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CHANGE# 1

# IR-4 PROTOCOL AMENDMENT FORM*

**Project Title:** Propiconazole / Stone Fruits (post-harvest) PR No.: 09787

**Field I.D. No.:** All

**Laboratory I.D. No.:** 09787.07-TIR06

**Description of Change: Replace Sections 15C, 18C and 29 with the following:**

## 15C. CHERRY POST-HARVEST APPLICATION TREATMENTS AND TIMING:

| Trt# | Treatment | Target Rate of active ingredient | Application Type |
|---|---|---|---|
| 01 | Untreated | Not Applicable | Not Applicable |
| 07 | Propiconazole | 0.1125 lb ai / 100 gallons water + wax fruit, including a post-harvest fruit wax, such as Decco Lustr 251 | High Volume spray, Drench or Dip |

*The nominal formulation concentration of the test substance will be used in calculating application rates (see Protocol Section 13 for the nominal concentration). (0.1125 lb = 113.4 grams (4 oz.) Propiconazole 45WP)

Make one application to fruit as a post-harvest high volume spray, a drench or a dip application. For each post-harvest treatment, use a post-harvest fruit wax, such as Decco Lustr 251, in the fungicide solution at the labeled rate. For the High Volume spray the above fungicide solution should be applied to fruit at the rate of 100 gallons of spray solution per 200,000 lbs fruit. For the Drench application, the above fungicide solution can be slowly poured over the fruit, the fruit turned over, and then immediately slowly poured over the fruit again. Do not allow drying between drenching of each side. The dip application should apply the above propiconazole rates in 100 gallons of solution (water + wax) by dipping the fruit in the fungicide/wax solution for 30 (±3) seconds and then allowing the fruit to drain. All fruit should be allowed to surface-dry prior to sample collection.

## 18C. CHERRY POST-HARVEST RESIDUE SAMPLE INVENTORY:

| SAMPLE ID | TRT# | TREATMENT | POST-HARVEST TREATEMENT | MINIMUM WEIGHT PER SAMPLE | CROP FRACTION |
|---|---|---|---|---|---|
| A | 01 | Untreated | NA | 2 lbs. | Cherries without pits & stems |
| B | 01 | Untreated | NA | 2 lbs. | Cherries without pits & stems |
| Q | 07 | Propiconazole | HV, Drench or Dip | 2 lbs. | Cherries without pits & stems |
| R | 07 | Propiconazole | HV, Drench or Dip | 2 lbs. | Cherries without pits & stems |

## 29. ANALYTICAL METHODOLOGY:

REFERENCE METHODS:

**Parent Propiconazole Method**

Syngenta Analytical Method REM 130.11, "Residue Analytical Method for the Determination of Residues of Propiconazole (CGA64250) in Crop Samples. Final Determination by LC-LC-MS/MS.", T. Clark, 12/1/05.

**Triazole Metabolite Method**

Determination of 1,2,4-Triazole, Triazole Alanine and Triazole Acetic Acid Residues in Plant and Animal Matrices, Morse Laboratories, Inc., Analytical Method # Meth-160, dated June 27, 2003.

**Common Moiety Method**

Determination of Total Residues of Propiconazole in Crops as 2,4-Dichlorobenzoic Acid by Capillary Gas Chromatography", Ciba-Geigy Corporation, Greensboro, NC 27419, Analytical Method AG-454B, John Toth, P.J. Manuli, 12/20/89.

REFERENCE METHOD MODIFICATIONS/METHOD VALIDATION:

The above listed Reference Methods may be modified if needed for the test matrix. The Reference Methods, along with any modifications must be validated on one crop matrix (peaches, plums or cherries) prior to residue sample analysis of that crop fraction. To validate the parent propiconazole method fortify some of the control samples in triplicate with propiconazole at a minimum of 3 concentration levels between the lowest level of method validation (0.05 ppm or lower) and 5 ppm. To validate the triazole metabolite method fortify some of the control samples in triplicate with 1,2,4-Triazole (T), Triazole Alanine (TA), and Triazole Acetic Acid (TAA) at a minimum of 3 concentration levels between the lowest level of method validation (0.05 ppm or lower) and 0.5 ppm. To validate the common moiety method fortify some of the control samples in triplicate with propiconazole at a minimum of 3 concentration levels between the lowest level of method validation (0.05 ppm or lower) and 5 ppm. A minimum of 6 fortification samples (recovery spikes) at the lowest level of method validation (LLMV) is required prior to completion of the analytical phase of the study.

The acceptable recovery range is 70-120%. **Documented approval from the Study Director** is needed for recoveries outside of this range. Document the exact procedures for sample analysis. This validated step-by-step Working Method should incorporate all changes from the Reference Method. **Provide the Study Director** with a copy of this Working Method and results of method validation prior to treated sample analysis. If the Working Method has been used successfully on the test matrix or a similar matrix, the Study Director may waive the requirement for method validation. **Contact the Study Director for details**.

SAMPLE ANALYSIS:

All treated samples and at least one untreated sample per trial will be analyzed for residues of propiconazole, T, TA and TAA following the Parent Propiconazole working method and the Triazole Metabolite working method. One-third (1/3) of the treated samples should be analyzed using the Common Moiety working method. Samples for common moiety method should be selected from different trials or different treatments in the same trial.
**Contact the Study Director** if residues above the lowest level of method validation for each matrix are detected in the untreated samples. Any changes or modifications to the Working Method **require Study Director approval**. Whenever possible, **notify the Study Director** prior to occurrence. Any change or modification to the Working Method should be documented in the raw data and discussed in the final report. A typical analytical set (or run) should consist of calibration standards, untreated sample(s), concurrent recovery sample(s), and treated sample(s). Each analytical set must begin and end with a calibration standard. Additional calibration standards should be injected with sample analysis to ensure goodness of fit to the standard curve.

Over the course of residue sample analysis, adequate concurrent recovery samples that bracket the actual residues should be analyzed. At least one concurrent fortification sample should be analyzed per analytical set.

**The Study Director should be immediately notified** if concurrent recoveries deviate from the acceptable recovery range of 70% to 120%. All efforts will be made to resolve existing recovery problems before continuing forward with additional analytical sets. If residues in samples are above the highest Working Method validation concentration,

additional recovery samples at levels above actual residues must be run in triplicate as soon as practical. A minimum of 6 fortification samples (recovery spikes) at the lowest level of method validation (LLMV) is required for each analyte prior to completion of the analytical phase of the study.

STORAGE STABILITY ANALYSIS:

The registrant has adequate storage stability data. A storage stability study is not required. For sample integrity purposes, the analytical laboratory may set up the following. As soon as possible after receipt of samples, a minimum of six sub samples of peach from the untreated control shall be fortified with propiconazole at 0.5 ppm and T, TA and TAA at 0.1 ppm each. Three samples will be analyzed after the appropriate storage period (generally, greater than the longest interval that an individual sample was stored between collecting the sample in the field/processing facility and analysis, **unless otherwise specified by the Study Director**). The remaining samples will be retained for long-term storage.

If analysis of treated/control samples is completed within 30 days of harvest analysis of storage fortification samples may not be required. If appropriate, **contact Study Director**.

STATISTICAL METHOD(S):

Utilize regression analysis to determine the linearity of the standard curve ($r^2$) or the goodness of fit if the standard curve is non-linear. Criteria for acceptance of the standard curve(s) or other statistical methods shall be determined by Laboratory Research Director and documented in the raw data.

**Reason for Change:**

This change is needed due to an inadvertent error in signing an earlier revision of the protocol.

**Impact on Study:**

None

Authorization:

David Thompson 3/7/2007

Dr. David C. Thompson Date
Study Director

3/7/2007

Dr. Daniel L. Kunkel Date
Sponsor Representative

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