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Gas Chromatographic Determination of Decamethrin Residues in Quail and Quail Eggs

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Previous studies (DAVID 1981, 1982a,b) have shown that technical or formulated decamethrin (NRDC 161) exerts pathological effects on birds. Decamethrin acted upon the brain, inducing behavior abnormalities (tremors, paralysis), and upon the genital tract, reducing principally the embryonic germ potential at early stages of development.

The aim of the present work was to analyse for residues of decamethrin in chicks, adult quails and their eggs after experimental contamination. The treatment was applied either directly by spraying of the shell of the unincubated eggs or indirectly by ingestion of contaminated feed by the parent quails.

MATERIALS AND METHODS

Laboratory-raised quails were used ("Géromoise" breed of Coturnix coturnix japonica).

Samples of formulated pesticide and excipient alone were donated by Roussel-Uclaf Procida, Paris. Any base-catalysed stereoconversions were forestalled by slightly acidifying the solutions used with 0.001% acetic acid.

In the first experimental series, quails were fed for two months with ration sprinkled daily with technical (D) or formulated decamethrin (DE) at the concentration of 10 mg of active substance per 100 g of feed; i.e. 100 ppm. The daily intake of contaminated feed was 30 g; i.e. 3 mg of decamethrin per day and per quail (DAVID 1981, 1982a). Control birds were fed with clean ration (C) or with excipient alone (E) and its main constituent xylene (X) added to diet at the same concentration.

In the second series, unincubated eggs were sprayed with a 2 or 0.05% aqueous suspension of commercial decamethrin (DEs), excipient (Es) or xylene (Xs). The 2% formulated pesticide solution was equivalent to 40 times that encountered in agricultural practice; the amount of active material sprayed on the eggshell was about 25 µg for each egg. The second concentration (0.05%) corresponded to that used against most of

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arthropods enemies of cultivated plants; i.e., 12.5 mg of active substance per liter of water (0.6 µg per egg) (HERVE et al. 1977, PASTRE et al. 1978). After being allowed to dry in open air, eggs were placed in a 38°C ventilated incubator.

Gas chromatographic determination was conducted with a nickel-63 electron capture detector and 3% OV-1 coated on AW DMCS-treated, 80-100 mesh chromosorb W column (3 mm X 1.20 m borosilicate glass). Column, injector and detector temperatures were 230, 245 and 295°C, respectively. Flow rate of argon was 70 mL/min.

Under these operating conditions, chromatographic injection of 1 ng of active substance gave a peak area of 18270 mm^2 for the technical and 14950 mm^2 for the formulated decamethrin, with a retention time of 365 ± 5 sec. At this moment, there was no signal with excipient and xylene. The retention time of the organochlorinated pesticide aldrin (internal standard) was 540 ± 5 sec.

In all the experiments, the amount of residual decamethrin was determined by comparison of the treated sample chromatograms with that of the 1 ng purified pyrethroid.

Extracts of control (C), excipient (E) and xylene (X) contaminated batches of eggs and quails were analysed under the same operating conditions for comparison.

RESULTS AND DISCUSSION

1° - Rate of penetration of decamethrin into eggs.

a) Contamination of feed (Table 1).

Eggs laid by quails fed with technical decamethrin contaminated ration (Df) contained 46 ppb of residual pesticide. With the formulated decamethrin (DEf), the rate of contamination (51 ppb) was close to that found previously. There were pyrethroid residues neither in control eggs nor in excipient or xylene treated samples.

b) Contamination by spraying (Table 2).

After egg spraying with aqueous suspension of 2% commercial decamethrin, the amount of pesticide which passed through the shell into the vitellus and albumin was relatively low; i.e. 17 ppb after 14 days of incubation. With the 0.05% solution, the residual level was still lower (3 ppb).

Thus, even with the higher concentration, the rate of contamination of the sprayed eggs was distinctly lower than that of the eggs laid by quails fed with treated ration.

Eggs of second generation (non-treated again); i.e.,

TABLE 1

Feed contamination with technical (Df) or commercial (DEf) decamethrin, excipient (Ef) and xylene (Xf). Mean weights (g) and pyrethroid residual rates with standard deviation (SD) in adult quails and their egg contents. (C = control series; each mean = 24 determinations)

	C	Xf	Ef	DEf				Df			
	Weight	Weight	Weight	Weight	ng	ppb	± (SD)	Weight	ng	ppb	± (SD)
Egg contents	10.04	10.86	10.53	10.58	562	51	(5)	10.98	505	46	(7)
Fat tissue	9.00	16.00	19.10	15.60	17360	1113	(220)	16.10	15750	978	(132)
Muscular tissue	108.00	113.50	111.20	117.90	2000	17	(11)	122.40	2690	22	(11)
Bone tissue	27.00	28.30	27.80	29.50	3650	124	(30)	30.60	7610	249	(44)
Brain	0.81	0.86	0.80	0.94	18	19	(12)	0.82	89	108	(30)
Heart	2.31	2.14	2.05	2.45	426	174	(24)	2.33	1190	511	(80)
Liver	7.40	7.90	6.70	7.80	491	63	(35)	7.40	89	12	(7)
Lungs	1.95	1.68	1.88	2.20	64	29	(12)	1.92	50	26	(4)
Kidneys	2.15	2.04	2.34	2.17	37	17	(6)	2.13	60	28	(4)
Testicles	4.08	5.85	5.90	5.46	5	1	(2)	5.90	30	5	(3)
Ovary	1.10	1.73	1.83	1.78	128	72	(26)	1.65	94	57	(11)
Genital tract (♀)	8.50	8.60	11.30	10.50	73	7	(2)	10.20	82	8	(2)
Total (1)	165.40	180.50	181.40	187.40	24150	129	(28)	192.60	27630	144	(28)
Feathers	10.50	10.50	10.50	10.50	19890	1894	(280)	10.50	242060	23050	(2300)
Digestive tract	16.90	20.50	18.30	18.70	30210	1607	(212)	18.40	68880	3723	(485)
Total (2)	27.40	31.00	28.80	29.20	50100	1710	(240)	28.90	310940	10720	(1080)
Total (1 + 2) (whole quail)	192.80	211.50	210.20	216.60	74260	343	(40)	221.50	338560	1528	(200)

those laid by quails issued from eggs sprayed with the 2% pesticide suspension, contained only trace amounts of decamethrin.

The newly hatched chicks retained a residual level higher than the eggs (67 and 11 ppb after 2% and 0.05% spraying). This increase would be due to the vitellus resorption and principally to the direct contamination of the chick by rubbing with the treated shell during hatching.

TABLE 2

Contamination by spraying of eggshell with a 2% or 0.05% aqueous suspension of commercial decamethrin (DEs). Mean weight (g) of eggs without shell during the first 14 days of development and chicks one day after hatching. Residual levels of pyrethroid into vitellus and albumin. (Egg F2 = unincubated egg of second generation, non-treated again; n = number of samples analysed; tr = trace amounts of pesticide < 1 ppb).

	DEs (2%)					DEs (0.05%)				
	n	Weight	ng	ppb	±(SD)	n	Weight	ng	ppb	±(SD)
Egg(1/5 d)	18	9.74	68	7	(1.4)	18	10.05	10	1	(0.4)
Egg(12/14 d)	12	9.28	316	34	(5.2)	14	9.78	59	6	(1.2)
Egg(1/14 d)	38	9.61	163	17	(4.2)	40	9.95	30	3	(1.1)
Chick(1 d)	14	8.28	551	67	(9.8)	16	8.23	91	11	(1.9)
Egg F2	20	10.26	4	tr		18	10.65	tr	tr	

2° - Rate of penetration of decamethrin into organs and tissues of adult quails.

For each experimental series, the quantitative analysis was carried upon 24 determinations (12 males and 12 females).

a) Contamination of feed (Table 1).

In case of technical decamethrin diet (Df), the pyrethroid levels varied from one bird to another. The least contaminated were quails showing an aversion for the treated ration. Others, less selective, retained more residues. On the average, the total amount of pesticide reached 1528 ppb.

With the formulated decamethrin (DEf), the

contamination level (343 ppb) was distinctly lower, though the only slight repellent effect of the product.

At first glance, the results seemed paradoxical. Thus, the residual rates of both technical and commercial decamethrin were similar in eggs and greatly different in adult birds. This difference would be due to the gradually accustoming of quails to the technical decamethrin diet. The pesticide stimulated their appetite (3.7 ppm in the digestive tract) and induced abundant salivation responsible for the great residual levels in feathers (23 ppm). The state of dependency produced by the purified pyrethroid was distinctly less marked with the commercial product, probably because the aversion of quails for the excipient odor (DAVID 1981). In this case, the feather and digestive duct residues were significantly lower (1.9 and 1.6 ppm, respectively). Excluding feathers and gut, the average amounts of technical and commercial decamethrin in the whole quails were similar (144 and 129 ppb). The other most contaminated samples were the fat, the heart with its blood contents and the bone tissue. The brain retained also significant amount of residual pesticide, which yielded the pathological behavior of the contaminated quails; i.e. salivation and tremors which, according to ALDRIDGE et al. (1978) are suggestive of an action of the pyrethroid on the brain by modification of acetylcholine and cyclic nucleotic levels.

b) Contamination by spraying (Table 3).

The adult quails issued from eggs sprayed with a 2% aqueous suspension of commercial decamethrin, only retained traces of pesticide. The organs and tissues still significantly contaminated were brain, skeleton and gonads. Nevertheless, the residual rate remained inferior to 1 ppb for the whole bird.

In young second generation quails (non-treated again) trace amounts of pyrethroid were only detected in fat, brain and bone tissue.

Comparative studies of the total mean weights in both control (C) and experimental quails (Xf, Ef, Df, DEf) shown that all the chemicals added to feed induced a significant weight increase. The latter was principally due to that of fat, bone and muscular tissues as also testicles, female genital system (duct and ovary) and, to a lesser degree, digestive tract (DAVID 1982b). Chromatographic measurements shown that the weight variations were not in relation with the contamination rates in the biological samples.

Likewise, the analysis indicated that the amounts of pesticide able to pass through the shell and contaminate the vitellus and albumin, and consequently the chicks and quails issued from the sprayed eggs, were relatively small. Though low, these residual levels strongly reduced the quail embryonic germ potential (DAVID 1982a).

TABLE 3

Contamination by eggshell spraying with a 2% aqueous suspension of xylene (Xs), excipient (Es) and commercial decamethrin (DEs). Mean weights (g) of some organs and tissues of control (C) and experimental adult quails. Residual levels of pyrethroid in the DEs series. (tr = trace amounts of pesticide < 1 ppb).

	C	Xs	Es	DEs (2%)			
	Weight	Weight	Weight	Weight	ng	ppb±(SD)	
Fat tissue	9.00	13.00	12.00	12.00	6	tr	
Muscular t.	108.00	101.40	103.00	103.50	tr	tr	
Bone tissue	27.00	26.10	25.80	25.80	79	3	(1.2)
Brain	0.81	0.83	0.85	0.81	2	2	(0.8)
Heart	2.31	2.22	2.26	2.28	1	tr	
Liver	7.40	7.20	7.50	7.40	tr	tr	
Lungs	1.95	1.95	2.14	1.78	4	2	(0.8)
Kidneys	2.15	1.82	1.85	1.47	3	2	(0.6)
Testicles	4.08	4.54	4.38	4.83	tr	tr	
Ovary	1.10	1.88	1.83	1.92	9	5	(1.8)
Genital tr.♀	9.50	10.60	11.20	11.30	5	tr	
Feathers	10.50	10.50	10.50	10.50	5	tr	
Digestive tr.	16.90	17.10	17.10	19.20	4	tr	
Whole quail	192.80	190.60	191.70	193.80	109	tr	

The rate of residual pesticide was higher in feed contamination than in spraying experiments but, because of its relatively short persistence, the pyrethroid residues detected in the second generation were very small in any case.

In the other hand, the very low amount of decamethrin in the liver suggested a fast hepatic metabolism of the original chemical.

At last, among the tested biological samples, the pesticide was retained preferentially in the brain and gonads, which was according to the pathological troubles induced by the pyrethroid and precedently observed in quails.

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