



Department of Pesticide Regulation



Brian R. Leahy
Director

MEMORANDUM

Edmund G. Brown Jr.
Governor

TO: Lisa Ross, Ph.D., Environmental Program Manager I

FROM: David Kim
Staff Environmental Scientist
Environmental Monitoring
(916) 324-4340

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SUBJECT: RESULTS FOR THE MONITORING OF IMIDACLOPRID AND
CYFLUTHRIN USED IN THE ASIAN CITRUS PSYLLID ERADICATION
PROGRAM IN IMPERIAL COUNTY (STUDY# 258)

INTRODUCTION

In August 2008, the California Department of Food and Agriculture's (CDFA) Pest Detection/Emergency Projects Branch detected the Asian citrus psyllid (ACP) in San Diego and Imperial counties. ACP is an invasive insect pest that can spread Huanglongbing (HLB) disease, a bacterial disease of citrus trees. The disease produces bitter, unmarketable fruit; there is no known treatment except tree removal. Worldwide, HLB disease has been found in the United States (Florida), Mexico, South America, Asia and Africa.

Subsequent to the find of ACP in San Diego and Imperial counties, detections were confirmed in Los Angeles, Orange, San Bernardino, Ventura and Riverside counties. Widespread ACP detections in Mexico (along the California border) prompted an eradication program in Mexico.

In November, 2009, CDFA began an extensive ACP eradication program utilizing the pesticides imidacloprid and cyfluthrin. At the request of CDFA, the Environmental Monitoring Branch of the Department of Pesticide Regulation (DPR) has developed a protocol¹ for monitoring imidacloprid and cyfluthrin treatments, and DPR staff is overseeing the pesticide monitoring.

Monitoring results summarized in this document include imidacloprid and cyfluthrin treatments in Imperial County for three sites and two treatment dates, March 17, 2009 and May 12, 2009. Air, vegetation (fruit and leaf), soil and surface water monitoring results are presented.

Description of Application

Over 50,000 properties have been treated in Imperial, San Diego, and Los Angeles counties under the ACP eradication program. Treatment consisted of a soil drench of imidacloprid around citrus tree trunks followed by a foliar application of cyfluthrin to all citrus trees on each property. Soil drench applications of Merit[®] 2F, with 21.4 percent active ingredient (a.i.) of

¹ Protocol available at: http://www.cdpr.ca.gov/docs/emon/epests/asiancitruspsyllid/acp_monitoring_prc.



imidacloprid², were delivered at a dilution rate of 16 ounces per 100 gallons of water (two gallons per inch of trunk growth) through a Bean Spray Gun with a #10 tip attached to a 300 foot hose connected to the application truck tank. Foliar applications of Tempo[®] SC Ultra (Bayer), 11.8 percent a.i. of β -cyfluthrin, were made at a dilution rate of 2.2 ounces Tempo[®] SC Ultra per 100 gallons of water. The pesticide was delivered through a Wheaton[®] Treegun equipped with a #8 nozzle tip attached to a 300 foot hose connected to the application truck tank. All applications were performed or supervised by CDFA staff.

MATERIALS AND METHODS

The materials and methods used for monitoring imidacloprid and cyfluthrin treatments in Imperial County are described below. Air, vegetation and soil were sampled at various pesticide application intervals: pre-treatment (background), treatment, and post-treatment. Surface water was sampled downstream of the treatment area. The pesticide application tank was also sampled to establish pesticide concentrations at the time of treatment. Table 1 identifies the number of samples collected and analyzed for imidacloprid and cyfluthrin for each sampling medium at each treatment site. Table 2 identifies the analytical methods used for each sampling medium. All samples were analyzed by CDFA's Center for Analytical Chemistry.

Sampling Sites

Three sampling sites were selected for Imperial County; these sites are identified as I 1 and I 2, located in Calexico, and I 5 in Heber; surface water samples were collected from the New River (Figure 1). Treatment for sites I 1 and I 2 took place on March 17, 2009; treatment for site I 5 occurred on May 12, 2009.

Air Sampling

All air samples were collected using XAD- 2 tubes (SKC# 226-30-02) and SKC air samplers (SKC# 224-PCXR8) calibrated at approximately 3 liters-per-minute. Air sampling equipment was located outdoors in an open area. Samples were collected at the following treatment intervals: 1) 12-18 hours prior to pesticide application; 2) the duration of the application plus one hour; and 3) the interval immediately following the application period sample (sample #2), plus 24 hours. Samples were stored on dry ice until delivered to the laboratory for analysis.

Leaf Sampling

Whole leaves were collected in close proximity to air monitoring sites. Two samples were collected: one prior to foliar application and the second after the spray had dried (one hour after treatment). Before- and after-treatment samples were collected from the same trees. Samples

² The mention of commercial products, their source, or use in connection with this eradication project is not to be construed as an actual or implied endorsement of such products.

were placed in a 4-ounce glass jar sealed with a Teflon[®]-lined lid, stored on wet ice, and delivered to the laboratory within 24 hours.

Fruit Sampling

Fruit samples were collected at the time of pesticide treatment if the fruit was ripe; this was done to confirm tolerances³ were not exceeded. Each sample was a composite of multiple fruit samples collected from a single property or tree. Samples were collected at various intervals when mature fruit was available; background samples were collected prior to pesticide application, post-application samples were collected after spray residue had dried. All samples were collected in paper bags and stored on wet ice until delivered to the laboratory.

Soil Sampling

Soil was sampled at treatment sites to measure the concentration of imidacloprid and cyfluthrin in soil before and after treatment. Each sample consisted of three randomly selected soil cores, taken to a depth of 1 inch. Cores were collected using a 2-1/2 inch (28.56 square centimeter [cm²]) diameter stainless steel tube and composited into one wide mouth Mason[®] jar with an aluminum foil lined lid. All samples were stored on dry ice (or frozen at -20°C) until delivered to the laboratory.

Surface Water Sampling

Surface water samples were collected near the treatment site. Samples were collected in one liter amber glass bottles with Teflon[®] lined lids. Surface water samples were stored and transported refrigerated (on wet or blue ice) until delivered to the laboratory.

Tank Mixture Sampling

Tank mixtures were sampled to establish the concentration of imidacloprid and cyfluthrin in the spray material. Samples were collected from treatment spray guns during or immediately after treatment. Samples consisted of half filled 500 milliliter Nalgene[®] wide mouth bottles; each bottle was triple bagged and kept on wet ice or refrigerated until delivered to the laboratory.

Quality Control

The CDFA Center for Analytical Chemistry analyzed all samples collected for this monitoring study. Standard operating procedures for continuing quality control (QC) measures are specified in QA/QC 001.00 (<http://www.cdpr.ca.gov/docs/emon/pubs/sops/qaqc001.pdf>). Continuing QC samples are evaluated by laboratory chemists and adjustments are made to the analytical equipment on an as-needed basis to ensure analytical integrity.

³ In this context, the term *tolerances* refers to the U.S EPA limits placed on the amount of pesticide residue that can be left on foods marketed in the United States. For more information regarding pesticide tolerances, see <http://www.epa.gov/opp00001/regulating/tolerances.htm> and <http://www.epa.gov/opp00001/food/viewtols.htm>.

RESULTS AND DISCUSSION

Air

A total of 12 air samples were collected. Three samples were collected for each pesticide at site I 1 for the March 17, 2009, treatment; similarly, three samples were collected for each pesticide at the I 5 site for the treatment on May 12, 2009. All samples contained no detectable amount of either imidacloprid or cyfluthrin in the pre-treatment, treatment and post-treatment samples (Table 3).

Acute inhalation screening levels have been developed by DPR in consultation with the Office of Environmental Health Hazard Assessment for imidacloprid and cyfluthrin at 150 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) and $1.04 \mu\text{g}/\text{m}^3$, respectively. Imidacloprid reporting limits were low enough to ensure air concentrations did not exceed acute screening levels. Pre- and post-treatment reporting limits for cyfluthrin were also adequate. In contrast, due to a short sampling period during cyfluthrin treatment (under three hours), the reporting limits were $1.1 \mu\text{g}/\text{m}^3$. However, homeowners were not present in the yard during treatment and therefore not exposed to cyfluthrin residues at this time. In addition, cyfluthrin was not detected in samples collected after treatment and the acute screening level was not exceeded.

Leaf Samples

Pre- and post-treatment whole leaf samples were collected at monitoring sites I 1 and I 2 for the March 17, 2009, treatment and analyzed for total residue (Table 4). All pre-treatment samples contained no detectable residues of either imidacloprid or cyfluthrin; a single post-treatment sample from site I 1 contained 0.06 parts per million (ppm) of cyfluthrin.

Fruit Samples

Fruit samples (lemon rind and pulp) from treatment site I 1 and I 2 contained no detected imidacloprid residues for the March 17, 2009, treatment (Table 5).⁴ Samples collected at site I 5 for the May 12, 2009, treatment contained imidacloprid residues of 0.23 and 0.02 ppm for post-treatment composite samples collected 29 and 72 weeks post-treatment, respectively. Imidacloprid is a soil applied systemic pesticide that takes time to be taken up by plant roots and distributed to fruit hence residues are not detected immediately after application. In contrast, cyfluthrin is applied to plant surfaces and residues may be detected immediately after treatment. Cyfluthrin was detected (0.08 ppm) in a sample collected after the March 17, 2009, treatment at

⁴ Complete fruit sample results for all Imperial County sites are described in the July 29, 2011, memorandum from David Kim to Lisa Ross, subject line: *Preliminary results for the 2009-2010 fruit monitoring of imidacloprid and cyfluthrin used in the Asian citrus psyllid eradication program in Imperial, San Diego, and Los Angeles counties*. Memorandum available at http://www.cdpr.ca.gov/docs/emon/epests/asiancitrispsyllid/acp_fruit_prelimin_results_july_2011.pdf.

site I 1 (0 weeks post-treatment), but was not detected in multiple samples from sites I 2 (March 17, 2009, treatment) and I 5 (May 12, 2009, treatment) for various post-treatment periods.

No fruit samples exceeded United States Environmental Protection Agency (U.S. EPA) tolerances for citrus (0.70 and 0.20 ppm for imidacloprid and cyfluthrin, respectively).

Soil Samples

Soil samples were collected from monitoring sites I 1 and I 2 (Table 6). Post-treatment samples from site I 1 exhibited residue levels of 26.9 ppm for imidacloprid and 0.06 ppm for cyfluthrin. Post-treatment samples from site I 2 contained 46.6 ppm imidacloprid and 0.11 ppm cyfluthrin.

Surface Water Samples

Surface water samples were collected from the New River. Two samples were collected: one sample was analyzed for imidacloprid, the other for cyfluthrin. A trace⁵ amount of imidacloprid was detected; cyfluthrin was not detected (Table 7).

No urban irrigation runoff was observed and urban rain runoff sampling was not attempted due to limited rainfall. Agricultural runoff samples collected from the New River and agricultural drains in Imperial County contained imidacloprid concentrations between not detected (ND) to 3.29 parts per billion (ppb) (Starner and Goh 2011).⁶

Tank Mix

Tank samples averaged concentrations of 0.029 and 0.0014 percent a.i. of imidacloprid and cyfluthrin, respectively, for treatment sites I 2 and I 5 (Table 8). Theoretical calculation of percent a.i. was 0.027 percent imidacloprid and 0.0020 percent cyfluthrin.

CONCLUSION

Imidacloprid and cyfluthrin monitoring for treatments in Imperial County on March 17, 2009, and May 12, 2009, yielded the following results:

- Pre-treatment, treatment and post-treatment air samples from treatment sites I 1 and I 5 contained no detected residues of imidacloprid or cyfluthrin.

⁵ A trace detection is defined as detection of the analyte above the method detection limit, but below the reporting limit. (The reporting limit for imidacloprid in surface water for this study was 0.05 parts per billion.) The determination of a trace detection is based upon the chemist's professional judgment.

⁶ Journal article *Detections of the neonicotinoid insecticide imidacloprid in surface waters of three agricultural regions of California, USA, 2010-2011* available at <http://www.springerlink.com/content/566t681j31233742/>.

- The post-treatment leaf sample from treatment site I 1 showed no detected imidacloprid residue, but contained 0.06 ppm of cyfluthrin. Pre- and post-treatment samples from site I 2 contained no detected residues of imidacloprid; samples from I 2 were not analyzed for cyfluthrin.
- No whole fruit samples (lemon rind and pulp) exceeded U.S. EPA tolerances for citrus. Fruit samples from treatment site I 1 and I 2 contained no detected imidacloprid residues for the March 17, 2009, treatment. Samples collected at site I 5 for the May 12, 2009, treatment contained imidacloprid residues of 0.23 and 0.02 ppm for post-treatment composite samples collected 29 and 72 weeks post-treatment, respectively. Cyfluthrin was detected (0.08 ppm) in a sample collected at site I 1 0 weeks post-treatment (for the March 17, 2009, treatment), but was not detected in multiple samples from sites I 2 (March 17, 2009, treatment) and I 5 (May 12, 2009, treatment) for various post-treatment periods.
- Post-treatment soil samples collected at sites I 1 and I 2 contained imidacloprid residue levels of 26.9 and 46.6 ppm, respectively. Post-treatment samples analyzed for cyfluthrin contained 0.06 and 0.11 ppm for sites I 1 and I 2, respectively.
- Two surface water samples were collected from the New River—one was analyzed for imidacloprid, the other for cyfluthrin. The sample analyzed for imidacloprid exhibited a trace detection; no cyfluthrin was detected in the sample analyzed for cyfluthrin.
- Tank samples collected at sites I 2 and I 5 over two treatment periods for imidacloprid yielded an average concentration of 0.029 percent a.i.; the theoretical calculation of the concentration was 0.027 percent.
- Tank samples collected at sites I 2 and I 5 over two treatment periods for cyfluthrin yielded an average concentration of 0.0014 percent a.i.; the theoretical calculation of the concentration was 0.0020 percent.

Table 1. Number of samples collected in Imperial County for imidacloprid and cyfluthrin in air, vegetation, soil, surface water and the application tank.

Sampling Medium	Treatment Site	Number of Samples Taken for Each Pesticide	
		Imidacloprid	Cyfluthrin
Air	I 1	3	3
	I 2	0	0
	I 5	3	3
Leaf	I 1	2	1
	I 2	2	0
	I 5	0	0
Fruit	I 1	2	2
	I 2	2	2
	I 5	3	2
Soil	I 1	2	2
	I 2	2	2
	I 5	0	0
Surface Water	New River	1	1
Tank Mixture	I 1	0	0
	I 2	1	1
	I 5	1	1

Table 2. Analytical methods used for imidacloprid and cyfluthrin in all sampling media. Reporting limits presented in micrograms per cubic meter ($\mu\text{g}/\text{m}^3$), parts per million (ppm), parts per billion (ppb) and percent.

Sampling Medium	Imidacloprid		Cyfluthrin	
	Analytical Method	Reporting Limit	Analytical Method	Reporting Limit
Air	[‡] EM 12.3	[†] 0.02 – 0.18 $\mu\text{g}/\text{m}^3$	EM 16.0 (Modified)	[†] 0.1 – 1.1 $\mu\text{g}/\text{m}^3$
Leaf	EM 12.4	0.1 ppm	EM 12.5 (Modified)	0.05 ppm
Fruit	^{‡‡} EM 12.5 PDP-SM-1 RES-SM-11	^{††} 0.01 – 0.05 ppm	^{‡‡} EM 12.5 PDP-SM-1 RES-SM-11	^{††} 0.01 – 0.05 ppm
Soil	EM 12.6	0.01 ppm	EM 52.9 (Modified)	0.01 ppm
Surface Water	EM 33.5	0.05 ppb	EMON-SM-05-023	0.02 ppb
Tank Mixture	EM 33.5	Percent	EMON-SM-05-023	Percent

[‡] Analytical methods protocols available at: http://www.cdpr.ca.gov/docs/emon/pubs/em_methd_main.htm

[†] The reporting limit for air samples varies from 0.02 to 0.18 $\mu\text{g}/\text{m}^3$ for imidacloprid and from 0.1 to 1.1 $\mu\text{g}/\text{m}^3$ for cyfluthrin due to the variation in sample collection duration (sample volume)

^{‡‡} List of all analytical methods used for fruit analysis during 2009-2010 monitoring

^{††} Reporting limits are between 0.01 and 0.05 ppm, depending on sample and analytical method used

Table 3. Results of air sampling in Imperial County for imidacloprid and cyfluthrin treatments on March 17, 2009 and May 12, 2009. Results are presented in micrograms per cubic meter ($\mu\text{g}/\text{m}^3$).

DPR Sample Number	Treatment Site	Sample Date	Sample Type	Amount Detected ($\mu\text{g}/\text{m}^3$)	Reporting Limit ($\mu\text{g}/\text{m}^3$)	
Imidacloprid	0121	I 1	03/17/2009	Pre-Treatment	†ND	0.02
	0122	I 1	03/17/2009	Treatment	ND	0.11
	0125	I 1	03/17/2009	Post-Treatment	ND	0.01
	0206	I 5	05/12/2009	Pre-Treatment	ND	0.02
	0220	I 5	05/12/2009	Treatment	ND	0.11
	0208	I 5	05/12/2009	Post-Treatment	ND	0.18
Cyfluthrin	0124	I 1	03/17/2009	Pre-Treatment	ND	0.1
	0123	I 1	03/17/2009	Treatment	ND	1.0
	0120	I 1	03/17/2009	Post-Treatment	ND	0.2
	0210	I 5	05/12/2009	Pre-Treatment	ND	0.2
	0215	I 5	05/12/2009	Treatment	ND	1.1
	0209	I 5	05/12/2009	Post-Treatment	ND	0.1

† Not detected; concentration below the reporting limit

Table 4. Results of leaf samples collected in Imperial County for imidacloprid and cyfluthrin treatments on March 17, 2009. Results are presented in parts per million wet weight (ppm).

DPR Sample Number	Treatment Site	Sample Type	Imidacloprid		Cyfluthrin	
			Amount Detected (ppm)	Reporting Limit (ppm)	Amount Detected (ppm)	Reporting Limit (ppm)
0127	I 1	Pre-Treatment	†ND	0.1	----	----
0130	I 2	Pre-Treatment	ND	0.1	----	----
0133	I 1	Post-Treatment	ND	0.1	0.06	0.05
0135	I 2	Post-Treatment	ND	0.1	----	----

† Not detected; concentration below the reporting limit

Table 5. Results of whole fruit (lemon rind and pulp) samples collected in Imperial County for imidacloprid and cyfluthrin treatments on March 17, 2009 and May 12, 2009. Results are presented in parts per million (ppm).

	Treatment Date	Treatment Site	Sample Date	Weeks After Treatment	Amount Detected (ppm)	Reporting Limit (ppm)	U.S. EPA Tolerance (ppm)
Imidacloprid	03/17/2009	I 1	03/17/2009	0	†ND	0.05	0.70
	03/17/2009	I 1	12/15/2010	89	ND	0.01	
	03/17/2009	I 2	03/17/2009	Background	ND	0.05	
	03/17/2009	I 2	03/17/2009	0	ND	0.05	
	05/12/2009	I 5	12/08/2009	29	0.23	0.01	
	05/12/2009	I 5	10/07/2010	72	0.02	0.02	
	05/12/2009	I 5	12/15/2010	81	ND	0.01	
Cyfluthrin	03/17/2009	I 1	03/17/2009	0	ND	0.05	0.20
	03/17/2009	I 1	03/17/2009	0	0.08	0.05	
	03/17/2009	I 2	03/17/2009	Background	ND	0.05	
	03/17/2009	I 2	03/17/2009	0	ND	0.05	
	05/12/2009	I 5	12/08/2009	29	ND	0.05	
	05/12/2009	I 5	10/07/2010	72	ND	0.01	

† Not detected; concentration below the reporting limit

Table 6. Results of soil sampling in Imperial County for imidacloprid and cyfluthrin treatments on March 17, 2009. Results are presented in parts per million (ppm).

DPR Sample Number	Treatment Site	Sample Type	Imidacloprid		Cyfluthrin	
			Amount Detected (ppm)	Reporting Limit (ppm)	Amount Detected (ppm)	Reporting Limit (ppm)
0126	I 1	Pre-Treatment	†ND	0.01	ND	0.01
0131	I 2	Pre-Treatment	ND	0.01	ND	
0132	I 1	Post-Treatment	26.9	0.01	0.06	
0134	I 2	Post-Treatment	46.6	0.01	0.11	

† Not detected; concentration below the reporting limit

Table 7. Results of surface water sampling in Imperial County for imidacloprid and cyfluthrin. Samples were taken from the New River on March 10, 2009. Reporting limits presented in parts per billion (ppb).

DPR Sample Number	Imidacloprid		Cyfluthrin	
	Amount Detected	Reporting Limit	Amount Detected	Reporting Limit
0116	†Trace	0.05 ppb	----	----
0118	----	----	††ND	0.02 ppb

† Analyte detected above the method detection limit, but below the reporting limit

†† Not detected; concentration below the reporting limit

Table 8. Results of tank sampling in Imperial County for imidacloprid and cyfluthrin treatments on March 17, 2009 and May 12, 2009.

	DPR Sample Number	Treatment Site	Tank Serial Number	Tank Sample Date	Amount Detected (Percent)	Average Concentration (Percent)
Imidacloprid	0136	I 2	1201776	03/17/2009	0.031	0.029
	0212	I 5	1201743	05/12/2009	0.027	
Cyfluthrin	0139	I 2	----	03/17/2009	0.0013	0.0014
	0213	I 5	1201743	05/12/2009	0.0014	

Figure 1. Monitoring sites I 1 and I 2 (Calexico), I5 (Heber), and the New River.

