General Method for the Analysis of Iprodione-Related Residues: Common Moisty Method

Rhône-Poulenc Ag Company
2 T. W. Alexander Drive
Research Triangle Park, NC 27709

Prepared by:
Horizon Laboratories, Inc.
1610 Business Loop 70W
P. O. Box 30608
Columbia, MO 65202

Iprodione/Plants/General Method
July 15, 1994
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Method for the Analysis of Iprodione-Related Residues: General Common Moiety Method

I. INTRODUCTION AND SUMMARY

A. Scope

Applications of ROVRAL® fungicides to agricultural crops may result in residues in/on harvested plant commodities. Prior metabolism studies have shown that the principal residues in plants are Iprodione [RP-26019, 3-(3,5-dichlorophenyl)-N-(1-methylethyl)-2,4-dioxo-1-imadazolidinecarboxamide, the ROVRAL® active ingredient] and two related compounds termed RP-30228 [3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide] and RP-32490 [3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide].

Traditional methods for the analysis of Iprodione-related residues in crops measure these compounds as individual species. Some care must be exercised when using these methods as they are considered complex and, depending upon the matrix, difficult; the methods use benzene as a solvent, rely on packed-column gas chromatography techniques, and require multiple injections for analysis of all components.

Iprodione, RP-30228, and RP-32490 share a common 3,5-dichloroaniline core structure; Iprodione and RP-32490 are imide-like derivatives of 3,5-dichloroaniline while RP-30228 is a urea-like derivative. All three compounds may be hydrolyzed by hot alkali to 3,5-dichloroaniline. Using this strategy, this method determines Iprodione, RP-30228, and RP-32490 as a single species (expressed as Iprodione-equivalents) in a variety of plant matrices, including fruits (succulent and dry), vegetative plant parts (wet and dried), and seeds (fatty/non-fatty, high starch/low starch).

B. Principle

Aliquots of plant material are weighed directly into glass vessels for subsequent alkaline hydrolysis. For some matrices that foam excessively during hydrolysis/distillation, a preliminary acetone extraction is required (e.g., samples with high fat and/or starch content such as cottonseed and dry beans). After

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1ROVRAL® is a fungicide developed by the Rhône-Poulenc Ag Company for use on a variety of edible crops.

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extraction and filtration to remove solids, the acetone extract is evaporated and the dry residue is quantitatively transferred to a glass vessel for hydrolysis.

RP-26019, RP-30228, and RP-32490 residues are hydrolyzed by overnight reaction with hot aqueous alkali in a tightly-sealed glass vessel. The hydrolysis product, 3,5-dichloroaniline, is distilled from the reaction mixture, partitioned into methylene chloride, then reacted with 2-chloropropionyl chloride (CPC) to yield N-(3,5-dichlorophenyl)-2-chloropropylamide (3,5-DCPA). Further purification is effected by Florisil® chromatography. Quantification of 3,5-DCPA in the final extract is performed by GC with electron capture detection. This procedure is sensitive to 0.05 ppm Iprodione-equivalent residue.

Figure 1 presents the chemical structures of Iprodione (RP-26019), RP-30228, and RP-32490.

II. MATERIALS AND METHODS

A. Equipment

Analytical Balance
Blender, High Speed
Boiling Stones
Compressed Air (GC)
Distillation Glassware
Filter Paper, GF/A
Fused-Silica Wool, Deactivated
Gas Chromatograph with Electron Capture Detector Wide-bore Capability Split-Splitless Injector

Suggested Manufacturers:

Ohaus GA110
Oster
Fisher Scientific, Cat. #09-191-12
Local Supply, Bottled
Fisher Scientific, (See Figure 2)
Whatman
Restek Corporation, Cat. #20790
Hewlett-Packard Model 5890 Series II

3Equivalent sources of the listed equipment and reagents may be used.

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Gas Chromatograph Column: Supelco "Sup-Herb"

General Laboratory Glassware

Glass Bottles

Glass Columns: 11 mm i.d. X 25 cm, equipped with a teflon stopcock and a 250 mL reservoir

Glass Wool

Helium Gas (GC)

Hydrogen Gas (GC)

Nitrogen Gas (GC)

Nitrogen Gas (Evaporations)

Oven

Rotary Evaporator
Single Pan Balance

B. Reagents and Standards

Antifoam B*: Sigma Chemical Company, Catalog #A-5757

Acetone: Fisher Optima Grade


Cyclohexane: Fisher HPLC Grade

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Diethylether (Ether): Fisher Anhydrous

6% Diethylether in Hexane: Dilute 30 mL of diethylether to 500 mL with hexane.

15% Diethylether in Hexane: Dilute 75 mL of diethylether to 500 mL with hexane.

Dry Ice*: Local Supplier
Ethyl Acetate: Fisher Optima Grade

Florisil*: 100-200 mesh, Fisher Scientific

Activate Florisil* overnight at 150°C. Cool and store in a desiccator; the reagent is stable for 12 months.

Hexane: Fisher Optima Grade
Methylene Chloride (DCM): Fisher Optima Grade

Potassium Hydroxide: Fisher Scientific

3N Aqueous KOH: Dissolve 168 g KOH pellets in 1 L of distilled water. Cool to room temperature.

Sodium Chloride: Fisher Scientific
Sodium Sulfate (anhydrous): Fisher Scientific, (ACS grade)

Water: Distilled or Deionized

Iprodione, RP-26019, 3-(3,5-dichlorophenyl)-N-(1-methyl-ethyl)-2,4-dioxo-1-imidazolidinecarboxamide, available from Rhône-Poulenc Ag Company (RPAC').

RP-30228, 3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide, available from RPAC.

RP-32490, 3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide, available from RPAC.

N-(3,5-dichlorophenyl)-2-chloropropylamide (3,5-DCPA), available from RPAC.

*RPAC is an acronym for the Rhône-Poulenc Ag Company.
C. Analytical Procedure

C.1 Sample Preparation

Remove debris and foreign materials from the sample. Homogenize the sample thoroughly with Dry Ice* in a grinder, then freeze pending analysis.

C.2 High-Fat and/or High-Starch Matrices: Extraction

a. Place a 10.0-gram sample into a one-quart blender jar (Note 1). Quality control fortifications are made at this point.

b. Add 200 mL of acetone to the sample and blend at high speed for 3 minutes.

c. Vacuum-filter the extract through 2 GF/A filters (Note 2); re-extract the solids and top filter with 150 mL of acetone as noted in C.2.b., then filter. Wash the jar and the filter cake with a total of 30-40 mL of acetone. Combine the extracts and washes into a 500-mL flask; discard the solids.

d. Vacuum-evaporate (i.e., rotary-evaporate) the extract to near dryness.

e. Quantitatively transfer the evaporated residue to a new 125-mL Wheaton glass bottle using acetone as the transfer solvent. Evaporate the acetone under a slow stream of nitrogen gas. Proceed to Step C.4.

C.3 Other Matrices

a. Place a 10.0-gram sample into a new 125-mL Wheaton glass bottle (Note 1). Quality control fortifications are made at this point.

C.4 Hydrolysis/Distillation

a. Suspend the sample in 40 mL of 3N aqueous KOH. Tightly cap the bottle (teflon-lined cap), then place in an oven set at 100°C ± 5°C (Caution: See Note 3!). After ca. 30 minutes, remove the sample from the oven and further tighten the cap (Note 4). Return the sample to the oven for overnight, unattended hydrolysis (Note 5).
b. When hydrolysis is complete (12-15 hours), remove the sample from the oven and cool to room temperature (Caution: The sample must be cooled to room temperature-or-below before opening the hydrolysis bottle. The bottle contents are pressurized when hot!). Quantitatively transfer the sample into a 1-L round-bottomed boiling flask with 3x25 mL of water. Add an additional 300-350 mL of water to the flask (Note 6).

c. Add 15 drops of Antifoam-B® (1.5 mL for cottonseed matrix) to the sample. Add 15-20 small boiling stones, then connect the flask to the distillation apparatus described in Figure 2.

d. Bring the extract to a rolling boil. Collect ca. 100 mL of distillate in a beaker or Erlenmeyer flask. Caution: DO NOT HEAT A CLOSED DISTILLATION SYSTEM (See Note 7 and Figure 2).

C.5 Dichloromethane Partition

a. Transfer the distillate into a 1-L separatory funnel using ca. 50 mL of distilled water and 100 mL of dichloromethane (DCM). Add 250 mL of distilled water to the sample.

b. Mix the phases thoroughly (ca. 30 seconds). After phase separation, percolate the lower DCM layer through a tightly-packed glass-wool plug (pre-washed with DCM) into a flask. Repeat the partition sequence two more times with 100 mL of DCM each time. Pool the DCM extracts; discard the aqueous phase (Note 8).

c. Add 5 drops of 2-chloropropionyl chloride (CPC) to the DCM solution (Caution: Severe lachrymator)(Note 9). Allow to react at room temperature for 30 minutes. The reaction product is N-(3,5-dichlorophenyl)-2-chloropropylamide (3,5-DCPA).

d. Rotary-evaporate the DCM extract to dryness. Add 10 mL of cyclohexane, then rotary-evaporate to dryness. Repeat the cyclohexane addition/evaporation sequence once more, then rotary-evaporate the sample to dryness (Note 10).
C.6 Florisil® Chromatography

a. Prepare a Florisil® clean-up column as follows: Place 1 gram of fully-activated Florisil® in an 11 mm i.d. glass column containing a glass wool plug. Top the Florisil® with a small amount (ca. 0.5 cm) of anhydrous sodium sulfate. Do not pre-equilibrate the column with solvent. The column is now ready for use.

b. Dissolve the dry residue from C.5.d in 10 mL of 6% ether in hexane. Transfer 4 mL of the solution to the top of the Florisil® column, taking care not to disturb the column surface. Percolate the sample into the column (ca. 2 drops/second), then wash the column sides with a total of 8 mL of 6% ether in hexane. Percolate the rinse through the column. Discard the eluate (Note 11).

c. Elute 3,5-DCPA from the column with 35 mL of 15% ether in hexane (Note 12).

d. Rotary-evaporate the eluant to dryness. Dissolve the dry residue in a known volume of ethyl acetate (generally 5 mL for LOQ residues). Dilute with ethyl acetate as necessary to maintain the analyte concentration within the standard curve range. Submit the sample for GC/ECD analysis as described in Step D.

D. Gas Chromatographic Analysis

D.1 Equipment

A gas chromatograph equipped with an Electron Capture Detector is required. Split-splitless injection and wide-bore capabilities are suggested.

GC Column: Supelco Sup-Herb (Catalog #2-5322, 15 m length, 0.53 mm i.d., 0.5 μM film). Other columns may be substituted if they give satisfactory resolution between the 3,5-DCPA analyte and any interferences.
D.2 Suggested Operating Conditions

Temperatures:

Injector: 230°C, 2 mm dia. glass insert with 0.5 cm loosely-packed deactivated fused-silica wool plug.

Detector: 300°C

Column Temperature:

Initial: 95°C, hold 1 min.
Ramp Rate 1: 40°C/min to 180°C, hold 5 min.
Ramp Rate 2: 40°C/min to 280°C, hold 5 min.

Carrier Gas: He, 7.1 mL/min at 95°C, head pressure = 3.0 psig. Constant flow off.

Injector Purge: He @ 3.6 mL/min.

Split Vent: He @ 24 mL/min, on @ 0.75 min.

Detector make up: N, @ 55 mL/min.

Anode Purge: N, @ 5.6 mL/min.

Injection: 2 μL, Split/Splitless

E. Calibration Procedures

E.1 Preparation of Standard Solutions

a. Stock solutions of RP-26019 and RP-30228 are made in ethyl acetate; stock solutions of RP-32490 are made in 24% (v/v) acetone in ethyl acetate. Stock concentrations are approximately 1.0 mg/mL. Stability of these stock solutions during prolonged storage have not been evaluated.

Note: Solutions of RP-26019 and RP-30228 are stored in a freezer. However, RP-32490 solutions must be stored at room temperature since

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this compound precipitates during prolonged freezer storage.

b. Dilutions of RP-26019, RP-30228, and RP-32490 standards are made at appropriate concentrations for fortification standards. These dilutions are made in ethyl acetate.

c. Stock solutions of N-(3,5-dichlorophenyl)-2-chloropropylamide (3,5-DCPA) are made in ethyl acetate. GC standards are prepared in ethyl acetate from the stock solution. Stability of this analyte during prolonged freezer storage has not been evaluated.

E.2 Detector Calibration

The sensitivity of the ECD detector is monitored by injecting 3,5-DCPA standards before, between, and after the samples. The suggested mass range is 20 pg to 400 pg injected. 3,5-DCPA must be detectable at the chosen minimum concentration. For RP-26019 and RP-30228, a minimum standard of 61 pg 3,5-DCPA injected and a final dilution volume of 5 mL for a 10 gram sample results in a calculated limit of quantification (LOQ) of 0.05 ppm Iprodione equivalent for both analytes. For RP-32490, a minimum standard of 70.2 pg 3,5-DCPA injected and a final dilution volume of 5 mL for a 10 gram sample results in a calculated limit of quantification (LOQ) of 0.05 ppm Iprodione equivalent.

Under the conditions of this assay, 3,5-DCPA elutes from the GC column at ca. 7.1 minutes. The GC/ECD limit of detection for 3,5-DCPA is ca. 5 pg injected.

F. Methods of Calculation

F.1 Injection Sequence

Run sequences are started and ended with one or two standards; standard injections are made throughout the run, generally with no more than two to three sample injections between each standard. A standard curve of 3,5-DCPA concentration (ng/mL) versus peak height or area is constructed using a method of curve generation appropriate for the GC/ECD instrumentation. The construction may be linear, quadratic or logarithmic.
F.2 Calculations

Calculate ppm values for Iprodione-related residues using the following equation:

\[
(\text{ng/mL Final Extract} \times \text{(ml Final Extract)} \times \frac{10 \text{ mL Florisil Total Volume}}{\text{ml Florisil Allocut Volume}}) \times 0.1 \times \frac{1 \text{ pg}}{1000 \text{ ng}} = \text{ppm}
\]

The conversion factor corrects for molecular weight differences between 3,5-DCPA and the starting Iprodione-related compounds. The appropriate factors are: RP-26019, 1.31; RP-30228, 1.31; and RP-32490, 1.14. The molecular weights for RP-26019, RP-30228, RP-32490, and 3,5-DCPA are 310.2 g/mole, 330.1 g/mole, 288.1 g/mole, and 252.5 g/mole, respectively.

For fortified-control samples, use the following equation to calculate the percent recovery:

\[
\text{Recovery} = \frac{(\text{ppm Found} - \text{ppm Control})}{\text{ppm Fortified}} \times 100
\]

G. Interferences

G.1 Sample Matrices

In sample matrices tested to date, there are only minor interferences at the quantification limit of 0.05 ppm. Chromatograms from these matrices contain several peaks, but the retention time for 3,5-DCPA is free of matrix-derived interferences. The GC parameters should be optimized to maximize resolution between 3,5-DCPA and any potential interference.

Despite the substantial clean-up procedures employed in this method, extensive sample injections do cause some undesirable chromatographic effects, most notably reduced instrument sensitivity towards 3,5-DCPA. This problem is corrected by cleaning the injector insert and replacing the deactivated fused-silica wool plug (Note 13).
G.2 Other Pesticides

A specificity study has not been conducted for this method.

G.3 Solvents and Reagents

The solvents specified in this procedure do not present any interferences at the stated LOQ. However, do not use cotton in place of glass wool where the latter is indicated unless the cotton has been tested for interferences; numerous ECD sensitive compounds which interfere with 3,5-DCPA analysis may be co-extracted from cotton.

G.4 Glassware

No interferences are detected from the labware at the stated LOQ. All glassware is pre-rinsed with acetone, then dried prior to use. Glass vessels are recommended for all steps without substitutions with plastic.

III. METHODS VALIDATION

A. Experimental Design

The method was validated in blueberries, raspberries, prunes, cottonseed, dry bean hay, dry bean seed, succulent bean hay, and succulent bean pods-with-seeds. Generally, the method was validated on UTC* matrices for each Iprodione-related compound at 3 fortification levels on 2 separate days; the fortification levels were ca. 0.05 ppm, 0.5 ppm, and 5.0 ppm. Thus, each validation study consisted of 20 samples.

B. Analytical Reference Materials

Example reference materials used during the conduct of the method validation studies are described in Table 1.

*UTC = Untreated control.

*Exceptions: Succulent bean matrices were validated only with RP-26019 fortified at 0.05 ppm and 0.25 ppm, in duplicate. Cottonseed was validated at 0.05 ppm, 0.25 ppm, and 0.50 ppm for all three ROPRAL*-related residues.

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C. **Control Matrices**

The method was validated using the following UTC samples provided by RPAC:

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Sample Number</th>
<th>RPAC Trial #</th>
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<tbody>
<tr>
<td>Blueberries</td>
<td>RM5468 &amp; RM5704</td>
<td>92-034 &amp; 92-052</td>
</tr>
<tr>
<td>Raspberries</td>
<td>RM5897</td>
<td>92-063</td>
</tr>
<tr>
<td>Prunes</td>
<td>RM5764</td>
<td>92-056</td>
</tr>
<tr>
<td>Cottonseed</td>
<td>RL2393</td>
<td>91-044</td>
</tr>
<tr>
<td>Dry Bean Hay</td>
<td>RM09944</td>
<td>93-0216</td>
</tr>
<tr>
<td>Dry Bean Seeds</td>
<td>RM09948</td>
<td>93-0216</td>
</tr>
<tr>
<td>Succulent Bean Hay</td>
<td>RN10084</td>
<td>93-0234</td>
</tr>
<tr>
<td>Succulent Bean</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pods-with-seeds</td>
<td>RN10052</td>
<td>93-0231</td>
</tr>
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</table>

D. **Validation Results**

Analyte recoveries obtained during the method validation studies are summarized in Table 2. The recoveries are sorted by matrix, compound, fortification level, and extraction technique; each value is the mean of N independent determinations within a parameter.

Briefly, 115 total samples were analyzed, comprised of 8 plant matrices, 3 fortified compounds (RF-26019, RF-30228, and RP-32490), 3 fortification levels (LOQ, mid-range, and high-range), and 2 extraction techniques (pre-extraction with acetone versus hydrolyzed directly). The overall mean recovery was 96.6% ± 6.71%, considering all matrices, compounds, fortification levels, and extraction techniques. The mean recovery of analyte from various plant matrices ranged from 93.9% to 109.7%; this data reflects the uniform elimination of matrix effects by the method during the hydrolysis/distillation step. Similarly, recovery was independent of fortified compound, indicating that the 12-15 hour oven-hydrolysis period is sufficient for quantitative conversion of all Iprodione-related analytes to 3,5-dichloroaniline. Recovery at the LOQ (0.05 ppm) was quantitative; at higher fortification levels, recovery of analyte was biased slightly low but still averaged greater than 90%. Pre-extraction of matrix with acetone slightly reduced the recovery of

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*Pre-extracted with acetone (cottonseed and dry bean seeds) versus hydrolyzed directly without pre-extraction (all others).*

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analytes compared to direct hydrolysis, which likely reflects the additional sample manipulation and concomitant loss of residue(s).

Thus, the data demonstrate exceptional method ruggedness. The method is remarkably insensitive to compound, matrix, fortification level, and extraction technique effects and consistently yields recoveries which are nearly quantitative and within current EPA guidelines for performance of residue methods (i.e., 70% → 120%). However, during routine applications of the method at other laboratories, acceptance criteria for individual data sets should be based upon independently-derived evaluations of method performance.

Based upon data obtained from the method validation studies, the performance of this method is summarized as follows:

(1) Recoveries of RP-26019, RP-30228, and RP-32490 from all tested matrices averaged 96.6%.

(2) Effects on analyte recovery due to compound, matrix, fortification level, and extraction technique were absent.

(3) Method precision is estimated at 6.9% of the overall mean analyte recovery (μ = 96.6%).

Original raw data and the original reports for the methods validation research are archived at the RPAC archives in Research Triangle Park, NC. Reports which provided data for this general method are itemized in Table 3, References.
IV. NOTES

Note 1: Iprodione-related residues in certain matrix-types (e.g., cottonseed and dry bean seeds) must be extracted into acetone prior to base hydrolysis. If hydrolyzed without pre-extraction, these samples foam excessively during the subsequent distillation step. The foaming cannot be controlled by addition of antifoam reagents. Excess foam is typical of samples containing high fat and/or starch, but the need to pre-extract a sample is based solely upon the presence or absence of excess foam during the subsequent distillation step. This must be determined empirically by direct hydrolysis and distillation of control matrix prior to methods validation or any sample analyses.

Note 2: Filter aids such as Celite® or cellulose are not necessary.

Note 3: The Wheaton bottles listed in Section II(A) are highly recommended for this assay. These bottles have thick walls with sufficient strength to withstand internal pressures generated during the overnight oven-hydrolysis at 100°C. Do not overfill the bottles! Use the recommended 40 mL solvent volume and discard each bottle after a single use! In the developer's laboratory, bottle failure is associated with overfill and/or extended bottle use. Failure is not explosive; instead, failed bottles crack around the bottom and leak hot alkali onto internal oven surfaces. Bottle failure has not been observed with new bottles when the recommended 10 g sample/40 mL alkali volume is used. Some investigators place the glass vessels in stainless steel or Pyrex® spill trays housed in the oven; however, ovens with heating cycles based upon time rather than temperature may overheat the spill tray, causing excess heat and pressure in the glass vessels. Thus, use of spill trays should be limited to ovens with temperature-based heating cycles.

Note 4: The hydrolysis product, 3,5-dichloroaniline, is volatile. Bottles and caps used for the hydrolysis must remain sealed and leak-proof to prevent loss of the analyte. Use Teflon®-lined caps only; do not use rubber- or polyethylene-based liners since they adsorb the 3,5-dichloroaniline hydrolysis product.

Note 5: RP-26019, RP-30228, and especially RP-32490 are resistant to hydrolysis by hot alkali. Recoveries are less-than-quantitative when distillation proceeds concurrently with hydrolysis. Thus, alkaline hydrolysis is performed overnight in sealed glass containers, then the sample is
distilled for quantitative recovery of the 3,5-dichloroaniline hydrolysis product.

Note 6: With some matrix-types (e.g., cottonseed), the subsequent distillation step proceeds uneventfully until the latter stages when excess foam may occur; this problem is mitigated by using more water diluent (i.e., 350 mL rather than 300 mL) during distillation.

Note 7: Joints in the distillation apparatus must be firmly sealed to prevent loss of the volatile 3,5-dichloroaniline, except do not seal the collection flask to the condenser fitting (Figure 2). Instead, leave this fitting open to ambient pressure. Do not heat a closed distillation system!

Foam and/or boiling extract should not "bump" or reflux over into the collection flask. If this occurs excessively (>2 mL), the distillate may be transferred back into the reflux flask (cooled!) and re-distilled. The distillation requires ca. 2 hours to collect 300 mL. The pH of the distillate ranges from near-neutral to slightly basic. The 3,5-dichloroaniline product is recovered quantitatively in the condensate; it is not necessary to trap the aniline as the salt via addition of acid to the collection flask.

Note 8: If emulsions form, dissolve ca. 1-2 grams of sodium chloride in the aqueous phase, then mix vigorously.

Note 9: Each lot of CPC should be pre-qualified prior to use since substandard lots have been noted by developers of this method. To pre-qualify the reagent, pipet 10-15 µg of 3,5-dichloroaniline (in acetone) into ca. 300 mL of DCM. Add 5 drops of CPC reagent and allow to react for 30 minutes. Evaporate the sample as noted in Step C.5.d, dissolve the product in an appropriate volume of ethyl acetate, then analyze by GC/ECD. The yield should exceed 90%.

Substandard lots of CPC probably contain water. Water slowly hydrolyzes CPC, yielding 2-chloropropionic and hydrochloric acids. When added to sample extracts, these acids form salts with 3,5-dichloroaniline, rendering the latter unavailable for reaction with CPC.

Substandard lots of CPC may be cleaned up as follows: Caution: Perform all operations in a fume hood! In a separatory funnel, dilute ca. 100 mL of CPC with 100 mL of DCM. Wash the DCM with ca. 100 mL of 5% aqueous sodium bicarbonate (Caution: Do not cap the separatory

Iprodione/Plants/General Method
July 15, 1994

Rhône-Poulenc Ag Company
Study EC-94-288 Good No. 8768
Page 84
funnel. Instead, vigorously swirl the reagents in the un-capped separatory funnel. The reaction between bicarbonate and acids contained in the CPC reagent yields carbon dioxide which can rapidly pressurize and cause failure of a sealed separatory funnel. The pH of the aqueous bicarbonate layer should remain neutral to slightly-alkaline (pH paper); repeat the bicarbonate wash if the pH is acidic. Percolate the DCM layer through a cone of anhydrous sodium sulfate; discard the aqueous layer. Distill the DCM layer. DCM distills at ca. 39°C to 41°C; discard this distillate. Collect CPC which distills at temperatures greater than 95°C, then pre-qualify the product as noted above. Store the product in a refrigerator.

Note 10: Addition of cyclohexane and subsequent rotary-evaporation removes excess CPC from the sample.

Note 11: The pre-wash with 6% diethylether in hexane elutes several matrix- and reagent-related chromatographic interferences from the sample.

Note 12: Fractionation parameters for each batch of Florisil® and type of glass column must be independently evaluated. The 11 mm diameter columns noted in Section II(A) are strongly recommended since they yield 1 gram Florisil® columns with sufficient height/diameter ratio for adequate separation of 3,5-DCPA from interferences. Subtle differences between batches of Florisil® and variable column characteristics (i.e., variable column height/diameter ratios) may cause dramatic differences in 3,5-DCPA elution profiles from those presented herein.

Note 13: For reproducible chromatography, new deactivated fused-silica wool plugs in the injector insert must be "primed" with 4 to 6 injections of sample extract.
V. TABLES
Table 1. Standard Reference Materials.

<table>
<thead>
<tr>
<th>STANDARD NAME</th>
<th>LOT NUMBER</th>
<th>PURITY</th>
<th>PHYSICAL CHARACTERISTICS</th>
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</thead>
<tbody>
<tr>
<td>Iprodione, BP-26019, 3-(3,5-dichlorophenyl)-N-(1-methylethyl)-1,4-dioxo-1-</td>
<td>029812 Batch</td>
<td>99.9%</td>
<td>White Powder</td>
</tr>
<tr>
<td>indanazolidinecarboxamide, CAS 16774-19-7</td>
<td>/TV2851/0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(EA)30025DB)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BP-10128, 3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-indazolidine-</td>
<td>030412 Batch</td>
<td>99.6%</td>
<td>White Powder</td>
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<tr>
<td>carboxamide, CAS 63827-89-8</td>
<td>/MN766-787 (EA)30259F2)</td>
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<tr>
<td>BP-12490, 3-(3,5-dichlorophenyl)-2,4-dioxo-1-indazolidinecarboxamide.</td>
<td>030009 Batch</td>
<td>97.1%</td>
<td>White Crystals</td>
</tr>
<tr>
<td>/GD3089 (EA)2456F1)</td>
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<td>DCPA, N-(3,5-dichlorophenyl)-2-chloropropylamine</td>
<td>M110591</td>
<td>10%</td>
<td>White Crystals</td>
</tr>
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</table>

Ipodione/Plants/General Method
July 15, 1994
Table 2.

Recovery of Iprodione-Related Residues, Sorted by Plant Matrix, Fortified Compound, Fortification Level, and Extraction Technique.

<table>
<thead>
<tr>
<th>Sorted Parameter</th>
<th>N</th>
<th>Mean % Recovery</th>
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<td><strong>By Matrix</strong></td>
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<tr>
<td>Cottonseed</td>
<td>18</td>
<td>93.9</td>
</tr>
<tr>
<td>Raspberries</td>
<td>18</td>
<td>95.0</td>
</tr>
<tr>
<td>Dry Bean Seed</td>
<td>18</td>
<td>95.2</td>
</tr>
<tr>
<td>Succulent Bean Hay</td>
<td>4</td>
<td>95.2</td>
</tr>
<tr>
<td>Blueberries</td>
<td>18</td>
<td>96.4</td>
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<tr>
<td>Prunes</td>
<td>17</td>
<td>97.9</td>
</tr>
<tr>
<td>Dry Bean Hay</td>
<td>18</td>
<td>98.7</td>
</tr>
<tr>
<td>Succulent Bean Pods</td>
<td>4</td>
<td>109.7</td>
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<tr>
<td><strong>By Compound</strong></td>
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<td></td>
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<tr>
<td>Iprodione (RP-26019)</td>
<td>44</td>
<td>97.5</td>
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<tr>
<td>RP-30228</td>
<td>36</td>
<td>95.7</td>
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<tr>
<td>RP-32490</td>
<td>35</td>
<td>96.4</td>
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<tr>
<td><strong>By Fortification Level</strong></td>
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<td></td>
</tr>
<tr>
<td>0.05 ppm (LOQ)</td>
<td>40</td>
<td>99.1</td>
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<tr>
<td>0.25 - 0.50 ppm</td>
<td>45</td>
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<tr>
<td>5.0 ppm</td>
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<td><strong>By Extraction Technique</strong></td>
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<tr>
<td>Pre-Extracted with Acetone</td>
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<td>94.5</td>
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<td>Not Pre-Extracted with Acetone</td>
<td>79</td>
<td>97.5</td>
</tr>
</tbody>
</table>

N = 115
Mean = 96.6
S.D. (u) = 6.71

(a) S.D. = Standard deviation.
Table 3. References.


2. RPAC Report #44336, Ground Application of ROVRAL® WP Fungicide to Blueberries to Determine the Magnitude of Residue Present After Harvest, USA92R25, 1994.

3. RPAC Report #44360, Ground Application of ROVRAL® 4 Flowable Fungicide to Plums Followed by Processing to Establish Residue Concentration or Reduction Factors in Fresh Prunes, USA92R27, 1994.


VI. FIGURES
Figure 1. Chemical Structures of Iprodione (RP-26019), RP-30228, RP-32490, and 3,5-DCPA.

- **Iprodione**
  - **RP-26019**
  - 3-(3,5-dichlorophenyl)-N-(1-methyl ethyl)-2,4-dioxo-1-imidazolidinecarboxamide

- **30228**
  - 3-(1-methyl ethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide

- **32490**
  - 3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide

- **DCPA**
  - N-(3,5-dichlorophenyl)-3-chloropropyl amide
Figure 2. Distillation Unit Setup

14 320F Teflon Sleeve

11 472 10G 1000 ml Heating Mantle

K169500 2440 Distilling Adapter

K452000 2430 Condenser

Water Out

Water In

This joint is not sealed!

09 521 110 Transformer

Note: Fisher Scientific part numbers.
Figure 1. Example Chromatography
Example Standard Curve

3,5-DCPA
B:021094.WQ1

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<th>log Height</th>
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<tr>
<td>1.1</td>
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- Slds

<table>
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<th>Response (Height Units)</th>
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$r^2 = 0.9988 \quad r = 0.9994$

$log(\text{response}) = 0.9942[\log(\text{ng/mL})] + 2.9833$
NOTE!

Table recopied for legibility.

Ipodione/Plants/General Method
July 15, 1994
HL Study #10074
GC012594.5  60 ng/mL

NOTE!
Table recopied for legibility.

Iprodione/Plants/General Method
July 15, 1994
NOTE!
Table recopied for legibility.
NOTE!
Table recopied for legibility.
NOTE!

Table recopied for legibility.

Iprodione/Plants/General Method
July 15, 1994
Rhone-Poulenc Study Number USA91G41
Cottonseed Control (RL2393)
10092-1 5 ml
Injection volume 2 μl

NOTE!
Table recopied for legibility.

Rhone-Poulenc Ag Company
Study EC-94-288 GooD No. 8768
Page 102
**Rhone-Poulenc Study Number USA91G4**
Cottonseed Spike 0.05 ppm RP26019
10092-2 5 ml
Injection volume 2 μl

**Cottonseed Control + 0.05 ppm RP26019**
ML2393, Trial 91-044

Recovery = 110.2%

---

**NOTE!**
Table recopied for legibility.

### METHOD NAME: K*DCPA.XPT

#### SAMPLE 4

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<th>AMOUNT NAME</th>
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<td>241955</td>
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TOTAL HEIGHT = 135071
HUL FACTOR = 1.0000E+00

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**NOTE!**
Table recopied for legibility.

### METHOD NAME: K*DCPA.XPT

#### SAMPLE 4

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<td>PV</td>
<td>10308</td>
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<td>PB</td>
<td>241955</td>
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TOTAL HEIGHT = 135071
HUL FACTOR = 1.0000E+00

---

Ipodione/Plants/General Method
July 15, 1994
NOTE!
Table recopied for legibility.

Rhône-Poulenc Ag Company
Study EC-94-288 GOOD No. 8768
Page 39 of 50
### Table

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**METHOD NAME**: M\$DCPA.MET

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**TOTAL HEIGHT = 75186.**

**MUL FACTOR=1.0000E+00**

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Note: Table recopied for legibility.
NOTE!

Table recopied for legibility.
NOTE!
Table recopied for legibility.
RUN 9 1319  FEB 15, 1994  17:33:16

START

1F

7.690
7.413

STOP

RUN 9 1319  FEB 15, 1994  17:33:16

METHOD NAME: N*DCPA.NET

NO CALIB PEAKS FOUND

HEIGTHS

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TOTAL HEIGHT= 9205
NUL FACTOR=1.00008±00

NOTE!
Table recopied for legibility

RUN 9 1319  FEB 15, 1994  17:33:16

METHOD NAME: N*DCPA.NET

NO CALIB PEAKS FOUND

HEIGTHS

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<th>TYPE</th>
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<tbody>
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TOTAL HEIGHT= 9205
NUL FACTOR=1.00008±00

IpÌrdione/Plants/General Method
July 15, 1994

RhÌ`ne-Poulenc Ag Company
Study EC-94-288 GOoD No. 8768
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Page 108
NOTE!

Table recopied for legibility.
RUN 9 1279-002

RUN 9 1291  FEB 9, 1994  17:07:11 2
START

Raspberries
UTC Control
RMS377, Trial 92-067

Ipodione Eq. = 0.0126 ppm

STOP

NOTE!
Table recopied for legibility.

Iprodione/Plants/General Method
July 15, 1994

Rhône-Poulenc Ag Company
Study EC-94-288 GOO D No. 8768
Page 110
**RASPBERRIES**

Control + 0.05 ppm BP26019

**RMS677, Trial 92-063**

Recovery = 90.11

---

**NOTE!**

Table recopied for legibility.

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**METHOD NAME: M*DCPA.NET**

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<td>7.692 PB</td>
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<td>8.475 MH</td>
<td>55536</td>
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TOTAL HEIGHT = 66118

**MUL FACTOR=1.00000E+00**

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**METHOD NAME: M*DCPA.NET**

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<td>6.045 PV</td>
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<td>8.475 MH</td>
<td>55536</td>
<td>.041</td>
<td>1519</td>
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TOTAL HEIGHT = 66118

**MUL FACTOR=1.00000E+00**

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**Ipfredone/Plants/General Method**

July 15, 1994

---

**Rhône-Poulenc Ag Company**

Study EC-94-288 GOoD No. 8768

Page 46 of 50
HL Study #100086
10086-01R 5 mL
UTC Hay
RN10084

Succulent Bean Hay
UTC Control
RN10084, Trial 93-0241

Iprodione Eq. = 0.0400 ppm

<table>
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<tr>
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</tbody>
</table>

NOTE!
Table recopied for legibility.

Iprodione/Plants/General Method
July 15, 1994

Rhone-Poulenc Ag Company
Study EC-94-288 GooD No. 8768
Page 47 of 50
HL Study #10086
10086-02R 5 mL
0.0505 ppm Spike

Succulent Bean Bay
Control + 0.0505 ppm RP26019
RW10086, Trial 93-0234

Recovery = 93.01

NOTE!
Table recopied for legibility.

Iprodione/Plants/General Method
July 15, 1994

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Study EC-94-288 GOoD No. 8768
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Page 113
**HL Study #10086**

**10086-06** 5 mL

UTC Seed Pod

RN10052

Succulent Bean Pods-With-Seeds

UTC Control

RN10052, Trial 93-0231

Iprodione Eq. = 0.0497 ppm

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**NOTE!**

Table recopied for legibility.

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**Iprodione/Plants/General Method**

July 15, 1994
HL Study #10086
10086-08 5 mL
0.0505 ppm Spike

Succulent Bean Pods-With-Seeds
Control + 0.0505 ppm BP26019
RL10052, Trial 93-0231

Recovery = 110.94

NOTE!
Table recopied for legibility.

 Ipodione/Plants/General Method
July 15, 1994