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Degradation of Disulfoton in Soil and Its Translocation into Asparagus

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Granular disulfoton at 0.5 and 4.0 kg of a.i./ha was applied as side-dressings to asparagus grown in a sandy loam soil to control the European asparagus aphid, *Brachycolus asparagi*. In soil, disulfoton (DS) oxidized rapidly to its sulfoxide (DSO) and sulfone (DSO_2), but only minute amounts of the sulfoxide and sulfone of the oxygen analogue (DOASO and $DOASO_2$) were present. At the realistic rate of 0.5 kg of a.i./ha the parent compound was detected in soil for about 42 days. Total residues (DS + DSO + DSO_2 + DOASO + $DOASO_2$) remained at the level of 1–2 ppm (dry weight) in the side-dress band for about 56 days and decreased to 0.06 ppm in 147 days. In the plants, residues of the sulfoxides and sulfones of both the parent compound and the oxygen analogue were detected after 14 days. However, disulfoton and its oxon were never detected. The total residues (DSO + DSO_2 + DOASO + $DOASO_2$) increased steadily to a maximum in about 70–85 days and declined thereafter. After 147 days asparagus ferns from plots treated at 4.0 kg of a.i./ha still contained 17.1 ppm (fresh weight) of total residue. However, only 0.01 ppm of the oxygen analogue sulfone was found in spears collected from the same plots the following growing season. No residue was detected in spears from plots treated at 0.5 kg of a.i./ha. Disulfoton at both application rates reduced the aphid populations to levels below the spray threshold of 0.5 aphid/g of asparagus fern tissue throughout the growing season. No damage in plants was observed in the treated plots whereas the asparagus in the control plots suffered from moderate to heavy damage.

The European asparagus aphid, *Brachycolus asparagi* Mordvilko, is a newly arrived and highly damaging pest of asparagus in the western United States and Canada. A toxin injected by the aphid at feeding causes severe deformity (bonzai or witches' broom-type growth) and even death of the plants (Forbes, 1981). An outbreak of this pest in Washington State and in the Okanagan Valley of British Columbia has been observed since 1979 and has caused considerable economic damage. Currently, malathion, mevinphos, and carbaryl are registered in Canada for control of the asparagus beetle, *Criocerus asparagi* (L.), and the spotted asparagus beetle, *Criocerus duodecimpunctata* (L.). However, they are ineffective against the asparagus aphid because these insecticides act on contact and have short residual activity. In order to suppress the aphid population throughout the growing season, several applications of these chemicals would be needed. These would probably disturb the balance of the agroecosystem because the chemicals are highly toxic to beneficial insects such as bees and aphid predators, and the applications might cause physical damage to the crop if tractor-mounted boom sprayers were used.

Disulfoton [*O,O*-diethyl *S*-[2-(ethylthio)ethyl] phosphorodithioate, Di-Syston] appeared to us to be a more suitable candidate. It is a widely used systemic insecticide, well suited for the control of sap-feeding insects. The advantage of using this compound is that beneficial insects can forage relatively safely in treated fields because its action is mostly systemic.

This paper describes the translocation of disulfoton and its metabolites from soil to asparagus fern after in-furrow application of Di-Syston 15 G at 0.5 and 4.0 kg of a.i./ha. Also, the efficacy of disulfoton for the control of asparagus aphid is discussed in terms of total residues [disulfoton

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Table I. Residues of Disulfoton and Its Metabolites in Immature Asparagus Ferns and in Soil after In-Furrow Application of Di-Syston 15 G at 0.5 kg of a.i./ha

time after application, days	residues, ppm (%[a])						
	DS	DSO	DSO_2	DOASO	$DOASO_2$	total	Ca/Cs[b]
14 A[c]	ND[f]	0.18 (25.7)	0.24 (34.3)	0.10 (14.3)	0.18 (25.7)	0.70	0.3
S[d]	1.00 (46.9)	0.65 (30.5)	0.39 (18.3)	0.09 (4.3)	trace[e]	2.13	
21 A	ND	0.41 (20.5)	0.71 (35.5)	0.19 (9.5)	0.69 (34.5)	2.00	1.5
S	0.13 (9.8)	0.20 (15.0)	0.94 (70.7)	0.05 (4.5)	trace	1.33	
42 A	ND	0.55 (15.8)	1.25 (35.8)	0.30 (8.6)	1.39 (39.8)	3.49	3.2
S	0.16 (14.7)	0.30 (27.5)	0.59 (54.1)	0.04 (3.7)	trace	1.09	
56 A	ND	0.91 (8.6)	4.48 (42.3)	0.58 (5.5)	4.62 (43.6)	10.6	10.4
S	trace	0.25 (24.5)	0.75 (73.5)	0.02 (2.0)	trace	1.02	
70 A	ND	0.34 (2.4)	4.70 (33.0)	0.49 (3.4)	8.70 (61.1)	14.2	83.5
S	ND	0.02 (11.8)	0.15 (88.2)	trace	ND	0.17	
77 A	ND	0.07 (1.4)	0.80 (16.8)	0.37 (7.8)	3.53 (74.6)	4.70	10.3
S	trace	0.06 (13.0)	0.40 (87.0)	trace	trace	0.46	
91 A	ND	0.06 (1.8)	0.70 (20.2)	0.39 (11.2)	2.32 (66.9)	3.47	10.5
S	ND	0.05 (15.2)	0.28 (84.8)	trace	trace	0.33	
98 A	ND	0.02 (0.8)	0.26 (10.9)	0.21 (8.8)	1.89 (79.4)	2.38	23.8
S	ND	0.02 (20.0)	0.06 (60.0)	0.02 (20.0)	trace	0.10	
105 A	ND	0.02 (1.1)	0.25 (13.4)	0.15 (8.1)	1.44 (77.4)	1.86	26.6
S	ND	0.01 (14.3)	0.06 (85.7)	trace	trace	0.07	
133 A	ND	trace	0.06 (7.4)	0.06 (7.4)	0.69 (85.2)	0.81	7.4
S	ND	trace	0.11 (100)	trace	trace	0.11	
147 A	ND	trace	trace	0.03 (7.1)	0.39 (92.9)	0.42	7.0
S	ND	trace	0.06 (100)	trace	ND	0.06	

[a] Percent of total residue (DS + DSO + DSO_2 + DOASO + $DOASO_2$). [b] Ca/Cs = ratio of disulfoton residues in asparagus fern/disulfoton residues in soil. [c] A = asparagus fern. [d] S = soil. [e] Trace = less than 0.01 ppm (fresh weight in asparagus fern and dry weight in soil). [f] ND = not detectable. The limit of detection for disulfoton and its metabolites was 2.5 ppb (fresh weight in asparagus fern and dry weight in soil).

sulfoxide (DSO) + disulfoton sulfone (DSO_2) + disulfoton oxon sulfoxide (DOASO) + disulfoton oxon sulfone ($DOASO_2$)] accumulated in the asparagus tissue.

MATERIALS AND METHODS

A field trial to evaluate disulfoton for the control of asparagus aphid was conducted at the Research Station of Agriculture Canada in Summerland, British Columbia. Asparagus of cv. Mary Washington was seeded in May 1980 in an Osoyoos sandy loam soil (Orthic brown chernozem; pH 6.8, organic content 1.2%, sand 68.3%, silt 25.2%, and clay 5.3%). The treatment plots were 6 m long by 2 rows wide, with a 1-m buffer strip between plots in each row. Three treatment plots were used for each test and for the control. In order to ensure aphid infestation, we collected wild asparagus aphids from nearby infested asparagus stands and released them throughout the experimental field on May 15, 21, and 27, June 2 and 8, and July 17, 1981.

On May 16, 1981, Di-Syston 15 G (Chemagro Limited, Mississauga, Ontario, Canada) granules were applied at 0.5 and 4.0 kg of a.i./ha as side-dressings. The granules were sprinkled into bands 2 cm wide at the bottom of trenches 10 cm deep, located 15 cm from each side of the asparagus row.

Before and at various intervals after insecticide application, four randomly selected asparagus ferns in each plot were cut at ground level for residue determination. Accompanying soil samples consisting of two cores (150 mm deep × 25.4 mm diameter) were taken from the application furrows of each plot. On April 29, 1982, 10 marketable asparagus spears were randomly collected from each plot. All samples were stored in plastic bags at −20 °C until analyzed within 90 days. Samples of soil and asparagus tissue from the control plots were fortified with 1 ppm each of disulfoton and its five toxic metabolites and stored under identical conditions for 118 days. Duplicates were extracted and analyzed at 0 day and after 7, 14, 28, 56, and 118 days. No significant changes were observed in the concentrations of the individual compounds, and the standard deviation for the 12 analyses was about 5% of the mean.

Asparagus ferns were cut up with shears and the spears were macerated in a Braun vegetable shredder. The separate samples were then mixed thoroughly in a plastic bag. Aliquots of 10 g of ferns, 20 g of spears, or 20 g of the well-mixed moist soil were extracted and analyzed for disulfoton and its toxic metabolites according to the method of Szeto and Brown (1982).

Efficacy of the insecticide was determined by counting the asparagus aphids extracted from 20 asparagus sprigs collected randomly from each treatment and control plot at various intervals before and after insecticide application. Insect damage was assessed by visually examining 10 plants in each plot and the damage was ranked as follows: 0 = no damaged fern tips; 1 = 1–3 damaged tips (light); 2 = 4–10 damaged tips (moderate); 3 = more than 10 damaged tips (severe); 4 = one or more bonzai spears (very severe). The average of the rankings for the total of 30 plants was considered as the damage index.

RESULTS AND DISCUSSION

Residues of disulfoton, its oxygen analogue, sulfoxide, oxon sulfoxide, sulfone, and oxon sulfone in asparagus and soil from plots treated with Di-Syston 15 G at 0.5 kg of a.i./ha and in asparagus from plots treated at 4.0 kg of a.i./ha are given in Tables I and II, respectively. No residue was ever detected in asparagus or soil from the control plots.

After in-furrow application at 0.5 kg of a.i./ha the disulfoton oxidized rapidly in soil, mostly to its sulfoxide and sulfone. The oxygen analogue was never detected in soil, and only minute amounts of the oxon sulfoxide and the oxon sulfone were found (Table I). The concentrations of disulfoton were 1 ppm (dry weight of soil) after 14 days, decreased to 0.13 ppm in 21 days and trace (<0.01 ppm dry weight) in 56 days, and became nondetectable in 91 days. The percentage of disulfoton in total residue (DS + DSO + DSO_2 + DOASO + $DOASO_2$) decreased from 47% after 14 days to 15% after 42 days while that of the

Table II. Residues of Disulfoton and Its Metabolites in Immature Asparagus Fern after In-Furrow Application of Di-Syston 15 G at 4.0 kg of a.i./ha

time after application, days	residues, ppm (%[a])				
	DSO	DSO_2	DOASO	$DOASO_2$	total
14	0.36 (33.0)	0.35 (32.1)	0.13 (11.9)	0.25 (23.0)	1.09
21	1.81 (27.2)	2.49 (37.4)	0.46 (6.9)	1.90 (28.5)	6.66
42	6.90 (17.6)	17.7 (45.3)	3.03 (7.7)	11.5 (29.4)	39.1
56	5.17 (13.3)	19.0 (48.8)	1.60 (4.2)	13.1 (33.7)	38.9
70	3.18 (8.6)	17.4 (47.3)	1.83 (5.0)	14.4 (39.1)	36.8
77	5.14 (8.8)	26.3 (45.1)	3.87 (6.6)	23.0 (39.5)	58.3
85	4.34 (7.2)	27.5 (45.3)	3.36 (5.5)	25.5 (42.0)	60.7
91	1.59 (4.6)	14.1 (41.1)	0.92 (2.7)	17.7 (51.6)	34.3
98	0.60 (2.2)	8.02 (28.5)	2.59 (9.2)	16.9 (60.1)	28.1
105	1.02 (3.7)	11.8 (40.7)	2.04 (7.0)	14.1 (48.6)	29.0
119	0.27 (1.5)	6.78 (35.9)	1.54 (8.1)	10.3 (54.5)	18.9
147	0.17 (0.7)	1.67 (9.8)	2.50 (14.6)	12.8 (74.9)	17.1

[a] Percent of total residue (DSO + DSO_2 + DOASO + $DOASO_2$).

sulfone increased from 18% after 14 days to 54% after 42 days. The initial decline of total residues in the side-dress band was relatively slow. There was 2.13 ppm present after 14 days which decreased to 1.02 ppm after 56 days. Thereafter they disappeared at a much faster rate, possibly as a result of the lateral and downward movements of toxicant from the point of application and only 0.06 ppm remained after 147 days.

Our results suggest that the major degradation pathway of disulfoton in soil was oxidation of the parent compound to the sulfoxide and sulfone, followed by the degradation of these intermediates. Menzer et al. (1970) studied the degradation of disulfoton and phorate in soil. Using thin-layer chromatography they found large amounts of the sulfones of the parent insecticides and the oxygen analogues, but only minute amounts of the oxygen analogues and the sulfoxides of the parent compounds and the oxygen analogues were detected. Our results differed from those of Menzer et al. in that no oxygen analogue was ever detected in soil and only trace amounts of the oxygen analogue sulfone were found. This could be due to the different methods of insecticide application. Disulfoton was applied broadcast in their studies. Suett (1975) also studied the persistence and degradation of disulfoton in soil after the chemical was incorporated by rotovating to a depth of 100 mm. He detected neither the oxygen analogue nor its sulfoxide and sulfone in soil. Our results were in general agreement with Suett's although, unlike Suett, we detected minute amounts of the oxygen analogue sulfoxide and oxygen analogue sulfone in soil. The apparent discrepancy could be explained by the difference in the limits of detection of the two studies. The highest concentration of the oxygen analogue sulfoxide was 0.09 ppm and only trace amounts of the oxygen analogue sulfone were detected in our studies (Table I). Since the limits of detection in Suett's studies were 0.05 ppm for the oxygen analogue sulfoxide and 0.01 ppm for the oxygen analogue sulfone, most of the residues of these two metabolites reported in our studies would not have been detected by Suett's analytical methods, except the 0.09 ppm of oxygen analogue sulfoxide present in the soil sample collected 14 days after application.

The residues of the sulfoxides and the sulfones were higher in soil than in the plant 14 days after the in-furrow application at 0.5 kg of a.i./ha, but thereafter residues in plant tissues increased as those in soil gradually declined (Table I). After 70 days the plant residues reached a maximum of 14.2 ppm and then gradually declined. After in-furrow application at 0.5 or 4.0 kg of a.i./ha in soil, disulfoton and its oxygen analogue were never detected in the plant tissues and the plant residues consisted only of the sulfoxides and the sulfones (Tables I and II). The parent sulfoxide residues as a percent of the total (DSO + DSO_2 + DOASO + $DOASO_2$) decreased with time whereas the parent sulfone gradually increased to a maximum after 56 days and decreased thereafter. The oxygen analogue sulfoxide as a percent of the total residue varied somewhat but remained relatively constant during the asparagus growing season and showed no definite trend to increase or decrease, whereas the oxygen analogue sulfone increased steadily. After 147 days most of the residues detected in plant tissues were the oxygen analogue sulfone. Our residue data support the metabolic scheme of disulfoton in plants postulated by Metcalf et al. (1957). Menzer and Ditman (1968) studied the residues in spinach grown in disulfoton-treated soil. They reported that oxidative metabolites of disulfoton were present. Only in a few instances were there significant amounts of the parent compound. The residues present in the largest quantities were the sulfoxide and the sulfone of the parent compound and the sulfone of the oxygen analogue. The oxon and its sulfoxide were present in only a few instances and then only in trace amounts. The difference between their findings and ours could be due to the different families of plants used and the methods of insecticide application. In the studies of Menzer and Ditman disulfoton was applied as a broadcast before seeding.

The total residues of the toxic metabolites of disulfoton in asparagus tissues correlated with the application rate in soil. They were at their maximum of 14.2 ppm 70 days after application at 0.5 kg of a.i./ha and 60.7 ppm 85 days after application at 4.0 kg of a.i./ha. Thereafter they decreased to 0.42 and 12.8 ppm, respectively, after 147 days (Tables I and II). Using ^{32}P-labeled disulfoton, Ridgway et al. (1965) studied the effect of application method on uptake of disulfoton by the cotton plant. They reported that after in-furrow application at 1.12 kg of a.i./ha, total uptake of radioactivity increased each week for the first 3 weeks but that after 3 weeks no additional uptake was detected. Also, they showed that application of disulfoton deep in the soil as a side-dressing increased uptake by cotton plants. However, the chemical identities were not determined. In our studies, total uptake as indicated by the total residues in asparagus increased for 70–85 days depending upon the rates of application (Tables I and II). The difference in the two findings could be due to the different families of plants used. Other factors, such as permeability and the persistence of disulfoton in different soils, and the methods of application could also affect uptake.

Samples of marketable asparagus spears harvested the following season were analyzed for residues. No residues

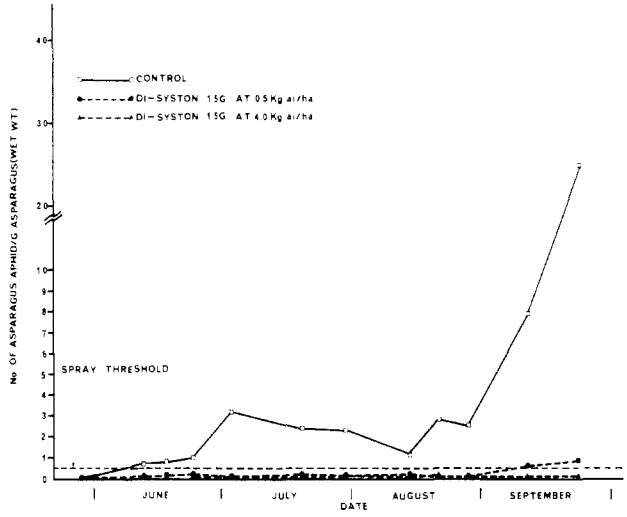

Figure 1. Population of *Brachycolus asparagi* in plots treated with Di-Syston 15 G at 0.5 and 4.0 kg of a.i./ha and in the control.

were detected in samples from plots treated at 0.5 kg of a.i./ha, and the limit of detection was 2 ppb for disulfoton and its toxic metabolites. Although the ferns collected at the end of the previous season from plots treated at 4.0 kg of a.i/ha had contained 17.1 ppm of total residue, the marketable spears in spring contained only about 0.01 ppm of the oxygen analogue sulfone.

In-furrow application of disulfoton at 0.5 or 4.0 kg of a.i./ha gave excellent control of the European asparagus aphid. The population of the pest in the treated plots remained below the spray threshold of 0.5 aphid/g of tissue during most of the growing season whereas that in the control consistently was above the spray threshold and exploded in late September when the ferns started to senesce (Figure 1). Although the aphid population in plots treated at 0.5 kg of a.i./ha was slightly higher than the spray threshold by late September, a chemical spray was not necessary because it was already near the end of the growing season. The damage indices were 0.10 and 0.07, respectively, for plots treated at 0.5 and 4.0 kg of a.i./ha, indicating that little damage occurred in the plants. By comparsion, asparagus in the control suffered from moderate to very heavy damage as evidenced by the damage index of 3.05. Based on the residue and efficacy data obtained in our studies, it is apparent that the biological activity of disulfoton against the European asparagus aphid resulted from the toxic sulfoxides and sulfones, derived from the oxidation of the parent compound.

Registry No. DSO, 2497-07-6; DSO_2, 2497-06-5; DOASO, 5286-73-7; $DOASO_2$, 4891-54-7; disulfoton, 298-04-4.

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Determination of Carbosulfan and Carbofuran Residues in Plants, Soil, and Water by Gas Chromatography

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Analytical procedures are described for the determination of carbosulfan and carbofuran residues in various crops, in soil, and in water. The quantitative methods involve extraction of residues using hexane–2-propanol or methanol–buffered water followed by column cleanup using Florisil, gel permeation, and Darco–Attaclay plus aluminum oxide, each column alone or in combination with each other. Residues of carbosulfan and carbofuran are detected by gas–liquid chromatography using a nitrogen-selective detector. Good recoveries are achieved with check samples fortified at the 0.05-ppm level with crops and soil and at the 0.01-ppm level with water.

Carbosulfan, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl [(dibutylamino)thio]methylcarbamate (Figure 1), is a carbofuran derivative being developed as a pesticide by FMC Corp. Carbosulfan, as the parent compound, and carbofuran, as a major metabolite, have been reported in soil (Clay et al., 1980) and in cotton and corn (Umetsu et al., 1979, 1980) following treatment with carbosulfan. Analytical procedures were developed by FMC Corp. that are capable of determining the residues of carbosulfan and carbofuran in the same sample extract.

Various studies (Umetsu et al., 1979, 1980; Clay et al., 1980) have shown that carbosulfan can be extracted from soil and crop samples by using organic solvents. The same studies showed that carbosulfan, when exposed to acidic media, was unstable and decomposed to carbofuran.

Methanol has been successfully used in a metabolism study (Knaak et al., 1970) to quantitatively extract carbofuran from bean plants. Chloroform was used to extract carbofuran in an apple, potato, and sugar beet residue study (Butler and McDonough, 1968). Investigations in this laboratory showed the hexane–2-propanol (IPA) and methanol–pH 8 buffer solutions to be as efficient to extract carbofuran from various matrices.

The analytical procedure was, therefore, designed to use organic solvents to extract carbosulfan, as the parent chemical, and carbofuran, as a major metabolite, from

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