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

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I. INTRODUCTION AND SUMMARY

A. Scope

Applications of ROVRAL®,[1] 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 (eg., samples with high fat and/or starch content such as cottonseed and dry beans). After

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

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

Equipment	Suggested Manufacturers:[2]
Analytical Balance	Ohaus GA110
Blender, High Speed	Oster
Boiling Stones	Fisher Scientific, Cat. #09-191-12
Compressed Air (GC)	Local Supply, Bottled
Distillation Glassware	Fisher Scientific, (See Figure 2)
Filter Paper, GF/A	Whatman
Fused-Silica Wool, Deactivated	Restek Corporation, Cat. #20790
Gas Chromatograph with Electron Capture Detector Wide-bore Capability Split-Splitless Injector	Hewlett-Packard Model 5890 Series II

[2]Equivalent sources of the listed equipment and reagents may be used.

Gas Chromatograph Column: Supelco "Sup-Herb"	Supelco, 15 M.X 0.53 mm i.d., 0.5 µM film thickness
General Laboratory Glassware	Various
Glass Bottles	Wheaton Media/Lab Bottle, Graduated, Wheaton #219815, Fisher #06-404F, Teflon-lined Cap
Glass Columns: 11 mm i.d. X 25 cm, equipped with a teflon stopcock and a 250 mL reservoir	Fisher Scientific
Glass Wool	Fisher Scientific, Cat. # 11-388
Helium Gas (GC)	Local Supply, Bottled, 99.999%
Hydrogen Gas (GC)	Local Supply, Bottled, 99.999%
Nitrogen Gas (GC)	Local Supply, Bottled, 99.999%
Nitrogen Gas (Evaporations)	Local Supply, Bottled, 99.9%
Oven	Capable of sustained operation at 100°C ± 5°C
Rotary Evaporator	Fisher Scientific
Single Pan Balance	Ohaus E400

B. Reagents and Standards

Antifoam B®: Sigma Chemical Company, Catalog #A-5757
Acetone: Fisher Optima Grade

2-Chloropropionyl Chloride: Aldrich Chemical Company, 98%, Catalog #15,713-9. Caution: Severe lachrymator. Store in a refrigerator (See Note 9).

Cyclohexane: Fisher HPLC Grade

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-methylethyl)-2,4-dioxo-1-imadazolidinecarboxamide, available from Rhône-Poulenc Ag Company (RPAC[3]).

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.

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[3]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. 300 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_2 @ 55 mL/min.
Anode Purge:	N_2 @ 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

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:

(1)

$$ppm = (ng/mL\ Final\ Extract) \times (mL\ Final\ Extract) \times \frac{10\ mL\ Florisil\ Total\ Volume}{4\ mL\ Florisil\ Aliquot\ Volume} \times \frac{1}{10\ grams} \times \frac{1\ ug}{1000\ ng} \times Conversion\ Factor$$

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 330.2 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:

$$\%\ Recovery = \left(\frac{(ppm\ Found) - (ppm\ Control)}{ppm\ Fortified}\right) \times 100$$

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[4] 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[5]. 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.

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[4]UTC = Untreated control.

[5]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 ROVRAL®-related residues.

C. Control Matrices

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

Matrix	Sample Number	RPAC Trial #
Blueberries	RM5468 & RM5704	92-034 & 92-052
Raspberries	RM5897	92-063
Prunes	RM5764	92-056
Cottonseed	RL2393	91-044
Dry Bean Hay	RN09944	93-0216
Dry Bean Seeds	RN09948	93-0216
Succulent Bean Hay	RN10084	93-0234
Succulent Bean Pods-with-seeds	RN10052	93-0231

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[4]; 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 (RP-26019, RP-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 analytes 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

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

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 (eg., 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 (eg., 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

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 → 6 injections of sample extract.

V. TABLES

Table 1. Standard Reference Materials.

STANDARD NAME	LOT NUMBER	PURITY	PHYSICAL CHARACTERISTICS
Iprodione, RP-26019, 3-(3,5-dichlorophenyl)-N-(1-methylethyl)-2,4-dioxo-1-imadazolidinecarboxamide, CAS #36734-19-7	029812 Batch #TV2852/D (EA2002SD8)	99.9%	White Powder
RP-30228, 3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolinecarboxamide, CAS #63637-89-8	030142 Batch #JM786-787 (EA2025RP2)	99.6%	White Powder
RP-32490, 3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide.	030009 Batch #GD8309 (EA2026RF1)	97.1%	White Crystals
DCPA, N-(3,5-dichlorophenyl)-2-chloropropylamide	MW110593	100%	White Crystals

Table 2.

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

Sorted Parameter	N	Mean % Recovery
By Matrix		
Cottonseed	18	93.9
Raspberries	18	95.0
Dry Bean Seed	18	95.2
Succulent Bean Hay	4	95.2
Blueberries	18	96.4
Prunes	17	97.9
Dry Bean Hay	18	98.7
Succulent Bean Pods	4	109.7
By Compound		
Iprodione (RP-26019)	44	97.5
RP-30228	36	95.7
RP-32490	35	96.4
By Fortification Level		
0.05 ppm (LOQ)	40	99.1
0.25 - 0.50 ppm	45	95.4
5.0 ppm	30	94.9
By Extraction Technique		
Pre-Extracted with Acetone	36	94.5
Not Pre-Extracted with Acetone	79	97.5
N =	115	
Mean =		96.6
S.D. (a) =		6.71

(a) S.D. = Standard deviation.

Table 3. References.

1. RPAC Report #44356, Ground Application of ROVRAL® WP Fungicide to Raspberries to Determine the Magnitude of Residue Present After Harvest, USA92R26, 1994.

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.

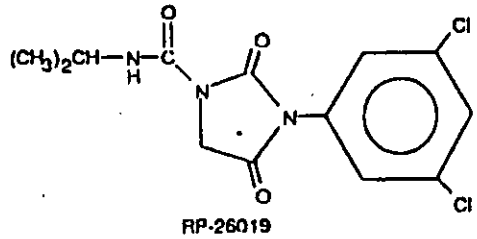
4. RPAC Report #44334, Determination of the Magnitude of Residues in/on Dry Beans Treated by Ground Application of ROVRAL® 4 Flowable Fungicide, US93R01R, 1994.

5. RPAC Report #44364, Determination of the Magnitude of Residues in/on Succulent Beans Treated by Ground Application of ROVRAL® 4 Flowable Fungicide, US93R08R, 1994.

6. RPAC Report #44333, ALIETTE®/ROVRAL® 15G/Cotton/Magnitude of Residue, USA91G41, 1994.

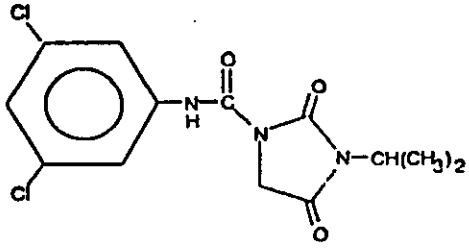
VI. FIGURES

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



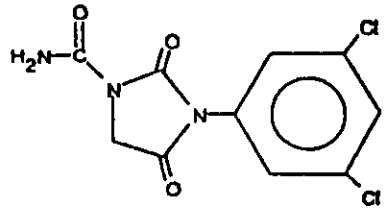
RP-26019

3-(3,5-dichlorophenyl)-N-(1-methylethyl)-2,4-dioxo-1-imidazolidinecarboxamide



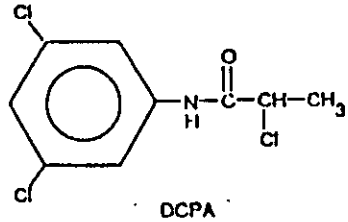
RP-30228

3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide



RP-32490

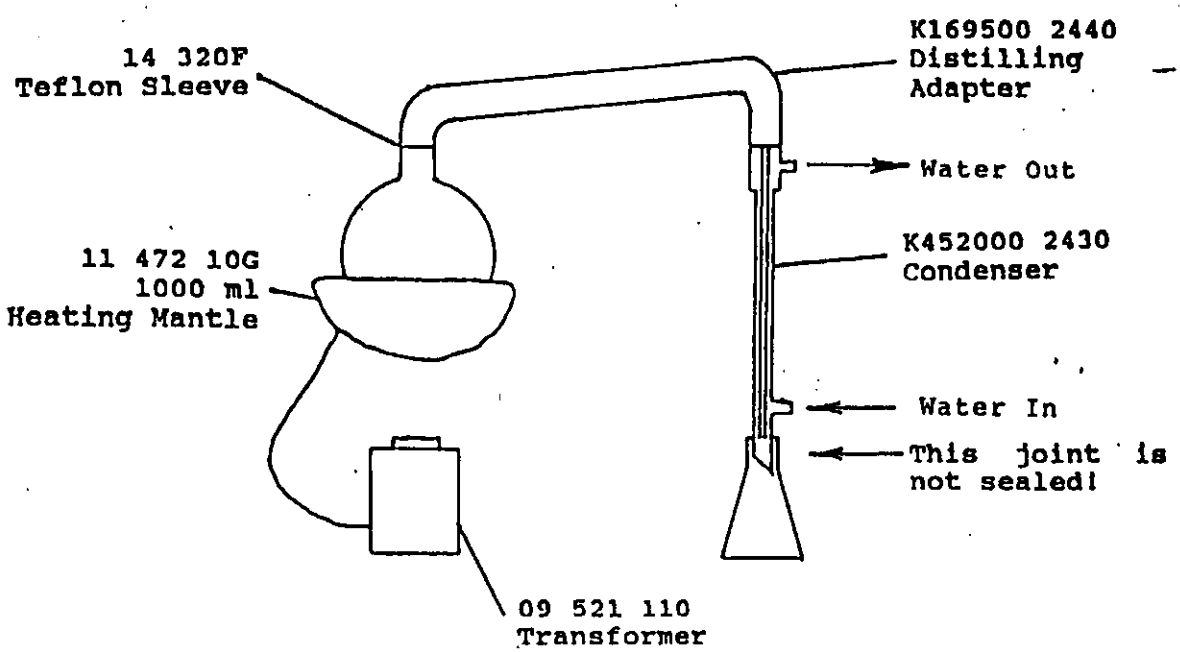
3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide



DCPA

N-(3,5-dichlorophenyl)-2-chloropropylamide

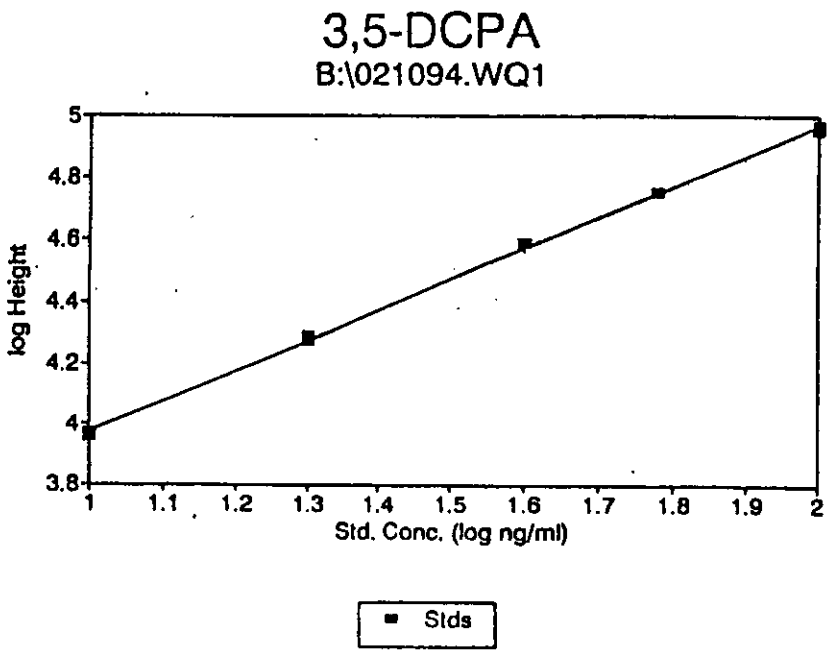
Figure 2. Distillation Unit Setup



Note: Fisher Scientific part numbers.

Figure 3. Example Chromatography

Example Standard Curve

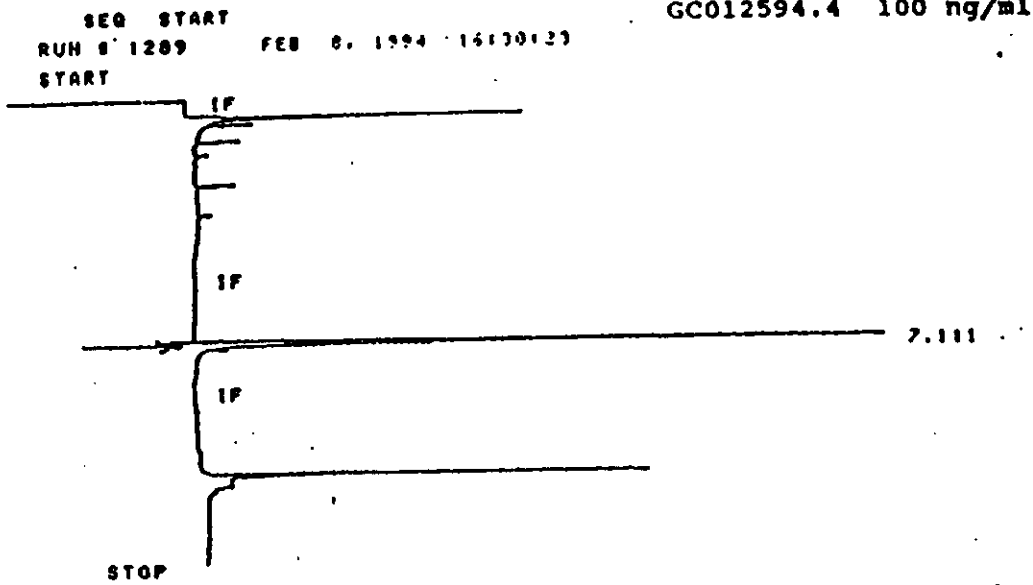


Concentration (ng/mL)	Response (Height Units)
100	93657
10	9413
20	19712
40	39336
60	56665
20	18936
10	9124
100	89742

$r^2 = 0.9988$ $r = 0.9994$

log(response) = 0.9942[log(ng/mL)] + 2.9833

HL Study #10074
GC012594.4 100 ng/ml



RUN# 1289 FEB 8, 1994 16:30:23

SAMPLE# 1

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.111	PB	415532	.079	87326	1	.000	DCPA

TOTAL HEIGHT= 87326
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

(E) DAT 1/13/95
Table not readable

RUN# 1289 FEB 8, 1994 16:30:23

SAMPLE# 1

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

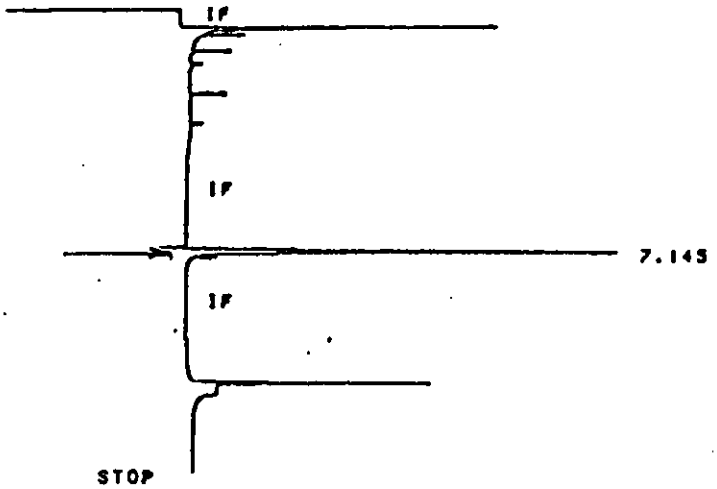
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.111	PB	415532	.079	87326	1	.000	DCPA

TOTAL HEIGHT= 87326
MUL FACTOR=1.0000E+00

RUN 8. 1298-002

HL Study #10074
GC012594.5 60 ng/mL

RUN # 1299 FEB 8, 1994 19:33:13
START



RUN# 1299 FEB 8, 1994 19:33:18

SAMPLE# 11

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.145	PB	257498	.080	53853	1	.000	DCPA

TOTAL HEIGHT= 53858
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

(2) DAT 1/13/95 Table not readable

RUN# 1299 FEB 8, 1994 19:33:18
SAMPLE# 11
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

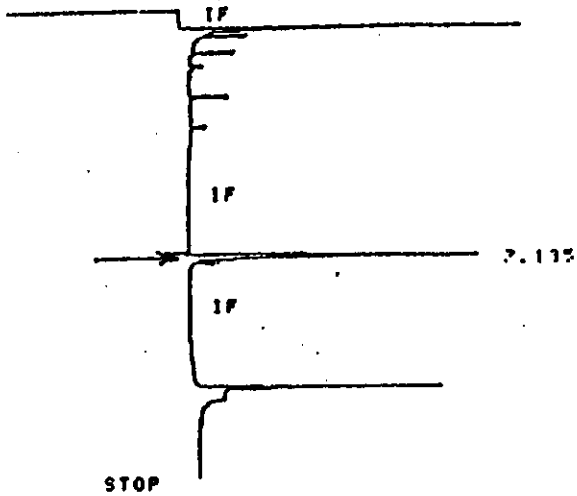
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.145	PB	257498	.080	53858	1	.000	DCPA

TOTAL HEIGHT= 53858
MUL FACTOR=1.0000E+00

RUN # 1295-002

HL Study #10074
GC012594.6 40 ng/mL

RUN # 1296 FEB 8, 1994 18:38:32
START



RUN# 1296 FEB 8, 1994 18:38:32

SAMPLE# 8

METHOD NAME: M*DCPH.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.135	PB	177321	.079	37472	1	.000	DCPH

TOTAL HEIGHT= 37472
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

RUN# 1296 FEB 8, 1994 18:38:32

SAMPLE# 8

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.135	PB	177321	.079	37472	1	.000	DCPA

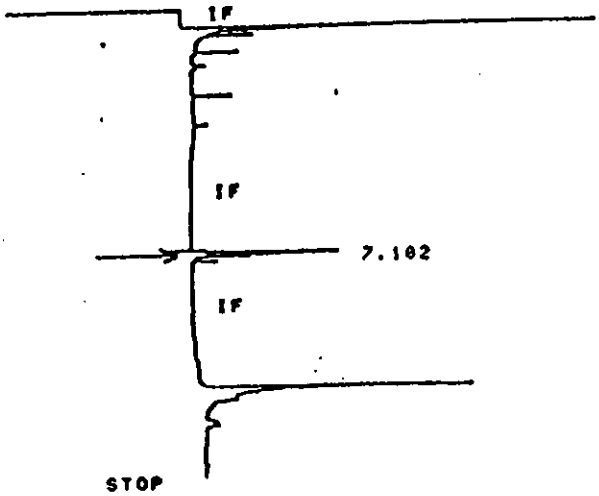
TOTAL HEIGHT= 37472
MUL FACTOR=1.0000E+00

(E) DAT 1/13/95
Table not readable

RUN # 1292-002

HL Study #10074
GC012594.7 20 ng/mL
~~Injection Volume: 2 µL~~ ⑤ with 2-28-94

RUN # 1293 FEB 8, 1994 17:43:45
START



RUN# 1293 FEB 8, 1994 17:43:45

SAMPLE# 5

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.102	PB	90399	.080	18816	1	.000	DCPA

TOTAL HEIGHT= 18816
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

RUN# 1293 FEB 8, 1994 17:43:45

SAMPLE# 5

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.102	PB	90399	.080	18816	1	.000	DCPA

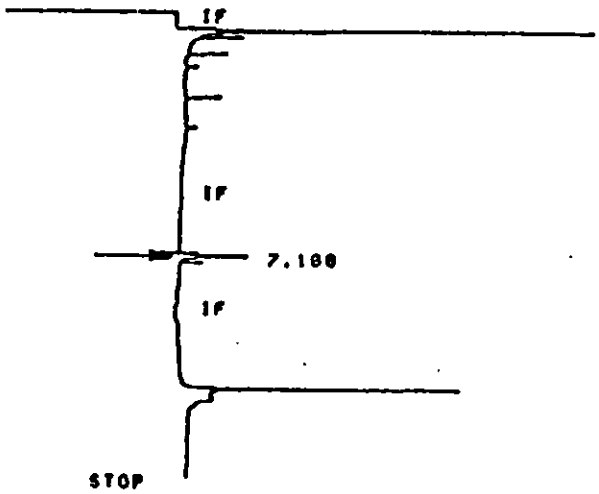
TOTAL HEIGHT= 18816
MUL FACTOR=1.0000E+00

⑤ DAT 1/13/95
Table not readable

RUN # 1289-002

HL Study #10074
GC012594.8 10 ng/ml

RUN # 1290 FEB 8, 1994 16:48:56
START



RUN# 1290 FEB 8, 1994 16:48:56

SAMPLE# 2

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.100	PB	45190	.084	8981	1	.000	DCPA

TOTAL HEIGHT= 8981
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

(E) DAT 1/13/95
Table not readable

RUN# 1290 FEB 8, 1994 16:48:56
SAMPLE# 2
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

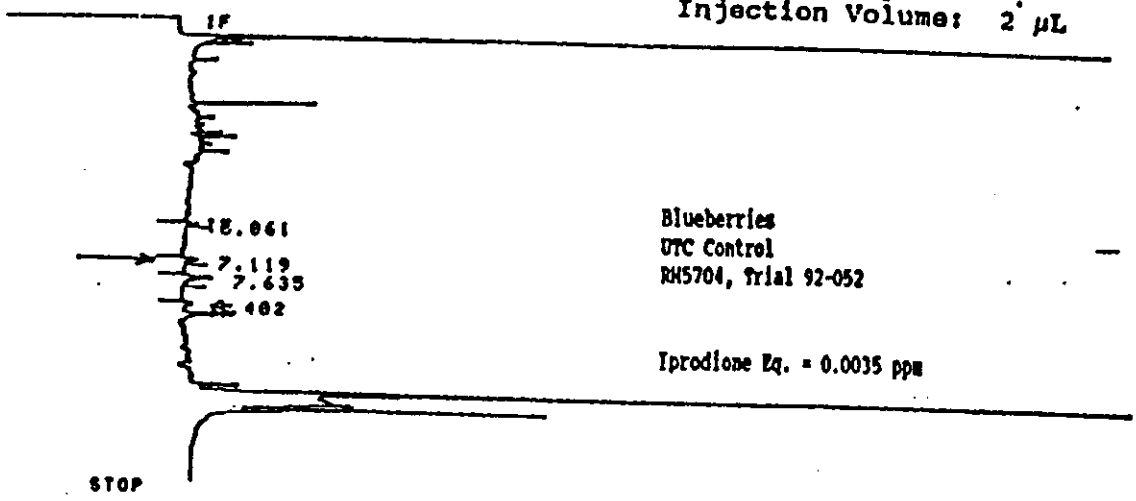
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.100	PB	45190	.084	8981	1	.000	DCPA

TOTAL HEIGHT= 8981
MUL FACTOR=1.0000E+00

RUN # 1127-002

Rhone-Poulenc AL Study #10075
10075-11R 5 mL
UTC Blueberry
Injection Volume: 2 µL

RUN # 1128 FEB 2, 1994 17:33:46
START



Blueberries
UTC Control
RH5704, Trial 92-052

Iprodione Eq. = 0.0035 ppm

RUN# 1128 FEB 2, 1994 17:33:46

SAMPLE# 3

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.061	BB	6648	.059	1870		.000	
7.119	PB	7952	.078	1709	IP	.000	DCPA
7.635	BB	21966	.100	3661		.000	
8.402	I BH	7407	.088	1398		.000	

TOTAL HEIGHT= 8638
MUL FACTOR=1.0000E+00

NOTE !

Table recopied for legibility.

RUN# 1128 FEB 2, 1994 17:33:46
SAMPLE# 3
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.061	BB	6648	.059	1870		.000	
7.119	PB	7952	.078	1709	1R	.000	DCPA
7.635	BB	21966	.100	3661		.000	
8.402	I BH	7407	.088	1398		.000	

TOTAL HEIGHT= 8638
MUL FACTOR=1.0000E+00

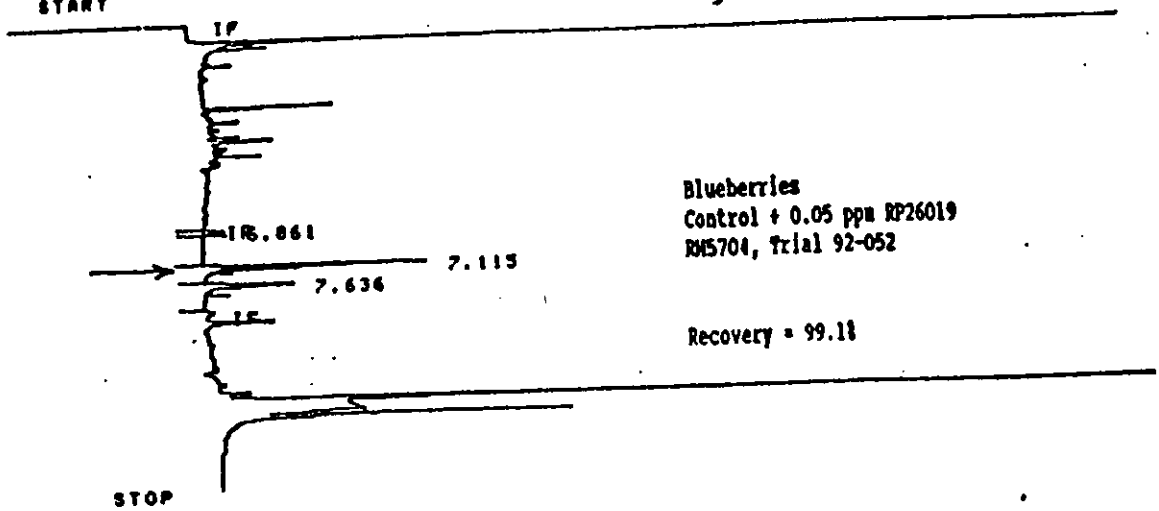
E. DLA 1/13/95

Table not readable

RUN # 1128-002

Rhone-Poulenc Study #10075
10075-12R 5 mL
0.05 ppm Spike
Injection Volume: 2 µL

RUN # 1129 , FEB 2, 1994 17:52:04
START



Blueberries
Control + 0.05 ppm RP26019
RM5704, Trial 92-052

Recovery = 99.1%

STOP

RUN# 1129 FEB 2, 1994 17:52:04

SAMPLES 4

METHOD NAME: M*DCPA.MET

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
ESTD-HEIGHT							
6.061	BB	9031	.058	2607		.000	
7.115	PB	129013	.077	27904	1R	.000	DCPA
7.636	BB	61916	.092	11180		.000	

TOTAL HEIGHT= 41691
MUL FACTOR=1.0000E+00

(E) DAS 1/13/95
Table not readable

RUN# 1129 FEB 2, 1994 17:52:04
SAMPLES 4
METHOD NAME: M*DCPA.MET

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
ESTD-HEIGHT							
6.061	BB	9031	.058	2607		.000	
7.115	PB	129013	.077	27904	1R	.000	DCPA
7.636	BB	61916	.092	11180		.000	

TOTAL HEIGHT= 41691
MUL FACTOR=1.0000E+00

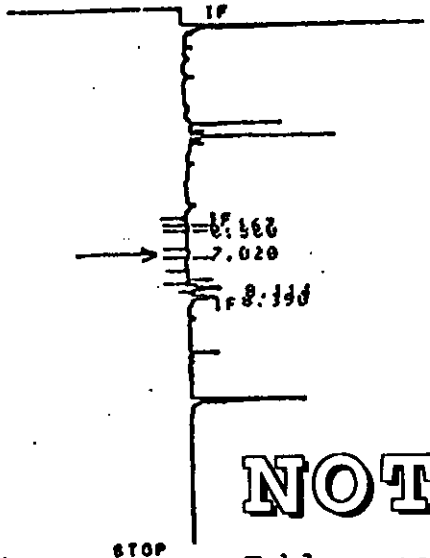
NOTE!

Table recopied for legibility.

Rhone-Poulenc Study Number USA91G41
Cottonseed Control (RL2393)
10092-1 5 ml
Injection volume 2 µl

RUN # 4172-002

RUN # 4173 MAY 27, 1994 16:17:00
START



Cottonseed
UTC Control
RL2393, Trial 91-044

Iprodione Eq. = 0.0012 ppm

RUN# 4173 MAY 27, 1994 16:17:00
METHOD NAME: H:DCPA.MET SAMPLE# 3

ESTD-HEIGHT RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.167	PB	7394	.103	1199		.000	
6.360	BB	5843	.074	1308		.000	
7.020	PB	8846	.090	1633	IP	.000	DCPA
8.114	BV	110113	.098	18663		.000	
8.390	I VB	103994	.105	16552		.000	

TOTAL HEIGHT= 39355
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

RUN# 4173 MAY 27, 1994 16:17:00

SAMPLE# 3

METHOD NAME: H:DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.167	PB	7394	.103	1199		.000	
6.360	BB	5843	.074	1308		.000	
7.020	PB	8846	.090	1633	IP	.000	DCPA
8.114	BV	110113	.098	18663		.000	
8.390	I VB	103994	.105	16552		.000	

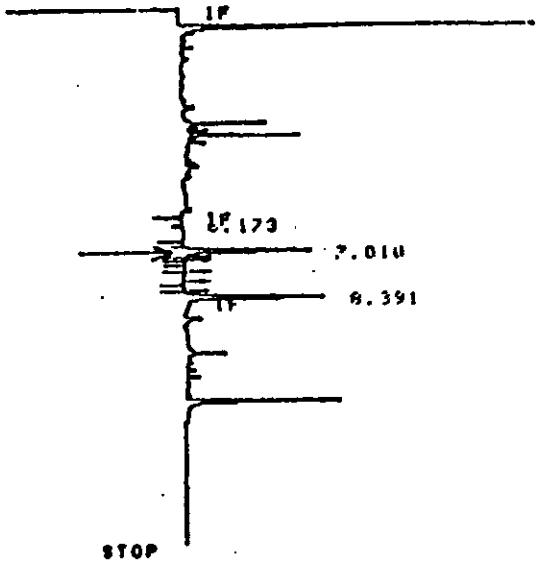
TOTAL HEIGHT= 39355
MUL FACTOR=1.0000E+00

DAT 1/13/95 Table not readable

Rhone-Poulenc Study Number USA91G4
Cottonseed Spike 0.05 ppm RP26019
10092-2 5 ml
Injection volume 2 µl

RUN # 4173-002

RUN # 4174 MAY 27, 1994 16:37:29
START



Cottonseed
Control + 0.05 ppm RP26019
RL2393, Trial 91-044

Recovery = 110.2%

RUN# 4174 MAY 27, 1994 16:37:29 ← NOTE !

SAMPLE# 4

METHOD NAME: M+DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.173	PV	10308	.116	1480		.000	
7.010	PB	314955	.082	63706	IP	.000	DCPA
8.391	I BM	393690	.093	70685		.000	

TOTAL HEIGHT= 135871
MUL FACTOR=1.0000E+00

Table recopied for legibility.

RUN# 4174 MAY 27, 1994 16:37:29
SAMPLE# 4

METHOD NAME: M+DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.173	PV	10308	.116	1480		.000	
7.010	PB	314955	.082	63706	IR	.000	DCPA
8.391	I BM	393690	.093	70685		.000	

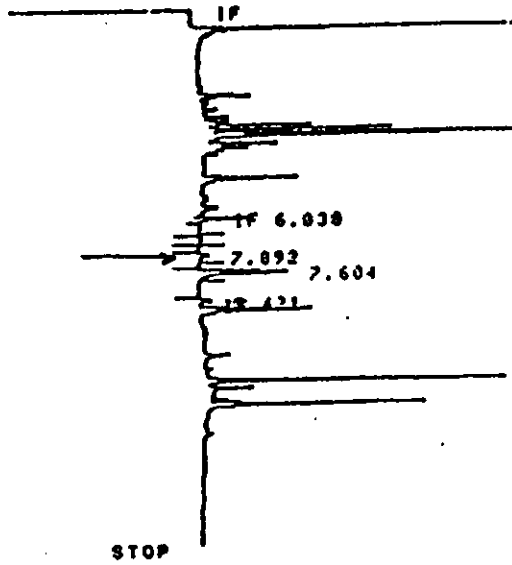
TOTAL HEIGHT= 135871
MUL FACTOR=1.0000E+00

E.
DAT 1/13/95
Table not readable

RUN # 1800-002

HL Study #10080
10080-21 5 mL
UTC Hay at pod fill
RN09944①.

RUN # 1801 FEB 23, 1994 16:31:40
START



Bean Hay at Pod Fill
UTC Control
RN09944, Trial 93-0216

Iprodione Eq. = 0.0044 ppm

RUN# 1801 FEB 23, 1994 16:31:40

SAMPLE# 3

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.038	BP	24917	.041	10029		.000	
7.092	BB	15744	.099	2656	1R	.000	DCPA
7.604	PB	126617	.092	22855		.000	
8.421	I BH	14344	.080	3000		.000	

TOTAL HEIGHT= 38540
MUL FACTOR=1.0000E+00

NOTE!
Table recopied for legibility.

RUN# 1801 FEB 23, 1994 16:31:40
SAMPLE# 3
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAM
6.038	BP	24917	.041	10029		.000	
7.092	BB	15744	.099	2656	1R	.000	DCP
7.604	PB	126617	.092	22855		.000	
8.421	I BH	14344	.080	3000		.000	

TOTAL HEIGHT= 38540
MUL FACTOR=1.0000E+00

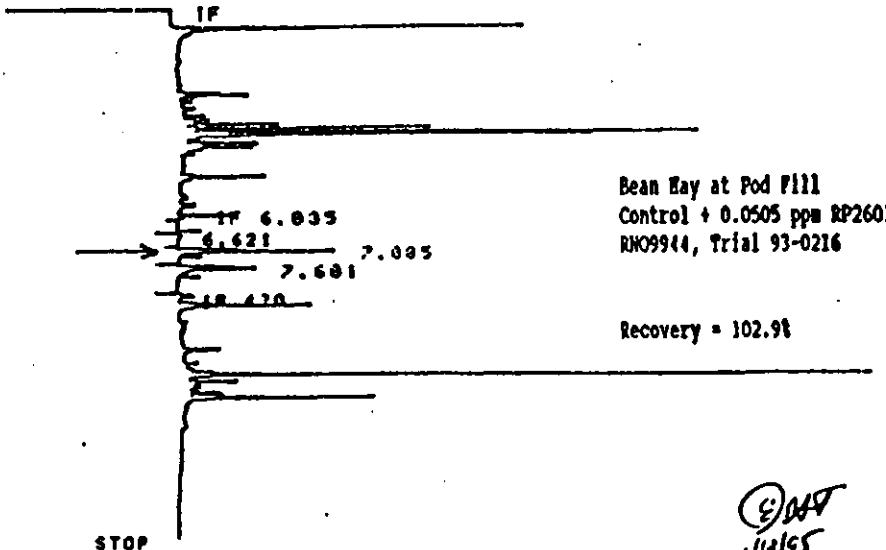
① DCW/TM 2-28-94
② DAT 1/13/95
Table not readable

RUN 8 1801-002

HL Study #10080
10080-22 5 mL
0.05 ppm Spike
0.0505

RUN 8 1802 FEB 23, 1994 16:52:05

START



Bean Hay at Pod Fill
Control + 0.0505 ppm RP26019
RH09944, Trial 93-0216

Recovery = 102.9%

RUN8 1802 FEB 23, 1994 16:52:05

SAMPLE# 4

METHOD NAME: M+DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.035	BP	24642	.038	10847		.000	
6.621	BP	9134	.126	1205		.000	
7.085	PB	182702	.079	38605	1R	.000	DCPA
7.601	BB	116224	.096	20156		.000	
8.420	I PM	20427	.078	4373		.000	

TOTAL HEIGHT= 75186
MUL FACTOR=1.0000E+00

RUN8 1802 FEB 23, 1994 16:52:05

SAMPLE# 4

METHOD NAME: M+DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.035	BP	24642	.038	10847		.000	
6.621	BP	9134	.126	1205		.000	
7.085	PB	182702	.079	38605	1R	.000	DCPA
7.601	BB	116224	.096	20156		.000	
8.420	I PM	20427	.078	4373		.000	

TOTAL HEIGHT= 75186
MUL FACTOR=1.0000E+00

DAT 1/13/95 Table not readable

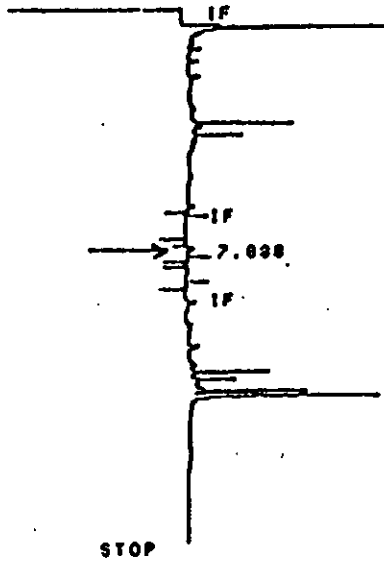
NOTE!

Table recopied for legibility.

RUN # 1742-002

HL Study #10080
10080-11 5 mL
UTC Dried Seed
RN09948

RUN # 1743 FEB 22, 1994 17:09:33
START



Dry Bean Seed
UTC Control
RN09948, Trial 93-0216

Iprodione Eq. = 0.0039 ppm

RUN# 1743 FEB 22, 1994 17:09:33

SAMPLE# 3

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.088	PB	14913	.096	2582	1P	.000	DCPA

TOTAL HEIGHT= 2582
MUL FACTOR=1.0000E+00

NOTE !

Table recopied for legibility.

(E) DAT 1/13/95
Table not readable

RUN# 1743 FEB 22, 1994 17:09:33
SAMPLE# 3

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

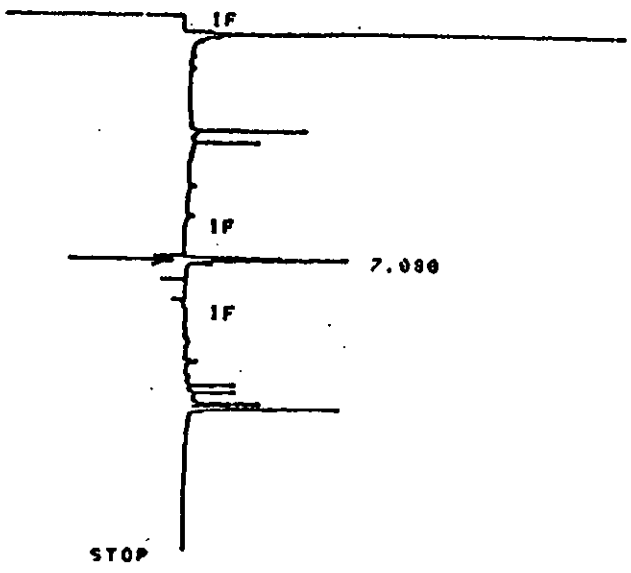
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.088	PB	14913	.096	2582	1R	.000	DCPA

TOTAL HEIGHT= 2582
MUL FACTOR=1.0000E+00

RUN # 1743-002

HL Study #10080
10080-12 5 mL
0.005 ppm Spike
0.0505 0.0505
0.04
Form 4690

RUN # 1744 FEB 22, 1994 17:29:53
START



Dry Bean Seed
Control + 0.0505 ppm RP26019
RH09948, Trial 93-0216

Recovery = 106.2%

RUN# 1744 FEB 22, 1994 17:29:53

SAMPLE# 4

METHOD NAME: M:DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.080	PB	195164	.078	41554	1P	.000	DCPA

TOTAL HEIGHT= 41554
MUL FACTOR=1.0000E+00

NOTE !

Table recopied for legibility.

RUN# 1744 FEB 22, 1994 17:29:58

SAMPLE# 4

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.080	PB	195164	.078	41554	1R	.000	DCPA

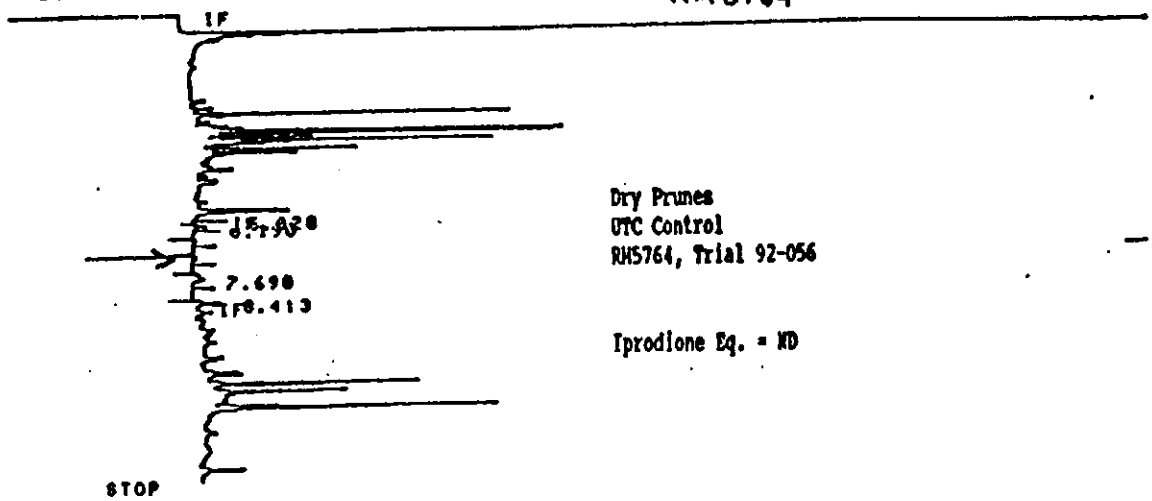
TOTAL HEIGHT= 41554
MUL FACTOR=1.0000E+00

(E) DAT 1/13/95
Table not readable

RUN # 1518-002

HL Study #10078
10078-21 5 mL
UTC Dried Fruit
RM5764

RUN # 1519 FEB 15. 1994 17:33:14
START



Dry Prunes
UTC Control
RH5764, Trial 92-056

Iprodione Eq. = ND

RUN# 1519 FEB 15. 1994 17:33:14

SAMPLE# 3

METHOD NAME: N*DCPA.MET

NO CALIB PEAKS FOUND
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
6.028	2401	BP	.031	26.06365
6.190	1870	PB	.066	20.31505
7.690	1533	BB	.145	16.65399
8.413	3401 I	BH	.083	36.94731

TOTAL HEIGHT= 9205
MUL FACTOR=1.0000E+00

NOTE !

Table recopied for legibility

RUN# 1519 FEB 15, 1994 17:33:14
SAMPLE# 3

METHOD NAME: N*DCPA.MET

NO CALIB PEAKS FOUND
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
6.028	2401	BP	.031	26.06365
6.190	1870	PB	.066	20.31505
7.690	1533	BB	.145	16.65399
8.413	3401 I	BH	.083	36.94731

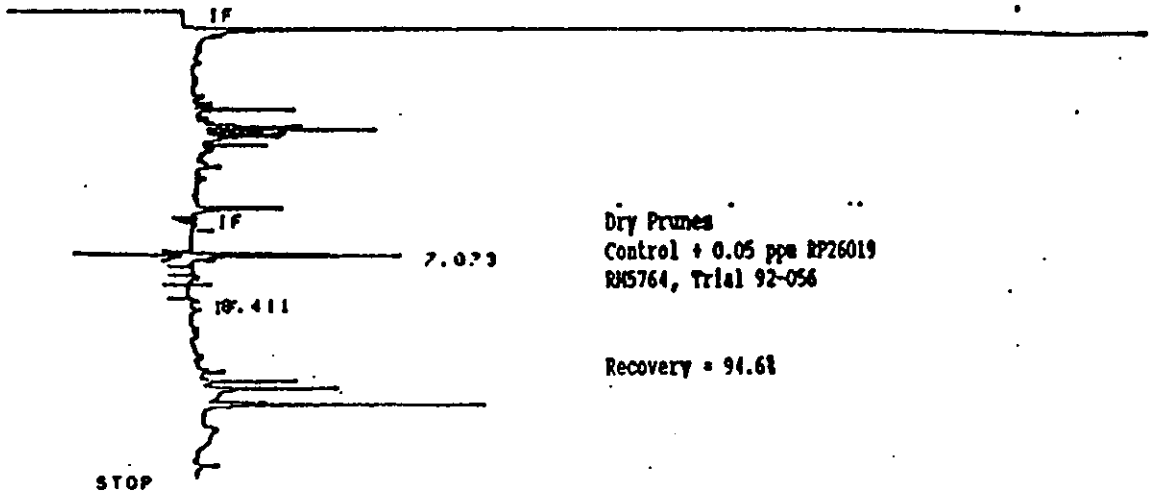
TOTAL HEIGHT= 9205
MUL FACTOR=1.0000E+00

(E) DAS 1/13/95 Table not readable

RUN # 1519-002

HL Study #10078
10078-22 5 mL
0.05 ppm Spike

RUN # 1520 FEB 15, 1994 17:51:35
START



Dry Prunes
Control + 0.05 ppm RP26019
RM5764, Trial 92-056

Recovery = 94.6%

RUN# 1520 FEB 15, 1994 17:51:35

SAMPLE# 4

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.073	PB	128413	.078	27580	1	.000	DCPA
8.411	I BH	7059	.091	1289		.000	

TOTAL HEIGHT= 28869
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

E DHT 1/13/95 Table not readable

RUN# 1520 FEB 15, 1994 17:51:35
SAMPLE# 4
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

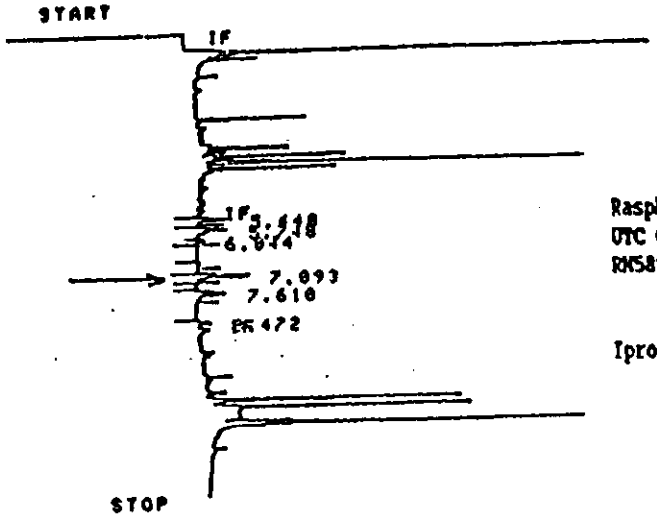
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.073	PB	128413	.078	27580	1	.000	DCPA
8.411	I BH	7059	.091	1289		.000	

TOTAL HEIGHT= 28869
MUL FACTOR=1.0000E+00

RUN # 1290-002

HL Study #10074
10074-1 5 mL
UTC Raspberry

RUN # 1291 FEB 8, 1994 17:07:12
START



Raspberries
UTC Control
RH5897, Trial 92-063

Iprodione Eq. = 0.0126 ppm

RUN# 1291 FEB 8, 1994 17:07:12

NOTE!

SAMPLE# 3

Table recopied for legibility.

METHOD NAME: M:DCPM.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
5.440	PB	13498	.056	4033		.000	
5.748	PP	20064	.086	3897		.000	
6.044	PB	4297	.060	1186		.000	
7.093	PB	32720	.079	6939	1	.000	DCPM
7.610	BB	23023	.104	3698		.000	
8.472	I BH	6996	.063	1849		.000	

TOTAL HEIGHT= 21602
MUL FACTOR=1.0000E+00

RUN# 1291 FEB 8, 1994 17:07:12
SAMPLE# 3

METHOD NAME: M:DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
5.440	PB	13498	.056	4033		.000	
5.748	PP	20064	.086	3897		.000	
6.044	PB	4297	.060	1186		.000	
7.093	PB	32720	.079	6939	1	.000	DCPA
7.610	BB	23023	.104	3698		.000	
8.472	I BH	6996	.063	1849		.000	

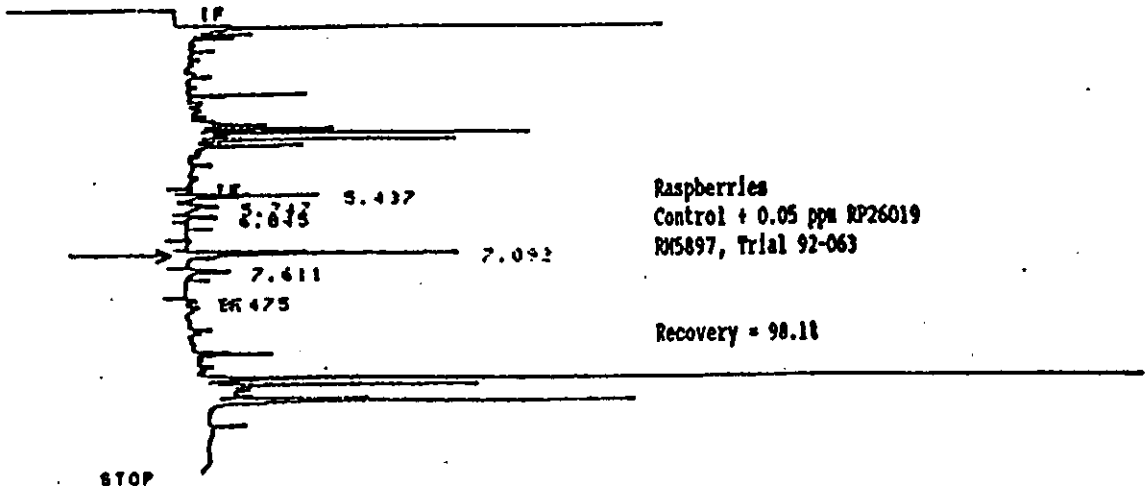
TOTAL HEIGHT= 21602
MUL FACTOR=1.0000E+00

EDD 1/13/95 Table not readable

RUN # 1291-002

HL Study #10074
10074-2 5 mL
0.05 ppm Spike

RUN # 1292 FEB 8, 1994 17:25:29
START



Raspberries
Control + 0.05 ppm RP26019
RM5897, Trial 92-063

Recovery = 98.1%

RUN# 1292 FEB 8, 1994 17:25:29

SAMPLE# 4

METHOD NAME: M*DCPM.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
5.437	PV	56114	.056	16737		.000	
5.747	VP	26182	.102	4274		.000	
6.045	PV	14813	.059	4208		.000	
7.092	PB	156069	.077	33820	1	.000	DCPA
7.611	BB	34151	.102	5560		.000	
8.475	I BH	5536	.061	1519		.000	

TOTAL HEIGHT= 66118
MUL FACTOR=1.0000E+00

NOTE !

Table recopied for legibility.

(E) DOD 1/13/95 Table not readable

RUN# 1292 FEB 8, 1994 17:25:29

SAMPLE# 4

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
5.437	PV	56114	.056	16737		.000	
5.747	VP	26182	.102	4274		.000	
6.045	PV	14813	.059	4208		.000	
7.092	PB	156069	.077	33820	1	.000	DCPA
7.611	BB	34151	.102	5560		.000	
8.475	I BH	5536	.061	1519		.000	

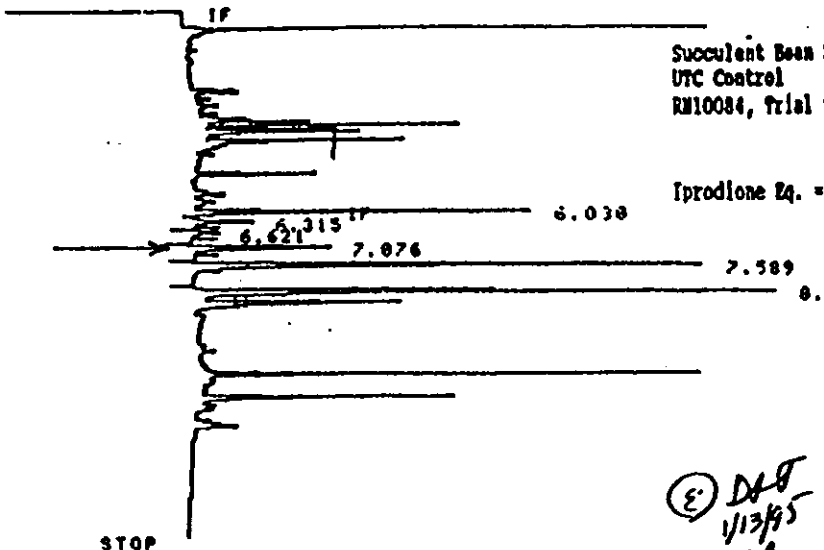
TOTAL HEIGHT= 66118
MUL FACTOR=1.0000E+00

RUN # 3040-002

HL Study #10086
10086-01R 5 mL
UTC Hay
RN10084

RUN # 3041 MAR 25, 1994 15:34:24

START



Succulent Bean Hay
UTC Control
RN10084, Trial 93-0234

Iprodione Eq. = 0.0480 ppm

RUN# 3041 MAR 25, 1994 15:34:24
METHOD NAME: M:OCPA.MET SAMPLE# 3

EST-R'T	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.030	BP	75089	.026	47437		.000	
6.315	PB	64286	.067	15948		.000	
6.621	BB	31515	.077	6851		.000	DCPA
7.076	PB	181894	.080	37766	1R	.000	
7.589	PB	686463	.089	128170		.000	
8.355 I	BM	763937	.086	147565		.000	

TOTAL HEIGHT= 383737
MUL FACTOR=1.0000E+00

(E) DAT 1/13/95 Table not readable

RUN# 3041 MAR 25, 1994 15:34:24

SAMPLE# 3

METHOD NAME: M:OCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.030	BP	75089	.026	47437		.000	
6.315	PB	64286	.067	15948		.000	
6.621	BB	31515	.077	6851		.000	
7.076	PB	181894	.080	37766	1R	.000	DCPA
7.589	PB	686463	.089	128170		.000	
8.355 I	BM	763937	.086	147565		.000	

TOTAL HEIGHT= 383737
MUL FACTOR=1.0000E+00

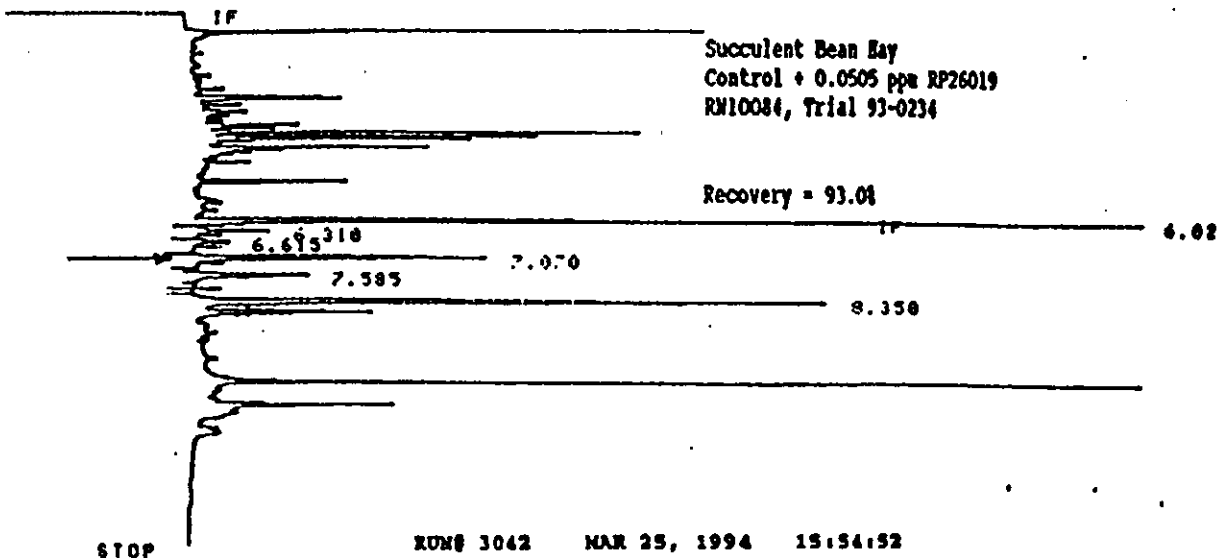
NOTE!

Table recopied for legibility.

RUN # 3041-002

HL Study #10086
10086-02R 5 mL
0.0505 ppm Spike

RUN # 3042 MAR 25, 1994 15:54:52
START



RUN# 3042 MAR 25, 1994 15:54:52
SAMPLE# 4
METHOD NAME: M*DCPA.MET

RUN# 3042 MAR 25, 1994 15:54:52
SAMPLE# 4
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
6.310	BB	70959	.067	17751		.000	
6.615	BB	36474	.073	8296		.000	
7.070	PB	360364	.080	75812	1R	.000	DCPA
7.585	PP	153953	.089	28703		.000	
8.350	I BB	821643	.086	158505		.000	

TOTAL HEIGHT= 288567
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

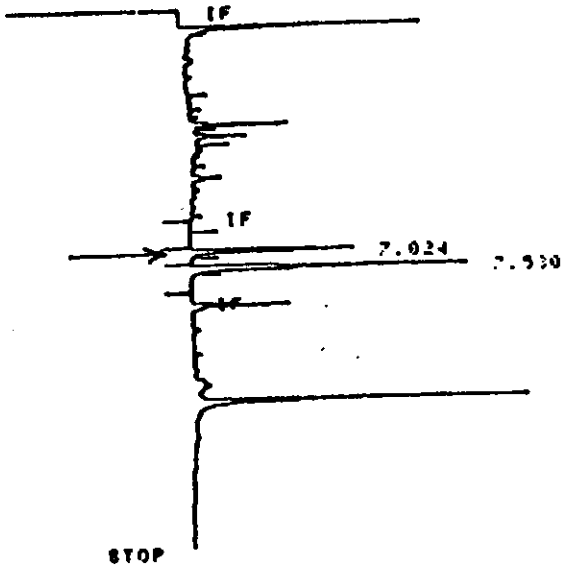
(E) DAT 4/13/95 Table not readable

Iprodione/Plants/General Method
July 15, 1994

HL Study #10086
10086-06 5 mL
UTC Seed Pod
RN10052

RUN # 2955-002

RUN # 2956 MAR 23, 1994 20:31:45



Succulent Bean Pods-With-Seeds
UTC Control
RN10052, Trial 93-0231

Iprodione Eq. = 0.0497 ppm

RUN# 2956 MAR 23, 1994 20:31:45

SAMPLE# 14

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.024	PB	194700	.080	40568	1R	.000	DCPA
7.530	PB	371777	.091	68304		.000	

TOTAL HEIGHT= 108872
MUL FACTOR=1.0000E+00

NOTE!

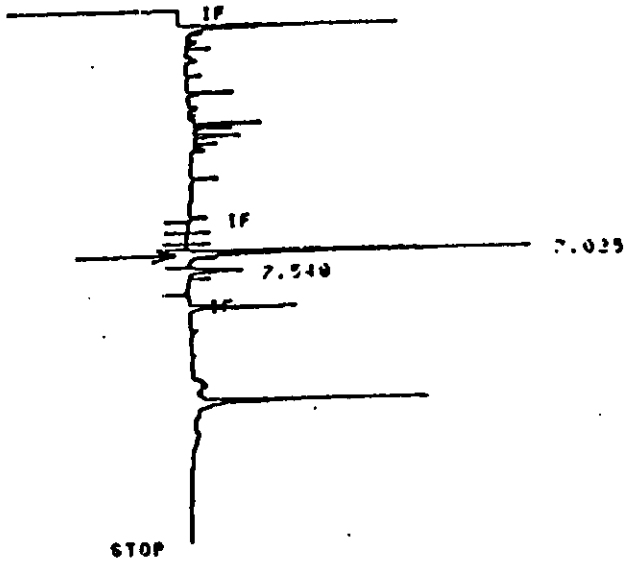
Table recopied for legibility.

(E) DOT 1/13/95 Table not readable

HL Study #10086
10086-08 5 mL
0.0505 ppm Spike

RUN # 2958-002

RUN # 2959 MAR 23, 1994 21:33:19
START



Succulent Bean Pods-With-Seeds
Control + 0.0505 ppm RP26019
RM10052, Trial 93-0231

Recovery = 110.9%

RUN# 2959 MAR 23, 1994 21:33:19

SAMPLE# 17

METHOD NAME: M*DCPA.MET

ESTD-HEIGHT RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.025	PB	412840	.080	85589	1R	.000	DCPA
7.540	BB	79796	.091	14620		.000	

TOTAL HEIGHT= 100209
MUL FACTOR=1.0000E+00

NOTE!

Table recopied for legibility.

RUN# 2959 MAR 23, 1994 21:33:19
SAMPLE# 17
METHOD NAME: M*DCPA.MET

ESTD-HEIGHT RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	AMOUNT	NAME
7.025	PB	412840	.080	85589	1R	.000	DCPA
7.540	BB	79796	.091	14620		.000	

TOTAL HEIGHT= 100209
MUL FACTOR=1.0000E+00

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Table not readable