00343
MDL= 3.14 * SD		MDL	0.00868			0.00959			0.01076
		RL	0.05			0.05			0.04
Spike/analyte	Dimethoate	Avg.	Methyl Parathion	Avg.	DEF	Avg.			
0.05/ ppb Spk 1	0.03922	0.03874	0.03898	0.04111	0.04046	0.04079	0.04293	0.04358	0.04326
0.05/ ppb Spk 2	0.04397	0.04344	0.04371	0.04610	0.04631	0.04621	0.04628	0.04591	0.04610
0.05/ ppb Spk 3	0.03692	0.03638	0.03665	0.03906	0.04019	0.03963	0.04186	0.04259	0.04223
0.05/ ppb Spk 4	0.03869	0.03900	0.03885	0.04044	0.03966	0.04005	0.04388	0.04400	0.04394
0.05/ ppb Spk 5	0.03068	0.03089	0.03079	0.03278	0.03343	0.03311	0.03993	0.04046	0.04020
0.05/ ppb Spk 6	0.03617	0.03964	0.03791	0.03637	0.03720	0.03679	0.03932	0.03886	0.03909
0.05/ ppb Spk 7	0.03801	0.03736	0.03769	0.03748	0.03708	0.03728	0.03696	0.03786	0.03741
		SD	0.00383			0.00406			0.00301
MDL= 3.14 * SD		MDL	0.01202			0.01276			0.00946
		RL	0.04			0.03			0.05

Method Validation Data

Analyte	Spike ppb	Set 1			Set 2			Set 3				
				Avg.			Avg.			Avg.		
Diazinon	0.01	83.4	82.7	83.1	90.1	90.4	90.3	94.5	107.0	100.8	SD	6.082
	0.025	89.2	90.9	90.1	85.6	91.3	88.5	93.6	85.2	89.4	Mean	90.2
	0.05	101.0	94.6	97.8	90.0	89.8	89.9	89.3	93.4	91.4	UCL	108.5
	0.1	85.0	85.5	85.3	86.2	87.9	87.1	89.3	89.4	89.4	UWL	102.4
	0.25	92.9	93.5	93.2	80.4	81.0	80.7	88.3	86.3	87.3	LWL	78.1
	0.5	93.7	93.7	93.7	98.1	98.8	98.5	89.4	87.0	88.2	LCL	72.0
Disulfoton	0.01	84.1	83.1	83.6	112.0	105.0	108.5	114.0	114.0	114.0	SD	10.855
	0.025	73.6	72.3	73.0	80.5	78.4	79.5	85.2	83.8	84.5	Mean	85.4
	0.05	74.1	73.2	73.7	84.6	86.0	85.3	90.7	87.4	89.1	UCL	117.9
	0.1	85.3	85.7	85.5	81.2	79.2	80.2	85.0	83.1	84.1	UWL	107.1
	0.25	79.8	78.7	79.3	76.1	74.7	75.4	83.5	82.4	83.0	LWL	63.7
	0.5	79.7	78.1	78.9	95.2	94.6	94.9	85.0	84.2	84.6	LCL	52.8
Chlorpyrifos	0.01	111.0	109.0	110.0	98.5	101.0	99.8	102.0	102.0	102.0	SD	7.133
	0.025	98.1	97.6	97.9	89.4	90.4	89.9	86.1	87.6	86.9	Mean	92.9
	0.05	97.7	98.4	98.1	90.7	93.9	92.3	89.8	90.0	89.9	UCL	114.3
	0.1	88.1	88.4	88.3	87.2	87.4	87.3	87.3	86.8	87.1	UWL	107.2
	0.25	93.8	94.0	93.9	81.0	81.2	81.1	87.0	86.4	86.7	LWL	78.6
	0.5	94.8	93.8	94.3	98.9	99.6	99.3	88.0	87.1	87.6	LCL	71.5
Malathion	0.01	88.0	87.8	87.9	90.6	94.4	92.5	99.4	97.0	98.2	SD	4.642
	0.025	93.0	96.8	94.9	91.6	90.3	91.0	89.2	88.4	88.8	Mean	91.8
	0.05	99.0	98.9	99.0	91.7	92.8	92.3	90.3	89.1	89.7	UCL	105.7
	0.1	89.0	91.2	90.1	88.2	86.6	87.4	91.2	88.0	89.6	UWL	101.1
	0.25	95.7	95.7	95.7	81.8	82.3	82.1	89.8	88.2	89.0	LWL	82.5
	0.5	97.3	96.0	96.7	99.0	99.1	99.1	89.0	87.3	88.2	LCL	77.8

Method Validation Data (continued)

Methidathion	0.01	97.3	91.2	94.3	81.2	81.7	81.5	92.1	95.1	93.6	SD	8.648
	0.025	107.0	103.0	105.0	84.4	81.5	83.0	90.4	83.6	87.0	Mean	91.6
	0.05	107.0	101.0	104.0	88.7	86.2	87.5	83.3	81.9	82.6	UCL	117.6
	0.1	103.0	99.6	101.3	85.0	83.9	84.5	87.9	87.7	87.8	UWL	108.9
	0.25	106.0	104.0	105.0	80.1	80.5	80.3	93.0	92.0	92.5	LWL	74.3
	0.5	101.0	100.0	100.5	95.5	95.8	95.7	84.4	82.5	83.5	LCL	65.7

Fenamiphos	0.01	75.7	73.1	74.4	77.3	78.6	78.0	77.6	76.9	77.3	Sd	6.793
	0.025	86.5	85.6	86.1	78.3	77.0	77.7	77.4	78.4	77.9	Mean	84.4
	0.05	93.0	90.7	91.9	90.3	82.1	86.2	84.5	79.2	81.9	UCL	104.8
	0.1	93.0	91.4	92.2	83.8	82.8	83.3	81.3	83.5	82.4	UWL	98.0
	0.25	96.3	94.0	95.2	77.8	77.2	77.5	85.5	86.3	85.9	LWL	70.8
	0.5	94.8	92.8	93.8	95.1	94.9	95.0	82.7	82.2	82.5	LCL	64.0

DDVP	ppb	Set 1	Avg.		Set 2	Avg.		Set 3	Avg.			
	0.01	86.0	74.8	80.4	81.3	80.1	80.7	77.3	93.9	85.6	SD	7.765
	0.025	90.9	89.3	90.1	74.6	81.7	78.2	89.3	81.8	85.6	Mean	86.4
	0.05	84.0	85.6	84.8	81.6	81.2	81.4	81.7	82.3	82.0	UCL	109.7
	0.1	109.0	107.0	108.0	85.0	84.1	84.6	86.0	89.0	87.5	UWL	101.9
	0.25	99.2	91.7	95.5	76.6	76.5	76.6	85.2	85.4	85.3	LWL	70.8
0.5	92.4	89.0	90.7	94.4	96.4	95.4	84.1	81.0	82.6	LCL	63.1	

Fonofos	0.01	95.5	89.9	92.7	92.2	87.6	89.9	108.0	84.3	96.2	SD	4.794
	0.025	90.3	92.8	91.6	82.6	82.2	82.4	85.6	84.2	84.9	Mean	88.6
	0.05	86.4	84.9	85.7	85.8	83.4	84.6	87.0	82.8	84.9	UCL	103.0
	0.1	86.5	87.2	86.9	86.6	85.5	86.1	89.3	88.0	88.7	UWL	98.2
	0.25	92.5	91.1	91.8	80.3	85.6	83.0	87.1	86.4	86.8	LWL	79.0
	0.5	91.7	90.4	91.1	101.0	101.0	101.0	88.8	86.3	87.6	LCL	74.3

Dimethoate	0.01	97.5	87.7	92.6	72.0	74.7	73.4	106.0	97.2	101.6	SD	14.969
	0.025	137.0	136.0	136.5	84.3	82.5	83.4	79.9	83.8	81.9	Mean	90.2
	0.05	102.0	98.7	100.4	79.1	80.2	79.7	73.7	73.6	73.7	UCL	135.1
	0.1	105.0	101.0	103.0	81.4	81.6	81.5	83.9	84.3	84.1	UWL	120.1
	0.25	99.7	97.8	98.8	76.0	76.4	76.2	88.9	88.3	88.6	LWL	60.3
	0.5	93.8	92.9	93.4	94.2	92.6	93.4	82.6	80.7	81.7	LCL	45.3

Methyl Parathion	0.01	89.3	81.8	85.6	82.3	76.5	79.4	93.9	82.3	88.1	SD	12.244
	0.025	106.0	99.0	102.5	80.5	81.0	80.8	83.5	81.0	82.3	Mean	89.4
	0.05	103.0	96.9	100.0	76.7	78.7	77.7	74.4	75.3	74.9	UCL	126.1
	0.1	120.0	116.0	118.0	79.9	80.0	80.0	86.1	88.8	87.5	UWL	113.9
	0.25	108.0	105.0	106.5	75.6	75.6	75.6	90.3	91.5	90.9	LWL	64.9
	0.5	104.0	104.0	104.0	92.9	94.1	93.5	82.8	82.1	82.5	LCL	52.7

DEF	0.01	82.6	76.6	79.6	76.2	78.8	77.5	83.2	82.9	83.1	Sd	6.175
	0.025	91.5	93.5	92.5	93.8	83.5	88.7	82.9	83.6	83.3	Mean	88.0
	0.05	94.8	92.5	93.7	83.5	82.8	83.2	83.4	83.3	83.4	UCL	106.5
	0.1	91.6	91.9	91.8	88.7	87.6	88.2	87.1	87.8	87.5	UWL	100.4
	0.25	98.2	97.1	97.7	83.9	83.2	83.6	88.4	87.7	88.1	LWL	75.7
	0.5	96.5	94.9	95.7	99.4	99.9	99.7	88.5	86.6	87.6	LCL	69.5

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Determination of Azoxystrobin, Kresoxim methyl, Pyraclostrobin and Trifloxystrobin in Surface Water by Ultra Performance Liquid Chromatography Coupled to Tandem Mass Spectrometry

1. Scope:

This section method (SM) provides stepwise procedure for azoxystrobin, kresoxim methyl, pyraclostrobin, and trifloxystrobin analysis in surface water. It is followed by all authorized EA personnel.

2. Principle:

The azoxystrobin, kresoxim methyl, pyraclostrobin, and trifloxystrobin are extracted from the surface water sample with methylene chloride. The extract is passed through sodium sulfate to remove residual water. The anhydrous extract is evaporated on a rotary evaporator and then a solvent exchange is performed with methanol. The extract is concentrated to a final volume of 1 mL and then vialled into an autosampler vial for analysis on Ultra Performance Liquid Chromatography (UPLC) coupled to a positive electrospray ionization triple quadrupole mass spectrometry (ES-LC/MS/MS).

3. Safety:

- 3.1 All general laboratory safety rules for sample preparation and analysis shall be followed.
- 3.2 Methylene chloride is a regulated and controlled carcinogenic hazardous substance. It must be stored and handled in accordance with California Code of Regulations, Title 8, Subchapter 7, Group 16, Article 110, Section 5202.

4. Interferences:

There were no matrix interferences for azoxystrobin, kresoxim methyl, pyraclostrobin, and trifloxystrobin at the time of method development.

5. Apparatus and Equipment:

- 5.1 Rotary Evaporator (Buchi/Brinkman or equivalent)
- 5.2 Nitrogen Evaporator (Meyer N-EVAP Organomation Model #112 or equivalent)
- 5.3 Balance (Mettler PC 4400 or equivalent)
- 5.4 Vortex-vibrating mixer

5.5 UPLC equipped with a triple quadrupole mass spectrometry and ES ion source.

.6. Reagents and Supplies:

- 6.1 Azoxystrobin CAS#131860-33-8
- 6.2 Kresoxim methyl CAS#143390-89-0
- 6.3 Pyraclostrobin CAS#175013-18-0
- 6.4 Trifloxystrobin CAS#141517-89-0
- 6.5 Methylene Chloride, nanograde or equivalent pesticide grade
- 6.6 Water, MS grade, Burdick & Jackson or equivalent
- 6.7 Methanol, MS grade, Burdick & Jackson or equivalent
- 6.8 Formic Acid, HPLC grade
- 6.9 Ammonium formate, reagent grade or equivalent
- 6.10 Separatory funnel, 2 L
- 6.11 Boiling flask, 500 mL
- 6.12 Sodium Sulfate, ACS grade
- 6.13 Funnels, long stem, 60°, 100 mm I.D.
- 6.14 Volumetric Pipette, 0.5 mL
- 6.15 Graduated conical tubes with glass stopper, 15 mL
- 6.16 Glass wool, Pyrex® fiber glass slivers 8 microns
- 6.17 Disposable Pasteur pipettes, and other laboratory ware as needed
- 6.18 Recommended analytical column:
Waters Acquity BEH C18 1.7µm, 2.1 x 100 mm
- 6:19 Aqueous Solution: For 500 mL, mix 470 ± 2mL water, 25 ± 0.5 mL methanol, 4.50 ± 0.25 mL 1 M ammonium formate and 0.5 ± 0.05 mL formic acid.
- 6.20 Organic Solution: For 500mL, mix 450 ± 2mL methanol and 45 ± 0.5 mL water with 4.50 ± 0.25 mL 1 M ammonium formate and 0.5 ± 0.05 mL formic acid.

7. Standards Preparation:

7.1 The individual stock standards of 1.0 mg/mL were obtained from the CDFA/CAC Standards Repository. The standards were diluted to 10 µg/mL with methanol for identification purposes.

A combination standard of 1 µg/mL was prepared from the individual 10 µg/mL standards with methanol. The standard was also used to dilute the following concentrations: 0.025, 0.05, 0.1, 0.25 and 0.5 µg/mL in methanol for instrument calibration.

7.2 Keep all standards in the designated refrigerator for storage.

7.3 The expiration date of each standard is six months from the preparation date.

8. Sample Preservation and Storage:

Store all samples waiting for extraction in a separate refrigerator (4 ± 3 °C).

9. Test Sample Preparation:

9.1 Background Preparation

The Department of Pesticide Regulations (DPR) provides the background water for matrix blank and spikes.

9.2 Preparation of blank and spike

Matrix blank: Weigh out 1000 g of background water and follow the test sample extraction procedure.

Matrix spike: Weigh out 1000 g of background water. Spike a client requested amount of fungicides into the background water, mix well and let it stand for 1 minute. Follow the test sample extraction procedure.

9.3 Test Sample Extraction

9.3.1 Remove samples from the refrigerator and allow them to reach ambient temperature.

9.3.2 Record the weight of water samples to 0.1 g by subtracting the weight of the sample container before and after water has been transferred into a separatory funnel.

9.3.3 Shake with 100 ± 5 mL of methylene chloride for 2 minutes. Vent frequently to relieve pressure.

9.3.4 After phases have separated, drain the lower methylene chloride layer through 25 ± 4 g of anhydrous sodium sulfate and glass wool into a 500 mL boiling flask.

- 9.3.5 Repeat steps 9.3.3 & 9.3.4 two more times using 80 ± 5 mL of methylene chloride and shake for 1 minute each time. Combine the extracts in the same boiling flask.
- 9.3.6 After draining the final extraction, rinse the sodium sulfate with 25 ± 5 mL of methylene chloride.
- 9.3.7 Evaporate the sample extract to 2 - 4 mL on a rotary evaporator using a water bath at 35 ± 2 °C and 15 – 20 inch Hg vacuum. Add 2-4 mL of methanol and rotoevaporate to 1-2 mL. Transfer the extract to a calibrated 15 mL graduated test tube.
- 9.3.8 Rinse flask 3 more times with 2 - 4 mL of methanol and transfer each rinse to the same test tube.
- 9.3.9 Evaporate the sample extract to a volume slightly less than 1 mL in a water bath at 40 ± 2 °C under a gentle stream of nitrogen. Then bring to a final volume of 1.0 mL with methanol, mix well and transfer to an autosampler vial. Submit extract for LC-MS analysis.

10. Instrument Calibration:

- 10.1 The calibration standard curve consists of a minimum of three levels. The lowest level must be at or below the corresponding reporting limit. The current working standard levels are 0.025, 0.05, 0.1, 0.25, and 0.5 µg/mL.
- 10.2 Calibration is obtained using a linear or quadratic regression with the correlation coefficient (r) equal to or greater than 0.995, with all levels weighted 1/x.

11. Analysis:

11.1 Injection Scheme

The LC-MS needs to be conditioned with standard or a sample extract 2 to 5 runs before running the following sequence: A set of calibration standards, a matrix blank, a matrix spike, a set of up to 12 test samples, then a set of standards, etc.

11.2 UPLC-MS/MS

11.2.1 UPLC Instrument: Waters Acquity Ultra Performance LC
Column: Waters Acquity BEH C18 1.7µm, 2.1 x 100 mm
Column Temperature: 60 °C
Mobile Phase: Gradient
Solvent 1: Aqueous Solution
Solvent 2: Organic Solution
Gradient:

<u>Time(min)</u>	<u>Flow rate (mL/min)</u>	<u>Solvent 1</u>	<u>Solvent 2</u>
0	0.60	90.0	10.0
0.5	0.60	90.0	10.0
7.0	0.60	10.0	90.0
8.80	0.60	10.0	90.0
9.50	0.60	90.0	10.0
10.5	0.60	90.0	10.0

Injection Volume: 1.0 µL

11.2.2 Mass Spectrometry and Operating Parameters
Model: Waters Xevo Triple Quadrupole
Ion ProbeType: Electrospray Ionization (ES)
Ion Mode: ES+
Source Temp: 150 °C

Compound	Retention Time (min)	Precursor ion	Product Ion	Dwell (s)	Cone(V)	Collision Energy/-ev
Azoxystrobin	5.53	404.21	344.21	0.028	6.0	24.0
		404.21	372.22	0.028	6.0	14.0
Kresoxim-methyl	6.41	314.16	115.99	0.028	14.0	12.0
		314.23	267.14	0.028	18.0	6.0
Pyraclostrobin	6.68	388.19	163.10	0.028	10.0	26.0
		388.19	194.08	0.028	10.0	12.0
Trifloxystrobin	7.04	409.23	145.00	0.028	10.0	44.0
		409.23	186.06	0.028	10.0	18.0

Quantitation ions are in bold.

12. Quality Control:

12.1 Method Detection Limits (MDL)

Method Detection Limit (MDL) refers to the lowest concentration of the analyte that a method can detect reliably. To determine the MDL, 7 well water samples are spiked at 0.1ppb and processed through the entire method along with a blank. The standard deviation derived from the spiked sample recoveries was used to calculate the MDL for each analyte using the following equation:

$$\text{MDL} = tS$$

Where t is the Student t test value for the 99% confidence level with n-1 degrees of freedom and S denotes the standard deviation obtained from n replicate analyses. For the n=7 replicates used to determine the MDL, $t=3.143$.

The results for the standard deviations and MDL are in Appendix 1.

12.2 Reporting Limit (RL)

Reporting limit (RL) refers to a level at which reliable quantitative results may be obtained. The MDL is used as a guide to determine the RL. The RL is chosen in a range 1-5 times the MDL, as per client agreement. The reporting limit for azoxystrobin, kresoxim-methyl, pyraclostrobin, and trifloxystrobin is 0.05 ppb.

12.3 Method Validation

The method validation consisted of five sample sets. Each set included five levels of fortification and a method blank. All spikes and method blanks were processed through the entire analytical method. Spike levels and recoveries for the analytes are shown in Appendix 2.

12.4 Control Charts and Limits

Control charts were generated using the data from the method validation for each analyte. The upper and lower warning and control limits are set at ± 2 and 3 standard deviations of the percent recovery, respectively, shown in Appendix 2.

12.5 Acceptance Criteria

12.5.1 Each set of samples will have a matrix blank and a spiked matrix sample.

12.5.2 The retention time should be within ± 2 percent of that of the standards.

12.5.3 The recoveries of the matrix spikes shall be within the control limits.

12.5.4 The sample shall be diluted if results fall outside of the calibration curve.

13. Calculations:

Quantitation is based on an external standard (ESTD) calculation using either the peak area or height. The triple quadrupole LCMS software used a linear curve fit, with all levels weighted $1/x$. Alternatively, at the chemist's discretion, sample results may be calculated using the response factor for the standard.

$$\text{ppb} = \frac{(\text{sample peak area or ht}) \times (\text{std conc}) \times (\text{std vol. injected}) \times (\text{final vol of sample})(1000 \mu\text{L/mL})}{(\text{std. peak area or ht}) \times (\text{sample vol injected}) \times (\text{sample wt (g)})}$$

14. Reporting Procedure:

Sample results are reported out according to the client's analytical laboratory specification sheets.

15. Discussion and References:

A storage stability study was done with this project. The storage stability study consisted of a 1.0 ppb spike level and 3 replicates over a 28 day period. Twenty-one liters of background surface water were spiked and then transferred to twenty-one of the one liter amber bottles. These spiked samples were stored in the refrigerator until analyzed on 0, 2, 4, 7, 14, 21 and 28 days. Along with the storage spikes a blank and method control spike were also extracted. This storage study showed no significant degradation for these compounds within the 28 days. Results for the storage study are shown in Appendix 3.

16. References:

- 16.1 Hsu, J and White, J; *Determination of Azoxystrobin, Azoxystrobin Acid, Azoxystrobin Z-metabolite, Dicloran, Iprodione, Isoiprodione, Vinclozalin and 3,5-Dichloroaniline in Well Water*, 2010 Environmental Analysis Method, Center for Analytical Chemistry, CDFA
- 16.2 *EURL-FV Multiresidue Method using QuEChERS followed by GC-QqQ/MS/MS and LC-QqQ/MS/MS for Fruits and Vegetables*, 2009 European Union Reference Laboratory –Fruit and Vegetables,EURL-FV

Appendix 1

The Determination of Method Detection Limit (MDL) and Reporting Limit (RL)

Lab #	Spk\Analyte	Azoxystrobin	Kresoxim methyl	Pyraclostrobin	Trifloxystrobin
2011-0062	blk	nd	nd	nd	nd
2011-0063	0.1ppb spk 1	0.085	0.083	0.082	0.077
2011-0064	0.1ppb spk 2	0.092	0.090	0.091	0.075
2011-0065	0.1ppb spk 3	0.099	0.098	0.097	0.084
2011-0066	0.1ppb spk 4	0.088	0.085	0.084	0.073
2011-0067	0.1ppb spk 5	0.088	0.086	0.085	0.070
2011-0068	0.1ppb spk 6	0.102	0.098	0.099	0.084
2011-0069	0.1ppb spk 7	0.102	0.091	0.092	0.081
	SD	0.00718	0.00604	0.00658	0.00547
Reported	MDL	0.0225	0.0190	0.0207	0.0172
	RL	0.05	0.05	0.05	0.05

All concentrations are expressed in ppb.

Appendix 3

Storage Stability Study

Analyte	Recovery %	Day 0	Day 2	Day 4	Day 7	Day 14	Day 21	Day 28
Azoxystrobin	blank	nd	nd	nd	nd	nd	nd	nd
	QC Spk		83.4%	96.9%	98.8%	91.8%	84.0%	115.0%
	Spike 1	81.6%	87.4%	86.5%	79.9%	88.7%	77.1%	92.8%
	Spike 2	89.1%	79.2%	90.8%	78.3%	95.7%	92.7%	90.6%
	Spike 3	85.4%	83.4%	89.9%	76.6%	97.0%	92.9%	88.4%
Kresoxim-methyl	blank	nd	nd	nd	nd	nd	nd	nd
	QC Spk		81.5%	94.0%	95.3%	82.5%	85.0%	115.0%
	Spike 1	79.8%	83.1%	82.7%	74.2%	86.9%	69.4%	80.9%
	Spike 2	88.6%	76.5%	88.4%	71.8%	86.8%	81.8%	77.8%
	Spike 3	84.8%	77%	87.0%	70.3%	87.8%	82.9%	75.8%
Pyraclostrobin	blank	nd	nd	nd	nd	nd	nd	nd
	QC Spk		80.9%	94.1%	96%	88.4%	82.4%	114%
	Spike 1	78.5%	84.7%	83.2%	75.4%	86.8%	76.3%	89.5%
	Spike 2	86.5%	77.5%	88.1%	73.8%	92.5%	89.8%	88.1%
	Spike 3	83.9%	79.0%	86.4%	72.3%	94.8%	90.6%	86.9%
Trifloxystrobin	blank	nd	nd	nd	nd	nd	nd	nd
	QC Spk		72.3%	86.8%	82.9%	72.1%	75.5%	103.6%
	Spike 1	73.8%	70.0%	72.9%	65%	81.5%	61.1%	69.5%
	Spike 2	77.8%	61.1%	78.4%	62.5%	80.5%	72.8%	71.3%
	Spike 3	74.6%	65.1%	73.4%	62.6%	77.6%	72.1%	72.3%

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