Residues of Ethoprop and Disulfoton in Soils and Flue-cured Tobacco*

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INTRODUCTION

Ethoprop [O-ethyl S,S-dipropyl phosphorodithioate] is a non-systemic, non-fumigant nematicide and soil insecticide which has been registered since 1967 for use on tobacco in the United States. Disulfoton [O,O-diethyl S(2-(ethylthio)ethyl) phosphorodithioate] is a systemic acaricide and insecticide used for soil application to protect tobacco plants from insect attack. This compound received a label for tobacco use in 1970. Disulfoton has been shown to persist in soils for at least 4 weeks (1), and the sulfone analog persists for more than 64 days (2). Disulfoton is metabolized by soil microorganisms to its sulfone and sulfoxide analogs, the rate depending on soil type, moisture, humidity and temperature. Disulfoton and the metabolites are absorbed and translocated in plants where further metabolism takes place (3). Few data are available in the literature on residues of ethoprop in either soils or flue-cured tobacco.

Bowman et al. (4) found that residues of disulfoton metabolites in flue-cured tobacco from the second harvest varied from 0.82 ppm for the oxygen analog sulfoxide to 23.7 ppm for the sulfone. U.S. flue-cured tobacco samples sent to the Federal Republic of Germany in 1973 and analyzed for various insecticides contained ethoprop residues of 0.01 and 0.03 ppm and total disulfoton residues of 0.4 and 0.9 ppm (5).

This study was conducted to determine residue levels of ethoprop and disulfoton in flue-cured tobacco grown in ethoprop and disulfoton-treated soil.

MATERIALS AND METHODS

Insecticide Application and Sampling

Experiments were established in 1976 on Norfolk sandy loam on the Central Crops Research Station near Clayton, N. C., and on a Cecil sandy loam on the Upper Piedmont Research Station near Reidsville, N. C. Cultural practices

suggested by the North Carolina Cooperative Extension Service were followed. Ethoprop⁺ was applied April 6 at Clayton and May 7 at Reidsville at rates of 6.72 and 13.4 kg a.i.⁺⁺/ha. A combination treatment of 6.72 kg a.i./ha of ethoprop plus 3.36 kg a.i./ha of disulfoton was included to represent a common agricultural practice. Disulfoton⁺ was applied broadcast on the same dates at rates of 3.4 and 6.7 kg a.i./ha, then incorporated with the disk set to cut about 12 to 15 cm deep. Plots were four rows wide and 14 m long. There were four border rows between plots. Rows were spaced 1.14 m. There were four replications. Speight variety G-28 was transplanted April 22 at Clayton and May 12 at Reidsville.

Soil samples were taken approximately 3 weeks after transplanting and monthly thereafter from each location. Sixteen cores (15 by 2.5 cm) were collected from the center 10 m of the middle two rows of each plot and were placed in a plastic bag. The middles between rows were avoided during sampling. The samples were packed in solid carbon dioxide, transported to the laboratory, and stored at -18 °C until analyzed.

Tobacco was harvested four times at Clayton and five times at Reidsville; the first harvest was taken approximately 12 weeks after transplanting at Clayton and 10 weeks after transplanting at Reidsville. The leaf samples were removed by hand at the normal harvest time from the same area in the plot as the soil samples. The tobacco was cured in conventional barns. After curing, the samples were placed in plastic bags and transported to the laboratory. Samples were ground in a Wiley mill and thoroughly mixed, and a 0.5-liter subsample was saved for analysis. Subsamples were stored at -18 °C.

Soil Analyses

For analysis of ethoprop, 20 g of sifted soil were weighed into a tared 475 ml glass jar and 75 ml of chloroform were added. The samples were shaken mechanically for 15 min. Extracts were filtered through 50 g of anhydrous sodium sulfate into 500 ml flasks. After an additional 75 ml of chloroform was added, the soil was extracted for an ad-

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+ Formulations of pesticides: ethoprop, Mocap 10 G; disulfoton, Di-Syston 15 G.

++ active ingredient.

ditional 15 min; and the suspension was filtered. The filter containing the soil was rinsed with 50 ml of chloroform, and the combined extract was evaporated under vacuum at 45 °C to 2 to 3 ml. Samples were brought to a 10 ml volume with ethyl acetate.

Twenty-five-gram subsamples of sifted soil from plots containing disulfoton were weighed into tared 475 ml glass jars, and 100 ml of ethyl acetate were added to each. The samples were shaken mechanically for 15 min. Extracts were filtered through 50 g of anhydrous sodium sulfate into 500 ml flasks. An additional 100 ml of ethyl acetate was added, and the soil was extracted for an additional 15 min. The suspension was filtered, and the filter containing the soil was rinsed with 50 ml of ethyl acetate. The combined extract was evaporated under vacuum at 45 °C to 2 to 3 ml and brought to a 10 ml volume with ethyl acetate.

Tobacco Analyses

Fifteen grams of ground tobacco containing ethoprop were weighed into a Soxhlet thimble and extracted 3 hours with 150 ml of hexane at 10 turnovers per hour. The hexane extract was evaporated to 5 to 8 ml under vacuum at 45 °C and transferred to a glass column (20 by 2.5 cm) containing 20 g of deactivated Florisil (5% water) topped with 2.5 cm layer of sodium sulfate. Fifty milliliters of hexane were added and allowed to drain; then 100 ml of ethyl acetate were added to the column to elute the ethoprop. The column eluate containing the ethoprop was evaporated to 1 to 2 ml in a rotary evaporator; the extract was diluted to 10 ml with ethyl acetate and analyzed by gas-liquid chromatography (GLC).

Ten grams of ground tobacco containing disulfoton were placed in a Soxhlet thimble and extracted 4 hours with 250 ml of ethyl acetate. The extract was cooled and poured directly into a glass column (20 by 2.5 cm) containing, from bottom to top, 5 cm of anhydrous sodium sulfate, 20 g of 5% deactivated Florisil, and another 5 cm of anhydrous sodium sulfate. The column was pre-rinsed with 50 ml of hexane. The eluate was concentrated to 2 to 3 ml under vacuum at 45 °C and diluted to 10 ml with ethyl acetate.

Gas Chromatography and Recovery

The gas chromatograph was either a Tracor MT 220 or Tracor Model 222 equipped with a flame photometric detector (FPD) operated in the phosphorus mode. The FPD on the Tracor Model 222 chromatograph has had the stainless steel base replaced by an aluminum bracket and utilizes a stainless steel capillary transfer line connected between the column exit port and the flame base. The two other flame photometric detectors in this laboratory were modified to reflect these changes. The column used for soil residues of ethoprop was 183.0 cm by 6.4 mm inside diameter U-shaped glass containing 4% SE-30 + 6% QF-1 on GasChrom Q (80/100). Because of an interference peak, a 183.0 cm by 6.4 mm inside diameter column packed with 5% Carbowax 20 M on GasChrom Q (60/80) was used for tobacco extracts. Temperatures were: oven,

180 °C for soil, 195 °C for tobacco; detector, 195 °C for soil and tobacco; and inlet port, 200 °C for both soil and tobacco. Nitrogen was the carrier gas, and flow rates were 120 ml/min for soils and 110 ml/min for tobacco. Gases to the detector were hydrogen and air at flow rates of 50 and 80 ml/min, respectively. Data were quantitated by the peak height method.

Disulfoton residues from soil and tobacco samples were determined on a 122.0 cm by 2.0 mm inside diameter U-shaped glass column containing 10% OV-101 on Supelcoport (80/100). Temperatures were: oven, 170 °C; detector, 190 °C; and inlet, 200 °C. Nitrogen was the carrier gas with a flow rate of 60 ml/min. Gases to the detector were hydrogen and air at flow rates of 200 and 100 ml/min, respectively.

The efficiency of the analytical method was determined by adding known amounts of ethoprop or disulfoton and its metabolites to untreated soil and tobacco and analyzing the fortified samples by the same method. Two fortified samples were analyzed with each set of 16 unknown samples. Residues of disulfoton and its metabolites were calculated by triangulation, and calibration curves of peak area versus the amounts of each component were constructed.

RESULTS AND DISCUSSION

Chromatograms of ethoprop in soil and tobacco were relatively free of interferences (Fig. 1). Figure 2 shows the mixed standard of disulfoton, its oxygen analog, the oxygen analog of disulfoton sulfoxide, and disulfoton sulfone. Typical chromatograms of these compounds in soil and tobacco samples are shown in Figures 3 and 4, respectively. It can be seen from these three figures that the longer retention times of the sulfoxide oxygen analog and sulfone metabolites made data calculation by triangulation a necessity.

Figure 1. Chromatograms of ethoprop in soil and tobacco.

- A. Ethoprop standard (containing 0.2 µg/ml) chromatographed on 4 % SE-30 + 6 % QF-1 column.
- B. Soil sample containing 1.03 ppm ethoprop (application rate: 13.4 kg a.i./ha; 1st sampling).
- C. Ethoprop standard (containing 0.2 µg/ml) chromatographed on 5.0 % Carbowax 20 M column.
- D. Tobacco sample containing 0.08 ppm (application rate: 13.4 kg a.i./ha; 1st harvest).

Figure 2. Chromatogram of analytical standards of disulfoton and three metabolites.

The average recoveries of ethoprop were 82 percent from soil and 85 percent from tobacco at spiking levels of 1 to 100 ppm in soil and 0.01 to 20 ppm in tobacco (Table 1). The lowest detectable limit was 0.01 ppm. Recoveries of disulfoton and 5 metabolites from untreated soil are shown in Table 2. Within each component the recoveries of varying amounts were consistent. The poorest recovery from soil was the disulfoton oxygen analog, averaging 66 percent from added amounts varying between 0.01 and 0.8 ppm. Disulfoton and the sulfone had the highest recoveries, averaging 86 and 84 percent, respectively. Table 3 shows recoveries of known amounts of disulfoton, and 5 metabolites from flue-cured tobacco. As was seen with the data from soil, recoveries of the oxygen analogs

Table 1. Recoveries of known amounts of ethoprop added to untreated soil or flue-cured tobacco*.

Sample type	Amount added (ppm)	Number of samples	Range (%)	Average recovery (%)
Soil	1	5	77-90	81
	5	4	83-94	91
	10	5	70-90	82
	25	2	78-81	80
	100	2	69-79	74
Tobacco	0.01	2	70-80	75
	0.05	2	84-88	86
	0.1	4	84-100	93
	0.2	1		92
	0.3	1		90
	0.5	1		67
	1.0	2	70-87	79
	1.5	1		85
	2.5	1		97
	3.0	2	78-85	82
	5.0	3	82-86	84
	10.0	3	69-87	79
	20.0	1		93

* Amounts shown were added to 20 g of untreated soil or 15 g of untreated tobacco. Recoveries based on equivalent amounts added to conical tubes and brought to 10 ml with ethyl acetate.

of disulfoton and disulfoton sulfoxide were lower than the other compounds. However, the average recoveries of all compounds were higher from untreated tobacco compared to untreated soil.

Ethoprop levels for the first sampling of soil taken about 30 days after application were approximately the same for corresponding treatments at the two locations (Table 4). The 13.4 kg/ha application rate averaged 1.13 ppm over locations, compared to 0.54 ppm for the 6.72 kg/ha rate. Data for subsequent samplings showed marked dif-

Figure 3. Chromatograms of three metabolites of disulfoton extracted from soil (application rate: 6.7 kg a.i./ha, 1st sampling).

Figure 4. Chromatogram of disulfoton and three metabolites extracted from flue-cured tobacco (application rate: 6.7 kg a.i./ha, 1st harvest).

Table 2. Recoveries of known amounts of disulfoton and five metabolites added to untreated soil*.

Compound	Amount added (ppm)	Number of samples	Range (%)	Average recovery (%)
Disulfoton	0.04	1		83
	0.05	2	89-98	94
	0.75	2	74-81	78
	1.00	2	86-87	87
	2.00	2	84-88	86
	3.00	2	89-93	91
	5.00	3	81-94	85
Disulfoton oxygen analog	0.01	4	65-88	71
	0.02	2	65	65
	0.05	2	64-65	65
	0.10	2	66-68	67
	0.25	2	63-64	64
	0.80	2	63	63
Disulfoton sulfoxide	0.01	4	80-92	84
	0.05	4	78-92	85
	0.10	4	78-84	82
	0.50	2	76-84	80
Disulfoton sulfoxide oxygen analog	0.01	4	70-78	74
	0.05	4	64-74	69
	0.10	4	69-73	71
	0.50	2	68-72	70
Disulfoton sulfone	0.04	4	80-86	83
	0.10	4	80-86	83
	0.25	2	84	84
	0.40	4	79-94	87
Disulfoton sulfone oxygen analog	0.04	4	78-81	80
	0.10	4	61-82	75
	0.25	2	78-82	80
	0.40	4	70-82	76

* Amounts shown were added to 25 g untreated soil. Recoveries based on equivalent amounts added to conical tubes and brought to 10 ml with ethyl acetate.

Table 3. Recoveries of known amounts of disulfoton and five metabolites added to untreated flue-cured tobacco*.

Compound	Amount added (ppm)	Number of samples	Range (%)	Average recovery (%)
Disulfoton	0.1	4	78-100	86
	0.25	4	82-95	89
	0.50	4	90-96	93
	0.75	4	82-84	84
	1.00	4	83-90	87
	1.50	4	81-91	85
Disulfoton oxygen analog	0.01	4	80-100	95
	0.025	4	50-88	72
	0.050	4	63-89	78
	0.10	4	92-97	94
	0.50	4	74-80	77
	1.00	4	76-88	82
Disulfoton sulfoxide	0.04	4	51-88	75
	0.08	4	83-99	90
	0.125	4	84-95	88
	0.25	6	84-92	87
Disulfoton sulfoxide oxygen analog	0.04	4	77-89	85
	0.08	4	56-78	66
	0.125	4	67-83	74
	0.25	6	69-87	76
Disulfoton sulfone	0.04	4	78-100	89
	0.025	4	79-91	85
	0.05	4	73-88	78
	0.10	6	83-99	89
Disulfoton sulfone oxygen analog	0.01	4	66-87	80
	0.025	4	80-88	84
	0.05	4	77-80	78
	0.10	6	75-85	80
	0.50	6	75-88	83

* Amounts shown were added to 10 g untreated flue-cured tobacco. Recoveries based on equivalent amounts added to conical tubes and brought to 10 ml with ethyl acetate.

Table 4. Ethoprop residues in soils taken from two locations.

Location	Rate (kg/ha)	Sampling*		
		first (ppm)	second (ppm)	third (ppm)
<i>Central Crops Research Station (Clayton, N.C.)</i>	0.0		0.01	0.01
	6.72	0.42	0.04	0.02
	13.4	0.96	0.09	0.04
	6.72 **	0.58	0.13	0.03
<i>Upper Piedmont Research Station (Reidsville, N.C.)</i>	0.0	0.04	0.02	0.02
	6.72	0.50	0.41	0.10
	13.4	1.30	0.74	0.23
	6.72 **	0.65	0.40	0.18

* Each value an average of four replications. Soils sampled May 10, June 16, and July 20, 1976 at Central Crops Research Station. Soils sampled June 14, July 16, and August 23, 1976 at Upper Piedmont Research Station. The untreated control plot was not sampled the first time.

** Combination treatment with disulfoton.

Table 5. Residues of disulfoton and three metabolites in soils from Central Crops (Clayton) and Upper Piedmont (Reidsville) Research Stations.

Disulfoton or metabolite	Rate (kg/ha)	Sampling*		
		first (ppm)	second (ppm)	third (ppm)
<i>Central Crops:</i>				
Disulfoton	3.4	0.02	0.02	0.02
	6.7	0.03	0.02	0.02
	3.4 **	0.02	0.01	< 0.01
Disulfoton oxygen analog	3.4	0.01	< 0.01	< 0.01
	6.7	< 0.01	< 0.01	< 0.01
	3.4 **	< 0.01	< 0.01	< 0.01
Disulfoton sulfoxide oxygen analog	3.4	0.03	< 0.01	< 0.01
	6.7	< 0.01	0.01	< 0.01
	3.4 **	< 0.01	< 0.01	< 0.01
Disulfoton sulfone	3.4	0.18	0.02	0.02
	6.7	0.34	0.11	0.11
	3.4 **	0.22	0.05	0.02
<i>Upper Piedmont:</i>				
Disulfoton	3.4	0.02	< 0.01	< 0.01
	6.7	0.02	< 0.01	< 0.01
	3.4 **	0.02	0.02	< 0.01
Disulfoton oxygen analog	3.4	< 0.01	< 0.01	< 0.01
	6.7	< 0.01	< 0.01	< 0.01
	3.4 **	< 0.01	< 0.01	< 0.01
Disulfoton sulfoxide oxygen analog	3.4	0.03	0.02	< 0.01
	6.7	0.02	< 0.01	< 0.01
	3.4 **	< 0.01	< 0.01	< 0.01
Disulfoton sulfone	3.4	0.26	0.13	0.08
	6.7	0.68	0.32	0.10
	3.4 **	0.19	0.07	0.03

* Each value an average of four replications. Residues of disulfoton sulfoxide and disulfoton sulfone oxygen analog were below the detectable limit of 0.01 ppm. Soils sampled May 10, June 16, and July 20, 1976 at Central Crops Research Station. Soils samples June 14, July 16, and August 23, 1976 at Upper Piedmont Research Station.

** Combination treatment with ethoprop.

ferences in ethoprop residues between locations. Residue levels declined gradually at the Upper Piedmont Research Station; the drop was more rapid at the other location and the concentration was near the low detectable limit at the last sampling.

Residues of disulfoton and the three metabolites found in soil from both locations are shown in Table 5. Small amounts (0.03 ppm or less) of disulfoton, its oxygen analog, and the sulfoxide oxygen analog were found in many of the samples from both locations. Residues of disulfoton sulfone were higher than those for the other three compounds and averaged 0.18 and 0.22 ppm, respectively, for the 3.4 kg/ha applied alone and in combination with ethoprop at the Central Crops Station. The average was 0.34 ppm for the 6.7 kg/ha rate. Corresponding residue levels for the Reidsville location were 0.26, 0.19, and 0.68 ppm. Total residues (disulfoton plus meta-

bolites) in soil were greater at the Upper Piedmont Station than from the Central Crops Station over the three sampling periods. These data are similar to those for ethoprop plots included in the same experiment (Table 3).

At Clayton 12.1 cm of rain fell in June and 1.1 cm during July compared to 13.0 cm in June and 7.7 cm (rainfall plus irrigation) in July at the Upper Piedmont Research Station. Moisture percentages were higher in soil from the Upper Piedmont Station than in that from the Central Crops Research Station at all three sampling times. At the second and third samplings, there was a 5 and 10fold difference, respectively. The difference in soil moisture level may have affected the persistence of the insecticides; however, most pesticides persist longer in dry soils than in moist soils where conditions are most favorable to microbial activity. Both insecticides persisted longest in soil at the Upper Piedmont Research Station where soil moisture was higher most of the growing season. Hence, other factors probably contributed to the difference.

In leaf samples from the first harvest at Reidsville, ethoprop residues were 0.20 and 0.27 ppm, respectively, for the 6.72 and 13.4 kg/ha rate (Table 6). Residues declined in each successive harvest, and in fifth-priming samples concentrations were less than 0.01 ppm. Levels of ethoprop in leaf samples from the Clayton test were much lower than those in samples from Reidsville. Values for the 6.72 and 13.4 kg/ha rate at Clayton averaged only 22 and 36%, respectively of the corresponding values for Reidsville.

Residues of ethoprop from the 6.72 kg/ha rate were less than 0.05 ppm when averaged over all samplings and locations. Therefore, average residues from ethoprop applied at the recommended rate approached the low limit of detection (0.01 ppm), and the pesticide probably would not be detectable in tobacco products.

Disulfoton residues varied widely among leaf samples from different locations, rates of application, and harvest dates. Residues from the 3.4 kg/ha rate applied alone or in combination with ethoprop were higher than those from the 6.7 kg/ha rate applied alone in several samplings (Table 7). We have no good explanation for this variation. Perhaps the broadcast application, the incorporation by disking, and the bedding operation produced uneven distribution in the beds and, hence, led to variation in the exposure of the tobacco roots to the chemical.

Residues of the metabolite, disulfoton sulfone, were higher than those for the parent compound, disulfoton, and the other metabolites, averaging 6.37 ppm in samples from Reidsville and 1.65 ppm in samples from Clayton for the first harvest at the 3.4 kg/ha rate. When total residues were compared between locations, those from the Upper Piedmont Station were highest. Our values for Reidsville samples were similar in magnitude to those obtained by Bowman et al. (4).

Total residues of disulfoton were found in much higher amounts in tobacco leaf than residues of ethoprop, suggesting that it, in comparison to ethoprop, is absorbed more readily by tobacco plants, is more persistent in plants, or is affected less during curing than ethoprop is.

Table 6. Ethoprop residues (at 13 % moisture) in flue-cured tobacco from two locations.

Location	Rate (kg/ha)	Harvest number *				
		1 (ppm)	2 (ppm)	3 (ppm)	4 (ppm)	5 (ppm)
<i>Central Crops Research Station (Clayton, N.C.)</i>	0.0	0.03	< 0.01	< 0.01	< 0.01	
	6.72	0.04	0.01	0.01	0.01	
	13.4	0.08	0.05	0.03	0.03	
	6.72**	0.02	0.02	0.02	0.02	
<i>Upper Piedmont Research Station (Reidsville, N.C.)</i>	0.0	0.09	0.06	0.04	0.02	< 0.01
	6.72	0.20	0.09	0.05	0.04	< 0.01
	13.4	0.27	0.17	0.06	0.04	< 0.01
	6.72	0.16	0.09	0.07	0.03	< 0.01

* Each value an average of four replications. Tobacco harvested July 15, July 23, August 5, and August 20, 1976 at Central Crops Research Station. Tobacco harvested July 22, August 10, August 23, September 8, and September 13, 1976 at Upper Piedmont Research Station.

** Combination treatment with disulfoton.

Table 7. Residues of disulfoton and three metabolites in flue-cured tobacco from Central Crops (Clayton) and Upper Piedmont (Reidsville) Research Stations.

Location	Disulfoton or metabolite	Rate (kg/ha)	Harvest number *				
			1 (ppm)	2 (ppm)	3 (ppm)	4 (ppm)	5 (ppm)
<i>Central Crops</i>	Disulfoton	3.4	0.04	0.02	0.06	0.02	
		6.7	0.04	0.02	< 0.01	< 0.01	
		3.4**	0.09	0.01	< 0.01	< 0.01	
	Disulfoton oxygen analog	3.4	0.04	0.03	0.07	0.02	
		6.7	0.05	0.03	< 0.01	< 0.01	
		3.4**	0.05	0.02	< 0.01	< 0.01	
	Disulfoton sulfoxide oxygen analog	3.4	0.81	0.14	0.96	0.03	
		6.7	0.46	0.32	0.17	< 0.01	
		3.4**	0.84	0.23	0.12	0.01	
	Disulfoton sulfone	3.4	1.65	0.46	0.38	0.04	
		6.7	1.34	1.18	0.31	0.06	
		3.4**	1.21	0.87	0.34	0.12	
	Disulfoton	3.4	0.05	0.02	< 0.01	0.04	0.02
		6.7	0.20	0.16	0.03	0.02	< 0.01
		3.4**	0.05	0.03	0.01	< 0.01	< 0.01
<i>Upper Piedmont</i>	Disulfoton oxygen analog	3.4	0.26	0.20	0.06	0.07	0.03
		6.7	0.14	0.15	0.03	0.02	< 0.01
		3.4**	0.05	0.02	0.01	< 0.01	< 0.01
	Disulfoton sulfoxide oxygen analog	3.4	3.57	2.97	1.05	0.14	< 0.01
		6.7	0.61	0.31	< 0.01	< 0.01	< 0.01
		3.4**	0.71	0.57	< 0.01	< 0.01	< 0.01
	Disulfoton sulfone	3.4	6.37	5.05	1.06	0.60	0.02
		6.7	5.54	4.91	0.16	0.08	< 0.01
		3.4**	2.76	1.39	0.77	0.27	< 0.01

* Each value en average of four replications. Residues of disulfoton sulfoxide and disulfoton sulfone oxygen analog were below the detectable limit of 0.01 ppm. Tobacco harvested July 15, July 23, August 5, and August 20, 1976 at Central Crops Research Station. Tobacco harvested July 22, August 10, August 23, September 8, and September 13, 1976 at Upper Piedmont Research Station.

** Combination treatment with ethoprop.

SUMMARY

Soil samples collected approximately 21 days after pre-transplant applications of ethoprop to flue-cured tobacco land contained residues averaging 0.54 and 1.13 ppm, respectively, for the 6.7 and 13.4 kg/ha rates. In contrast total residues of disulfoton (disulfoton plus three metabolites) averaged 0.28 and 0.54 ppm at 3.4 and 6.7 kg/ha rates. Concentrations of both compounds decreased with time at each of two locations. Residue levels of ethoprop in the first harvest of flue-cured tobacco from the Central Crops Research Station (Clayton, North Carolina) were 0.04, 0.02, and 0.08 ppm; from the Upper Piedmont Research Station (Reidsville, North Carolina), residues were 0.20, 0.16, and 0.27 ppm, respectively, for rates of 6.7, 6.7 combination (with disulfoton), and 13.4 kg/ha. Residues declined in succeeding harvests. Total residues of disulfoton plus three metabolites in the first sample of tobacco harvested from Central Crops averaged 2.52 and 1.89 ppm; and from Upper Piedmont residues averaged 10.24 and 6.50 ppm, respectively, for rates of 3.4 and 6.7 kg/ha. The residues declined in succeeding harvest and were 0.14 ppm or less by the fourth harvest at Central Crops and 0.08 ppm or less by the fifth harvest at Upper Piedmont.

ZUSAMMENFASSUNG

Etwa 21 Tage nach Applikation von Ethoprop in Mengen von 6,7 kg und 13,4 kg je ha vor dem Auspflanzen der Setzlinge ("flue-cured") wiesen Bodenproben durchschnittlich Rückstände von 0,54 ppm bzw. 1,13 ppm auf. Die Gesamtrückstände im Boden an Disulfoton einschließlich 3 Metaboliten beliefen sich dagegen im Durchschnitt bei Gaben von 3,4 kg und 6,7 kg je ha auf 0,28 ppm bzw. 0,54 ppm. An zwei Versuchsorten zeigte sich gleichermaßen ein Rückgang der Konzentration beider Verbindungen im Boden mit fortschreitender Zeit. Bei Gaben von 6,7 kg Ethoprop, 6,7 kg Ethoprop in Verbindung mit Disulfoton und 13,4 kg Ethoprop je ha machten die Rückstände im "flue-cured"-Tabak bei der ersten Ernte in *Central Crops Research Station*, Clayton, N.C., 0,04, 0,02 bzw. 0,08 ppm und in *Upper Piedmont Research Station*, Reidsville, N.C., 0,20, 0,16 bzw. 0,27 ppm aus. Die Rückstandsmengen gingen in den nachfolgenden Ernten zurück. Nach Applikation von 3,4 kg und 6,7 kg Disulfoton je ha zeigten die Tabakproben bei der ersten Ernte Gesamtrückstände an Disulfoton einschließlich 3 Metaboliten von durchschnittlich 2,52 ppm bzw. 1,89 ppm in Clayton und 10,24 ppm bzw. 6,50 ppm in Reidsville. Die Rückstände nahmen bei den folgenden Ernten ab und betrugen in Clayton bei der vierten Ernte 0,14 ppm und weniger und in Reidsville bei der fünften Ernte 0,08 ppm und weniger.

RÉSUMÉ

Des échantillons de terre, prélevés environ 21 jours après le traitement à l'éthoprop précédant la transplantation du tabac «flue-cured» et effectué à raison de 6,7 et 13,4 kg par hectare, ont présenté des quantités moyennes de résidus de 0,54 ppm et 1,13 ppm respectivement. Comparativement, les résidus totaux de disulfoton (y compris 3 métabolites) atteignaient, après une application de 3,4 et 6,7 kg par hectare, une moyenne de 0,28 et 0,54 ppm respectivement. On a constaté une diminution de la concentration des deux composés dans le sol, en fonction du temps, dans deux centres de recherche. Après des traitements comprenant 6,7 kg d'éthoprop, une combinaison de 6,7 kg d'éthoprop et de disulfoton et 13,4 kg d'éthoprop par hectare, les résidus dans le tabac «flue-cured» de la première récolte atteignaient 0,04, 0,02 et 0,08 ppm respectivement à la *Central Crops Research Station*, Clayton, N. C., et 0,20, 0,16 et 0,27 ppm à la *Upper Piedmont Research Station*, Reidsville, N. C. Les résidus ont diminué lors des récoltes subséquentes. À la suite d'applications de 3,4 kg et de 6,7 kg de disulfoton par hectare, les échantillons de tabac de la première récolte contenaient en moyenne des résidus totaux de disulfoton (y compris 3 métabolites) de 2,52 ppm et 1,89 ppm respectivement à Clayton et de 10,24 ppm et 6,50 ppm à Reidsville. Les résidus ont diminué lors de récoltes subséquentes. À Clayton, ils atteignaient 0,14 ppm et moins lors de la quatrième récolte, à Reidsville, 0,08 ppm et moins à la cinquième récolte.

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