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SUBJECT: RESULTS FOR THE MONITORING OF IMIDACLOPRID AND CYFLUTHRIN USED IN THE ASIAN CITRUS PSYLLID ERADICATION PROGRAM IN LOS ANGELES 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 protocol[1] for monitoring imidacloprid and cyfluthrin treatments, and DPR staff is overseeing the pesticide monitoring.

This document summarizes monitoring results for two imidacloprid and cyfluthrin treatments (September 24, 2009 and October 6, 2009) at two sites in Los Angeles County. Air, vegetation (fruit and leaf) and soil 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.

imidacloprid[2], 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 Los Angeles County are described below. Air, fruit and soil were sampled at various pesticide application intervals: pre-treatment (background), treatment, and post-treatment. 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

Two sampling sites were selected for Los Angeles County; these sites are identified as LA 1, located in the city of Los Angeles, and LA 5, in South El Monte (Figure 1).

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.

Fruit Sampling

Fruit samples were collected at the time of pesticide treatment if the fruit was ripe; this was done to confirm tolerances[3] 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; background samples were collected prior to pesticide application, post-application samples were

[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.

[3] In this context, the term *tolerances* refers to the United States Environmental Protection Agency 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.

collected when fruit was mature. 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 soil core 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 [cm^2]) 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.

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
No quality control samples were analyzed in conjunction with the sample analyses presented in this summary document.

RESULTS AND DISCUSSION

Air
A total of 12 air samples were collected (Table 3). Three samples were collected for each pesticide at the LA 1 site for the September 24, 2009, treatment; similarly, three samples were collected for each pesticide at the LA 5 site for treatment date October 6, 2009. All samples contained no detectable amount of either imidacloprid or cyfluthrin in the pre-treatment, treatment and post-treatment samples; reporting limits were below the acute human health screening levels of 150 micrograms per cubic meter ($\mu g/m^3$) for imidacloprid and 1.04 $\mu g/m^3$ for cyfluthrin.

Fruit Samples
Fruit samples (orange rind and pulp) were collected only at the LA 1 site on treatment date September 24, 2009 (Table 4). For this site, two samples were collected and analyzed for imidacloprid: one pre-treatment and one 19-week post-treatment sample.[4] The reporting limit

[4] Complete fruit sample results for multiple sites in Los Angeles County 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.

for chemical analysis of imidacloprid in citrus was 0.01 parts per million (ppm). Both samples were reported as non-detect, therefore no fruit samples exceeded the United States Environmental Protection Agency (U.S. EPA) tolerances for imidacloprid on citrus of 0.70 ppm.

Soil Samples
Soil samples were collected from both monitoring sites and analyzed for imidacloprid. Pre-treatment samples exhibited no detections; samples taken post-treatment exhibited residue levels of 119 ppm for the September 24, 2009, treatment at LA 1 and 20.7 ppm for the October 6, 2009, treatment at LA 5 (Table 5).

Tank Mix
Tank samples averaged concentrations of 0.026 and 0.0014 percent a.i. of imidacloprid and cyfluthrin, respectively (Table 6). Theoretical calculation of percent a.i. was 0.027 percent imidacloprid and 0.0020 percent cyfluthrin.

CONCLUSION

Imidacloprid and cyfluthrin monitoring in Los Angeles County for treatments on September 24, 2009 and October 6, 2009, yielded the following results.

- Pre-treatment, treatment and post-treatment air samples from treatment sites LA 1 and LA 5 contained no detected residues of imidacloprid or cyfluthrin.
- No fruit samples exceeded U.S. EPA tolerances for citrus. Whole fruit samples (orange rind and pulp) collected from treatment site LA 1 analyzed for imidacloprid contained no detected residues for the September 24, 2009, pre-treatment sample and 19-week post-treatment samples collected at maturity.
- Soil samples taken during the two imidacloprid treatments yielded 119 ppm at LA 1 for the September 24, 2009, treatment and 20.7 ppm at LA 5 for the October 6, 2009, treatment.
- Tank samples taken at LA 1 and LA 5 over the two treatment periods for imidacloprid yielded an average concentration of 0.026 percent a.i.; the theoretical calculation of the concentration was 0.027 percent.
- Tank samples taken at LA 1 and LA 5 over the 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 Los Angeles County for imidacloprid and cyfluthrin in air, fruit, soil and the application tank.

Sampling Medium	Treatment Site	Number of Samples Taken for Each Pesticide	
		Imidacloprid	Cyfluthrin
Air	LA 1	3	3
	LA 5	3	3
Fruit	LA 1	2	0
	LA 5	0	0
Soil	LA 1	2	0
	LA 5	2	0
Tank Mixture	LA 1	1	1
	LA 5	2	2

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

Sampling Medium	Imidacloprid		Cyfluthrin	
	Analytical Method	Reporting Limit	Analytical Method	Reporting Limit
Air	‡EM 12.3	†0.01 – 0.09 $\mu g/m^3$	EM 16.0 (Modified)	†0.1 – 0.9 $\mu g/m^3$
Fruit	††EM 12.5 PDP-SM-1 RES-SM-11	0.01 ppm	No Samples	No Samples
Soil	EM 12.6	0.01 ppm	No Samples	No Samples
Tank Mixture	HPLC	Percent	HPLC	Percent

‡ Protocols for analytical methods available at: http://www.cdpr.ca.gov/docs/emon/pubs/em_methd_main.htm

† The reporting limit for air samples varies from 0.01 to 0.09 $\mu g/m^3$ for imidacloprid and from 0.1 to 0.9 $\mu g/m^3$ 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

Table 3. Results of air sampling for imidacloprid and cyfluthrin from two sites in Los Angeles County for treatments on September 24, 2009 and October 6, 2009. Results are reported in micrograms per cubic meter (µg/m^3).

	DPR Sample Number	Treatment Site	Sample Date	Sample Type	Amount Detected (µg/m^3)	Reporting Limit (µg/m^3)
Imidacloprid	0330	LA 1	09/23-24/09	Pre-Treatment	†ND	0.09
	0201	LA 1	09/24/09	Treatment	ND	0.01
	0326	LA 1	09/24-25/09	Post-Treatment	ND	0.01
	0354	LA 5	10/05-06/09	Pre-Treatment	ND	0.03
	0359	LA 5	10/06/09	Treatment	ND	0.01
	0357	LA 5	10/06-07/09	Post-Treatment	ND	0.09
Cyfluthrin	0334	LA 1	09/23-24/09	Pre-Treatment	ND	0.9
	0327	LA 1	09/24/09	Treatment	ND	0.1
	0333	LA 1	09/24-25/09	Post-Treatment	ND	0.1
	0355	LA 5	10/05-06/09	Pre-Treatment	ND	0.1
	0358	LA 5	10/06/09	Treatment	ND	0.1
	0356	LA 5	10/06-07/09	Post-Treatment	ND	0.9

† Not detected; concentration below the reporting limit

Table 4. Results of whole fruit (orange rind and pulp) samples from the LA 1 site analyzed for imidacloprid. 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)
09/24/09	LA 1	09/24/2009	Background	†ND	0.01	0.70
09/24/09	LA 1	02/08/2010	19	ND		

† Not detected; concentration below the reporting limit

Table 5. Results of soil sampling for imidacloprid from two sites in Los Angeles County for treatments on September 24, 2009 and October 6, 2009. Results are presented in parts per million (ppm).

DPR Sample Number	Treatment Site	Sample Date	Sample Type	Amount Detected (ppm)	Reporting Limit (ppm)
0336	LA 1	09/24/09	Pre-Treatment	†ND	0.01
0340	LA 1	09/24/09	Post-Treatment	119	
0346	LA 5	10/06/09	Pre-Treatment	ND	
0343	LA 5	10/06/09	Post-Treatment	20.7	

† Not detected; concentration below the reporting limit

Table 6. Results of tank sampling for imidacloprid and cyfluthrin for treatments on September 24, 2009 and October 6, 2009 in Los Angeles County.

	DPR Sample Number	Treatment Site	Tank Serial Number	Tank Sample Date	Amount Detected (Percent)	Average Concentration (Percent)
Imidacloprid	0211	LA 1	Tank 1:8L18569	09/24/09	0.024	0.026
	0372	LA 5	1609404	10/06/09	0.031	
	0375	LA 5	1682711	10/06/09	0.022	
Cyfluthrin	0214	LA 1	Tank 2:8L18569	09/24/09	0.0014	0.0014
	0371	LA 5	1665855	10/06/09	0.0011	
	0374	LA 5	1706297	10/06/09	0.0016	

Figure 1. Monitoring sites LA 1 (Los Angeles) and LA5 (South El Monte).