



# Department of Pesticide Regulation



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## MEMORANDUM

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

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### 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 APC 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<sup>1</sup> 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 at two sites in San Diego County on March 26, 2009. 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<sup>®</sup> 2F, with 21.4 percent active ingredient (a.i.) of

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<sup>1</sup> Protocol available at: [http://www.cdpr.ca.gov/docs/emon/epests/asiancitruspsyllid/acp\\_monitoring\\_prc](http://www.cdpr.ca.gov/docs/emon/epests/asiancitruspsyllid/acp_monitoring_prc).



imidacloprid<sup>2</sup>, 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<sup>®</sup> SC Ultra (Bayer), 11.8 percent a.i. of  $\beta$ -cyfluthrin, were made at a dilution rate of 2.2 ounces Tempo<sup>®</sup> SC Ultra per 100 gallons of water. The pesticide was delivered through a Wheaton<sup>®</sup> 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 San Diego County are described below. Air, vegetation 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**

This summary document describes monitoring results from two treatment sites in San Diego County. These sites are identified as SD 1 and SD 2, both located in Jamul (Figure 1). Both sites were treated on March 26, 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 (or leaf punches) 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 leaves were collected from the same

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<sup>2</sup> 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.

trees. Samples were placed in a 4-ounce glass jar sealed with a Teflon<sup>®</sup>-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 tolerances<sup>3</sup> 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 [cm<sup>2</sup>]) 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<sup>®</sup> 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.

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<sup>3</sup> 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 at monitoring sites SD 1 and SD 2. Three samples were collected for each pesticide at each treatment site. 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. Acute inhalation screening levels were not exceeded as imidacloprid and cyfluthrin were not detected in air samples and reporting limits were low enough to detect residues at these levels.

### Leaf Samples

Pre- and post-treatment leaf samples were collected at monitoring sites SD 1 and SD 2 for the March 26, 2009, treatment. Whole leaf samples were analyzed for total residue; leaf punch samples were analyzed for dislodgeable residue (Table 4). All pre-treatment samples contained no detectable residues of either imidacloprid or cyfluthrin; a single post-treatment whole leaf sample at site SD 2 contained 0.06 parts per million (ppm) of cyfluthrin (total residue).

### Fruit Samples

Whole fruit samples (orange rind and pulp) were collected at SD 1 and SD 2.<sup>4</sup> A single sample from site SD 1 contained 0.02 ppm of imidacloprid 35 weeks post-treatment (Table 5). 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 are detected immediately after treatment. One sample each from sites SD 1 and SD 2 contained 0.11 ppm of cyfluthrin 0 weeks post-treatment.

No fruit samples exceeded the United States Environmental Protection Agency (U.S. EPA) tolerances for citrus (0.70 and 0.20 ppm for imidacloprid and cyfluthrin, respectively). Reporting limits for both pesticides were low enough to detect residues above the tolerance limits for citrus.

In addition to whole fruit analysis (which is required for tolerance testing), oranges were peeled and analyzed for cyfluthrin residues. These samples were all non-detects indicating one can reduce exposure to surface applied pesticides by peeling fruit.

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<sup>4</sup> Complete fruit sample results for all San Diego 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](http://www.cdpr.ca.gov/docs/emon/epests/asiancitrispsyllid/acp_fruit_prelimin_results_july_2011.pdf).

### **Soil Samples**

Soil samples were collected from monitoring sites SD 1 and SD 2 and analyzed for imidacloprid. Post-treatment samples exhibited residues levels of 22.4 ppm and 12.2 ppm from sites SD 1 and SD 2, respectively (Table 6).

### **Tank Mix**

Sample analyses of percent a.i. in the tank mixture used at sites SD 1 and SD 2 exhibited levels of 0.028 and 0.0026 percent imidacloprid and cyfluthrin, respectively (Table 7). (The same tank mixture was used at both sites.) Theoretical calculation of percent a.i. was 0.027 percent imidacloprid and 0.0020 percent cyfluthrin.

## **CONCLUSION**

Imidacloprid and cyfluthrin monitoring in San Diego County for the March 26, 2009, treatments yielded the following results:

- Pre-treatment, treatment and post-treatment air samples from sites SD 1 and SD 2 contained no detected residues of imidacloprid or cyfluthrin.
- Pre- and post-treatment leaf samples from treatment sites SD 1 and SD 2 analyzed for imidacloprid exhibited no treatment residues; a post-treatment sample from SD 2 contained 0.06 ppm of cyfluthrin (total residue).
- No fruit samples (orange rind and pulp) exceeded the U.S. EPA tolerances for citrus. Fruit samples from treatment sites SD 1 and SD 2 exhibited the following residues: a single sample from Site SD 1 contained 0.02 ppm of imidacloprid 35 weeks post-treatment; single samples from sites SD 1 and SD 2 each contained 0.11 ppm of cyfluthrin 0 weeks post-treatment. Peeled oranges did not contain detectable amounts of cyfluthrin.
- Post-treatment soil samples collected at sites SD 1 and SD 2 contained imidacloprid residue levels of 22.4 and 12.2 ppm, respectively.
- The application tank used for imidacloprid treatments at sites SD 1 and SD 2 yielded a concentration of 0.028 percent a.i.; the theoretical calculation of the concentration was 0.027 percent.
- The application tank used for cyfluthrin treatments at sites SD 1 and SD 2 yielded a concentration of 0.0026 percent a.i.; the theoretical calculation of the concentration was 0.0020 percent.

**Table 1.** Number of samples collected in San Diego County for imidacloprid and cyfluthrin in air, vegetation, soil and the application tank.

Sampling Medium	Treatment Site	Number of Samples Taken for Each Pesticide	
		Imidacloprid	Cyfluthrin
Air	SD 1	3	3
	SD 2	3	3
Leaf	SD 1	4	4
	SD 2	4	4
Fruit	SD 1	4	4
	SD 2	2	3
Soil	SD 1	2	0
	SD 2	2	0
Tank Mixture	SD 1	1	1
	SD 2		

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

Sampling Medium	Imidacloprid		Cyfluthrin	
	Analytical Method	Reporting Limit	Analytical Method	Reporting Limit
Air	<sup>‡</sup> EM 12.3	<sup>†</sup> 0.01 – 0.06 $\mu\text{g}/\text{m}^3$	EM 16.0 (Modified)	<sup>†</sup> 0.1 – 0.6 $\mu\text{g}/\text{m}^3$
Leaf (Total Residue)	EM 12.5 (Modified)	0.1 ppm	EM 12.5 (Modified)	0.05 ppm
Leaf (Dislodgeable)	EM 12.4	0.25 $\mu\text{g}/\text{cm}^2$	WHS-SM-1	0.25 $\mu\text{g}/\text{cm}^2$
Fruit	<sup>++</sup> EM 12.5 PDP-SM-1 RES-SM-11	* 0.01 – 0.05 ppm	<sup>++</sup> EM 12.5 PDP-SM-1 RES-SM-11	0.05 ppm
Soil	EM 12.6	0.01 ppm	No Samples	No Samples
Tank Mixture	EM 33.5	Percent	HPLC	Percent

<sup>‡</sup> Analytical methods protocols available at: [http://www.cdpr.ca.gov/docs/emon/pubs/em\\_methd\\_main.htm](http://www.cdpr.ca.gov/docs/emon/pubs/em_methd_main.htm)

<sup>†</sup> The reporting limit for air samples varies from 0.01 to 0.06  $\mu\text{g}/\text{m}^3$  for imidacloprid and from 0.1 to 0.6  $\mu\text{g}/\text{m}^3$  for cyfluthrin due to the variation in sample collection duration (sample volume)

<sup>++</sup> 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 San Diego County for imidacloprid and cyfluthrin treatments on March 26, 2009. Results are presented in micrograms per cubic meter ( $\mu\text{g}/\text{m}^3$ ).

	DPR Sample Number	Treatment Site	Sample Type	Amount Detected ( $\mu\text{g}/\text{m}^3$ )	Reporting Limit ( $\mu\text{g}/\text{m}^3$ )
Imidacloprid	0183	SD 1	Pre-Treatment	<sup>†</sup> ND	0.01
	0177	SD 1	Treatment	ND	0.06
	0182	SD 1	Post-Treatment	ND	0.01
	0175	SD 2	Pre-Treatment	ND	0.01
	0179	SD 2	Treatment	ND	0.06
	0174	SD 2	Post-Treatment	ND	0.01
Cyfluthrin	0181	SD 1	Pre-Treatment	ND	0.1
	0176	SD 1	Treatment	ND	0.6
	0178	SD 1	Post-Treatment	ND	0.1
	0180	SD 2	Pre-Treatment	ND	0.1
	0173	SD 2	Treatment	ND	0.6
	0172	SD 2	Post-Treatment	ND	0.1

<sup>†</sup> Not detected; concentration below the reporting limit

**Table 4.** Results of leaf samples (total residue and dislodgeable or surface residues) collected in San Diego County for imidacloprid and cyfluthrin treatments on March 26, 2009. Reporting limits are presented in micrograms per square centimeter ( $\mu\text{g}/\text{cm}^2$ ) and parts per million wet weight (ppm).

DPR Sample Number	Treatment Site	Sample Type	Type of Analysis	Imidacloprid		Cyfluthrin	
				Amount Detected (ppm)	Reporting Limit	Amount Detected (ppm)	Reporting Limit
0163	SD 1	Pre-Treatment	Total Residue	<sup>†</sup> ND	0.1 ppm	ND	0.05 ppm
0165	SD 1	Pre-Treatment	Dislodgeable	ND	0.25 $\mu\text{g}/\text{cm}^2$	ND	0.25 $\mu\text{g}/\text{cm}^2$
0168	SD 1	Post-Treatment	Dislodgeable	ND	0.25 $\mu\text{g}/\text{cm}^2$	ND	0.25 $\mu\text{g}/\text{cm}^2$
0191	SD 1	Post-Treatment	Total Residue	ND	0.1 ppm	ND	0.05 ppm
0161	SD 2	Pre-Treatment	Total Residue	ND	0.1 ppm	ND	0.05 ppm
0167	SD 2	Pre-Treatment	Dislodgeable	ND	0.25 $\mu\text{g}/\text{cm}^2$	ND	0.25 $\mu\text{g}/\text{cm}^2$
0169	SD 2	Post-Treatment	Dislodgeable	ND	0.25 $\mu\text{g}/\text{cm}^2$	ND	0.25 $\mu\text{g}/\text{cm}^2$
0189	SD 2	Post-Treatment	Total Residue	ND	0.1 ppm	0.06	0.05 ppm

<sup>†</sup> Not detected; concentration below the reporting limit



**Table 5.** Results of whole fruit (orange rind and pulp) samples collected in San Diego County for imidacloprid and cyfluthrin treatments on March 16, 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	03/26/2009	SD 1	03/25/2009	Background	†ND	0.05	0.70
	03/26/2009	SD 1	03/26/2009	0	ND		
	03/26/2009	SD 1	12/03/2009	35	0.02	0.01	
	03/26/2009	SD 1	12/14/2010	88	ND		
	03/26/2009	SD 2	03/25/2009	Background	ND	0.05	
	03/26/2009	SD 2	03/26/2009	0	ND		
Cyfluthrin	03/26/2009	SD 1	03/25/2009	Background	ND	0.05	0.20
	03/26/2009	SD 1	03/26/2009	0	**ND		
	03/26/2009	SD 1	03/26/2009	0	0.11		
	03/26/2009	SD 1	12/03/2009	35	ND		
	03/26/2009	SD 2	03/25/2009	Background	ND		
	03/26/2009	SD 2	03/26/2009	0	**ND		
	03/26/2009	SD 2	03/26/2009	0	0.11		

† Not detected; concentration below the reporting limit

\*\* Analysis of fruit pulp only; fruit peeled before analysis

**Table 6.** Results of soil sampling in San Diego County for imidacloprid treatments on March 26, 2009. Results are presented in parts per million (ppm).

DPR Sample Number	Treatment Site	Sample Type	Amount Detected (ppm)	Reporting Limit (ppm)
0162	SD 1	Pre-Treatment	†ND	0.01
0188	SD 1	Post-Treatment	22.4	
0160	SD 2	Pre-Treatment	ND	
0190	SD 2	Post-Treatment	12.2	

† Not detected; concentration below the reporting limit

**Table 7.** Results of tank sampling in San Diego County for imidacloprid and cyfluthrin treatments on March 26, 2009.

DPR Sample Number	Treatment Site	Tank Serial Number	Tank Sample Date	Imidacloprid Detected (Percent)	Cyfluthrin Detected (Percent)
0170	SD 1	1201743	03/26/2009	0.028	0.0026
0171	SD 2				

**Figure 1.** Monitoring sites SD 1 and SD 2: Jamul.

