

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 1 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

1. Purpose:

To provide standard procedures for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS Pesticide Data Program (PDP) samples for the peach acute dietary risk survey.

2. Scope:

This standard operating procedure (SOP) shall be followed by the analytical laboratory conducting pesticide residue studies for the PDP peach acute dietary risk survey, the New York Department of Agriculture & Markets Food Laboratory (NY1). All samples shall be shipped to this location. States should coordinate sampling dates with the receiving laboratory. Monthly sampling rates are 2x January-March, 2000, with imports targeted, and 1x May-September, 2000, during which time primarily domestic samples should be available. During the double sampling period (January-March, 2000), a total of 124 samples are targeted per month, apportioned by State as follows:

| | |
|---------|---------|
| CA – 28 | NY - 18 |
| CO – 4 | OH - 12 |
| FL – 14 | TX – 16 |
| MD – 8 | WA - 8 |
| MI – 12 | WI - 4 |

During the single sampling period (May-September, 2000), a total of 62 samples are targeted per month, apportioned by State as follows:

| | |
|---------|--------|
| CA – 14 | NY - 9 |
| CO – 2 | OH - 6 |
| FL – 7 | TX – 8 |
| MD – 4 | WA - 4 |
| MI – 6 | WI - 2 |

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 2 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

3. Outline of Procedures:

- 6.1 Sample Receipt
- 6.2 Sample Preparation
- 6.3 Sample Storage
- 6.4 Sample Analysis
- 6.5 Data Reporting
- 6.6 Sample Disposal

4. References:

USDA/AMS PDP Federal/State Meeting, October 26-28, 1999
Federal/Industry Meeting, October 4, 1999
USDA/EPA Meeting, August 4, 1999
USDA, AMS SOP, PDP-LABOP-10, Acute Dietary Risk Survey, Organophosphates and Carbamates in Apples, 05/01/99

5. Summary:

For each five pound sample, an individual peach shall be selected for single serving analysis. The remaining peaches shall be homogenized and analyzed as a composite sample. Both single serving and composite samples shall be analyzed for the identified compounds (refer to Table 1). The single serving sample shall not be subject to any applicable re-extraction or presumptive tolerance violation (PTV) reporting requirements.

6. Procedure:

This standard operating procedure (SOP) represents minimum PDP requirements for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS, PDP samples for the peach acute dietary risk survey, and is presented as a general guideline. The analytical laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in that laboratory.

- 6.1 Sample Receipt
-

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 3 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

- a. Each sample shall be comprised of five pounds of peaches. The laboratory shall randomly select an individual peach of adequate size for single serving analysis. The remaining peaches shall comprise the composite sample.
- b. Those samples, or portions thereof, received in a damaged condition (refer to SOP PDP-LABOP-01, subsection 5.1) shall be discarded and not analyzed. Condition and disposal shall be documented on the Sample Information Form (SIF).
- c. The composite shall use the standard sample identification number. The single serving sample shall be identified by the use of CX as the commodity code in the sample identification number when logging in samples. An example sample identification number would be CA-00-01-0114-CX-NY1 (as *distinct from the composite, which would be coded CA-00-01-0114-PC-NY1*). The individual peach does not need to be physically designated at time of login.
- d. The laboratory shall maintain a log of samples received. Refer to SOP PDP-LABOP-01, subsection 5.1.

6.2 Sample Preparation

- a. Randomly select an individual peach which should provide at least the minimum amount of homogenate required for analysis. Wash and prepare the individual peach per PDP-LABOP-03. When pitting the sample, be sure to preserve as much flesh as possible.
- b. Combine remaining peaches for the composite sample and prepare according to PDP-LABOP-03.
- c. Homogenize individual peach in a food chopper which will ensure a finely chopped, homogeneous mixture. For the composite sample, mechanically chop peaches just until a visually homogeneous mixture is attained.

6.3 Sample Storage

An adequate portion of the homogenized composite sample, as processed in 6.2.c,

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 4 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

shall be held in reserve if re-analysis and/or confirmation is needed. This portion shall be distributed among several small containers (polypropylene or styrofoam recommended) rather than one large container and stored at 0°C or less. The laboratory internal SOP shall specify “adequate portion” and distribution. Retention of reserve single serving homogenate after weighing of analytical portion is not required.

6.4 Sample Analysis

a. Weighing of Analytical Portion

An appropriate amount of homogenized sample shall be weighed for analysis. The laboratory internal SOP shall define the sample weight and the necessary precision which, for a 50 gram sample, shall not be more than +/- 0.25 grams.

At this point, the analytical portion for the individual and/or composite sample may be stored in an appropriate, labeled small container and stored at 0°C or less, until time of analysis.

Note: If individual or composite samples are expected to be frozen prior to analysis, a single freezer spike, as detailed in subsection 6.4.b, must be completed for this study.

b. Sample Set Requirements

A sample set is the group of samples which are spiked individually with the designated process control and extracted on a single day along with the required QC samples. Each set shall consist of no more than 20 analytical samples, where an analytical sample is defined as either a composite or an individual peach. Required QC samples per set consist of a reagent blank, peach matrix blank, peach matrix spike(s), and peach freezer spike(s) where applicable (refer to note in subsection 6.4.a).

The matrix spike(s) shall be spiked at approximately 2xLOQ and shall contain at least the following marker pesticides: azinphos methyl, carbaryl, DDE p,p', diazinon, dicloran, endosulfan I, ethion, iprodione, methomyl, permethrins, propargite, terbacil, and tetrachlorvinphos.

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 5 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

The freezer spike(s) shall be spiked at approximately 2xLOQ with all required compounds. The spike shall be held for a period of time equal to or greater than the longest expected time of storage prior to analysis. This data shall be reported to Manassas via established RDE procedures.

Each analytical sample shall be spiked with the appropriate process controls at approximately 5xLOQ. All components of sample sets shall be subject to the sample analytical process as detailed in the method SOPs.

c. Analytical Requirements

Each composite plus the individual peach shall undergo analysis for the identified required compounds as detailed in Table 1.

Table 1. Required Compounds

| | | |
|---------------------------|-----------------------|----------------------|
| Azinphos methyl | Captafol | Captan |
| Carbaryl | Chlorpyrifos | DDE p,p' |
| Diazinon | Diazinon O-analog | Dichlorvos |
| Dicloran | Dicofol (p,p') | Dieldrin |
| Endosulfan I | Endosulfan II | Endosulfan sulfate |
| Esfenvalerate | Ethion | Ethion mono oxon |
| Fenamiphos | Fenamiphos sulfone | Fenamiphos sulfoxide |
| Fenbuconazole | Fenvalerate | Heptachlor epoxide |
| Iprodione | Lindane | Malathion |
| Malathion O-analog | Metalaxyl | Methidathion |
| Methiocarb | Methomyl | Methoxychlor p,p' |
| Metolachlor | Mevinphos E/Z | Myclobutanil |
| Norflurazon | Norflurazon desmethyl | o-Phenylphenol |
| Oxyfluorfen | Parathion | Parathion methyl |
| Parathion methyl O-analog | Parathion O-analog | Permethrin cis/trans |
| Phosalone | Phosmet | Piperonyl butoxide |
| Pronamide | Propargite | Propiconazole |

**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 6 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

Table 1. Required Compounds

| | | |
|-------------|-------------------|-------------|
| Simazine | Tebuconazole | TEPP |
| Terbacil | Tetrachlorvinphos | Tetradifon |
| Triadimefon | Trifluralin | Vinclozolin |

Analytical standard for ethion mono oxon is available through the US EPA Standards Repository. Other compounds are commercially available through PDP approved vendors and may be purchased under the blanket purchasing agreements by contacting the Residue Branch. These standards may also be requested from the EPA Standards Repository. The EPA Standard Repository contact is Dick Griffith, EPA, Fort Meade, MD (410-305-2905), or Chuck Stafford (410-305-2914).

All new compounds and compounds added since peaches were removed from the program shall undergo validation/evaluation requirements as specified in SOP PDP-QC-07. All compounds requiring validation shall be considered as “related to the marker compounds” for validation purposes.

6.5 Data Reporting

- a. Process control and marker matrix spikes for composites shall be subject to the criteria and reaction procedures set forth in SOP PDP-QC-04, Acceptability Criteria for Process Control and Fortification Recoveries. Individual peaches shall also be subject to this criteria, except where re-extraction is indicated.
- b. No presumptive tolerance violation (PTV) reporting requirements, as specified in SOP PDP-DATA-02 apply to individual peaches.



**United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program**

| | | |
|--|---------------|---------------------|
| SOP No.: PDP-LABOP-11 | | Page 7 of 8 |
| Title: Peach Acute Dietary Risk Survey | | |
| Revision: Original | Replaces: N/A | Effective: 01/01/00 |

c. All data shall be transmitted following established RDE procedures.

6.7 Sample Disposal

Stored homogenates of composites for each set shall be disposed of when all requirements for acceptability criteria have been met and results have been successfully transmitted via RDE to USDA, AMS, Residue Branch. Disposal shall be documented (e.g., freezer log, sample log, extraction worksheet) and shall contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.



