Residues in Cattle Treated with DDT for Control of Horn Flies on Pasture

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A number of animals were used in experiments to evaluate the efficacy of pesticides in controlling horn flies on cattle during the summer grazing period on pasture. On completion of the field tests some of the animals treated with DDT were marked for individual identification during the fattening period on high energy rations. Samples of fat removed at slaughter were analyzed to estimate the residues of DDT, TDE and DDE remaining in these animals at prime market weight.

Methods and Materials

Grazing Procedure. The test cattle were yearlings that grazed between June 3 and October 4 on irrigated pasture in which the vegetation was known to be free of any direct application of pesticides. They were rotated between fields at weekly intervals to equalize exposure to other environmental conditions between treated and untreated groups.

Treatments. The experimental group of interest in this test had been treated with a coarse spray of 0.25 per cent formulation of DDT wettable powder applied at 350 p.s.i. Each animal received 2 measured quarts of the aqueous suspension distributed on the head, neck, back and flanks. A total of 11 treatments were applied at weekly intervals beginning on June 27 and terminating on September 26 with the exception of the second and fourth weeks of August.

Control animals in the previous experiment included one group that received no treatment and one that received a spray of water equal in volume, pressure

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and frequency to that of the DDT spray. A third group received regular subcutaneous injections of eserine as a stressor agent instead of the pesticide.

Finishing Procedures. Fifteen Hereford heifers, comprising eleven treated with DDT, one with water, one with eserine and two with no treatment, were selected from test groups in the pest-control experiment. They were identified individually with permanent marks and transferred to a feedlot at a local packing plant. All the animals were placed on a high-energy grain ration in a large pen with approximately 80 commercial yearlings being finished for slaughter. Three other Hereford heifers of approximately the same weight among the 80 commercial yearlings previously assigned to the pen were also identified individually with permanent marks for comparison with the 15 experimental cattle. Selected animals in the pen were slaughtered each week as they reached the preferred finishing weight of 1000 lb.

Sampling Procedure. The heaviest experimental heifer was slaughtered 58 days after its final exposure to DDT while staff was unavailable to secure fat samples. Two fat samples were obtained from each of the remaining 14 experimental heifers and 3 commercial animals as they reached slaughter weight at approximately 1000 lb. One sample represented perirenal fat and the other was taken from the dorsal surface of the pelvic cavity. Similar samples of fat were taken from a random assortment of unfinished slaughter-run cattle on the same day to provide additional information on background levels of DDT residues in commercial cattle. All fat samples were frozen immediately and kept in cold storage until analyzed.

Analysis. Since the objective was to establish only the levels of acid-stable DDT and its derivatives, a slight modification of the well established Davidow procedure (3) was used for the cleanup of the fat samples. This modified procedure was also shown to be applicable to o,p'-DDT and TDE and to p,p'-TDE and DDE with recoveries of 85 per cent or better. All n-hexane used had been previously treated with oleum/concentrated sulphuric acid (1:1) overnight, washed free of acid and filtered through anhydrous potassium carbonate. The final product could be concentrated 20 times if needed and still give a satisfactory background.

Extraction. Beef fat (20.0 g) was cut into small pieces (approx. 5 mm cubes), and extracted with 100 ml oleum-purified n-hexane for 5 hr with a Virtis Extract-omatic shaker. The funnel contents were filtered with suction and the filtrate was made up to 100 ml. A 10.0 ml aliquot was used to determine the fat content. Two other aliquots were cleaned up by the modified Davidow procedure in which the oleum-treated hexane was used for elution instead of carbon tetrachloride. The hexane eluate (300 ml) was filtered through anhydrous potassium carbonate, reduced in volume and made up to 200 ml. Usually, with relatively high levels of DDT, the solution could be used directly for analysis by gas chromatography. Concentrations were calculated by bracketing peaks from one-fifth to one-half full-scale deflection with standard solutions giving approximately the same peak height. The standard solutions contained 200 ng/ml of the o,p' and p,p' isomers of DDT and TDE and 20 ng/ml of p,p'-DDE.

Operating Conditions for Gas Chromatography. A 6' x ¼" Pyrex glass column was packed with 11 per cent QF1 + OV17 mixed phase (1.30 wt ratio) on Gas Chrom Q with 80/100 mesh (Applied Science Laboratories). It was conditioned at 250°C for 16 hr with no flow, and for 4 hr at 250°C and for 72 hr at 200°C with 60 ml/min N_2 flow. The column was operated at 206°C and 78 ml/min N_2 (purge 18 ml/min). Injection volume 2λ (on column).

The gas chromatograph (Micro Tech 220) was equipped with a Ni^{63} pin-cup electron capture detector. It operated at a detector temperature of 250°C, and electrometer setting of 8×10^{-10} amps (FSD). Retention times of standards in minutes were 7.8 for p,p'-DDE; 9.3 for o,p'-TDE; 11.2 for o,p'-DDT; 12.1 for p,p'-TDE and 14.6 for p,p'-DDT. Additional evidence for the compound assignments made was afforded by use of an OV17 column and thin-layer chromatography on silica gel plates (4).

Results and Discussion

The cumulative dose of DDT was approximately the same for the experimental heifers as for commercial cattle treated according to the recommendations for control of horn flies (Table 1).

TABLE 1

Comparison between experimental heifers and commercial animals of exposure to DDT for the usual range in weight of yearlings on pasture in summer at Lethbridge, Alberta.

Body weight, lb.	No. of treatments	Concentration, per cent	Vol./ head	Cumulative dose, mg/kg
Experimental Animals				
450	11	0.25	2 qt.	306
800	11	0.25	2 qt.	172
Commercial Animals				
450	3	0.5	1 gal.	333
800	3	0.5	1 gal.	187

Exposure of experimental animals was more frequent at a regular lower concentration for eleven weeks as compared with the usual requirement of three treatments over a period of 9 weeks in the registered use (1,2) for commercial operations.

The experimental heifers reached the finished stage for slaughter within 58 to 81 days of the last treatments on pasture. Total residues (p,p'-DDT, p,p'-DDE and p,p'-TDE) exceeded the tolerance of 7 p.p.m. in all samples of fat except in one from an animal requiring 81 days to reach prime slaughter weight (Table 2). Although p,p'-DDT was degraded to levels below the tolerance level at 81 days in three animals, the level of p,p'-TDE remained high at approximately 7 p.p.m. The high levels of p,p'-TDE found are of some interest since this compound does not appear to be stored in the fat of rats (5). The possibility that it is a post-mortem artifact derived from p,p'-DDT on storage must be considered since this transformation is known to occur readily in other tissues (6). No o,p'-compounds were detected.

TABLE 2

Residues of DDT in the fat of experimental animals at time of slaughter

Animal number *	Days after treatment	Residues in fat (p.p.m.) pp'DDE	pp'TDE	pp'DDT	Total
DDT Group **					
114(L)	67	2.6	8.0	14.3	24.9
114(C)	67	2.5	8.0	13.7	24.2
112(L)	67	2.0	7.7	11.5	21.2
112(C)	67	2.8	6.7	16.7	26.2
85(L)	67	3.6	11.3	14.0	28.9
85(C)	67	2.7	6.3	15.3	24.3
118(L)	73	2.6	5.5	14.0	22.1
118(C)	73	2.2	5.8	11.2	19.2
6(L)	73	2.3	5.1	13.6	21.0
6(C)	73	2.3	5.6	12.5	20.4
15(L)	73	3.6	11.3	12.7	27.6
15(C)	73	3.9	12.5	11.0	27.4
80(L)	73	2.5	3.1	13.5	19.1
80(C)	73	2.8	5.1	8.4	16.3
113(L)	81	1.4	6.6	ND	8.0
109(L)	81	1.8	7.9	1.5	11.2
27(L)	81	1.5	4.4	1.0	6.9
Water					
66(L)	67	ND ***	ND	ND	ND
66(C)	67	ND	ND	ND	ND
No Treatment					
107(L)	73	ND	ND	ND	ND
107(C)	73	ND	ND	ND	ND
95(L)	73	ND	ND	ND	ND
95(C)	73	ND	ND	ND	ND
Eserine					
104(L)	73	ND	ND	ND	ND
104(C)	73	ND	ND	ND	ND

* (L) - pelvic sample (loin)
(C) - perirenal sample (caul)
** Eleven treatments - 2 qts. per treatment at .25% concentration on back and sides
*** Not detectable (< 0.1 p.p.m.)

TABLE 3

Residues of DDT in the fat of commercial animals selected from market stock to correspond with experimental animals and other slaughter types

Animal number *	Finishing period (days)	Residues in fat (p.p.m.)			
		pp'DDE	pp'TDE	pp'DDT	Total
Commercial Feedlot Heifers					
24(L)	59	2.2	8.6	14.2	25.0
24(C)	59	1.6	9.4	7.0	18.0
37(L)	65	1.5	3.1	9.1	13.7
37(C)	65	1.3	3.6	9.3	14.2
38(L)	65	1.3	2.0	8.3	11.6
38(C)	65	1.6	2.9	7.1	11.6
Slaughter Run (Canner and Sub-grade Stock)					
3-4 yr. steer (C)	--	ND **	ND	ND	ND
2 yr. steer (C)	--	ND	ND	ND	ND
2 yr. heifer (C)	--	ND	ND	ND	ND
2 yr. bull (C)	--	ND	ND	ND	ND
6-8 yr. cow (C)	--	ND	ND	ND	ND
Old cow	--	ND	ND	ND	ND
2 yr. heifer	--	ND	ND	ND	ND

* (L) - pelvic sample (loin)
(C) - perirenal sample (caul)
** Not detectable (< 0.1 p.p.m.)

Total residues of DDT and its degradation products were lower in the commercial than in the experimental heifers, but they also exceeded the tolerance level (Table 3). The history of the commercial animals before October 4 was incomplete. Residue analyses indicated, however, that yearlings off summer pasture in commercial operations probably reach a prime market weight in the feedlot before DDT from treatments for fly control is degraded to acceptable levels.

Residues were not detected in two animals receiving no treatments, nor in one animal receiving a water spray, nor in one animal receiving injections of eserine. Thus, the residue levels in cattle at the packing plant were not affected by any exposure to DDT after October 4.

Unfinished slaughter-run cattle were graded for canning, cutting or utility carcasses. Most of these animals were known to arrive directly from primary producers and were culled from basic herds normally gathered at this time of year for maintenance on winter range. Failure to detect any residues indicated little or no exposure of herds to DDT on remote summer range.

Summary

Residues were analyzed in the fat of yearlings after exposure on pasture to continuous protection from horn flies with spray treatments of DDT. Eleven applications of 2 quarts of a 0.25 per cent suspension per head produced total residues of DDT, TDE and DDE that exceeded the acceptable tolerance of 7 p.p.m. when animals had reached the prime slaughter-weight on a high-energy finishing ration. More information is needed on the mobilization of fat and the DDT stored within the depot to relate an acceptable tolerance for slaughter cattle to practical methods in beef production.

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