

Task-1R: Review of PYDRIN

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A 30, 60, and 120-day rotation crop study using carbon-14 labeled chlorophenyl- and phenoxyphenyl-SD 43775, Fan, H.Y., and P.W. Lee, Snell Chemical Company, May 1980, TIR-22-004-80, Acc. #242588, Tab 1.

Procedure

This rotational crop study was conducted with Hanford sandy loam soil samples (57.6% sand; 26.6% silt; 15.8% clay; 0.7% organic carbon; CEC 9.3 meq/100 g; and pH 6.1), fortified with either [^{14}C]chlorophenyl- or [^{14}C]phenoxyphenyl-Pydrin (SD 43775) at 0.224 ppm (equivalent to a Pydrin dosage rate of approximately 0.25 lb ai/A). At 30, 60, and 120 days posttreatment, soils were planted with seeds of 'Cajeme 71' wheat, 'Detroit red' table beets, or 'Prizehead' leaf lettuce. (No seeds were planted in the 120-day [^{14}C]phenoxyphenyl-Pydrin-treated soil). Wheat, beets, and lettuce were also planted in untreated (control) soils. Soil core samples were taken immediately after application and at each planting.

Mature lettuce plants were harvested close to the ground, cleaned, and homogenized. Mature beets were separated into aerial and root portions and homogenized. Wheat stalks were cut approximately 4 inches above the ground and divided into straw and heads. Straw was homogenized, and wheat heads were separated into grain and bran before homogenization of each component. Immature lettuce, beets, and wheat plants were also harvested at various intervals after germination and analyzed for total ^{14}C residues.

The study was conducted in an outdoor plant growth enclosure area at Modesto, California. Plants were fertilized (10-10-10 fertilizer) and watered ($\frac{1}{4}$ - $\frac{1}{2}$ inch every other day) during the study.

Methodology

Soil core samples were air dried, sieved (20 mesh), and analyzed for ^{14}C activity by combustion and liquid scintillation counting (LSC). The distribution of radioactivity in the soil (extractable versus unextractable) and nature of the degradation products were investigated. Soil samples were extracted with methanol, the methanol extracts were concentrated, and the volume was adjusted with saturated sodium chloride solution. This aqueous solution mixture was then extracted with chloroform, and the chloroform extract was dried, concentrated, and analyzed by two-dimensional thin-layer chromatography (TLC) for comparison against known standards. Radioactivity remaining in the soil following the methanol extractions was determined by combustion and LSC.

Plant residues were analyzed for ^{14}C residues by combustion and LSC. Wheat straw samples that showed the highest levels of ^{14}C residues were further analyzed to determine the nature and distribution of ^{14}C activity.

Studies were conducted to determine total extractable ^{14}C residues, organoextractable residues, water-soluble conjugates, and unextractable residues.

Results

The limits of detection for ^{14}C residues in soil and plant tissue samples (expressed as Pydrin equivalent) were 0.018 and 0.008-0.009 ppm, respectively. Plant and soil residue data are summarized in Table 1. TLC analysis of soil residues showed the presence of SD 48838 (benzeneacetic acid, 4-chloro- α -(1-methylethyl)-,cyano-(3-phenoxy-4-hydroxyphenyl)-methyl ester) and SD 47117 (benzeneacetic acid, 4-chloro- α -(1-methylethyl)-(aminocarbonyl)-(3-phenoxyphenyl)methyl ester) in both [^{14}C]chlorophenyl- and [^{14}C]phenoxyphenyl-Pydrin-treated soils. SD 44064 (benzeneacetic acid, 4-chloro- α -(1-methylethyl)) was found in the [^{14}C]chlorophenyl-Pydrin-treated soil. However, none of these metabolites represented more than 10% of the applied radioactivity (Table 2). Unextractable (bound) residues reached a maximum of 8% of the applied [^{14}C]chlorophenyl-Pydrin activity (at 60 days) and 12% of the applied [^{14}C]phenoxyphenyl-Pydrin activity (also at 60 days).

No ^{14}C residues were detected in lettuce plants during the study, and no ^{14}C residues were detected in beets (tops and roots) or wheat (straw, bran, and grain) grown in [^{14}C]phenoxyphenyl-Pydrin-treated soil. Residues were detected in beet tops (0.018 ppm maximum), beet roots (0.024 ppm maximum), wheat straw (0.061 ppm maximum), wheat bran (0.022 ppm maximum), and wheat grain (0.021 ppm maximum) grown in [^{14}C]chlorophenyl-Pydrin-treated soil (Table 1).

Studies to determine the nature of ^{14}C residues in wheat straw showed that approximately 68% of the activity was extractable with aqueous acetone (59% water-soluble conjugates and ~8.6% organosoluble). TLC analysis of the chloroform extract of water-soluble conjugates showed SD 44064 as the major metabolite. Almost all of the unextractable radioactivity was associated with the cellulose fraction of the straw. Residues in other plants (or plant parts) were not further analyzed due to the low levels of activity found.

Analysis of immature plants grown in [^{14}C]chlorophenyl-Pydrin-treated soil from the 30- and 60-day planting intervals showed that total ^{14}C residues were either below the limit of detection (lettuce) or were below those levels found in plants harvested at maturity.

Table 1. Distribution of the total [^{14}C]Pydrin equivalent residues in the soil, mature lettuce, beet, and wheat samples from the various planting intervals in the [^{14}C]chlorophenyl-Pydrin (I) and [^{14}C]-phenoxyphenyl-Pydrin (II) rotation crop study.

Sample	[^{14}C]Pydrin equivalent residues (ppm)				
	30-Day		60-Day		120-Day
	I	II	I	II	I
Soil ^a	0.093 ^b	0.109	0.107	0.076	0.060
Lettuce	ND ^c	ND	ND	ND	ND
Beet (Top)	0.015	ND	0.018	ND	0.080
(Root)	0.024	ND	0.015	ND	ND
Wheat (Straw)	0.013	ND	0.061	ND	0.032
(Bran)	ND	ND	0.022	ND	0.009
(Grain)	ND	ND	0.021	ND	0.009

^a The initial concentration of Pydrin in the soil was 0.224 ± 0.033 ppm.

^b Average of five replications.

^c Residue level was below the limit of detection.

Table 2. Distribution of the applied radioactivity in the methanol-extractable and unextractable fractions from Hanford sandy loam soil at various time intervals after application of [^{14}C]chlorophenyl and [^{14}C]phenoxyphenyl-Pydrin.

Sample	Percent applied radioactivity				
	[^{14}C]chlorophenyl-Pydrin			[^{14}C]phenoxyphenyl-Pydrin	
	30-Day	60-Day	120-Day	30-Day	60-Day
Methanol-extractable	35.2	39.8	20.4	37.1	21.9
SD 43775 ^a	29.3	27.3	15.4	31.9	20.1
SD 44064 ^a	1.0	9.2	1.1	--	--
SD 48838 ^a	0.9	0.4	1.1	1.2	0.3
SD 47117 ^a	2.0	2.1	1.4	1.9	1.0
Other ^b	2.0	0.8	1.4	2.1	0.5
Unextractable	6.3	8.0	6.4	11.6	12.0
Total	41.5	47.8	26.8	48.7	33.9

^a See text for chemical names.

^b Including radioactivity remaining in the aqueous phase after chloroform extraction, and radioactivity associated with the origin of TLC plate and minor metabolites.

Conclusions

Uptake of ^{14}C residues occurs in beets (top and root) and wheat (straw, bran, and grain) planted 30, 60, and 120 days after soil application of labeled Pydrin at a recommended rate (0.25 lb/A). The greatest uptake of ^{14}C occurs in wheat straw where 0.061 ppm [^{14}C]Pydrin equivalent is found in the mature crop planted 60 days after soil treatment, identified as SD 44064, the chlorophenyl acid moiety of Pydrin. Residues in lettuce remain below the limit of detection (<0.008 ppm). Levels of ^{14}C residues in soil decline to approximately 27% of the applied activity at the 120-day planting time; three soil metabolites are present, but none exceed 10% of the applied activity.

1979-Residue data for SD 43775 in rotational crops (wheat, radishes, and potato foliage) following various applications of SD 43775 to cotton and potatoes, an Arizona study, Barber, G.F., Shell Chemical Company, May 1980, TIR-22-167-79, Acc. #242588, Tab 2.

Procedure

Studies were conducted in which 'Produra' wheat, 'Globe' radishes, and 'Tennebec' potatoes were rotated on a sandy loam soil (organic matter <1.0%) following either 10 or 15 aerial applications of Pydrin (0.2 lb ai/A) to cotton. Radishes and wheat were planted 38 days after the last application to cotton, and potatoes were planted 80 days after the last application. Radishes, wheat, and potatoes were sampled 182, 224, and 217 days, respectively, after the last cotton treatment. Additional studies were conducted in which potato plants, rotated after 15 applications of Pydrin to cotton, were treated (ground spray) seven times with Pydrin (0.2 lb ai/A) and harvested 0 and 2 days after the last application.

Methodology

Green wheat, radishes (roots and tops), and potatoes (tubers and tops) were macerated and frozen until analysis. Samples were then extracted with hexane:IPA and analyzed for Pydrin residues by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in all plant samples was 0.01 ppm. Studies with fortified crop samples showed recoveries ranging from 78 to 112% at fortification levels ranging from 0.05 to 0.2 ppm.

Table 1 summarizes the Pydrin residue data from these rotational crop studies. Potato tubers, potato tops, wheat plants, and radish roots rotated after cotton receiving either 10 or 15 applications of Pydrin contained the compound at less than 0.01 ppm. Radish tops rotated after cotton treated with 10 applications of Pydrin showed residue levels of 0.01-0.02 ppm. Potato plants rotated after 15 applications of Pydrin to cotton and treated seven times with Pydrin showed no residues (<0.01 ppm) in tubers sampled 0 and 2 days after the last application. Potato tops, however, showed maximum residue levels of 3.4 ppm in samples taken zero days after the last treatment of potatoes rotated after 15 applications to cotton.

Table 1. Summary of Pydrin (SD 43775) residues in rotational crops.

Rotation crop sample	SD 43775 dosage (lb ai/A) each application	Number of applications		Interval, final treatment-to-sampling (days)	SD 43775 residue, (ppm)
		Cotton	Potatoes		
Potato tubers	None	-	-	-	<0.01
" "	0.2	10	7	0	<0.01
" "	0.2	15	7	0	<0.01
" "	0.2	10	7	2	<0.01
" "	0.2	15	7	2	<0.01
Potato tops	None	-	-	-	<0.01
" "	0.2	10	7	0	2.8
" "	0.2	15	7	0	3.4
" "	0.2	10	7	2	3.0
" "	0.2	15	7	2	3.0
Potato tubers	None	-	-	-	<0.01
" "	0.2	15	-	217	<0.01
" "	0.2	10	-	217	<0.01
Potato tops	None	-	-	-	<0.01
" "	0.2	15	-	217	<0.01
" "	0.2	10	-	217	<0.01
Wheat (whole)	None	-	-	-	<0.01
" "	0.2	15	-	224	<0.01
Radish roots	None	-	-	-	<0.01
" "	0.2	15	-	182	<0.01
Radish tops	None	-	-	-	<0.01
" "	0.2	10	-	182	0.02

Conclusions

Pydrin residues are not found (<0.01 ppm) in potatoes (tops or tubers), whole wheat, or radish roots rotated on a sandy loam soil following 10 or 15 applications of Pydrin to cotton at recommended rates (0.2 lb ai/A). Low residue levels (0.01-0.02 ppm) are found in radish tops. The highest levels of Pydrin residue (2.8-3.4 ppm) occur in potato tops (rotated after 10 or 15 applications of Pydrin to cotton) receiving seven direct applications and sampled within 0-2 days. Under the same conditions, however, no Pydrin residues (<0.01 ppm) are found in potato tubers.

1979-Residue data for SD 43775 in soil following various applications to cotton and potatoes, an Arizona study, Barber, G.F., Shell Chemical Company, May 1980, TIR-24-140-79-B, Acc. #242588, Tab 3.

Procedure

This study presents data on Pydrin (SD 43775) residues in soil following application to cotton and potatoes as described in report TIR-22-167-79 (previously reviewed in the data package in Tab 2). Soil core samples were taken to a depth of 12 inches 30, 80, 131, and 172 days after the last of either 10 or 15 applications of Pydrin to cotton (0.2 lb ai/A), and 0 days after the last of seven applications to potatoes (which had been rotated to the cotton plots following harvest). These sampling times approximate the planting and harvest dates of the rotational crops (see previous review of Tab 2).

Methodology

Soil core samples were cut into 4-inch sections (0-4, 4-8, and 8-12 inch depths) and frozen until analysis. Samples were screened (8 mesh) to remove stones and debris and extracted with acetone:hexane for analysis of Pydrin residue by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in soil samples was 0.01 ppm. Studies with fortified soil samples showed Pydrin recoveries ranging from 96 to 107% at fortification levels of 0.05-0.10 ppm.

Soil residue data are summarized in Table 1. The highest Pydrin residue level (0.10 ppm) was found in the 0-4 inch soil sample taken 0 days after the last of seven applications of Pydrin to potatoes, following 10 previous applications to cotton. Residue levels at the 4-8 and 8-12 inch depths were generally below the level of detection (<0.01 ppm).

Table 1. Summary of Pydrin residues in soil following application to cotton and potatoes.

Number of applications ^a	Final treatment-to-sampling interval (days)	Pydrin residues (ppm)		
		0-4 in.	4-8 in.	8-12 in.
10	30	0.02	ND ^b	ND
10	80	0.07	0.03	ND
10	172	0.01	ND	0.02
10+7 ^c	0	0.10	0.01	0.02
15	30	0.04	ND	ND
15	30	0.02	ND	ND
15	80	0.02	ND	ND
15	172	0.01	ND	ND
15+7 ^c	0	0.06	ND	ND

^a 0.2 lb ai/A

^b Not detected (<0.01 ppm).

^c Following the harvest of treated cotton, potatoes were planted in the same plots and subsequently received seven applications of Pydrin.

Note: Residue levels in control plots (untreated) were below the level of detection in all samples, with the exception of a 0.02 ppm finding in one 0-4 inch sample.

Conclusions

Pydrin does not accumulate in a sandy loam soil following repeated (10 or 15) applications (0.2 lb ai/A) to cotton, even when subsequently rotated to potatoes that receive additional Pydrin treatments (seven applications, 0.2 lb ai/A).

1979-Residue data for SD 43775 in rotation crops (soybeans and peanuts) following various applications of SD 43775 to cotton, soybeans and peanuts, a Louisiana study, Barber, G.F., Shell Chemical Company, May 1980, TIR-24-278-79, Acc #242588, Tab 4.

Procedure

'Bragg' soybeans and peanuts were planted in Louisiana in a silt loam soil (35% fine sand; 57% silt; 5.3% clay) 237 days after the last application of Pydrin (SD 43775; 2.4 EC) on cotton. The cotton was treated with 10 or 15 applications at 0.2 lb ai/A during 1978. Soybeans and peanuts in rotation with cotton were left untreated or were treated with four and three applications (soybeans and peanuts, respectively), at 0.2 lb ai/A in 1979. Plant samples were collected at maturity in peanuts and the green pod stage of soybeans, 323 days after the last pesticide application on cotton and 21 days after the last application on soybeans or peanuts. Samples were stored frozen until analysis.

Methodology

Samples were extracted in hexane:IPA and filtered. Extracts were partitioned with water to remove the IPA and with CH_3CN to remove the hexane portion, and CH_3CN was then exchanged for hexane. The resulting solution was passed through an activated Florisil column, and the Pydrin was eluted with hexane:ethyl acetate. A gas-liquid chromatograph equipped with a ^{63}Ni electron capture detector was used to analyze the samples.

Results

Results are shown in Table 1. Analytical recovery studies showed recoveries of 73-108% with a mean of 94% in crops spiked with Pydrin at 0.05-1.0 ppm. The control plot residues were higher than the treated plot residues, indicating that samples of soybeans and peanuts that had no additional pesticide in 1979 were contaminated. Samples that had been treated with additional pesticide in 1979 showed residues at 4.4-6.4 ppm and 0.8-8.3 ppm in soybean and peanut plants, respectively, with no detectable residues (<0.01 ppm) in either soybean seeds or peanut meats.

Table 1. Pydrin residues found in rotational crops. Application rate was 0.2 lb ai/A of 2.4 EC.

Sample	Number of applications			Interval, final application to sampling (days)	Residue (ppm)
	1978	1979			
	Cotton	Soybeans	Peanuts		
Soybean plants	--	--	--	323	0.24 ^a
"	10	--	--	323	0.04
"	15	--	--	323	0.12
"	--	4	--	21	5.8
"	10	4	--	21	6.4
"	15	4	--	21	4.4
Soybean seeds	--	4	--	21	ND ^b
"	10	4	--	21	ND
"	15	4	--	21	ND
Peanut plants	--	--	--	323	0.07 ^a
"	10	--	--	323	0.03
"	15	--	--	323	0.06
"	--	--	3	21	0.8
"	10	--	3	21	2.0
"	15	--	3	21	8.3
Whole peanuts	--	--	3	21	ND
"	10	--	3	21	0.04
"	15	--	3	21	0.02
Peanut meats	--	--	3	21	ND
"	10	--	3	21	ND
"	15	--	3	21	ND

^a Samples appear to be contaminated. Re-extraction confirmed these results.

^b Not detectable, <0.01 ppm.

Conclusions

Rotational soybean and peanut plants that do not receive any direct application of Pydrin are not expected to accumulate residues at levels higher than 0.12 ppm if they are harvested around 300 days after the last Pydrin application (a total of 3 lb ai/A applied the previous year). Rotational treated green soybean seeds and mature peanut meats do not accumulate Pydrin (<0.01 ppm) if the last pesticide application is 20 days before harvest, but the whole plants do accumulate residues after the additional pesticide has been applied (mean values of 5.5 ppm in soybean plants and 3.7 ppm in peanut plants).

1979 - Residue data for SD 43775 in soil receiving 10 or 15 applications SD 43775 to cotton in 1978 followed by 3 or 4 applications to peanuts or soybeans in 1979, a Louisiana study, Barber, G. F., Shell Chemical Company May 1980, TIR-24-391-78-B, Acc #242588, Tab 5.

Procedure

This study presents data on Pydrin (SD 43775) residues in a silt loam soil following application to cotton, peanuts and soybeans as described in report TIR-24-278-79 (previously reviewed in this data package in Tab 4). The soil was sampled at three times: just prior to the 1979 planting; midseason, 237 days after the last application of Pydrin (cotton, 1978); and at harvest, 323 days after the last application of Pydrin to cotton (control) or 21 days after the last application to plants that were treated again in 1979. Samples were core increments of 0-3, 3-6, and 6-12 inches that were stored frozen until analysis was performed.

Methodology

The soil was screened through a 4-mm sieve and extracted twice with high-frequency vibration in the presence of acetone:hexane (1:1). The acetone was evaporated on a steam table, and the extract was washed with water and then purified on an activated Florisil column using hexane:ethyl acetate eluant. The eluate was analyzed by gas-liquid chromatography using a ⁶³Ni electron capture detector.

Results

The minimum detectable concentration of Pydrin in soil was 0.01 ppm. Studies with fortified soil samples showed recoveries ranging from 56 to 93% at fortification levels ranging from 0.05 to 0.1 ppm. Soil residues in the surface 3 inches ranged from 0.01 to 0.22 ppm with a mean value of 0.06 ppm. Experimental noise masked treatment effect. The 3-6 inch layer had lower residue levels ranging from <0.01 to 0.11 ppm with a mean value of 0.03 ppm. The 6-12 inch cores had levels <0.01 ppm. The data are shown in Table 1.

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Table 1. Pydrin residues in a silt loam soil in Louisiana after multiple application at 0.2 lb ai/A.

Number of applications			Interval, final application to sampling (days)	Residue (ppm)	
1978	1979			0-3 in.	3-6 in.
Cotton	Peanuts	Soybeans			
--	--	--	--	ND	ND
10	--	--	323	0.03	0.02
15	--	--	323	0.03	0.01
10	--	--	237	0.22	0.06
15	--	--	237	0.05	0.11
--	3	--	21	0.02	ND ^a
10	3	--	21	0.13	0.02
15	3	--	21	0.10	0.04
--	--	4	21	0.04	ND
10	--	4	21	0.04	0.01
15	--	4	21	0.07	0.02

^aNot detectable, <0.01.

Conclusions

No particular pattern existed to show the effect of treatment, but residue levels of approximately 0.06 ppm could be expected in a silt loam soil from 21 to 323 days after multiple Pydrin applications. There is no evidence of Pydrin leaching in this soil.

1979 - Residue data for SD 43775 in rotational crops (green wheat) following 10 or 15 applications of SD 43775 to cotton the previous year, an Oklahoma study, Barber, G. F., Shell Chemical Company, May 1980, TIR-24-209-79, Acc # 242588, Tab 6.

Procedure

Studies were conducted in which 'Tam-101' wheat was rotated on a clay loam (0.5% organic matter; other characteristics not given) following either 10 or 15 applications of Pydrin at 0.2 lb ai/A to 'Lankart-57' cotton. Wheat was planted 39 days after the last application to cotton, and green wheat samples were collected for analysis of Pydrin residues 135, 150, and 169 days after the last application to cotton corresponding to 2-4 inch, 4-8 inch, and headed wheat, respectively. Studies were conducted at Hobart, Oklahoma, in 1979.

Methodology

Wheat samples (frozen during storage) were macerated whole, extracted with hexane:IPA, and analyzed for Pydrin residues by gas-liquid chromatography (GLC) with an electron capture detector.

Reported Results

The minimum detectable concentration of Pydrin residues in green wheat samples was 0.01 ppm. Studies with fortified wheat samples showed recoveries ranging from 76 to 81% at a fortification level of 0.05 ppm. Residue levels in green wheat at all sampling intervals and after 0 (control), 10, and 15 applications of Pydrin were reported as 0.01 ppm (minimum detectable concentration).

Conclusions

Pydrin residues do not exceed the minimum detectable concentration (0.01 ppm) in green wheat rotated on a clay loam soil following 10 or 15 applications of Pydrin to cotton at 0.2 lb ai/A.

1979 - Residue data for SD 43775 in rotational crops (potatoes) following various applications of SD 43775 to cotton and potatoes, an Oklahoma study, Barber, G. F., Shell Chemical Company, May 1980, TIR-24-209-79-B, Acc # 242588, Tab 7.

Procedure

Studies were conducted in which Irish potatoes were rotated on a clay loam (1.5% organic matter) following either 10 or 15 applications of Pydrin (0.2 lb ai/A) to cotton. Potatoes were planted 202 days after the last application to cotton and received seven applications of Pydrin from 52 to 86 days after planting. Studies were also conducted in which rotated potato plants remained untreated or were rotated and treated without previous application of Pydrin to cotton.

Methodology

Potato vines, roots, and tubers were macerated and extracted with hexane: IPA. Pydrin residues were analyzed by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in potato tubers, peels, vines, and roots was reported as <0.01 ppm. Studies with fortified samples of tubers, peels, vines, and roots showed recoveries ranging from 72 to 86% at fortification levels ranging from 0.05 to 0.2 ppm. Whole tubers, peeled tubers, potato vines, and roots showed Pydrin residue levels <0.01 ppm under all conditions. The only samples showing Pydrin residue levels >0.01 ppm were peels from potatoes sampled 7 days after the last of seven Pydrin applications. Values of 0.015 and 0.014 were reported in these peel samples when potatoes were rotated after 15 or 0 applications to cotton, respectively. Potatoes rotated after 10 applications to cotton and subsequently treated seven times showed peel residue levels <0.01 ppm.

Conclusions

Pydrin residues do not accumulate in potatoes rotated following repeated applications of Pydrin (10 or 15) to cotton (0.2 lb ai/A), and receiving seven additional applications of Pydrin prior to sampling. The highest Pydrin residue levels (0.015 ppm) occur in potato peels, but residue levels in whole tubers, peeled tubers, potato vines, and roots are otherwise less than 0.01 ppm.

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1979 - Residue data for SD 43775 in soil receiving seven ground spray applications of SD 43775 to potatoes following ten or fifteen applications the previous year to cotton, an Oklahoma study, Huston, C. K., Shell Chemical Company, May 1980, TIR-24-381-78-C, Acc # 242588, Tab 8.

Procedure

This study presents data on Pydrin (SD 43775) residues in a clay loam soil following application to cotton and potatoes as described in report TIR-24-209-79-B (previously reviewed in this data package in Tab 7). Soil core samples (0-3, 3-6, and 6-12 inch depths) were taken 6 days after the last of seven Pydrin applications (0.2 lb ai/A) to potatoes, which had been rotated following either 10 or 15 applications to cotton. The study was conducted at Hobart, Oklahoma.

Methodology

Soil samples were screened to remove stones and plant materials and were extracted with acetone:hexane. Pydrin residues were then determined by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in soil samples was 0.01 ppm. Studies with fortified soil samples showed recoveries ranging from 90 to 104% at fortification levels of 0.05-0.1 ppm.

No Pydrin residues (<0.01 ppm) were detected in the 3-6 and 6-12 inch soil samples. The highest residue level (0.12 ppm) was found in the 0-3 inch sample, collected 6 days after the last of seven application to potatoes which had been rotated following 15 applications to cotton (Table 1).

Table 1. Summary of Pydrin residues in a clay loam soil following application to potatoes rotated after cotton.

No. of applications		Soil sample	Pydrin residue (ppm) ^c
<u>1978^a</u>	<u>1979^b</u>		
0	0	0-3"	ND ^d
0	0	6-12"	ND
10	7	0-3"	0.07
10	7	3-6"	ND
10	7	6-12"	ND
15	7	0-3"	0.12
15	7	3-6"	ND
15	7	6-12"	ND
0	7	0-3"	0.08
0	7	3-6"	ND
0	7	6-12"	ND

^a0.2 lb ai/A to cotton during each application.

^b0.2 lb ai/A to potatoes during each application; potatoes rotated after cotton.

^cSamples taken 6 days after the last application to potatoes.

^dNot detectable, <0.01 ppm.

Conclusions

Pydrin does not accumulate (<0.01 ppm) in the 3-12 inch depths of a clay loam soil following repeated (10 or 15) applications (0.2 lb ai/A) to cotton, and subsequent rotation to potatoes that receive seven additional Pydrin treatments (0.2 lb ai/A). Higher residue levels (maximum 0.12 ppm reported) are found in the 0-3 inch soil layer, but the experimental design does not allow any conclusion with respect to the pattern of residue increases and/or declines in this soil segment.

1979-Residue data for SD 43775 in rotation crops (wheat, table beets, sugar beets, onions, carrots, and lettuce) following ten applications of SD 43775 to cotton the previous year, a California study, Barber, G.F., Shell Chemical Company, May 1980, TIR-24-149-79, Acc. #242588, Tab 9.

Procedure

Studies were conducted in which 'Mexicalli' wheat, 'Dark red' table beets, 'USH-10' sugar beets, 'Dessex' onions, carrots, and 'Climax' lettuce were rotated on a Holtville sandy loam (1.0% organic matter) following 10 applications of Pydrin at 0.2 lb ai/A to cotton. Crops were rotated 30 and 60 days after the final treatment to cotton and sampled for residue analysis 125 or 126 days after the final treatment to cotton. At sampling, wheat and onions were immature, but beets, carrots, and lettuce were market size. The study was conducted at Brawley, California, during 1978 and 1979.

Methodology

Samples were macerated and extracted with hexane:IPA. Pydrin residues were determined by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin residues in the crop samples was 0.01 ppm. Studies with fortified crop samples showed recoveries ranging from 79 to 112% at a fortification level of 0.05 ppm. Pydrin residues in wheat heads, beet (table and sugar) tops and roots, onions, carrot tops and roots, and lettuce heads were below the level of detection (<0.01 ppm) in all samples.

Conclusions

Pydrin residues are not found (<0.01 ppm) in wheat, table and sugar beets, onions, carrots, or lettuce rotated in a sandy loam soil following 10 applications of Pydrin to cotton at 0.2 lb ai/A (last application 30 or 60 days prior to vegetable planting).

1978-Residue data for SD 43775 in rotation crops (soybeans, sorghum, and sugar beets) following eight applications of SD 43775 the previous year to cotton, a California study, Huston, C.K., Shell Chemical Company, May 1980, TIR-24-266-78-B, Acc. #242588, Tab 11.

Procedure

Three rotational crops, 'Williams' soybeans, 'Pioneer 28' sorghum, and 'USH-10' sugar beets were planted in a fine sandy loam (organic matter <1%) following cotton. The previous year, Pydrin (SD 43775; 2:4 EC) had been applied eight times at 0.2 and 0.4 lb ai/A to the cotton during August and September. The rotational crops were planted 250-253 days and harvested 370 days after the final Pydrin treatment on the cotton. Soybeans were divided into pods and vines; sorghum, into heads and stalks; and sugar beets, into roots and tops. Samples were frozen at the laboratory before analysis.

Methodology

Samples were extracted with hexane:IPA in a Waring blender. Cleanup steps followed extraction, and the samples were analyzed by gas-liquid chromatography using a ^{63}Ni electron capture detector.

Results

No residues were detected (minimum detection concentration 0.01 ppm) in any of the samples.

Conclusions

Soybeans, sorghum, and sugar beets grown in fine sandy loam after Pydrin-treated cotton and harvested mature, 1 year after the last Pydrin application, contain the compound at less than 0.01 ppm.

Table 1. Pydrin residues in California sandy loam soil following eight applications on cotton.

Interval, final application to sampling (days)	Treatment ^a (lb ai/A)	Residue (ppm)		
		0-3 in.	3-6 in.	6-12 in.
--	--	ND ^b	ND	ND
49	0.2	0.28	0.04	ND
204	0.2	0.16	0.03	ND
258	0.2	0.02	0.01	0.01
370	0.2	ND	ND	ND
49	0.4	0.30	0.18	0.01
204	0.4	0.32	0.16	0.03
258	0.4	0.03	0.03	0.04
370	0.4	0.01	0.02	ND

^a Eight applications at the rate shown.

^b Not detectable, <0.01 ppm.

Conclusions

Pydrin residue levels of approximately 0.3 ppm can be expected in the surface 0-3 inches of a sandy loam soil 50 days after the last application, when 1.6-3.2 lb ai/A is the total amount applied. Residue levels will decline with time, though more slowly when higher application rates (0.5 lb ai/A) are used. Some leaching may occur, especially in soil that is treated at high application rates.

1978-Residue data for SD 43775 in rotation crops (carrots, onions, beets, and lettuce) following ten applications of SD 43775 to cotton, a Texas study, Barber, G.F., Shell Chemical Company, May 1980, TIR-24-401-78, Acc. #242588, Tab 13.

Procedure

Studies were conducted in which carrots, onions, beets, and lettuce were rotated on a sandy loam (58.8% sand; 23.6% silt; 17.6% clay; 1.6% organic matter) following 10 applications of Pydrin (SD 43775) to cotton. Rotational crops were planted 29 and 61 days after the 10th application of Pydrin (0.2 lb ai/A) to cotton. Lettuce plants were harvested 89 and 133 days after the last Pydrin application, carrots were harvested 134 and 201 days after the last application, and beets and onions were harvested 110 and 201 days, respectively, after the last application. Guthion was also applied during the study at 0.25 lb/A (9 times) and (0.5 lb/A) (15 times). Studies were conducted at Donna, Texas.

Methodology

Samples were frozen until analysis. Macerated samples were extracted with hexane:IPA, and Pydrin residues were determined by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in rotated crop samples was 0.01 ppm. Studies with fortified samples of lettuce, beets, carrots, and onions showed Pydrin recoveries ranging from 92 to 97% at fortification levels of 0.05 ppm Pydrin.

Pydrin residue levels in lettuce, table beets and beet tops, carrot roots and tops, and onions were below the limit of detection (0.01 ppm) in all samples.

Conclusions

Pydrin residues are not found (<0.01 ppm) in carrots, onions, beets, or lettuce rotated in a sandy loam soil following 10 applications of Pydrin to cotton at a recommended application rate (0.2 lb ai/A). Guthion was also applied during the study, but its effect on Pydrin residues in soil cannot be determined from the data presented, as controls (plots receiving no Guthion) apparently were not run.

1978-Residue data for SD 43775 in soil resulting from ten applications of SD 43775 to cotton, a Texas study, Nugent, K.D., Shell Chemical Company, May 1980, TIR-24-400-78, Acc. #242588, Tab 14.

Procedure

This study involved the analysis of Pydrin (SD 43775) soil residues following 10 applications of the insecticide to cotton, grown on a Hidalgo sandy clay loam soil (70% sand; 8% silt; 21.6% clay; 0.9% organic matter; pH 8.2). Pydrin was applied to cotton (blooming to mature stages) 10 times at 0.2 lb ai/A in the period from July 7, 1978, to August 23, 1978. Guthion at 0.25 lb ai/A (nine applications) and 0.5 lb ai/A (15 applications) was also applied. The study was conducted at Donna, Texas.

Soil core samples were taken to a depth of 12 inches 5, 29, 61, 89, 134, and 201 days after the last Pydrin application and were frozen until analysis.

Methodology

Soils were screened to remove stones and debris and were then extracted with acetone:hexane. Pydrin residues were determined by gas-liquid chromatography (GLC) with an electron capture detector.

Results

The minimum detectable concentration of Pydrin in soil was 0.01 ppm. Studies with fortified soil samples showed recoveries ranging from 78 to 91% at fortification levels ranging from 0.05 to 0.40 ppm.

Pydrin residues were not detected (<0.01 ppm) in soil samples taken below 3 inches at any treatment-to-sampling interval (ranging from 5 to 201 days). Pydrin residue levels of 0.28 and 0.03 ppm were, however, found in 0-6 inch samples taken 29 and 61 days, respectively, after the final treatment. Residue levels in 0-3 inch soil samples were highest (0.20 ppm) 5 days after the final Pydrin treatment, but declined to 0.01 ppm in samples taken after 201 days.

No data were provided on Guthion (which was also applied during this study) or its effect on Pydrin residues in soil.

Conclusions

Pydrin does not accumulate in a sandy clay loam soil following repeated (10) applications (0.2 lb ai/A) to cotton. Residue levels in the 0-3 inch soil layer decline as the interval from final treatment to sampling increases, with maximum residue levels of 0.20 ppm occurring at the 5 day interval and 0.01 ppm occurring at the 201 day interval. No residues are present (<0.01 ppm) in the 3-6 inch and 6-12 inch soil layers at any interval (although 0.28 ppm was found in a 0-6 inch soil segment at 29 days).

Guthion was also applied during the study, but its effect on Pydrin residues in soil cannot be determined from the data presented as controls (plots receiving Pydrin but no Guthion) were apparently not run.

Analysis - Pydrin Rotational Crop Studies

Laboratory studies show the occurrence of ^{14}C residues in mature beets (top and root) and wheat (straw, bran, and grain) seeded 30, 60, and 120 days after soil (sandy loam) application of ^{14}C labeled chlorophenyl-Pydrin at 0.25 lb ai/A. The greatest uptake of ^{14}C occurs in wheat straw where 0.061 ppm [^{14}C]Pydrin equivalent is found in the mature crop planted 60 days after soil treatment, identified as the chlorophenyl acid moiety of Pydrin. Residues in lettuce were below the limit of detection (<0.008 ppm) when grown in ^{14}C -labeled chlorophenyl Pydrin-treated soils. No ^{14}C residues were detected in beets, wheat, or lettuce grown in soil treated with ^{14}C labeled phenoxyphenyl Pydrin.

Levels of ^{14}C residues in the [^{14}C]chlorophenyl- and phenoxyphenyl-Pydrin-treated soils declined to 0.107 and 0.076 ppm (Pydrin equivalent), respectively, after 60 days, from initial concentrations of 0.224 ppm. Three soil metabolites are present, but none exceed more than 10% of the applied radioactivity.

Field studies show that Pydrin residues are not found (<0.01 ppm) in a variety of crops rotated following 10 or 15 applications of Pydrin to cotton at recommended rates (0.2 lb ai/A). An absence of residues under these conditions is reported in rotational potatoes, wheat, table and sugar beets, onions, carrots, lettuce, soybeans, and sorghum grown in a sandy loam soil after harvest of treated cotton. Residues are reported, however, in radish tops (0.01-0.02 ppm, sampled 182 days after the last of 10 applications to cotton) and in soybean (maximum 0.12 ppm, sampled 323 days after the last of 15 applications to cotton) and peanut plants (maximum 0.06 ppm, sampled 323 days after the last of 15 applications to cotton) rotated after cotton. Higher Pydrin residues occur when crops are rotated after harvest of treated cotton, and receive additional direct applications of Pydrin (0.2 lb ai/A). Under these conditions, Pydrin residues are found in potato tops (maximum 3.4 ppm, rotated after 15 applications to cotton and sampled immediately after the last of seven direct applications), in soybean (maximum 6.4 ppm, rotated after 10 applications to cotton and sampled 21 days after the last of 4 direct applications), and peanut plants (maximum 8.3 ppm, rotated after 15 applications to cotton and sampled 21 days after the last of 3 direct applications). No residues (<0.01 ppm), however, are found in soybean seeds and peanut meats under the same conditions, and no residues are found in tubers of rotated potatoes sampled immediately after the last of seven direct Pydrin applications, rotated after 15 applications to cotton.

Soil samples taken during rotational-crop studies show that Pydrin residues do not accumulate in soils following repeated applications to cotton and subsequently rotated to other crops which may receive additional Pydrin treatments. The highest residue reported, 0.32 ppm, occurred in the 0-3 inch depth of a sandy loam soil, sampled 204 days after the last of eight applications to cotton at twice (0.4 lb ai/A) the recommended application rate. Only 0.01 ppm was present in soil sampled 370 days after the last application. Pydrin residues below 3 inches in the soils studied (sandy loam, clay loam, silt loam, and sandy clay loam) are generally below 0.01 ppm (the limit of detection), with a maximum value of 0.18 ppm reported in the 3-6 inch segment of a sandy loam soil, 49 days after the last of eight applications to cotton at twice the recommended application rate. Residues decline to 0.02 ppm in a sample taken 370 days after the last application.