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MEMORANDUM

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SUBJECT: SUMMARY OF JAPANESE BEETLE ERADICATION PROGRAM
MONITORING FOR CARBARYL, IMIDACLOPRID AND CYFLUTHRIN IN
SACRAMENTO COUNTY, 2015

INTRODUCTION

In 2014, California Department of Food and Agriculture's (CDFA) Pest Detection/Emergency Projects Branch found Japanese beetles in traps in the Carmichael area of Sacramento County and initiated an eradication program. Adult Japanese beetles feed on the foliage, fruit and flowers of more than 300 plants; grubs feed mostly on grass roots, causing significant damage to lawns and pastures.

The Japanese beetle was first found in the United States in 1916 near Riverton, New Jersey. It has spread throughout most states east of the Mississippi River. Only partial infestations have been discovered west of the Mississippi River, most of which have been eradicated.

CDFA's eradication program relies on the application of the pesticides carbaryl, imidacloprid and cyfluthrin to control the Japanese beetle. At the request of CDFA, the Environmental Monitoring Branch of the Department of Pesticide Regulation (DPR) developed a protocol¹ for monitoring pesticide treatments, and DPR staff is overseeing the monitoring.

This document summarizes monitoring results for Japanese beetle eradication program treatments in Sacramento County from May through June 2015. Air, foliage turf/soil and treatment tank mixture were monitored for pesticide residue.

Description of Application

In 2015, treatment for Japanese beetle in Sacramento County consisted of foliar applications of cyfluthrin to citrus and other host plants and trees, carbaryl to other fruit trees, plus an

¹ Protocol available at:
http://www.cdpr.ca.gov/docs/emon/epests/japanesebeetle/japanese_beetle_protocol_final.pdf.



application of a liquid imidacloprid to turf/soil. Up to 6 cyfluthrin, 3 carbaryl, and 1 imidacloprid application occurred over the 3-month period, starting on May 18, 2015.

Specific pesticide products used for Japanese beetle eradication treatments are listed below.²

- Sevin[®] SL Carbaryl Insecticide (EPA Reg.# 432-1227)³ (carbaryl, applied at a concentration of 0.32 percent) for foliar treatment. Three applications per season began on the weeks of May 18th, July 6th, & August 17th.
- Merit[®] 2F Insecticide (EPA Reg.# 432-1312) (imidacloprid applied at 0.027 percent imidacloprid) for turf/soil treatment, one application began on the week of June 22nd.
- Tempo[®] SC Ultra Insecticide (EPA Reg.# 432-1363) (cyfluthrin, applied at a concentration of 0.005 percent) for foliar treatment, six applications per season began on the weeks of May 18th, June 8th, July 6th, July 20th, August 3rd, & August 17th.

Sampling Sites

Sampling sites were established in the Carmichael area of Sacramento County. The sites consisted of individual residences within the treatment area. Sites 1 and 2 received the first applications of carbaryl and cyfluthrin on May 19th, 2015, an application of imidacloprid on June 23, 2015, and the final treatment cyfluthrin on August 18, 2015. For sites 3, 4 & 5 only cyfluthrin dislodgeable residue samples were collected. Site 3 received the sixth cyfluthrin treatment on August 22, 2015 and sites 4 & 5 on August 28, 2015.

MATERIALS AND METHODS

The materials and methods used for monitoring carbaryl, imidacloprid and cyfluthrin treatments in Sacramento County during the Japanese beetle eradication program of 2015 are described in detail below. Air, foliage, fruit and turf/soil were sampled at various pesticide application intervals: pre-treatment (background), treatment, and post-treatment. The pesticide application tank was sampled to establish treatment concentrations of carbaryl, cyfluthrin and imidacloprid.

The number of samples collected and analyzed for carbaryl, imidacloprid and cyfluthrin for each sampling medium at each treatment site are identified in Table 1. Table 2 lists the analytical methods and reporting limits used for each sampling medium. All samples were analyzed by CDFA's Center for Analytical Chemistry.

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 United States Environmental Protection Agency pesticide product registration number.

Air Sampling

A personal air sample pump (SKC# 224-PCXR) calibrated to a flow rate from 2.5 to 3 liters per minute was mounted with XAD-2 resin tubes at each site. (These monitoring specifications were chosen based upon previous monitoring experience (Segawa, 2004; Kim, 2007) Air samples were collected at the following treatment intervals (sample intervals were run consecutively and did not overlap).

- **Pre-Treatment (Background):** These samples were collected just prior to the pesticide application; the air sampler was run for the duration of 18-22 hours.
- **Treatment:** These samples were collected during application, for a duration of about 7 hours, coinciding with the time of all treatments of residences in the treatment area for the day.
- **Post-treatment:** These samples were collected, for a duration of 20-24 hours, immediately after treatment samples were collected

All air samples were frozen (on dry ice or in a freezer) until delivered to the laboratory for analysis.

Foliage Sampling for Total Residue

Foliage samples were collected from two to three plant species per site and analyzed for carbaryl and cyfluthrin residues. Samples were collected where human contact would likely occur ~2-4 feet from the ground. Background samples were collected prior to pesticide application; post-application samples were collected after application residue had dried, 2-3 hours, from the same plants as the background samples. Total residue samples consisted of 30-60 grams of whole leaves placed in wide mouth Mason jars. Samples were frozen (on dry ice or in a freezer) until delivered to the laboratory. Leaves were ground up and analyzed for total residues and reported in ppm (parts per million).

Foliage Sampling for Dislodgeable Residue

Foliage samples were collected from three plant species per site and analyzed for dislodgeable cyfluthrin residues. Samples were collected before and after the sixth (final) application. Samples were collected where human contact would likely occur ~2-4 feet from the ground. Samples that were collected on multiple events were from the same plants as the previous event, eg. Site 5.1 was the same host plant for all 3 sampling events. Dislodgeable residue samples (>150 square centimeter [cm^2]) consisted of whole leaves placed in wide mouth Mason jars. Samples were refrigerated (on wet or blue ice) until delivered to the laboratory. Leaves were rinsed (shaken) in a water-based detergent solution within 24 hours of sample collection; the solution was then analyzed for dislodgeable residue and reported in micrograms per square centimeter ($\mu\text{g}/\text{cm}^2$). The leaf surface area was measured with a Li-Cor 3100 area meter.

Turf/Soil Sampling

Each turf/soil sample consisted of two or three randomly selected cores taken to a depth of 1 inch. Cores were collected using a 2-1/2 inch (28.56 cm²) diameter stainless steel tube and composited into one wide mouth Mason jar with an aluminum foil lined lid. Background samples were collected before treatment; post-treatment samples were collected 2-3 hours after the pesticide application when the turf was dry. Turf/soil core samples were refrigerated or frozen (on wet, blue or dry ice) until delivered to the laboratory.

Tank Mixture Sampling/Product Concentration

Tank mixture samples of carbaryl, cyfluthrin and imidacloprid were collected from treatment spray guns at the time of treatment to establish pesticide concentrations in the spray material. Samples consisted of half-filled 500 milliliter Nalgene[®] wide mouth bottles. The exterior of each bottle was rinsed to remove spilled product; bottles were then triple bagged and refrigerated (on wet or blue ice) until delivered to the laboratory. Tank sample results were compared to the amount/application rate specified on the product label to ensure the pesticide was mixed properly.

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.

RESULTS AND DISCUSSION

Air

No imidacloprid residues were detected in six samples (one sample at each of the two sites prior to, during, and after treatment) for the June 23rd treatments (Table 3). Similarly, no cyfluthrin or carbaryl residues were detected in 12 samples from the August 18th treatments (one sample at each of the two sites prior to, during, and after treatment for each pesticide).

Foliage Samples

Foliage samples were analyzed for total (ppm) residues of carbaryl and cyfluthrin from Sites 1 and 2 (Table 4) and dislodgeable foliar residues for cyfluthrin from sites 3, 4 and 5 (Table 5). Samples were collected before and after the final treatment for each insecticide, three and six total treatments for carbaryl and cyfluthrin respectively. Background samples (before any 2015 treatments) were collected from sites 1 and 2.

Sites 1 & 2 August 18, 2015 Treatments – Total (ppm) residues

Twelve samples were collected for the final (August 18, 2015) treatments and analyzed for cyfluthrin or carbaryl: four background samples were collected before the first treatment (May 19, 2015), four samples were collected the morning before the final treatment, four post-application samples were collected the afternoon after the final treatment. The background sample from site 1 exhibited 0.2 ppm of cyfluthrin, all other background samples were below the detection limit of 0.1 ppm. The pre-final-treatment samples showed cyfluthrin concentrations of 4.6 ppm and 18.6 ppm (11.6 ppm mean) for the two sites, showing residues remaining on the plant two weeks after the previous treatment and potential accumulation. However, there was no increase in the individual or mean cyfluthrin concentration of 2.2 and 19.4 ppm (10.8 ppm mean), after final treatment. The reason for this finding may be due to the small sample size, different plants sampled at each time, and the tank mix samples indicating concentrations of only 21% of the target concentration.

The carbaryl treated samples exhibited detections of 3.94 ppm and 9.11 ppm (6.5 ppm mean), before treatment and 65.7 ppm and 66.2 ppm (66.9 ppm mean), after the final treatment

Site 3 August 22, 2015 Treatment – Dislodgable Foliar Residues ($\mu\text{g}/\text{cm}^2$)

Three samples were collected and analyzed for cyfluthrin foliar dislodgable residues three days after the 6th and final treatment; the mean concentration of the “3 Days” post-treatment samples was 0.44 $\mu\text{g}/\text{cm}^2$ (Table 5).

Site 4 & 5 August 28, 2015 Treatment – Dislodgable Foliar Residue ($\mu\text{g}/\text{cm}^2$)

Sixteen samples were collected and analyzed for cyfluthrin foliar dislodgable residues: six samples were collected the morning before the final treatment (“2 Weeks”, Table 5), six samples were collected the afternoon after the final treatment (“0 Days”, Table 5), and four samples were collected one week after the final treatment (“1 Week”, Table 5). The mean concentrations for the three periods were 0.40 $\mu\text{g}/\text{cm}^2$ (before), 0.47 $\mu\text{g}/\text{cm}^2$ (after), and 0.42 $\mu\text{g}/\text{cm}^2$ (1 week after). Only 4 of the 6 samples were collected for the last period due to lack of access to host plants.

Fruit Samples

The stone and pome fruit trees at our sample sites did not produce sufficient fruit for sampling due to drought conditions, and therefore were not collected.

Turf/Soil Samples

Four turf/soil plugs were analyzed for imidacloprid (Table 6). Pre and post treatment samples were collected at Sites 1 and 2 for the June 23, 2015 treatment. No residues were detected in the pre-treatment sample; the two post-treatment samples had concentrations of 3.88 $\mu\text{g}/\text{cm}^2$ and 8.67 $\mu\text{g}/\text{cm}^2$ for sites 1 and 2 respectively (Table 6), the target application rate for this product is 4.6 $\mu\text{g}/\text{cm}^2$. For an average concentration of 6.27 $\mu\text{g}/\text{cm}^2$, or 137% of the target rate.

Tank Mix

Sites 1 and 2 were treated with the same application truck/crew, therefore only one tank mix sample was collected for each insecticide, after treatment. The tank mix resulted in a mixture of 0.0234% imidacloprid, (target application rate is 0.02675%), 0.306% carbaryl (target application rate is 0.323%), and 0.00104% cyfluthrin (target application rate is 0.0049%). Or, 87.48%, 94.74% and 21.22% of the target concentrations for imidacloprid, carbaryl and cyfluthrin respectively

CONCLUSION

Monitoring of the Japanese beetle eradication program pesticide treatments yielded the following results.

- No carbaryl, imidacloprid or cyfluthrin residues were detected in air at either site for various sample dates during application.
- Foliage samples analyzed for total residues of carbaryl contained a post-treatment detection mean of 6.5 ppm the morning before final (third) treatment, and 66.0 ppm after the final treatment.
- Foliage samples analyzed for total residues of cyfluthrin contained a post-treatment detection mean of 11.62 ppm the morning before final (sixth) treatment, and 10.79 ppm after the final treatment.
- Foliage samples analyzed for total dislodgeable residues of cyfluthrin contained post-treatment detection means of 42 $\mu\text{g}/\text{cm}^2$, 40 $\mu\text{g}/\text{cm}^2$, 47 $\mu\text{g}/\text{cm}^2$, and 44 $\mu\text{g}/\text{cm}^2$ for samples taken pre-final treatment (2 weeks post-5th treatment), and 2 hours, 3 days and 1 week post final treatment, respectively.
- The turf/soil plug samples analyzed for imidacloprid yielded a post-treatment mean detection of 6.27 $\mu\text{g}/\text{cm}^2$; no residues were detected in the pre-treatment samples.
- The tank concentrations were 94.74%, 87.48% and 21.22% of the target concentrations for carbaryl, imidacloprid, and cyfluthrin respectively. The low % of target concentration for cyfluthrin could have an impact on cyfluthrin residue results.

Table 1. Number of samples collected at two sites in Sacramento County for carbaryl, imidacloprid and cyfluthrin in air, foliage, fruit, turf/soil, and the application tank.

Sampling Medium	Treatment Site	Number of Samples Taken for Each Pesticide		
		Carbaryl	Imidacloprid	Cyfluthrin
Air	1	3	3	3
	2	3	3	3
Foliage	1	3	0	3
	2	3	0	3
	3	0	0	3
	4	0	0	7
	5	0	0	9
Fruit	1	0	0	0
	2	0	0	0
Turf/Soil	1	0	2	0
	2	0	2	0
Tank Mixture/ Product Concentration	1	0	0	0
	2	1	1	1

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

Sampling Medium	Carbaryl		Imidacloprid		Cyfluthrin	
	† Analytical Method	Reporting Limit	† Analytical Method	Reporting Limit	† Analytical Method	Reporting Limit
Air Background	EM 11.3	0.02 $\mu\text{g}/\text{m}^3$	EM 12.3 (Modified)	0.15 $\mu\text{g}/\text{m}^3$	EM 16.0 (Modified)	0.16 $\mu\text{g}/\text{m}^3$
Air Application	EM 11.3	0.04 $\mu\text{g}/\text{m}^3$	EM 12.3 (Modified)	0.43 $\mu\text{g}/\text{m}^3$	EM 16.0 (Modified)	0.43 $\mu\text{g}/\text{m}^3$
Air Post Application	EM 11.3	0.01 $\mu\text{g}/\text{m}^3$	EM 12.3 (Modified)	0.15 $\mu\text{g}/\text{m}^3$	EM 16.0 (Modified)	0.13 $\mu\text{g}/\text{m}^3$
Foliage Total	EM 12.3	0.01 ppm	No Samples	No Samples	EM 12.5 (Modified)	0.1 ppm
Foliage Dislodgeable	No Samples	No Samples	No Samples	No Samples	WHS-SM-1	‡ <0.005 $\mu\text{g}/\text{cm}^2$
Turf/Soil	No Samples	No Samples	EM 12.6 (Modified)	0.003 $\mu\text{g}/\text{cm}^2$	No Samples	No Samples
Tank Mixture	HPLC		HPLC		HPLC	

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

‡ The reporting limit for foliage samples varies due to the difference in leaf surface area for any given sample.

Table 3. Results of air sampling for carbaryl, imidacloprid and cyfluthrin. Results are presented in micrograms per cubic meter ($\mu\text{g}/\text{m}^3$).

A.I.	Treatment Site	Sample Date	Sample Type	Amount Detected ($\mu\text{g}/\text{m}^3$)	Reporting Limit ($\mu\text{g}/\text{m}^3$)
Carbaryl	1	8/17-8/18/2015	Background	†ND	0.02
	1	8/18/2015	Treatment	ND	0.04
	1	8/18-8/19/2015	Post-Treatment	ND	0.01
	2	8/17-8/18/2015	Background	ND	0.02
	2	8/18/2015	Treatment	ND	0.04
	2	8/18-8/19/2015	Post-Treatment	ND	0.01
Imidacloprid	1	6/22-6/23/2015	Background	ND	0.14
	1	6/23/2015	Treatment	ND	0.39
	1	6/23-6/24/2015	Post-Treatment	ND	0.14
	2	6/22-6/23/2015	Background	ND	0.15
	2	6/23/2015	Treatment	ND	0.47
	2	6/23-6/24/2015	Post-Treatment	ND	0.16
Cyfluthrin	1	8/17-8/18/2015	Background	ND	0.15
	1	8/18/2015	Treatment	ND	0.41
	1	8/18-8/19/2015	Post-Treatment	ND	0.13
	2	8/17-8/18/2015	Background	ND	0.17
	2	8/18/2015	Treatment	ND	0.45
	2	8/18-8/19/2015	Post-Treatment	ND	0.13

† Not detected; concentration below the reporting limit

Table 4. Results of foliage samples analyzed for total residues of cyfluthrin and carbaryl from two sites in Sacramento County. Results are presented in parts per million (ppm).

A.I.	Treatment Site	Sample Date	Sample Type	Amount Detected (ppm)	Reporting Limit (ppm)
Cyfluthrin	1	05/19/2015	Background	0.2	0.1
	1	08/18/2015	Pre-Final-Treatment	4.64	0.1
	1	08/18/2015	Post-Final-Treatment	2.18	0.1
	2	05/19/2015	Background	†ND	0.1
	2	08/18/2015	Pre-Final-Treatment	18.6	0.1
	2	08/18/2015	Post-Final-Treatment	19.4	0.1
Carbaryl	1	05/19/2015	Background	ND	0.005
	1	08/18/2015	Pre-Final-Treatment	9.11	1.0
	1	08/18/2015	Post-Final-Treatment	65.7	10
	2	05/19/2015	Background	ND	0.005
	2	08/18/2015	Pre-Final-Treatment	3.94	1.0
	2	08/18/2015	Post-Final-Treatment	66.2	10

† Not detected; concentration below the reporting limit

Table 5. Results of foliage samples analyzed for dislodgeable residues of cyfluthrin from three sites in Sacramento County. Results are presented in micrograms per square centimeter ($\mu\text{g}/\text{cm}^2$).

Site / Sample	Time Since Treatment	Sample Date	Number of Treatments	Amount Detected ($\mu\text{g}/\text{cm}^2$)	Leaf Area Collected (cm^2)	Amount Collected (grams)
4.1	0 Days ~2 hours after final treatment	08/28/2015	6 treatments prior to collection	0.29	311.9	6.6
4.2				1.09	202.4	6.8
4.3				0.51	290.6	11.6
5.1				0.13	204.5	3.1
5.2				0.21	339.6	7.5
5.3				0.57	251.4	3.5
			Mean	0.47		
3.1	3 Days 3 days after final treatment	08/25/2015	6 treatments prior to collection	0.32	264.0	4.3
3.2				0.36	426.3	12.7
3.3				0.63	334.5	8.9
			Mean	0.44		
4.3	1 Week 1 week after final treatment	09/04/2015	6 treatments prior to collection	0.40	269.4	9.9
5.1				0.04	150.6	4.5
5.2				0.74	249.9	5.0
5.3				0.49	268.1	4.2
			Mean	0.42		
4.1	2 Weeks Before Final treatment	08/28/2015	5 treatments prior to collection	0.17	419.9	9.2
4.2				1.29	278.0	9.1
4.3				0.14	402.8	15.5
5.1				0.02	276.8	3.9
5.2				0.39	339.8	7.0
5.3				0.39	186.8	1.7
			Mean	0.40		

Table 6. Results of turf/soil plug sampling for imidacloprid. Results are presented in micrograms per square centimeter ($\mu\text{g}/\text{cm}^2$).

Treatment Site	Sample Date	Sample Type	Amount Detected ($\mu\text{g}/\text{cm}^2$)	Reporting Limit ($\mu\text{g}/\text{cm}^2$)
1	6/23/2015	Background	[†] ND	< 0.05
2	6/23/2015	Background	ND	
1	6/23/2015	Post-Treatment	3.88	
2	6/23/2015	Post-Treatment	8.67	

[†] Not detected; concentration below the reporting limit