**California Environmental Protection Agency**
**Department of Pesticide Regulation**
**Environmental Monitoring Branch**
**P.O. Box 4015**
**Sacramento, California 95812-4015**

# PROTOCOL FOR MONITORING IMIDACLOPRID AND CYFLUTHRIN IN THE ASIAN CITRUS PSYLLID ERADICATION PROGRAM

May 15, 2009

## I. INTRODUCTION

The Asian citrus psyllid (ACP) is an invasive pest that can vector "Huanglongbing" (HLB), a disease of citrus trees. The California Department of Food and Agriculture (CDFA) Pest Detection/Emergency Projects Branch (PDEP) has detected the ACP in San Diego and Imperial counties (initial find in August 2008), and started an eradication program in September 2008. Extensive ACP detections in Mexico, along the California border, have prompted a similar eradication program in Mexico.

Current treatment includes soil applied systemic, and foliar applied contact insecticide treatments. Imidacloprid is applied as a soil drench around each host plant followed by a foliar application of cyfluthrin.

At the request of CDFA, the Environmental Monitoring Branch (EM) of the Department of Pesticide Regulation (DPR) has developed this protocol to monitor the imidacloprid and cyfluthrin pesticide treatments in San Diego and Imperial counties. Monitoring may be expanded to additional counties if requested by CDFA. Monitoring will provide information about the concentrations of imidacloprid and cyfluthrin in air, fruit and/or foliage, soil, and if obtainable, surface water runoff.

## II. PERSONNEL

This study will be conducted by the Environmental Monitoring Branch, under the general direction of Lisa Ross (Environmental Program Manager I). Key personnel are listed below.

Project Leader: David Kim
Field Coordinator: Laura Petro (CDFA)
Senior Scientist: Randy Segawa
Laboratory Liaison: Sue Peoples
Analyzing Laboratory: CDFA, Center for Analytical Chemistry

All questions from the media should be directed to Lea Brooks, (916) 445-3974, e-mail lbrooks@cdpr.ca.gov.

## III. OBJECTIVES

The objectives of this monitoring are to: 1) Measure the amount of imidacloprid and cyfluthrin in outdoor ambient air; 2) Characterize the concentrations of imidacloprid and cyfluthrin residue in ripe fruit and/or foliage before and after application; 3) Measure the concentrations of imidacloprid and cyfluthrin in soil; 4) Measure the concentrations of imidacloprid and cyfluthrin in surface runoff water following application and storm events; 5) Measure the amount of imidacloprid and cyfluthrin in the spray material.

## IV. MONITORING PLAN

Sampling sites will be located within the ACP treatment area of San Diego and Imperial counties, additional counties may be included if the treatment area expands to additional counties. Air sampling site selection is based on the following criteria: sites must be (1) located in the treatment area and contain ACP host plants; (2) accessible the day before, during, and after the application; and (3) located in a secure area where any disturbance of the air sampling equipment would be unlikely. Soil and foliage sampling sites require access to property only on the day of application. Fruit sampling sites require ripe fruit and access during sampling. Permission from owner or tenant to access private property must be granted before any samples are collected. Soil, foliage and fruit (if ripe) will be collected at all air sampling sites.

OBJECTIVE 1: To measure the amount of imidacloprid and cyfluthrin in outdoor ambient air. DPR uses screening levels to evaluate the possible health effects of exposure to a chemical, based on a chemical's toxicity. A concentration that is below the screening level is not considered to represent a significant health concern and would not generally undergo further evaluation, but also should not automatically be considered "safe."

Air Samples - Imidacloprid and cyfluthrin are relatively non-volatile pesticides, so little or no material is anticipated in air once the spray settles. Four to six sites located in San Diego and Imperial counties will be sampled to measure outdoor air concentrations of imidacloprid and cyfluthrin. Sites must be accessible at all hours, protected from any direct spray. Based on previous monitoring (Kim 2007, Segawa 2004) a personal air sample pump (SKC#224-PCXR), calibrated to 3 liters/min, mounted with a XAD-2 resin tube as the trapping medium, will be used at each site. The samples will be collected for a period prior to application (background, 12-24 hr), during application (1-4 hr), and for 1 day after application (post application, about 24 hr).

All air samples are stored and transported frozen (dry ice or freezer) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR and/or CDFA staff will collect the following number of samples.

4 sites x 3 sample periods x 2 chemicals/site = 24 samples

OBJECTIVE 2: To characterize the concentrations of imidacloprid and cyfluthrin residue in ripe fruit and/or foliage before and after application. These results will be used to determine if legal and effective concentrations are achieved. The maximum allowable concentration (tolerance) for imidacloprid in mature citrus fruit is 0.7 ppm and 0.2 ppm for cyfluthrin. This tolerance is based on analysis of the entire fruit, rind included.

Fruit– Fruit samples will be collected from one or two species (e.g., lemon and orange) at multiple sites within the two-county treatment area to confirm that tolerances are not exceeded. Background samples will be collected prior to application, and application samples will be collected after application residue has dried. Post application (interval) samples will be collected from selected sites treated at various intervals prior to citrus fruit harvest. The air sampling sites may be used at a later time as interval sampling if immature fruit is present at time of application. Suggested intervals between treatment and harvest range from 2 to 40 weeks, other intervals may be added depending on the pattern of concentrations observed.

Each sample is a composite of several fruit collected in a paper bag from a single property or tree. Samples are stored and transported refrigerated (wet or blue ice) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR and/or CDFA staff will collect the following number of samples.

Application Site: 4 sites x 2 periods/site x 2 samples/period = 16 samples
Interval sampling sites: 6 to 24 sites x 1 sample/period = 6 to 24 samples

Foliage – Foliage samples will be collected from one or two species (e.g., lemon and orange) at multiple sites within the two county treatment area to determine efficacy of the spray program. Background samples will be collected prior to application, and post-application samples will be collected after application residue has dried. Leaf Punches will be collected and analyzed for dislodgeable cyfluthryn residues. Whole leaf samples will be collected and analyzed for imidacloprid total residues. CDFA-PDEP is also collecting whole leaf samples to monitor imidacloprid residues over time.

Dislodgeable residue samples consist of 40 one-inch-diameter leaf punches collected into a 4-ounce glass jar and sealed with a Teflon®-lined lid. Samples are stored refrigerated (wet or blue ice) and delivered within 24 hours to CDFA Center for Analytical Chemistry staff. DPR and/or CDFA staff will collect the following number of samples.

4 sites x 2 periods/site = 8 samples

Whole leaf samples consist of a minimum of 25 grams of whole leaves collected into a quart mason jar with a foil lined lid. Samples are stored and transported refrigerated or frozen (wet, blue or dry ice) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR and/or CDFA staff will collect the following number of samples.

4 sites x 2 periods/site = 8 samples

OBJECTIVE 3: To measure the concentrations of imidacloprid and cyfluthrin in soil before and after treatment. These results will be used to determine if effective concentrations (imidacloprid) are achieved, and measure drift to the ground (cyfluthrin) after treatment.

Soil Samples - Samples will be collected using a 2-1/2 inch stainless steel tube, 28.56$cm^2$. Each sample will consist of three randomly selected soil cores, 1 inch deep. The background sample will be collected within 24-hours before treatment. The post treatment sample will be collected 1 to 5 hours after treatment.

Soil cores are composited into wide mouth Mason jars with aluminum foil lined lids. Samples are stored and transported refrigerated or frozen (wet, blue or dryice) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR and/or CDFA staff will collect the following number of samples.

4 sites x 2 sample periods/site = 8 samples

OBJECTIVE 4: To measure the concentrations of imidacloprid and cyfluthrin in surface water and or runoff water following application or storm events. These results will be used to determine if imidacloprid and cyfluthrin may adversely affect sensitive habitat or aquatic organisms.

Surface Water Samples – Surface water may be monitored during storm or irrigation runoff events to determine imidacloprid and cyfluthrin concentrations due to wash off from exposed surfaces. Surface water samples will be collected at sensitive sites and runoff water samples at a common discharge point from treated properties. If rain or irrigation runoff is not present when sampling personnel are available, runoff samples will not be collected.

Water samples are collected and stored in one liter amber glass bottles with Teflon® lined lids. Samples are stored and transported refrigerated (wet or blue ice) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR and/or CDFA staff will collect the following number of samples.

If runoff present: 4 sites x 2 collections/site = 8 samples

OBJECTIVE 5: To measure the amount of imidacloprid and cyfluthrin in the spray material. The results will be compared to the amount and/or rate specified on the pesticide product label to ensure that the pesticide is mixed properly.

Tank Mixture samples - DPR or CDFA staff will collect tank mixture samples. Samples will be collected, from the treatment spray guns, in plastic bottles.

Samples are stored and transported refrigerated (wet or blue ice) until received by CDFA Center for Analytical Chemistry laboratory staff for analysis. DPR or CDFA staff will collect the following number of samples.

4 sites x 2 chemicals/site = 8 samples

All movement of plant material outside the quarantine area will be transported in accordance with the CDFA issued permit #2618 (see attachment).

This monitoring plan includes current treatment areas of Imperial and San Diego counties. The number of samples and sites may increase if the treatment area expands into other counties or additional pesticides are used to retreat existing areas.

## V. CHEMICAL ANALYSIS/QUALITY CONTROL

CDFA's Center for Analytical Chemistry will perform the laboratory analysis for imidacloprid and cyfluthrin in all media. Quality control measures will include analysis of spikes, (samples with known amounts of imidacloprid and cyfluthrin), to verify the accuracy and precision of the methods, and sample blanks, (samples with no imidacloprid or cyfluthrin), to check for contamination, as described in Segawa et al. (1995). The quality control samples will comprise approximately 10% of the field samples.

All plant material will be frozen for at least 24 hours before disposal.

## VI. DATA ANALYSIS

Concentrations of imidacloprid and cyfluthrin in air will be reported in micrograms per cubic meter (µg/m3) and parts per trillion (ppt), water concentrations will be reported as both micrograms per liter (µg/L) and parts per billion (ppb), fruit samples will be reported as parts per million (ppm), foliage samples will be reported as micrograms per gram (ug/g) or micrograms per square meter (ug/$m^2$). When sample size permits, means, percentiles and frequency histograms will be presented. Tank sample results will be reported in percent active ingredient and compared to the target application rate. Air concentrations will be compared to the screening level. Water concentrations will be compared to aquatic toxicity levels. Foliage samples will be compared to effective levels. Fruit concentrations will be compared to tolerances. Samples used for tolerance purposes must be at the harvest stage, and in its unpeeled, natural form.

## VII. REFERENCES

Kim, D. 2007. Monitoring Results of Imidacloprid Application for Glassy-Winged Sharpshooter Control in a Residential Area of Santa Clara County. http://www.cdpr.ca.gov/docs/emon/epests/gwss/imid_gwss_07.pdf

Segawa, R, J. Walters, S. Fan. 2004. Preliminary Monitoring Results of Imidacloprid and Cyfluthrin Applications for Glassy-Winged Sharpshooter Control in a Residential Area of Solano County
http://www.cdpr.ca.gov/docs/emon/epests/gwss/gwss091704.pdf

Segawa, R. 1995. Chemistry and Laboratory Quality Control, Standard Operating Procedure QAQC001.00. California Department of Pesticide Regulation. Environmental Monitoring Branch. http://www.cdpr.ca.gov/docs/empm/pubs/sops/qaqc001.pdf

**Attachment – Permit 2618**

NO PERMIT WILL BE ISSUED TO MOVE AND USE LIVE INSECTS OR PLANT PESTS OR NOXIOUS WEEDS UNTIL A COMPLETED APPLICATION IS RECEIVED.

DEPARTMENT OF FOOD AND AGRICULTURE
PLANT HEALTH AND PEST PREVENTION SERVICES
1220 N STREET, ROOM A-316
SACRAMENTO, CALIFORNIA 95814

APPLICATION AND PERMIT TO MOVE AND USE LIVE PLANT PESTS OR INSECTS OR NOXIOUS WEEDS

**SECTION A TO BE COMPLETED BY APPLICANT**

1. NAME AND ADDRESS (Include Zip Code)
John Hooper
1220 N Street Room A-330
Sacramento CA 95814

2. TELEPHONE NUMBER/FAX NUMBER/EMAIL
916-654-1211

3. TYPE OF ORGANISM
☒ Arthropod ☐ Pathogen ☐ Noxious Weed
☐ Biocontrol Agent ☐ Other

| 4. SCIENTIFIC AND COMMON NAMES OF ORGANISMS | CLASSIFICATION (Order, Family, etc.) | LIFE STAGES | NUMBER OF SPECIMENS | MOVED OR SHIPPED FROM | WHAT HOST MATERIAL WILL ACCOMPANY PEST? |
|---|---|---|---|---|---|
| Asian Citrus Psyllid Host Plants | Rutaceae | ALL | Various | Balance of ACP Quarantine area | Fruit, stems, leaves, buds, blossoms |

| 5. ADDRESS OF USE LOCATION IF DIFFERENT THAN ITEM 1. | 6. NAME AND ADDRESS OF SUPPLIER | 7. DESTINATION COUNTY |
|---|---|---|
| 3292 Meadowview Rd Sacramento, CA 95830 Center for Analytical Chemistry | Various field staff CDFA and/or CDR contract staff | Sacramento |

| 8. APPROXIMATE DATE OF MOVEMENT | 9. NUMBER OF SHIPMENTS | 10. METHOD OF SHIPMENT |
|---|---|---|
| March 9-10, 2009 | | ☐ Mail ☒ Freight ☐ Baggage ☒ Auto |

11. INTENDED USE (Be specific; state whether use will be in a laboratory and/or greenhouse and/or in the field, and, in the case of pathogens, state whether use will include plant inoculation.)

Chem Lab will grind up and test for pesticide residues

| 12. METHODS TO BE USED TO PREVENT ESCAPE OF THE ORGANISMS | 13. METHOD OF FINAL DISPOSITION |
|---|---|
| See attached sheet | Freezing followed by trash |

14. I/We agree to comply with the conditions attached to this form, and understand that the permit is subject to other conditions which may be prescribed.

SIGNATURE OF APPLICANT John Hooper DATE 3/20/09

**SECTION B – TO BE COMPLETED BY STATE OFFICIAL**

PERMIT

(Permit not valid unless signed by an authorized official of Plant Health and Pest Prevention Services Division)

PERMIT NUMBER 2618

Under authority of Section 6305 of the Food and Agricultural Code, permission is hereby granted to the applicant named above to move and use the organisms described, except as deleted, subject to the conditions stated on, or attached to, this application. (See attached standard conditions.)

VIOLATION OF ANY OF THE CONDITONS OF THIS PERMIT SHALL BE SUFFICIENT CAUSE FOR ITS IMMEDIATE REVOCATION.

| 15. SIGNATURE OF STATE OFFICIAL | 16. DATE ISSUED | 17. EXPIRATION DATE |
|---|---|---|
| | March 27, 2009 | March 31, 2011 |

Attachments may accompany application if space on application is insufficient.

FORM 66-026 (6/05)
Plant Health and Pest Prevention Services

Copy to: County Agricultural Commissioner
Pest Exclusion Area Biologist
Plant Pest Diagnostics Branch
Supplier
File

FORM 66-026

Answer to question Number 12.

All plant samples will be visually inspected for the presence of Asian Citrus Psyllid (ACP) before shipment. Samples containing any suspect ACP life stages (egg, nymph, or adult) will be destroyed prior to shipment. Samples will be double contained inside sealed plastic bags, which are then placed inside sealed containers with ice. Samples will remain double contained until they are tested. Samples will be opened in a manner so as to prevent the escape of any ACP adults.

STANDARD CONDITIONS OF PERMIT

1. All organisms shall be shipped in sturdy, escape-proof containers and a copy of this permit shall accompany each shipment.

2. Arrival of each shipment shall be immediately reported to the office of the County Agricultural Commissioner and held for inspection prior to use (telephone: N/A ).

3. All packing material and shipping containers shall be sterilized or destroyed immediately after removing the organisms.

4. Organisms, and inoculated plants if any, shall be kept and used only within the laboratory or designated area at the permittee's address and/or the address specified in Item 5, Section A.

5. No living organisms kept under this permit, and inoculated plants if any, shall be removed from the confined, designated area except by prior approval from State and, if applicable, federal agricultural regulatory officials.

6. Without prior notice and during regular business hours, State and county agricultural regulatory officials shall be allowed to inspect the conditions under which the organisms are kept and used.

7. All necessary precautions must be taken to prevent escape of pests. In the event of pest escape, this office shall be immediately notified (916) 654-1017.

8. All organisms kept under this permit, and inoculated plants if any, shall be destroyed at the completion of the intended use, and not later than the expiration date, unless an extension is granted by this issuing office. Written request for an extension of the expiration date should be submitted at least 30 days in advance of the expiration date.