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OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Iprodione. Plum (Fresh Prune) Processing Study. Rereg. Case No. 2335.
Chemical No. 109801 MRID #43255702 DP Barcode D205006 CBRS #13956

FROM: Steven A. Knizner, Chemist *St A Knizner*
Chemistry Pilot Review Team
Chemistry Branch II - Reregistration Support
Health Effects Division (7509C)

THRU: Francis B. Suhre, Acting Chief *Francis Suhre*
Chemistry Branch II - Reregistration Support
Health Effects Division (7509C)

TO: William Wooge, PM Team 52
Special Review and Registration Division (7505C)

The Iprodione Phase 4 Review (C. Olinger, 3/15/91) required the registrant to conduct a processing study for plums (fresh prunes). In response, Rhone-Poulenc submitted data (MRID #43255702) from a processing study conducted in 1992 depicting the residues of iprodione, its isomer, and its metabolite in/on plums, washed plums, and dried prunes grown in WA. Data from this submission are summarized in the attached Data Evaluation Record produced by Dynamac Corp. under contract to the Agency.

A tolerance of 20.0 ppm has been established for the combined residues of iprodione, its isomer, and its metabolite in/on plums as a result of preharvest and postharvest applications [40 CFR §180.399(a)]. A tolerance of 20.0 ppm has also been established for the combined residues of iprodione, its isomer, and its metabolite in/on prunes [40 CFR §180.399(a)]. CBRS notes that in conjunction with the iprodione RED, the commodity entry "Prunes" should be changed to "Prunes, fresh".

For plums and prunes, a maximum of five foliar applications of iprodione at 0.5-1.0 lb ai/A may be made per growing season using ground equipment in a minimum of 20 gal water/A or aerial equipment in a minimum of 15 gal/A. Applications may be made at 7- to 14-day



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intervals. A 0-day PHI has been established. The 50% WP formulation (EPA Reg. No. 264-453, dated 7/7/94) is also registered for postharvest applications to plums. A single application of a solution containing 1 lb ai/100 gal may be made to 200,000 lb of plums as a wax spray or dip solution.

Conclusions

1. Adequate raw data pertaining to field trial information, application of the test substance, sample-handling procedures, and processing procedures including material balance, were provided.
2. Plums (fresh prunes) grown in WA were harvested on the day of the last of five foliar applications of the 4 lb/gal EC formulation (EPA Reg. No. 264-482) at 2.0 lb ai/A/application (2x the maximum seasonal application rate for preharvest applications) in 100 gal water/A using ground equipment. Applications began at the prebloom stage and were repeated at full bloom (6-day retreatment interval), petal fall (5-day interval), postbloom (96-day interval), and on the day of harvest (40-day interval). The plums did not receive a post-harvest application of iprodione.
3. Plums were received at the processing facility and were refrigerated (0-4 C) until processing was initiated the next day. The plums were briefly washed using a high pressure cold water spray to remove excess dirt. The washed plums were briefly dipped in hot water (93 C) and then dried in an air dryer at 60-74 C for 24-36 hours to reduce moisture content to ~18%.
4. The submitted recovery data indicate that the GC/ECD analytical method used is adequate for data collection of iprodione residues of concern in/on plums and plum processed commodities.
5. The combined residues of iprodione, its isomer, and its metabolite were 0.41' - 0.99 ppm in/on plums receiving a 2X preharvest treatment of iprodione.
6. The submitted data indicate that the combined residues of iprodione, its isomer, and its metabolite concentrated up to 6.6x in prunes processed from plums bearing detectable residues. The average concentration factor was approximately 4x. CBRS notes that the maximal theoretical concentration factor for prunes is 4x (Follow-up Guidance For maximum Theoretical Concentration Factors, EPA 737-R-93-001).
7. Establishment of a Section 409 tolerance for iprodione in/on dried prunes may be affected by current/future deliberations concerning the Delaney Clause.

Recommendations

In conjunction with the iprodione RED, the commodity entry "Prunes" in 40 CFR §180.399(a) should be changed to "Prunes, fresh".

The submitted plum (fresh prune) processing study is fully adequate and satisfies Guideline 171-4(l) requirements for plums. No additional plum (fresh prune) processing data are required.

Based on the 4X concentration factor and the existing fresh prune tolerance of 20 ppm, CBRS would have to recommend for establishment of a food additive tolerance (FAT in 40 CFR §185.3750) for the combined residues of iprodione, its isomer, and its metabolite in/on dried prunes at 80 ppm.

CBRS notes that the iprodione residue levels (0.41 - 0.99 ppm) in/on fresh prunes (2X treatment rate) in this processing study are significantly lower than the existing iprodione tolerance of 20 ppm for fresh prunes. The registrant should be advised that residue chemistry data supporting the iprodione fresh prune tolerance may significantly over-estimate actual residues from the registered use.

The appropriate level for the FAT for dried prunes would be 4X that of the reassessed fresh prune tolerance established by the Agency. In the absence of additional data, CBRS will, at the time of the iprodione RED, recommend for an 80 ppm FAT for dried prunes.

Note to CRM:

CBRS may be able to recommend for a lower tolerance for iprodione and its related regulated compounds in/on the rac fresh prunes, and consequently the food dried prunes, provided the registrant submits additional field trial data. In the current study (one field trial in WA), iprodione residues on plums (fresh prunes) ranged from 0.41 to 0.99 ppm when a 2X pre-harvest treatment rate was used, the isomer and metabolite were not detected (<0.025 ppm each).

The registrant has previously submitted data in conjunction with PP#8E3645 (MRID #40637201) generated by IR-4 depicting the magnitude of the residue in plums receiving pre- and post harvest applications of iprodione. This study consisted of one field trial in CA, in which 4-6 plum trees were treated both pre- and post-harvest (in wax) at the maximum label rate (1X). Two samples were taken for the pre-harvest treatment only, and residues of iprodione were approximately 0.2 ppm (0.23 ppm and 0.2 ppm), with the isomer and metabolite non-detectable (<0.025 ppm each). The registrant attributed the low levels of residues found for the pre-harvest application to the use of commercial type application equipment and larger plots (4/6 trees plot), whereas earlier trials used backpack sprayer application and smaller plots.

In the same submission, the registrant stated that "... fruit harvested for processing as prunes is immediately dehydrated after harvest and thus would not be treated with postharvest fungicides (See attached documentation.)". This documentation consisted of the "California Prune Food Service Buying Guide, Second Edition, April, 1974", published by the California Prune Advisory Board (CPAB). The publication noted that "Immediately after harvesting the orchard ripe fruit is taken to the dehydrator yard where it is washed, placed on large wooden rays and dehydrated...". CBRS also notes that the 50% WP formulation (EPA Reg. No.

264-453, dated 7/7/94) is registered for postharvest applications to plums only, not fresh prunes, whereas the preharvest label directions specify both plums and prunes. CBRS agrees that it is highly unlikely that fresh prunes would receive a post-harvest treatment of iprodione prior to drying.

Attachment.

cc: S.F., circ., R.F., List B File, S.Knizner, J. Frane (PSPS)
RDI: W. Smith, B. Cropp-Kohlligian, L. Edwards, P. Deschamp, C. Olinger, 1/11/95, M. Metzger, 1/18/95, F. Suhre 1/20/95
7509C:CBRS:CM#2:305-6903:SAK:sak:iprodion\plum:1/11/95

DATA EVALUATION RECORD

DP BARCODE(S): D205006

CBRS NO.: 13956

STUDY TYPE: Magnitude of the Residue in/on Plum Processed Commodities [Guideline Reference No. 171-4(1)].

STUDY SPONSOR: Rhone-Poulenc Ag Company (Research Triangle Park, NC).

MRID NOS.: 43255702 S. Murayama. ROVRAL®/Plum/Magnitude of Residue/Processed Commodities. Rhone-Poulenc Study Number: USA92R27. Study Completed on 5/26/94.

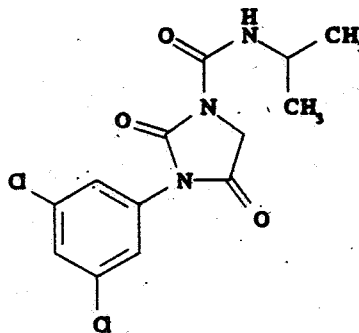
PERFORMING LABORATORIES: Field: Ron Britt and Associates, Inc. (Yakima, WA).
Processing: Wm. J. Englar and Associates, Inc. (Moses Lake, WA).
Analytical: Horizon Laboratories, Inc. (Columbia, MO).

TEST MATERIAL APPLIED TO CROP: Iprodione [3-(3,5-dichlorophenyl)-N-(1-methylethyl)-2,4-dioxo-1-imidazolidinecarboxamide] (CAS No. 36734-19-7).

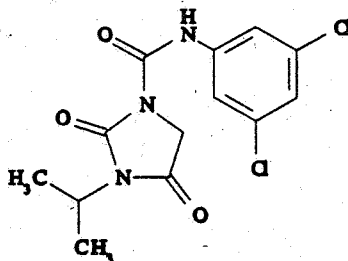
EPA REG. NO.: 264-482 (Rovral® 4F).

RESIDUES MEASURED:

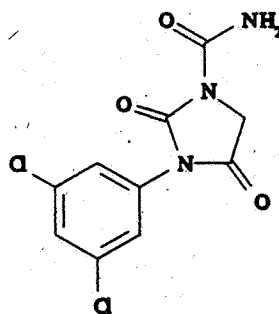
Iprodione



Iprodione isomer [3-(1-methylethyl)-N-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide]



Iprodione metabolite [3-(3,5-dichlorophenyl)-2,4-dioxo-1-imidazolidinecarboxamide]



DETAILED CONSIDERATIONS

Magnitude of the Residue in Plum Processed Commodities

Rhone-Poulenc Ag Company submitted data (1994; MRID 43255702) from a processing study conducted in 1992 depicting the residues of iprodione, its isomer, and its metabolite in/on plums, washed plums, and prunes grown in WA. Data from this submission are described and presented in this Data Evaluation Record.

Established tolerance: A tolerance of 20.0 ppm has been established for the combined residues of iprodione, its isomer, and its metabolite in/on plums as a result of preharvest and postharvest applications [40 CFR §180.399(a)]. No food additive tolerance has been established for prunes.

Use patterns: A REFS search conducted 9/27/94 identified four Rhone-Poulenc iprodione end-use products registered for use on plums: two 50% WP formulations (EPA Reg. No. 264-453, ROVRAL® Fungicide dated 7/7/94; and EPA Reg. No. 264-532, ROVRAL® 50 SP Fungicide, dated 7/6/94), a 4 lb/gal FIC formulation (EPA Reg. No. 264-482, ROVRAL® 4 Flowable Fungicide, dated 7/7/94), and a 50% EC formulation (EPA Reg. No. 264-524, ROVRAL® WG Fungicide, dated 5/xx/94; a copy of the label for this product was not

available for review). A maximum of five foliar applications at 0.5-1.0 lb ai/A may be made per growing season using ground equipment in a minimum of 20 gal water/A or aerial equipment in a minimum of 15 gal/A. Applications may be made at 7- to 14-day intervals. A 0-day PHI has been established.

The 50% WP formulation (EPA Reg. No. 264-453, dated 7/7/94) is also registered for postharvest applications to plums. A single application of a solution containing 1 lb ai/100 gal may be made to 200,000 lb of plums as a wax spray or dip solution.

Discussion of the data: Plums grown in WA were harvested on the day of the last of five foliar applications of the 4 lb/gal EC formulation (EPA Reg. No. 264-482) at 2.0 lb ai/A/application (2x the maximum seasonal application rate for preharvest applications) in 100 gal water/A using ground equipment. Applications began at the prebloom stage and were repeated at full bloom (6-day retreatment interval), petal fall (5-day interval), postbloom (96-day interval), and on the day of harvest (40-day interval).

Plum samples were harvested by hand, placed in boxes, and shipped to the processing facility the day of harvest at ambient temperature. The samples were received at the processing facility and were refrigerated (0-4 C) until processing was initiated the next day. The plums were briefly washed using a high pressure cold water spray to remove excess dirt. The washed plums were briefly dipped in hot water (93 C) and then dried in an air dryer at 60-74 C for 24-36 hours to reduce moisture content to ~18%. Adequate raw data pertaining to field trial information, application of the test substance, sample-handling procedures, and processing procedures including material balance, were provided.

After processing was completed, samples were stored frozen at -26 to -9 C for 12-17 days and then shipped frozen via ACDS freezer truck to Rhone-Poulenc (Research Triangle Park, NC) where they were stored frozen (<-10 C) for ~3.5 months. Samples were then shipped to Horizon Laboratories (Columbia, MO) where they were stored frozen at -20 to -10 C prior to analysis. The intervals between harvest and residue analysis were 540-541 days (~18 months); samples were analyzed within 1 day of extraction. Residues in/on treated and untreated plums and prunes were determined using a GC/ECD method in which iprodione, its isomer, and its metabolite are derivatized to a common moiety. The results of the plum processing study are presented in Table 1. Apparent iprodione residues of concern were nondetectable (<0.05 ppm) in/on one sample each of untreated plums, washed plums, and prunes.

The submitted data indicate that the combined residues of iprodione, its isomer, and its metabolite concentrated up to 6.6x in prunes processed from plums bearing detectable residues. The average concentration factor was approximately 4x. CBRS notes that the maximal theoretical concentration factor for prunes is 4x (see Follow-up Guidance For Maximum Theoretical Concentration Factors, EPA 737-R-93-001).

Table 1. Iprodione residues of concern found in/on plums and plum processed commodities following treatment at 2x the maximum seasonal rate.

Commodity	Number of samples	Residues (ppm iprodione equivalents)			Concentration/reduction factor		
Plums	3	0.41	0.93	0.99	--		
Washed plums	3	0.58	1.1	0.58	1.4	1.2	0.6
Prunes	3	2.7	3.7	2.5	6.6	4.0	2.5

Residue Analytical Methods

Plum commodity samples from the submitted processing study were analyzed for residues of iprodione, its isomer, and its metabolite using a GC/electron capture detection (ECD) method in which the analytes are hydrolyzed to 3,5-dichloroaniline and derivatized prior to analysis. The limit of quantitation was 0.05 ppm; the registrant estimated the limit of detection to be 0.004 ppm. The method included in this submission is entitled "Method for the Analysis of Rovral®-Related Residues: Common Moiety Method (Version 3.0 for Plums/Prunes)."

In this method, plum commodity samples which had been ground with dry ice were combined with 3 N aqueous KOH and heated at 100 C overnight to hydrolyze iprodione residues of concern to 3,5-dichloroaniline (DCA). The hydrolyzed samples were combined with water and distilled; the distillate was mixed with water and partitioned three times with dichloromethane. Following phase separation, the dichloromethane fractions were combined and DCA residues were derivatized to N-(3,5-dichlorophenyl)-2-chloropropylamide (DCPA) by the addition of 2-chloropropionyl chloride. The extract was rotary-evaporated to dryness, taken up in cyclohexane, and evaporated to dryness again. The dried residue was dissolved in 6% diethyl ether in hexane and purified on a Florisil column. DCPA was eluted from the column with 15% diethyl ether in hexane. The eluant was rotary-evaporated to dryness, dissolved in ethyl acetate, and analyzed by GC/ECD.

Method validation and concurrent method recoveries were conducted by Horizon Laboratories to determine the suitability of this method for residue data collection purposes. For method validation, samples of untreated prunes were each fortified with iprodione, its isomer (RP-30228), and its metabolite (RP-32490) at 0.05 ppm, 0.50 ppm, and 5.00 ppm. Apparent residues in/on unfortified samples were not included in the submission. For concurrent method recovery, samples of untreated plums, washed plums, and prunes were fortified with iprodione *per se*. Representative chromatograms, sample calculations, and standard curves were provided. Method validation and concurrent method recovery data are presented in Table 2. The submitted recovery data indicate that the GC/ECD method is adequate for data collection of iprodione residues of concern in/on plums and plum processed commodities. The registrant stated that because the method validation data for prunes indicated that there were no compound-related effects on recovery, concurrent method recoveries were only conducted for the parent iprodione.

Table 2. Method validation and concurrent method recovery of iprodione, its isomer (RP-30228), and its metabolite (RP-32490) from untreated samples of plum commodities fortified with each analyte and analyzed by GC/ECD.

Commodity	Fortification level (ppm)	% Recovery ^a		
		Iprodione	RP-30228	RP-32490
Method validation				
Prunes	0.05	93.7, 94.9	92.5, 100.8	104.1, 106.0
Prunes	0.50	97.5, 100.8	94.9, 96.8	100.7
Prunes	5.00	96.1, 99.8	91.6, 99.8	96.0, 98.2
Concurrent method recovery				
Plums	5.00	100.4	-- ^b	--
Washed plums	5.00	102.3	--	--
Prunes	0.05	94.6	--	--

^a Each recovery value represents one sample.

^b Concurrent method recovery was only conducted for the parent iprodione.

Storage stability data

Plum samples were harvested by hand, placed in boxes, and shipped to the processing facility the day of harvest at ambient temperature. The samples were received at the processing facility and were refrigerated (0-4 C) until processing was initiated the next day. After processing was completed, samples were stored frozen at -26 to -9 C for 12-17 days and then shipped frozen via ACDS freezer truck to Rhone-Poulenc (Research Triangle Park, NC) where they were stored frozen (<-10 C) for ~3.5 months. Samples were then shipped to Horizon Laboratories (Columbia, MO) where they were stored frozen at -20 to -10 C prior to analysis. The maximum storage interval prior to residue analysis of commodities collected from the respective field trials was 541 days (~18 months). Data depicting the frozen storage stability of iprodione, its isomer, and its metabolite in/on related commodities are currently under review (MRID 43273401, CBRS No. 14162, D206161). These data indicate that iprodione residues of concern are stable for up to 12 months in/on peaches and raisins.

IPRODIONE - Residues in Plums and Its Processed Commodities Formulation = 4 lb/gal FIC

MFLD	Year	Loc.	Number of Samples	Crop	RAC	PTI, days	Application			Retreatment Intervals, days	Total lb a/A	Residues, ppm Iprodione	Conc. factor	Chemical	Fortification ppm	% Recovery	Storage Interval, days	Temp., C	Sample Number	Harvest Date	Analysis Date
							Number	Method	Equip.												
43255702	92	WA	1	plums	plums	N/A	N/A	N/A	N/A	N/A	N/A	<0.05	--	Iprodione	5.00	100.4	540	<-7	5764-01	08/24/92	02/15/94
43255702	92	WA	1	plums	plums	0	5	follar	ground	100	5-96	0.41	--				540	<-7	5768-01	08/24/92	02/15/94
43255702	92	WA	1	plums	plums	0	5	follar	ground	100	5-96	0.99	--				540	<-7	5769-01	08/24/92	02/15/94
43255702	92	WA	1	plums	plums	0	5	follar	ground	100	5-96	0.93	--				540	<-7	5770-01	08/24/92	02/15/94
43255702	92	WA	1	plums	washed plums	N/A	N/A	N/A	N/A	N/A	N/A	<0.05	--	Iprodione	5.00	102.3	541	<-7	5764-11	08/24/92	02/16/94
43255702	92	WA	1	plums	washed plums	0	5	follar	ground	100	5-96	0.98	1.4				541	<-7	5768-11	08/24/92	02/16/94
43255702	92	WA	1	plums	washed plums	0	5	follar	ground	100	5-96	0.99	0.6				541	<-7	5769-11	08/24/92	02/16/94
43255702	92	WA	1	plums	washed plums	0	5	follar	ground	100	5-96	1.1	1.2				541	<-7	5770-11	08/24/92	02/16/94
43255702	92	WA	1	plums	prunes	N/A	N/A	N/A	N/A	N/A	N/A	<0.05	--	Iprodione	0.05	94.6	540	<-7	5764-09	08/24/92	02/16/94
43255702	92	WA	1	plums	prunes	0	5	follar	ground	100	5-96	2.7	6.6				540	<-7	5768-09	08/24/92	02/16/94
43255702	92	WA	1	plums	prunes	0	5	follar	ground	100	5-96	2.5	2.5				540	<-7	5769-09	08/24/92	02/16/94
43255702	92	WA	1	plums	prunes	0	5	follar	ground	100	5-96	3.7	4.0				540	<-7	5770-09	08/24/92	02/16/94

NOTES: Year = year trial was conducted
 N/A = Not applicable.
 Residue values are not corrected for concurrent method recoveries or residues in untreated samples.