THE EFFECT OF TREATING SOIL OR PASTURE WITH HEPTACHLOR GRANULES ON HEPTACHLOR RESIDUES IN SOIL, PASTURE AND STOCK

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Summary

Bare ground was treated with 5% heptachlor granules at rates of $\frac{1}{2}$ lb and 1 lb active ingredient per acre and then sown to pasture. Maximum residues of heptachlor epoxide found in the fat of sheep and in the milk of cows grazed on these pastures, after withholding periods of up to 27 weeks, were about 0.10 ppm.

The fat of ewes and wethers which had been grazed after withholding periods of 5 to 11 weeks on pastures that had been treated directly with heptachlor granules at rates of 2 to 3 oz active ingredient per acre, contained up to 0.20 ppm heptachlor epoxide. The fat of lambs running with the ewes contained up to 0.50 ppm heptachlor epoxide.

Heptachlor treatment of soil prior to sowing to pasture resulted in lower residues in stock than did the treatment of established pasture. Nevertheless, the use of modern analytical methods means that neither treatment would comply with a "nil" tolerance for heptachlor and its principal metabolite in animal fats.

INTRODUCTION

THE TRIALS described in this paper were designed to determine whether heptachlor treatment of soil prior to sowing pasture would comply with a "nil" tolerance for heptachlor and its principal metabolite in animal fats. For comparison, information is presented on residues in stock grazed on heptachlor-treated pasture.

EXPERIMENTAL AND RESULTS

ANALYTICAL

Residues of heptachlor and its principal metabolite, heptachlor epoxide, were determined separately by gas chromatography.

Results are expressed as parts per million (ppm) on a dry weight basis for soil and pasture and on a raw fat basis for fat.

Only heptachlor epoxide was detected in animal fat. In soil and pasture samples both heptachlor and heptachlor epoxide were detected, and in these cases the results have been added together and expressed as "heptachlor plus heptachlor epoxide" residues. The limits of sensitivity for heptachlor and heptachlor epoxide were 0.002 ppm in animal fat and 0.01 ppm in pasture.

SILVERSTREAM TRIAL WITH SHEEP

A 1.35 acre field (soil type, Waikanae silt loam), cultivated to produce a suitable seedbed, was divided into six approximately $\frac{1}{4}$ acre paddocks.

Heptachlor granules (5% active ingredient) were broadcast evenly by hand on two of the paddocks at the rate of $\frac{1}{2}$ lb (all rates in a.i. per acre) and on two other paddocks at the rate of 1 lb, the two untreated paddocks serving as control areas. The whole experimental site was then sown with a seed and fertilizer mixture. Treatments were applied in April, 1966 (autumn). Six days after seeding, the area was partly flooded from a nearby creek. However, subsequent analysis of soil samples showed that no heptachlor had been transferred to the control paddocks, although some insecticide appeared to have been leached from the heptachlor-treated paddocks.

Soil and pasture samples were taken at various times during the trial for residue analysis.

When pastures were established, 24 Romney crossbred ewe hoggets were divided into six groups of four animals. Each group was assigned to a paddock and the paddocks were all grazed according to the following time sequence:

Period after soil treatment						20 weeks
First grazing period						27 days
Spelled period						3 weeks
Period after soil treatment						27 weeks
Second grazing period						43 days
Spelled period						12 weeks
Period after soil treatment						45 weeks
Third grazing period						35 days
Period after soil treatment						52 weeks

At the end of each grazing period all sheep were weighed and omental fat was surgically removed for chemical analysis (Table 2). During the intervals between the grazing periods on experimental paddocks the sheep were run together on pasture free from heptachlor.

SOUTH ISLAND TRIALS WITH SHEEP

Samples of soil, pasture and sheep fat were obtained from field trials in the South Island that were being carried out to test the efficacy of heptachlor against DDT-resistant grass-grub populations. Trials on three farms in Marlborough were under the direction of R. L. Taylor, Farm Chemicals Co., Nelson, and J. M. Kelsey, Entomology Division, Department of Scientific and Industrial Research, directed trials on farms in Canterbury. Brief details of these trials are as follows:

Marlborough, Farm 1, Treatment (a): Approximately 100 acres of newly reclaimed swampland (soil type, Paynter peaty silt loam) were treated with 10% heptachlor granules at the rate of 1 lb and the insecticide was disced into the soil which was then sown with a ryegrass/white clover seeds mixture. This area was twice flooded after treatment, but analysis of soil samples indicated that the heptachlor had not been leached out to any extent.

Marlborough, Farm 1, Treatment (b): Six acres of very closely grazed ryegrass/clover pasture on a heavy silt soil were topdressed with 1¼% heptachlor granules at the rate of 2 oz. At the time of treatment there were patches of bare ground caused by severe grass-grub damage.

Marlborough, Farm 2: Five acres of dense, vigorous, slightly damp ryegrass/clover pasture, 2 to 3 in. long, were topdressed with 1¼% heptachlor granules at the rate of 3 oz. The soil was a medium silt.

Marlborough, Farm 3: 1¼% heptachlor granules were applied at the rate of 2 oz to two strips across a 5 acre, close-grazed, well established pasture. The soil was a medium silt.

Canterbury, Farm 4: In a replicated plot trial, heptachlor granules were applied to pasture on a light stoney silt soil at rates of ½ lb and 1 lb. The plots were grazed with sheep until 24 weeks after topdressing. Samples of soil and pasture were taken 24 and 31 weeks after topdressing, respectively.

Canterbury, Farm 5: Part of a farm in the Cheviot district was topdressed with heptachlor granules at the rate of 1 lb.

The treated areas on these farms were grazed at various times, and details of stock used, grazing times, and results of analyses for residues in soils and pastures and in samples of peri-renal fat taken from sheep at slaughter are presented in Tables 3 and 4.

KAITOKE TRIAL WITH LACTATING COWS

A 2.5 acre field (soil type, Pukuratahi silt loam) was prepared for treatment in the same way as the experimental area at Silverstream, and then fenced into two equal paddocks. Heptachlor granules (5%) were applied uniformly to the two paddocks at rates of ½ lb and 1 lb, respectively. Seed and fertilizer application similar to that at Silverstream was carried out 35 days after heptachlor topdressing. Seeds were sown in June, 1966 (winter).

When pasture was established, three cows were grazed continuously on each of the treated areas for two successive periods of 14 days, with a spelled period of four weeks in between. The first period started 27 weeks and the second period 33 weeks after the soil had been treated with insecticide. Milk was sampled from each cow after 1 and 14 days grazing on the treated areas during the first period, and after 5 and 14 days grazing during the second period. Heptachlor epoxide levels in these samples are shown in Table 5. When not on the experimental areas the cows were returned to the main Kaitoke herd and grazed on untreated areas of the farm. Two cows in the main herd served as control animals. Pasture samples (each approximately 2 lb in weight) were taken at the start and at the end of each grazing period for residue analysis. Levels of heptachlor and heptachlor epoxide in these samples were similar to those found in pasture samples from the Silverstream trial.

DISCUSSION

Total residues in soils (heptachlor plus heptachlor epoxide) at Silverstream and Canterbury (Farm 1) where insecticide treatments were replicated were quite variable. It is believed that these variations were due mainly to sampling errors associated with the use of an insecticide formulated as granules that disintegrate only slowly in the soil. While this precludes any quantitative comparisons of soil residues between the various trials, the results obtained provide evidence of the persistence of heptachlor in a variety of soil types.

In pastures grown in soil after the bare ground had been treated with heptachlor (Tables 1 and 3) total residues were low — not exceeding 0.2 ppm on a dry matter basis in any sample — but were persistent, being still detectable one year after treatment (Table 1). In view of the known persistence of heptachlor in soils and its translocation in small amounts

TABLE 1: SILVERSTREAM SHEEP GRAZING TRIAL
Heptachlor plus Heptachlor Epoxide in Soil and Pasture (ppm)
Weeks after Treatment and Type of Sample Analysed

<i>Treatment (lb)</i>	<i>3 Soil</i>	<i>20 Soil</i>	<i>20 Pasture</i>	<i>24 Pasture</i>	<i>27 Pasture</i>	<i>33 Pasture</i>	<i>47 Pasture</i>	<i>52 Soil</i>	<i>52 Pasture</i>
0 (control)	...	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
1/2	...	0.27	0.04	0.07	0.03	0.03	0.02	0.58	0.04
1	...	0.38	0.07	0.05	0.06	0.04	0.04	0.26	0.04

into the aerial parts of plants (Lichtenstein and Schulz, 1960), it would be expected that low levels of heptachlor and its epoxide would be detectable in pastures grown in heptachlor-treated soils for some years following treatment. No attempt was made in the trials to distinguish between systemic residues and residues due to surface contamination of the pasture plants, for, irrespective of which source was more significant, it is clear that the residues were at all times "available" to grazing stock (Tables 2, 4 and 5).

TABLE 2: SILVERSTREAM SHEEP GRAZING TRIAL
Heptachlor Epoxide in Omental Fat (ppm)

<i>Grazing Periods on Treated Plots (Weeks after Soil Treatment)</i>						
<i>Treatment (lb)</i>		<i>20-24 Sampled end of 24th wk</i>	<i>27-33 Sampled end of 33rd wk</i>	<i>46-52 Sampled start of 46th wk</i>	<i>Sampled end of 52nd wk</i>	
0 (control)		0.009		0.027		0.011
1/2		0.031		0.082		0.031
1		0.072		0.117		0.052
						0.084

Each figure is the mean of analyses of fat samples from 8 sheep. Pre-trial tests on biopsy omental fat showed that, initially, all sheep were free from heptachlor epoxide.

TABLE 3: SOUTH ISLAND TRIALS
Total Heptachlor plus Heptachlor Epoxide in Soils and Pastures (ppm)

<i>Farm and Type of Applica- tion</i>	<i>Rate a.i. per acre</i>	<i>Weeks after Treatment and Type of Sample Analysed</i>			
FARM 1		<i>14 wk after Sowing Pasture Seeds</i>		<i>25 wk after Sowing Pasture Seeds</i>	
Treatment (a) (bare ground)	1 lb	Soil	Pasture	Soil	Pasture
		2.08	0.18		0.06
Treatment (b) (pas- ture)	2 oz	<i>5 wk after Treatment</i>		<i>15 wk after Treatment</i>	
		Soil	Pasture	Soil	Pasture
		0.48	0.50		0.11
FARM 2		<i>5 wk after Treatment</i>		<i>16 wk after Treatment</i>	
(Pasture)	3 oz	Soil	Pasture	Soil	Pasture
		0.97	3.91		0.24
FARM 3		<i>5 wk after Treatment</i>			
(Pasture)	2 oz	Soil	Pasture	Soil	Pasture
		1.26	2.76		—
FARM 4 (Plot trials)		<i>24 wk after Treatment</i>		<i>31 wk after Treatment</i>	
		<i>Soil</i>		<i>Pasture</i>	
		<i>(Means of 5 replicates)</i>		<i>(Means of 5 replicates)</i>	
(Pasture)	8 oz		0.25		0.03
(Pasture)	1 lb		0.50		0.05
FARM 5		<i>63 wk after Treatment</i>			
(Pasture)	1 lb	Soil	Pasture	Soil	Pasture
		2.43	0.24		—

TABLE 4: SOUTH ISLAND TRIALS
Heptachlor Epoxide in Peri-renal Fat from Sheep and Lambs (ppm raw fat basis)

<i>Farm and Type of Application</i>	<i>Rate a.i. per acre</i>	<i>Details of Grazing</i>	<i>Sheep No.</i>	<i>ppm Heptachlor Epoxide</i>
FARM 1 Treatment (a) (bare ground) 	1 lb	Five 4- to 7-year-old ewes put on to pasture approximately 14 wk after treatment, grazed for 1 to 2 hr per day on treated pasture for a period of approximately 11 wk before slaughter.	1	0.018
			2	<0.002
			3	0.007
			4	0.010
			5	0.007
			6	0.010
			7	0.114
			8	0.107
			9	0.003
			10	<0.002
			11	<0.002
			12	0.002
			13	<0.002
Treatment (b) (pasture) Controls (no access to heptachlor- treated pasture) 	2 oz 0	One 6-tooth ewe put on to pasture approximately 17 wk after treatment, grazed for 5 wk continuously before slaughter. Two wethers put on to pasture 11 wk after treatment, grazed treated pasture for 31 days before slaughter. Two ewes from a flock not exposed to heptachlor-treated pasture. Three wethers not exposed to heptachlor-treated pasture.	14	0.182
			15	0.092
			16	0.140
			17	0.114
			18	0.045
			19	0.274
			20	0.503
			21	0.110
			22	0.186
			23	0.660
FARM 2 (pasture) 	3 oz	Ewes with lambs put on to pasture 5 wk after treatment. Allowed to graze treated pasture for 20 hr per day for periods of — (i) 3 wks (ii) 4 wks (iii) 6 wk taken off for 4 wk, put on continuously for one week. Six lambs run with the above flock of ewes, slaughtered 16 wk after the pasture was treated.	14	0.182
			15	0.092
			16	0.140
			17	0.114
			18	0.045
			19	0.274
			20	0.503
			21	0.110
			22	0.186
			23	0.660
FARM 5 (pasture) 	1 lb	6-tooth ewe put on to pasture approximately one year after treatment, grazed treated pasture continuously for 8 wk before slaughter.	14	0.182
			15	0.092
			16	0.140
			17	0.114
			18	0.045
			19	0.274
			20	0.503
			21	0.110
			22	0.186
			23	0.660

TABLE 5: KAITOKE DAIRY COW GRAZING TRIAL
Heptachlor Epoxide in Butterfat (ppm)

Treatment (lb)	Grazing Periods on Treated Plots (weeks after soil treatment)				
	27-29		2nd Pre- grazing samples	33-35	
	1-day samples	14-day samples		5-day samples	14-day samples
0 (control)	0.002	<0.002		0.002	0.004
1/2	0.006	0.066		0.018	0.048
1	0.009	0.102		0.028	0.067

There were 2 cows in the control group, 3 cows in each of the treated groups. Each figure is the mean of analyses of butterfat samples from all cows in each group.

Pre-trial tests showed that, initially, butterfat from all cows was free from heptachlor epoxide.

The levels of heptachlor and heptachlor epoxide were higher in pasture topdressed directly with insecticide than in pasture grown subsequent to heptachlor treatment of soils (Tables 1 and 3).

Only heptachlor epoxide was detected in sheep fat and in milk fat following exposure of the test animals to pastures carrying residues of heptachlor and its epoxide (Tables 2, 4 and 5).

The results would indicate, also, that the proportion of heptachlor epoxide stored in animal fat from the low intake levels obtaining in these trials was relatively greater than from the much higher rates of intake studied in experiments of Claborn *et al.* (1960). This phenomenon has been noted in intake/storage studies with other insecticides in rats, sheep and cattle (Lehman, 1952; Solly, unpubl.).

The trials also indicate that, as far as the grazing animal is concerned, neither the treatment of bare ground prior to sowing to pasture, nor the treatment of established pasture with heptachlor granules would comply with a "nil" tolerance for heptachlor and heptachlor epoxide in animal fats owing to the extreme sensitivity of present analytical methods.

Full details of these trials will be published elsewhere.

REFERENCES

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