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THE SUDAN GEZIRA BOARD
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Monitoring the Agricultural Inputs Programme 1985 - 1986

ADDENDUM VOLUME 3

HAZARDS TO THE ENVIRONMENT

THE INTEGRATED PEST MANAGEMENT CONCEPT

JULY 1986

HUNTING TECHNICAL SERVICES LIMITED
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Monitoring the Agricultural Inputs Programme 1985 - 1986

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ERRATA

A number of errors escaped correction in course of preparation of the Main Report volumes. These should be corrected as follows:

Glossary	Block area = 5,000 fd Group area = 35 - 40,000 fd
Summary	Page 1, line 2, insert comma after 1985/86 Page 1, line 15, insert comma after "downwind" Page 1, line 27, "Within" constraints. . . . Page 3, line 9, economic "return" on . . . . .
Chapter 1	Page 2, para. 1.2.1, line 4, "Sennar" Page 5, line 19, . . .the tenants, "with" extension
Chapter 3	Page 52, Table 3.9, line 2, delete "Mean" after Temik treated Page 61, Table 3.12, top col. 1, insert 1985 above Crop
Chapter 6	Page 83, organogram, delete "/" after Assistant Page 89, Table 6.3, line Um Gelala, column vehicles,amend to read 1:8,416
Chapter 7	Page 98, line 32, "selects" Page 107, line 6, "Saving" move to line 7 Page 108, Table 7.6, last column amend to read "cost/fd as % 'of value'" Page 111, line 8, . . .thinning, over-irrigation . . . .
Chapter 8	Page 124, Table 8.4, line 3, delete "/bicron" Page 127, Table 8.8 line 4, col. 2, "chalcidae"
Appendix B	Page 139, Table B.2, col. 2, line 1, $1{,}540^2$ Page 139, Table B.2, col. 2, line 2, $2{,}200^2$ Page 140, Table B.3, col. 5, last line insert 38,000 kg Page 140, Table B.3, col. 6, last line delete 38,000 kg Page 144, Table B.7, col. 1, line 11, 'Deltamethrin' Page 146, Center page formula - insert "division" sign line Page 147, line 5, . . .consistently "above" 70%
Annex A	Page 16, para. 1.3.2, line 8, "microscope" Page 16, para. 1.3.2, line 12, "numbers/cm^2" Page 16, para. 1.3.2, line 15, "handlens" Page 16, para. 1.3.2, line 18, "Figures AA.1-8" Page 16, para. 1.3.2, line 23, "handlens" Page 16, para. 1.3.2, line 27, "aerially applied" Page 19, Figure AA.11, line 3, "Ref. 17" Page 24, line 5, (horizontal "+" vertical ÷ 2) Page 32, bottom line, col. 6, "38%" Page 34, line 1, -text- . . .the "amounts" . . . . .
Annex D	Page 114, Table AD.12, line 5, col. 4, "None"

CHAPTER 1

INTRODUCTION

This Addendum has been necessary due to the delays in analysis of pesticide residues in soil, water and food samples for reasons outlined in Chapter 1.4 of the Main Report. The results of the analyses and the conclusions drawn regarding hazard to man, livestock and the environment from pesticides applied by air to cotton are now discussed in Chapter 2. The opportunity has also been taken to comment on aspects of the Integrated Pest Management concept and its implications for the Gezira and other production Boards, and to make a few suggestions on research work necessary for implementation of such a pest control policy. The programme proposed by ARC Cotton Research Coordinator for 1986-87 season (Ref. 1) has been taken into consideration here. Discussions were also held with the British ODA mission on a cotton pesticide research project that visited the Gezira in 1985 and early 1986.

CHAPTER 2

ASSESSMENT OF ENVIRONMENTAL HAZARD

2.1 INSECTICIDE DRIFT DOWNWIND

2.1.1 General

The subject has been discussed in detail in Chapter 8 of the Main Report and in its Annex A, Part 1. Fifteen spray drift studies were carried out. Results from eight on the Gezira were included in the main report, and those from six on other schemes are given here. Table 2.1, taken from Table AA.4 of the Annex summarises climate conditions and droplet distribution. One trial failed due to mechanical faults in application. Data on methodology for all studies are given in the main report. Interpretation of results for the six studies now reported can only be made as direct assessment of the quantitative g.l.c. analyses. The detailed droplet analyses, which in eight of the studies permitted interpretation of g.l.c. analysis as percentage recoveries of the amounts applied, could not be made for these six studies. In these basic monitoring studies it was not planned to place MgO_2 coated slides in the field, nor were rotating MgO tubes placed adjacent to the acrylic wool collectors, and direct correlation between g.l.c. analysis and field application was outside the scope of the basic monitoring programme.

The results of g.l.c. analyses are shown in graphical form in Figures 2.1 to 2.6 and in Table 2.2. The original data are given as Appendix A. In the Figures the axis showing the amounts of insecticide recovered (μg/m of wool collector) is shown as bands scaled at 1:1, 1:4, 1:17.5 and 1:90 ratios. This has been necessary to permit presentation of non-transformed data which has a wide spread. (It could be transformed to the square-root or logarithm if so desired, e.g. for statistical analysis). As indicated in Table 2.2 wool collectors were placed on the field borders, along the edge of the effective spray-swath of the aircraft, in five of the trials. The amounts of insecticide recovered from these collectors is arguably representative of the amounts that were applied in practice at the downwind edge of the field, assuming cross-field drift to compensate for loss of downwind drift from the field-edge swath. Table 2.3 shows downwind deposition of insecticide as a percentage of field-edge recovery.

2.1.2 Discussion of Results

The amounts of insecticide recovered at 500 - 600 m downwind in these studies were of a similar order of magnitude as in the eight Gezira studies (Table AA.3, Main Report Annex). The amounts recovered by the field-edge collectors were substantially below the estimations for in-field collection for the Gezira trials for the five insecticides represented in both groups of trials, indicating that the method for estimation of in-field deposit is reasonably accurate (Annex A, para. 1.3.1) since it would be expected that in-field levels would be greater than field edges.

The conclusions reached in the Main Report are supported by the additional data generated by the six studies carried out at Rahad, New Halfa and White Nile. Maximum fallout of chemical occurred within 100 m of the field except in the cases of studies 12 and 15. In 12 no sharp decline was noted but by 300 m downwind recovery levels were low. In this ULV study abnormally large droplets were collected on the in-field sensitive papers (Figure AA.13, Survey 4, Study 12) and it is possible this resulted in fewer but relatively larger involatile droplets and thus amounts of chemical, drifting downwind. The results from study 15 are suspect. There is no reason for the rise in recovery levels of both

TABLE 2.1 MONITORING - DROPLET DISTRIBUTION AND DOWNWIND DRIFT - RESUME OF RESULTS
(Taken from Annex A, Table A.A.4)

Study No.	Date	Location	Aircraft	Spray Equipment	Spray Volume per fd	Wind Direction Relative to Spray Run	Wind Strength mps	Meteorology Temp. °C	Meteorology R.H.%	Meteorology I.E.	Distribution/Droplet Collection Collectors Type	Vertical $\tilde{x}$ Drops Cm²	Vertical CV%	Horizontal $\tilde{x}$ Drops Cm²	Horizontal CV%
9	20.09	Rahad	Air Tractor	Micronair	ULV 1.0 L	45°	1.0-2.0	27	72	0.37	O/S	126	60	5	115
10	20.09	Rahad	Air Tractor	Micronair	ULV 1.0 L	45°	1.5-2.0	36	36	1.36	O/S	113	63	14	100
11[3]	24.09	Rahad	Pawnee	Micronair	ULV 1.0 L	90°	2.0-6.0	32	56	0.75	O/S	299	52	10	110
12[3]	12.10	New Halfa	Pawnee	Micronair	ULV 1.5 L	70°	3.0-4.0	32	44	0.92	O/S	280	25	20	180
13[1]	05.10	Rahad	Air Tractor	Whirljet 3	CLV 2.0g	0°	0.5-1.0	31	50	0.78	W/S	75	64	20	87
14	05.10	Rahad	Air Tractor	Whirljet 3	CLV 2.0g	45°	1.5-2.5	36	40	1.21	W/S	118	48	25	104
15[3]	14.11	Rahad	Air Tractor	Whirljet 3	CLV 2.0g	45°	2.5-4.0	30	25	1.22	O/S	152	33	29	48
16[3]	20.12	New Halfa	Pawnee	D8-45	CLV 2.0g	60°	1.0-3.0	25	31	0.98	O/S	97	40	42	59
17[2]	04.11	Rahad	Air Tractor	Whirljet 3	CLV 2.0g	90°	2.0-3.0	28.5	25	1.15	O/S	170	76	33	38
18[2 3]	04.11	Rahad	Air Tractor	Whirljet 3	CLV 2.0g	90°	2.0-3.0	28.5	25	1.15	O/S	387	21	73	48
19[3]	11.12	W. Nile	Turbo Thrush	Micronaire	CLV 2.0g	90°	2.0-2.5	28.5	45	0.94	-	-	-	-	-

Notes:

[1] *CV exclusive of sector between arrows on Figure A.A.10.*

[2] *Aerial Application for Whitefly Control Experiment (Annex A, Part 3).*

mps - Metres per second

EI - Evaporative Index

W/S - Water Sensitive Paper

O/S - Oil Sensitive Paper

CV% - Coefficient of Variation %

[3] *Downwind drift assessment studies*

TABLE 2.2 SUMMARY OF INSECTICIDE COLLECTION BY DOWNWIND WOOL STRAND COLLECTORS (μg/m)

Study No.	Date	Insecticide	Application rate g.a.i/fd[1]	Distance Downwind (m) 0	25	50	100	200	300	400	500	600	700
11	24.09	dimethoate	180	1032.2	133.5	50.9	25.4	14.9	11.4	6.2	-	12.9[2]	-
		fenvalerate	50	548.7	140.2	75.4	51.3	37.8	39.3	20.5	-	36.6[2]	-
12	12.10	chlorpyrifos	360	414.7	353.6	126.7	79.0	30.5	9.3	2.9	-	3.1	2.0
		cypermethrin	21	58.7	52.5	34.6	17.0	13.0	4.2	1.8	-	2.8	1.6
15	14.11	dicrotophos	210	145.3	101.3	40.0	16.0	28.2	31.7	20.3	16.7	10.9	-
		endosulphan	400	544.6	305.8	168.8	110.1	[729.2	-	463.0	364.0	291.5][3]	-
									250m				
16	20.12	triazophos[4]	175	-	599.3	382.7	200.1	-	143.1	-	70.0	-	-
		endosulphan	325	-	927.0	604.9	318.7	-	247.6	-	137.2	-	-
18	4.11	dicrotophos	230*	-	157.3	157.3	143.2	-	79.5	-	42.7	-	-
		endosulphan	385*	-	949.6	909.8	658.5	-	366.5	-	142.6	-	-
19	11.12	amitraz	200*	538.9	473.6	300.4	121.9	-	64.2	-	42.2	-	-
		endosulphan	265*	787.3	612.3	371.5	168.6	-	94.4	-	65.2	-	-

Notes:

[1] *Grams active ingredient/feddan. Rates assumed at standard recommendation except where marked * . These are actual field recorded rates.*

[2] *Collectors sited on top of 5 m high canal bank.*

[3] *[ ] Data in brackets considered questionable.*

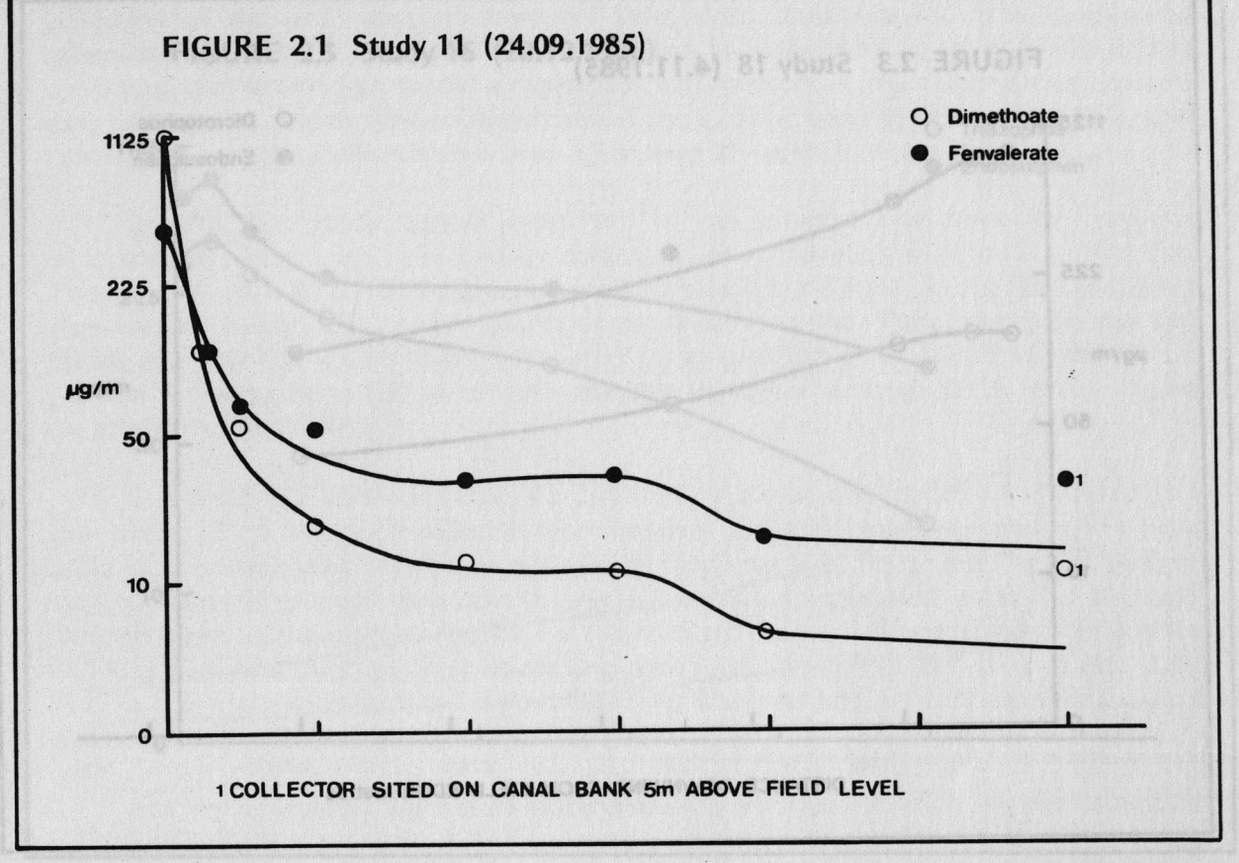
[4] *The control collector removed just prior to spraying showed abnormal contamination at 42.3 μg/m_3 indicating drift from application to another upwind field.*

TABLE 2.3 DOWNWIND DEPOSITION OF INSECTICIDE

Study No.	Insecticide	Downwind deposition as % of field edge recovery Distance Downwind m 25	50	100	200	250	300	400	500	600	700
11	dimethoate	12.9	4.9	2.5	1.4	-	1.1	0.6	-	1.2[1]	-
	fenvalerate	25.5	13.7	9.3	6.9	-	7.2	3.7	-	6.7[1]	-
12	chlorpyrifos	85.3	30.6	19.0	7.4	-	2.2	0.7	-	0.7	0.5
	cypermethrin	89.4	58.9	29.0	22.1	-	7.2	3.1	-	4.8	2.7
15	dicrotophos	69.7	27.5	11.0	19.4	-	21.8	14.0	11.5	7.5	-
	endosulphan	56.2	31.0	20.2	[?	-	?	?	?	?][2]	-
19	amitraz	87.9	55.7	22.6	-	11.9	-	-	7.8	-	-
	endosulphan	77.8	47.2	21.4	-	12.0	-	-	8.3	-	-

Notes: [1] *Collectors sited on top of 5 m high canal bank.*
[2] *[ ] Data unreliable.*

FIGURES 2.1 to 2.6 : Downwind Insecticide drift as micrograms active ingredient collected by one metre long vertical acrylic wool samplers (μg/m). (Vertical axis scale banded).



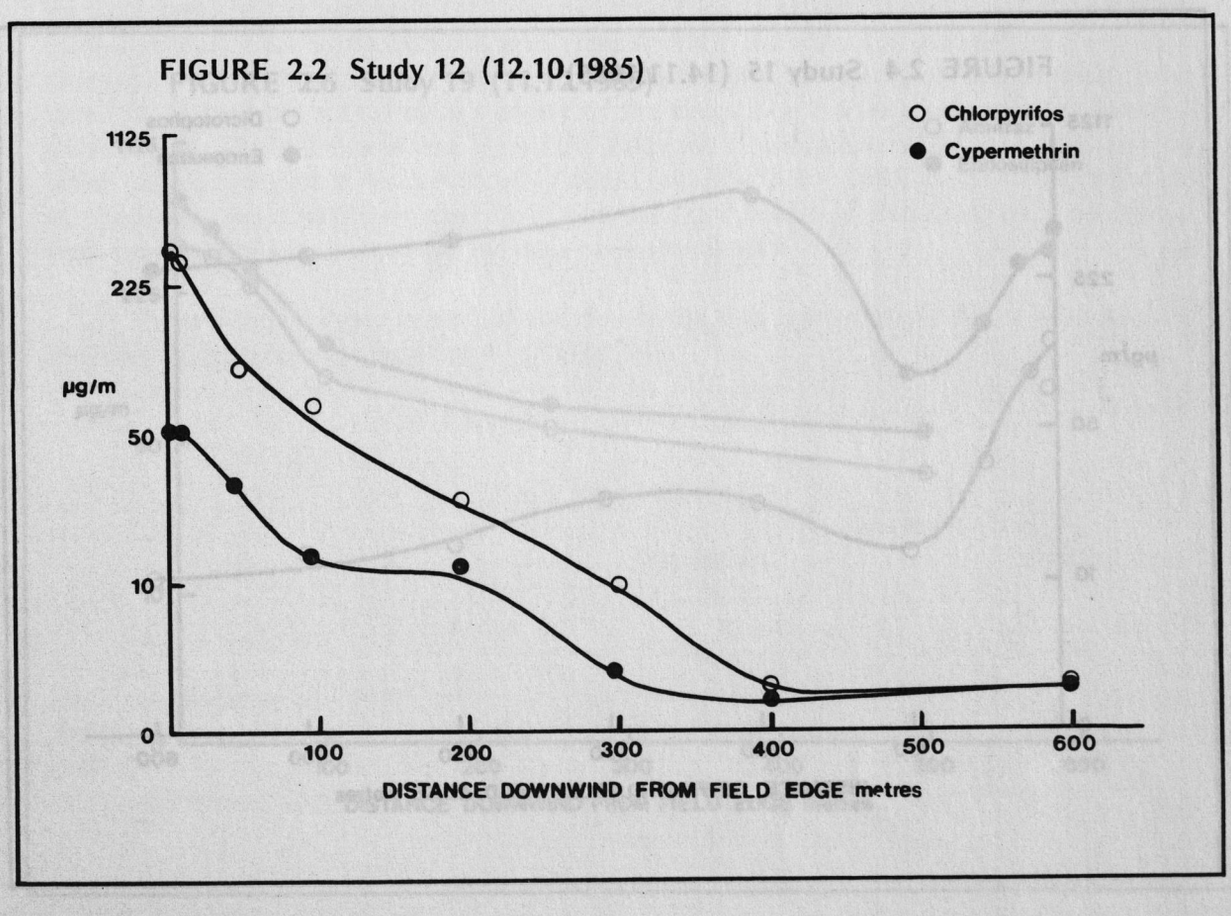


FIGURE 2.3 Study 18 (4.11.1985)

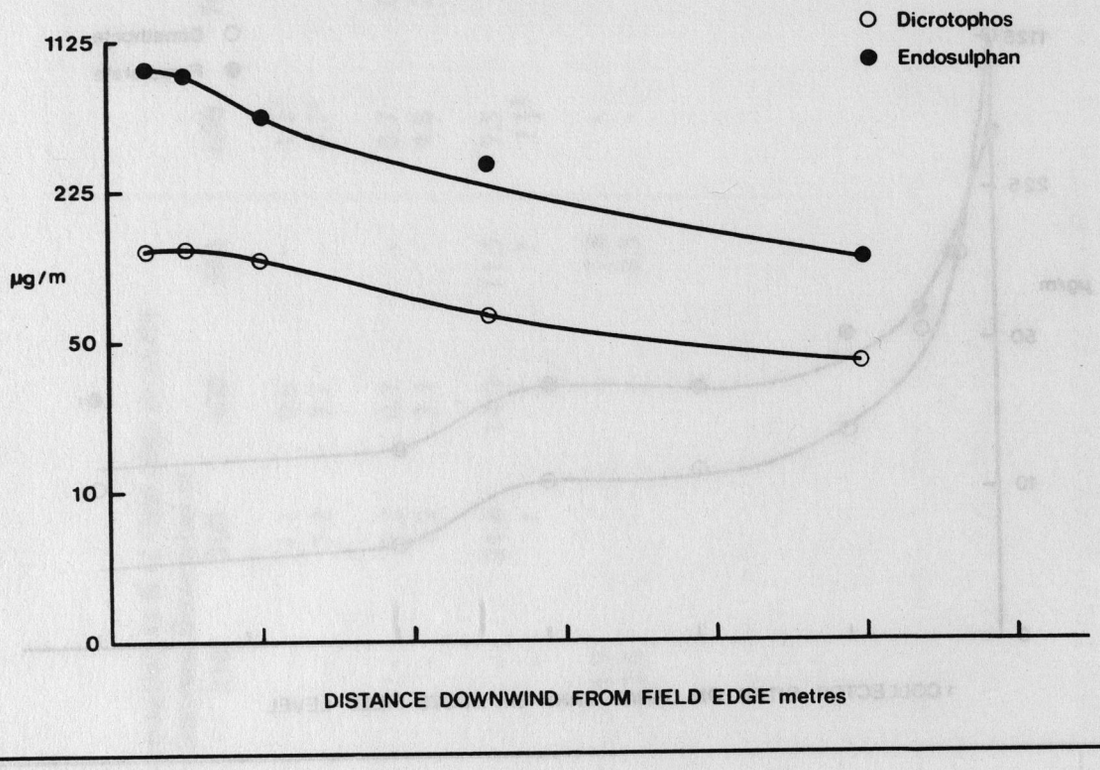


FIGURE 2.4 Study 15 (14.11.1985)

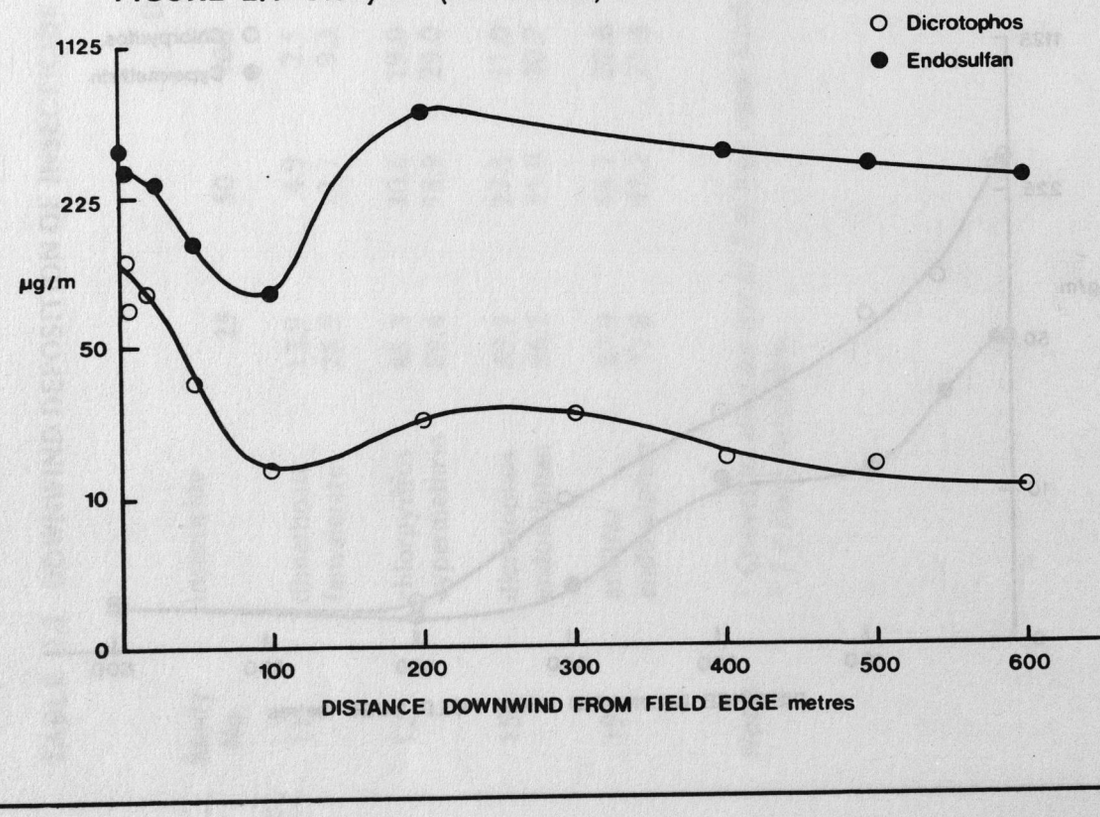


FIGURE 2.5 Study 16 (20.12.1986)

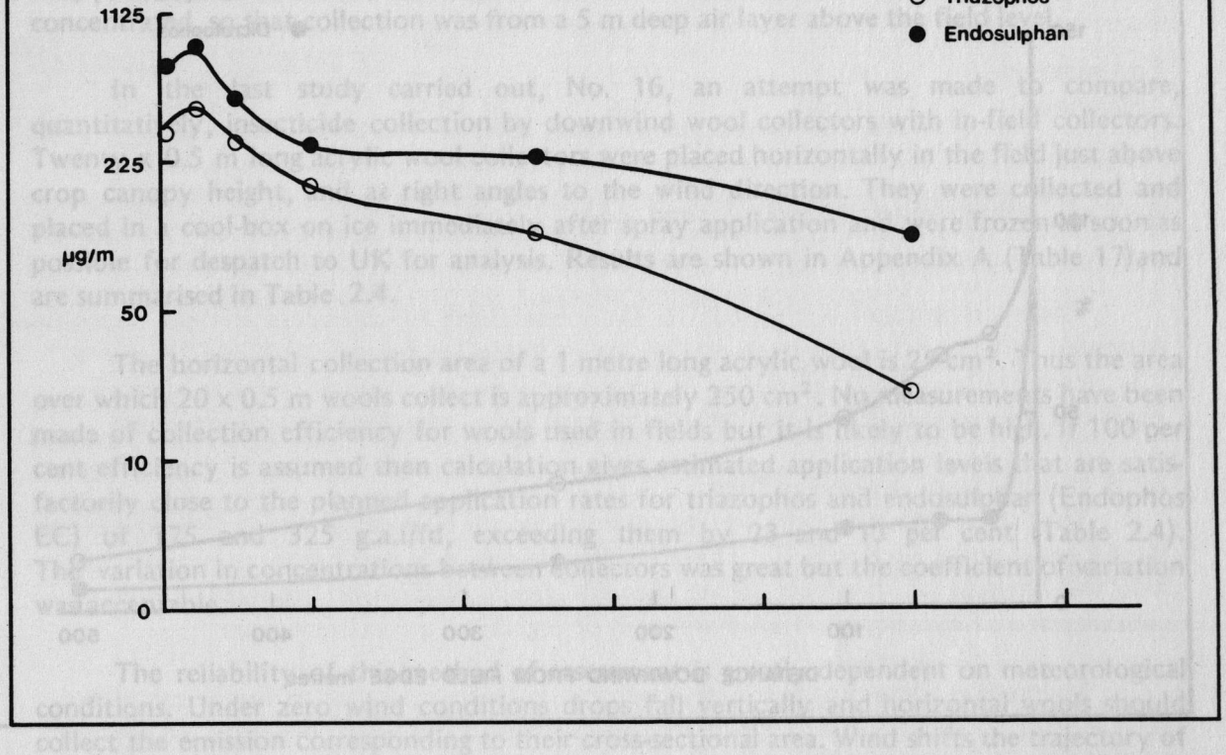


FIGURE 2.6 Study 19 (11.12.1985)

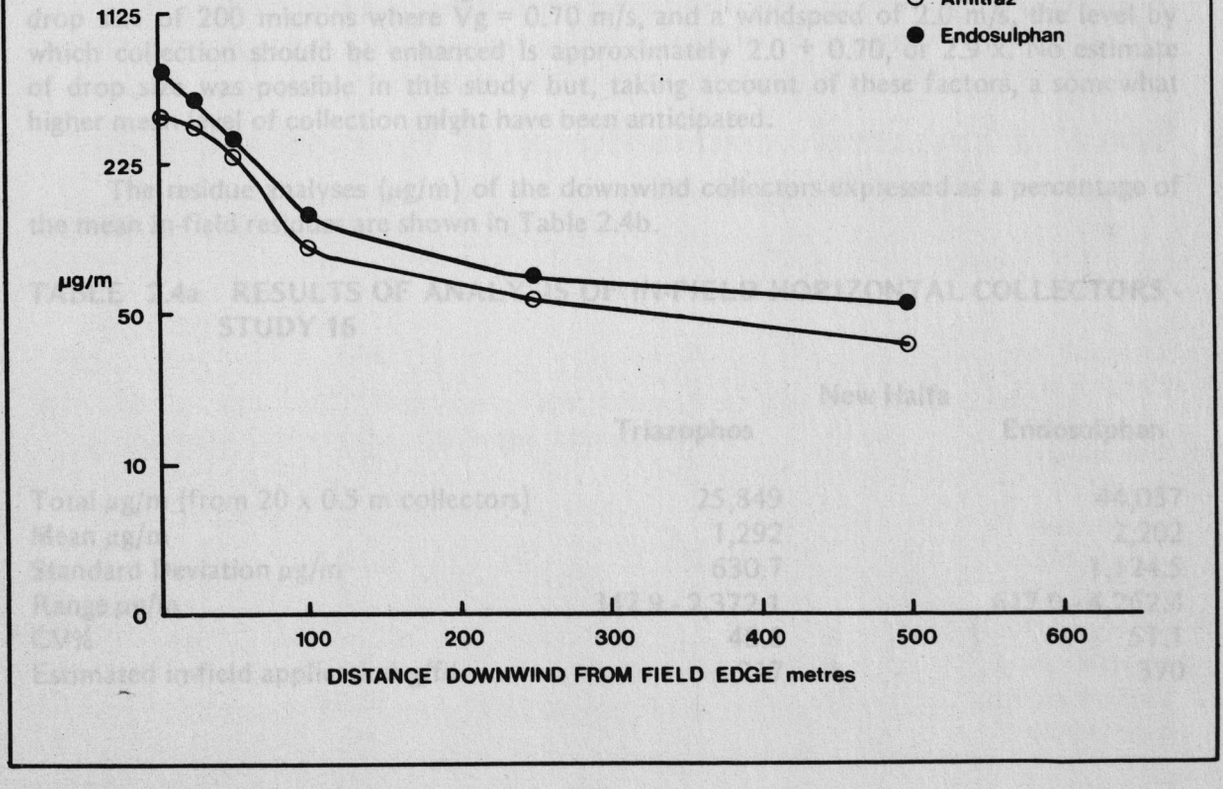
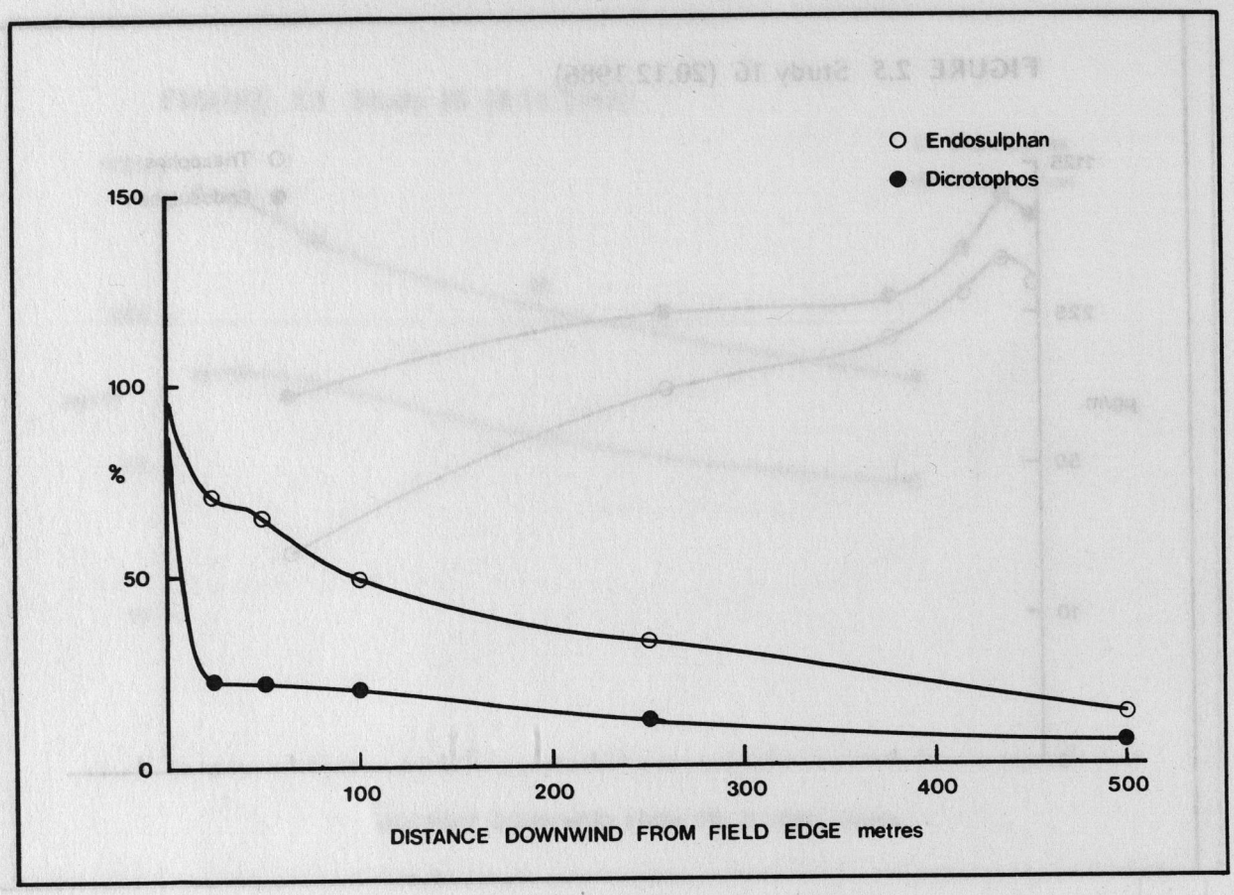


FIGURE 2.7 STUDY 18 (4.11.85)
Downwind insecticide drift as % of mean in-field deposition for one metre long vertically disposed wool strand collectors.



dicrotophos and endosulphan from 200 m downwind other than contamination during collection of the wool samplers from the field, since conditions for spraying appeared reasonably good and no abnormalities were noted in application. In study 11 the 600 m collectors were situated on top of a canal bank and recovery of insecticide showed a slight rise, presumably due to upward movement of the airflow, whereby drifting droplets were concentrated, so that collection was from a 5 m deep air layer above the field level.

In the last study carried out, No. 16, an attempt was made to compare, quantitatively, insecticide collection by downwind wool collectors with in-field collectors. Twenty x 0.5 m long acrylic wool collectors were placed horizontally in the field just above crop canopy height, and at right angles to the wind direction. They were collected and placed in a cool-box on ice immediately after spray application and were frozen as soon as possible for despatch to UK for analysis. Results are shown in Appendix A (Table 17)and are summarised in Table 2.4.

The horizontal collection area of a 1 metre long acrylic wool is 25 cm^2. Thus the area over which 20 x 0.5 m wools collect is approximately 250 cm^2. No measurements have been made of collection efficiency for wools used in fields but it is likely to be high. If 100 per cent efficiency is assumed then calculation gives estimated application levels that are satisfactorily close to the planned application rates for triazophos and endosulphan (Endophos EC) of 175 and 325 g.a.i/fd, exceeding them by 23 and 13 per cent (Table 2.4). The variation in concentrations between collectors was great but the coefficient of variation was acceptable.

The reliability of this method of assessment is greatly dependent on meteorological conditions. Under zero wind conditions drops fall vertically and horizontal wools should collect the emission corresponding to their cross-sectional area. Wind shifts the trajectory of the drops out of the vertical so that the wools collect an enhanced fraction of the emission computed from their cross-sectional area. The stronger the wind the greater the increase in drops collected. This enhanced fraction may be roughly calculated from the mean wind speed, $\bar{u}$, divided by the terminal velocity of the drops, $\bar{V}g$. For example with an 'average' drop size of 200 microns where $\bar{V}g = 0.70$ m/s, and a windspeed of 2.0 m/s, the level by which collection should be enhanced is approximately 2.0 ÷ 0.70, or 2.9 x. No estimate of drop size was possible in this study but, taking account of these factors, a somewhat higher mean level of collection might have been anticipated.

The residue analyses (µg/m) of the downwind collectors expressed as a percentage of the mean in-field residues are shown in Table 2.4b.

TABLE 2.4a RESULTS OF ANALYSIS OF IN-FIELD HORIZONTAL COLLECTORS - STUDY 16

	New Halfa	
	Triazophos	**Endosulphan**
Total µg/m (from 20 x 0.5 m collectors)	25,849	44,037
Mean µg/m	1,292	2,202
Standard Deviation µg/m	630.7	1,124.5
Range µg/m	342.9 - 2,372.1	617.0 - 4,262.4
CV%	48.8	51.1
Estimated in-field application g/fd	217	370

TABLE 2.4b DOWNWIND DEPOSITION AS A % OF IN-FIELD DEPOSITION ON ACRYLIC WOOL COLLECTORS (μg/m) - STUDY 16[1]

	Distance Downwind (m)				
	25	50	100	250	500
Endosulphan	42.1	27.5	14.5	11.2	6.2
Triazophos	46.4	26.6	15.5	11.1	5.4

Note: [1] *Data derived from Table 2.2 and 2.4a*

These percentages are similar to the percentages of estimated in-field deposition presented in Table 8.1 of the main report, and of field-edge recovery in Table 2.4 above. It is therefore concluded that the use of acrylic wool collectors gives a reliable method of assessing downwind drift and that the conclusions drawn regarding hazards are sound.

2.1.3 Hazards from Downwind Drift

The factors affecting drift and its potential hazard to man have been discussed in the Main Report under Section 8.1.3. The principle conclusion is that hazard is minimal and that the chances of ill-effects either through direct contact or indirectly through consumption of contaminated sorghum grain or vegetables are extremely small.

Theoretical contamination levels assuming worse case parameters can be calculated for sorghum grain crops, based on the recoveries from acrylic wool collectors. An average sorghum head in vertical plane occupies approximately 75 cm^2 and may have an average head weight of about 27 grams (mean of 14 varieties reported by Sudan workers). Impact and retention of small drifting droplets by a solid object as a sorghum head is low, due to diversion of the moving airstream. If however a high efficiency is assumed (worst case hypothesis) then a sorghum head would collect three times the amount of insecticide of a one metre long wool strand. Theoretical contamination levels have been calculated on this basis for 250 and 500 m distances and are shown in Table 2.5.

TABLE 2.5 THEORETICAL CONTAMINATION LEVELS OF SORGHUM HEADS BEFORE HARVEST - ppm

Insecticide	No. Studies	250 m	500 m
Endosulphan	8	25.1	14.1
Chlorpyrifos	2	4.6	0.6
Dicrotophos	1	8.8	4.7
Dimethoate	3	5.5	3.1
Quinalphos	1	14.9	5.9
Thiometon	1	3.8	5.0
Triazophos	2	17.9	11.0
Amitraz	2	15.0	5.6
Alphamethrin	1	0.7	0.3
Cypermethrin	2	2.1	1.0
Deltamethrin	1	0.03	0.01
Fenvalerate	1	4.1	2.3

Fields (Numbers) on the Gezira are 280 m wide. Sorghum may be in the adjacent number or the second, occasionally third, number away. Harvest is in the second half of November so that head contamination from four to six sprays is possible. In order to assess whether this level of field contamination might be potentially hazardous to the farmer consumer samples of heads were taken just before harvest and of grain from farm stores approximately ten weeks after harvest. These samples were analysed for residues and results are discussed in Section 2.3.

2.1.4 Drift of Insecticide Study 18 (Ref. Main Report, Annex A.1 and A.3)

The data given in the main report on g.l.c. analysis of insecticide drift, assessed as a proportion of the estimated in-field deposition of wool collectors, excluded that on the trial carried out at Rahad on whitefly control. For sake of completeness these data are given here in Tables 2.6 and 2.7 and were obtained from the standard application treatment. Table 2.2 should be referred to for the amounts of insecticide collected.

Recovery of insecticide drifting downwind was of the same order as the other studies, and showed a similar pattern of drift whereby the great proportion of chemical fall-out occurred within 100 m of the field-edge. Deposition of endosulphan was rather higher than the norm at 250 and 500 m but not very much so. These data further substantiate the conclusions given in the main report.

TABLE 2.6 SUMMARY OF SPRAY DROPLET SIZE AND COVER ON DOWNWIND SAMPLES (STUDY 18,WHITEFLY CONTROL)

Date		Distance Downwind (m)				
		25	50	100	250	500
		Rotary Mg0 Samplers				
4.11	N	2693	2430	1646	2110	456
	Vmd	68	50	53	47	48
	nmd	27	28	26	22	18
	Vol	68	63	51	37	8
		Paper Targets (no/cm²)				
4.11		425	560	352	260	55

Note: N denotes number drops/cm²; Vmd volume mean diameter; Nmd number mean diameter; Vol volume of counted drops in nl/cm².

TABLE 2.7 SUMMARY OF INSECTICIDE COLLECTION BY WOOL SAMPLERS AS PROPORTION OF ESTIMATED DEPOSITION ON AN EQUIVALENT TARGET IN THE SPRAYED AREA[1] (STUDY 18, WHITEFLY CONTROL)

Date	Insecticide	Estimated in-field deposition[2] (μg/m)	Downwind deposition as % of in-field deposition at:				
			25m	50	100	250	500
4.11.86	dicrotophos	713	22.1	22.1	20.1	11.2	6.0
	endosulphan	1359	69.9	66.9	48.5	27.0	10.5

Notes: [1] *Data derived from that presented in Tables 2.6 and 2.2.*
[2] *Calculated for a one metre vertically disposed wool strand, i.e. of the same dimension as the downwind samplers.*

2.2 SOIL RESIDUES

2.2.1 General

Results of soil sample analysis for Temik (aldicarb) residues have been presented in the main report (Ref. Section 3.2.9). Three other fields were sampled twice during the season, and soils were analysed for residues of the range of insecticides which had been applied during the season onto those fields. Results are presented in Table 2.8. Data on application is given in Tables 2.10 and 2.11.

2.2.2 Methodology

Soil samples were taken as two replicates from each field at each sampling date. Sample pits were randomly sited along a line walked at right angles to the field edge and aircraft flight path, inward from the downwind side and about 1/3rd along from the end. Four small pits were dug and bulked for each replicate, two were in the cotton row on the ridge and two were inter-row in the furrow, although by February the ridges had almost flattened. The eight pits extended about halfway across the field. Samples were taken at three depths, 0-3: 3-10: 10-20 cms, and care was taken to avoid contamination between depths. The samples were placed in cotton bags and transported to the laboratory where, after mixing and subsampling of 600-700 grams, the samples were wrapped in aluminium foil, placed in polythene bags and deep frozen. Transportation to UK for analysis was in insulated cool boxes. Details on the gas liquid chromatography procedures are given in Appendix B together with the original analytical data. Control samples were taken from untreated bush, untreated rough grazing and untreated fallow. Results from analyses are given in Table 2.8.

TABLE 2.8 RESIDUES OF INSECTICIDES IN CONTROL AND UNTREATED SAMPLES - ppm

	Control		Rough Grazing	Fallow		
Date Sampled	30/11		30/11	10/12		
Depth	0 - 3	3 - 20	0 - 20	0 - 3	3 - 10	10 - 20
Aldicarb	ND	ND	ND	-	-	-
Amitraz	ND	ND	-	-	-	-
Endosulphan	ND	0.03	0.24	ND	ND	ND
Chlorpyrifos	ND	ND	0.08	ND	ND	ND
Dimethoate			ND			
Profenophos			ND			
Quinalphos			ND			
Thiometon			ND			
Triazophos			ND			
Cypermethrin			ND			
Deltamethrin			ND			
Fenvalerate			ND			

2.2.3 Results and Pollution Hazard

Few of the insecticides left residues that were detectable by routine Gas Chromatography methods and two of those showing readily measurable residues, aldicarb and thiometon, were at low enough concentrations (ppb) not to pose an unacceptable hazard to

the soil micro-organisms. Both are quite highly soluble in water and thus mobile and degradeable (Ref. Main Report, Section 3.2.9). The level of endosulfan residues are high enough to give cause for some concern however.

High levels of endosulfan were recorded from Block 9 Seed Farm for samples taken on November 21st between one and two hours after application of endosulphan plus thiometon (Table 2.10). The level of 2.39 ppm thus reflects accurately the deposition on the soil surface immediately after this sixth application of endosulfan. With seven applications over the season the total surface deposit would be greater than 16.8 ppm, since a greater proportion of early spray would penetrate to the soil surface. Levels in the surface layer had declined to 0.295 ppm after 80 days. The residue levels at lower depths for the November 21st sample are cumulative of five sprays applied between September 30th and November 12th, from which downward movement of the insecticide will have occurred following irrigation, and show similar decline by February. Similar considerations apply to thiometon (Table 2.9).

This fall in residue levels between mid-season and late season sampling dates was found with all insecticides having detectable residues except in Block 37, where no detectable residues were found in the mid-season samples. It is clear from these results that insecticide deposited on the soil during spraying is broken down rapidly over the season and immediately afterward, to very low levels which in general are no threat to the environment in the long-term. A possible exception to this is endosulphan as evidenced by the levels of 240 ppb in rough grazing control and 30 ppb in the 'clean' control sampled from uncultivated bush several kilometers from the nearest sprayed area and presumably due to drift accumulations. There is also an indication that endosulfan may accumulate in the upper 0-3 cm layer in cultivated soils, which is in agreement with its low solubility in water ($\triangleq$ 0.33 mg/l for both isomers at 22°C). Stability to sunlight and heat under Sudan conditions is unknown but is likely to be good to moderate. It would be subject to loss by evaporisation due to relatively high volatility. Little data is available on the persistance of endosulfan in tropical soils but the concern stems from considerations such as its half-life. Five to 45 days has been recorded in Indian soil; from application of 1.5 kg a.i./haa 63% reduction from a 0.55 ppm residual concentration in two months has been recorded (Ref.5).

Information on effects on soil organisms is also sparse from tropical soils. Microbial growth (bacteria and fungi) is inhibited at higher concentrations (400 ppm) but not at low ones (240 ppm) in culture, and at low concentrations degradation by bacteria and fungi is accelerated (Ref. 6). Other workers have demonstrated that in pure culture many organisms can degrade the isomers and breakdown products of endosulfan. Micro-organisms from the rhizosphere of rice fields showed insignificant changes in population after 20 days exposure. (Conc. unknown). Nitrogen fixation by *Rhizobium* from *Phaseolus aureus* and breakdown of organic matter were unaffected.

The physical and biological results from the intensive repetitive spraying of endosulfan plus potentiating compounds practised by the Sudan cotton producing boards will depend on soil type and environmental factors. Under a less intensive system breakdown should be rapid, taking account of the relative solubility of endosulfan as compared with other organo-chlorines, its volatility and the temperature and light intensity of the Gezira.

It is concluded that no significant hazard exists to soils micro-organisms or processes from aldicarb, thiometon or the other insecticides as currently used on the Gezira excepting possibly from endosulfan. More work on effects on non-target soil organisms in these soils is necessary before drawing conclusions on this compound.

TABLE 2.9 RESIDUES OF INSECTICIDES IN SOIL - ppm (Mean of Two Bulked Samples)

	Block 37 Turabi						Block 7 Wad Shannan						Block 9 Seed Farm						Detection Limits
Season	Mid			Late			Mid			Late			Mid			Late			
Date Sampled	10/12			5/2			30/11			4/2			21/11			9/2			
Depth cm	0 - 3	3 - 10	10 - 20	0 - 3	3 - 10	10 - 20	0 - 3	3 - 10	10 - 20	0 - 3	3 - 10	10 - 20	0 - 3	3 - 10	10 - 20	0 - 3	3 - 10	10 - 20	ppm
Insecticide																			
Aldicarb	-	-	-	ND			0.15[1]	0.03[1]	0.04[1]	0.04	ND	ND	ND			ND			0.02
Amitraz	-	-	-	-	-	-	-	-	-	-	-	-	ND			ND			0.05
Endosulphan	ND			0.04	0.05	0.04	0.19	0.03	0.04	0.06	0.02	ND	2.39	0.12	0.04	0.295	0.02	ND	0.01
Chlorpyriphos	ND			ND			0.05	ND	0.025	ND						ND			0.02
Dimethoate	ND			ND			ND			ND						ND			0.1
Profenophos	ND			ND			ND			ND						ND			0.04
Quinalphos	ND			ND			ND			ND						ND			0.01
Thiometon	ND			ND			0.45	ND	0.41	ND			0.33	ND	ND	ND			0.01
Triazophos	ND			ND			0.04	ND	ND	ND			ND			ND			0.04
Cypermethrin	ND			ND			ND			ND						ND			0.02
Deltamethrin	ND			ND			ND			ND						ND			0.02
Fenvalerate	ND			0.05	ND	ND	ND			ND						ND			0.05

Notes: [1] *Sample date 15-10-85*

ND: *Not Detected below limit of detection (end column).*

-: *Not analysed.*

TABLE 2.10 INSECTICIDE APPLICATIONS ON SAMPLED FIELDS

No.	Block 37 Turabi		Block 7 Wad Shannan		Block 9 Seed Farm	
Air Sprayed						
1.	8-10	fenvalerate dimethoate	19/9	profenophos	30/9	endosulphan dimethoate
2.	20/10	endosulphan triazophos	30/9	deltamethrin dimethoate	14/10	endosulphan quinalphos
3.	5/11	endosulphan chlorpyriphos	9/10	deltamethrin dimethoate	22/10	endosulphan amitraz
4.	12/11	endosulphan triazophos	21/10	cypermethrin	2/11	endosulphan triazophos
5.	20/11	endosulphan quinalphos	6/11	endosulphan triazophos	12/11	endosulphan profenophos
6.	30/11	endosulphan chlorpyriphos	16/11	endosulphan thiometon	21/11	endosulphan thiometon
7.	15/12	endosulphan fenvalerate	25/11	endosulphan thiometon	5/12	endosulphan amitraz
8.	8/1	endosulphan fenvalerate	30/11	endosulphan chlorpyriphos		
9.		-	16/12	endosulphan profenophos		
10.		-	25/12	endosulphan chlorfenvinphos		
Soil Applied						
		-	2 - 10/10	aldicarb		-

TABLE 2.11 A.R.C. RECOMMENDED RATES g.a.i./fd

aldicarb	1,160	cypermethrin	15 - 25
amitraz	200	deltamethrin	7
endosulphan	400[1]	fenvalerate	50
chlorpyriphos	360		
dimethoate	180		
profenophos	280		
quinalphos	190		
thiometon	200		
triazophos	176		

Note: [1] *rate with: dimethoate 350 g thiometon 375 g*
quinalphos 375 g triazophos 325 g

2.3 INSECTICIDE RESIDUE IN FOODSTUFFS

2.3.1 General

The possibility that insecticide drift when spraying cotton will contaminate neighbouring sorghum crops or vegetables grown near to villages, which is an increasingly important farming activity, was assessed. Unfortunately only a total of 10 samples could be checked since analysis had to be made in the United Kingdom.

2.3.2 Methodology

Duplicate sets of samples were taken in the same Blocks as were sampled for soil residue analysis, and near the villages where blood cholinesterase levels were being monitored.

Mid-season sorghum samples were taken by cutting several heads just prior to harvest from edges of fields. The grains were cut from the main stem with minimal disturbance, bulked and packed in aluminium foil placed in plastic bags and frozen. As soon as possible the frozen heads were transported to UK in insulated cool boxes. In February, 10 weeks after harvest, grain samples were taken from farm stores in villages near to the sampled blocks and processed in the same way.

Vegetables were sampled from fields in the same areas where possible and frozen as for sorghum.

2.3.3 Results and Hazard Assessment

Results are summarised in Tables 2.12 and 2.13 with full data in Appendix C. The schedule of sprays applied are given in Table 2.10 for Blocks 37, 7 and 9, and in Table 2.14 for Blocks 5, 20 and 95.

These data indicate the potential for contamination of the sorghum samples quite accurately, but less so for vegetables. The Gezira recommended rates of application are shown in Table 2.11.

- Sorghum

  In one sample of grain from sorghum heads taken in the middle of the spray season relatively high residues of four chemicals were found. In the stored grain, however, all residues were very low or below the limit of detection. Of these four chemicals the levels of endosulphan, triazophos and chlorpyrifos were of the order indicated by theoretical levels estimated in Section 2.1.3. From Table 2.14 it will be seen that six sprays of endosulphan had been made during the previous months on Block 5 and one of these plus chlorpyrifos was given the day before sampling, while triazophos had been sprayed two weeks prior. This indicates a reasonable relationship between residues and field application, and with estimated contaminations (Table 2.5). However, residues of the fourth chemical, amitraz, cannot be related. It was not sprayed at all in Block 5 according to records and the high residue level is perplexing.

  The levels of amitraz found in stored grain at 0.05, 0.19 and 0.17 ppm are well below the Maximum Residue Limit set by CODEX at 0.5 mg/kg amitraz plus metabolites. Residues in grain after milling and cooking would be further reduced. The Acceptable Daily Intake has been set (1984 JMPR) at 0.003 mg/kg/day which provides a maximum permissable intake of 0.18 mg/day for a 60 kg person. Workers in Sudan may consume 600 grams of grain per day, which at the residue levels found would contain 0.084 mg, under half the permissable intake, without taking account of further breakdown in milling and cooking.

TABLE 2.12 RESIDUES IN SORGHUM GRAIN - ppm

Season	Control	Block 37 Turabi		Block 5/7 South		Detection Limits
		Mid	Late	Mid	Late	
Date Sampled	25/11/85	21/11/85	5/2/85	20/11/85	4/2/86	
Sample Nos.	28	29	36/38	30	35	
Insecticide						
Aldicarb	ND	-	-	ND	ND	0.02
Amitraz	0.08	0.20	0.05/0.19	26.25	0.17	0.05
Endosulfan	0.07	0.20	0.01/0.07	3.55	0.11	0.01
Chlorpyrifos	ND	ND	ND	1.40	ND	0.01
Chlorfenvinphos	ND	ND	ND	0.16	ND	0.01
Dimethoate	0.12	0.04	0.07/0.06	0.11	0.11	0.02
Fenitrothion	ND	ND	ND	ND	ND	0.01
Profenofos	ND	ND	ND	ND	ND	0.01
Quinalphos	ND	0.05	ND	0.30	ND	0.005
Thiometon	ND	ND	ND	0.03	ND	0.005
Triazophos	ND	0.02	0.02/ND	3.51	0.02	0.01
Cypermethrin	ND	ND	ND	0.03	0.07	0.02
Deltamethrin	ND	ND	ND	0.04	ND	0.02
Fenvalerate	0.02	ND	ND/0.04	ND	ND	0.02

Notes: [1] *Two varieties samples from two farm stores.*

ND: Not detected at limits of detection.

Control sample taken from untreated sorghum near Sennar, south of Gezira scheme.

TABLE 2.13 RESIDUES IN VEGETABLES - ppm

Site	Okra			Bird Peppers/Aubergine		Cucumber
	Block 95 Control	Block 95	Block 9	Block 20	Block 7	Block 9
Date Sampled	26/11/85	29/11/85	4/2/86	3/12/85	4/2/86	29/11/85
Insecticide						
Amitraz	0.05	ND	ND	0.14	ND	ND
Endosulfan	0.02	0.12	ND	0.25	ND	ND
Chlorpyrifos	ND	ND	ND	0.02	ND	ND
Chlorfenvinphos	ND	ND	ND	ND	ND	ND
Dimethoate	ND	ND	ND	ND	ND	ND
Fenitrothion	ND	ND	ND	ND	ND	ND
Profenofos	ND	ND	ND	0.06	ND	ND
Quinalphos	ND	ND	ND	ND	ND	ND
Thiometon	ND	ND	ND	ND	ND	ND
Triazophos	0.01	ND	ND	0.02	ND	ND
Cypermethrin	ND	ND	ND	ND	ND	ND
Deltamethrin	ND	ND	0.03	ND	ND	ND
Fenvalerate	ND	ND	ND	ND	ND	ND

Note: ND: Not detected at limits of detection.

TABLE 2.14 INSECTICIDE APPLICATIONS TO SAMPLED BLOCK AREAS

No.	Block 5 South		Block 95 South		Block 20 Messalamia	
1	28/9	Endosulfan* Dimethoate	26/9 OR	Profenofos Chlorpyrifos	24/9	Profenofos
2	10/10	Deltamethrin Dimethoate	6/10	Deltamethrin Dimethoate	7/10	Deltamethrin Dimethoate
3	21/10	Endosulfan Thiometon	18/10	Endosulfan Amitraz	16/10	Endosulfan Triazophos
4	29/10	Endosulfan Chlorfenvinphos	26/10	Deltamethrin	30/10	Endosulfan Quinalphos
5	4/11	Endosulfan Triazophos	1/11 OR 6/11	Fenvalerate Endosulfan & Triazophos	4/11	Endosulfan Chlorpyriphos
6	11/11	Endosulfan Quinalphos	23/11	Endosulfan Thiometon	11/11	Endosulfan Profenofos
7	20/11	Endosulfan Chlorpyriphos	29/11	Endosulfan* Chlorpyriphos	18/11	Endosulfan Chlorpyriphos
8	27/11	Endosulfan* Chlorpyriphos	4/12	Endosulfan* Amitraz	28/11	Endosulfan Amitraz
9	3/12	Endosulfan Dicrotophos	15/12	Endosulfan Chlorenvinphos	4/12	Endosulfan* Amitraz
10	19/12	Endosulfan Chlorfenvinphos	29/12	Endosulfan* Fenvalerate	11/12	Endosulfan* Chlorfenvinphos
11	3/1/86	Endosulfan Fenvalerate		-	22/12	Endosulfan* Amitraz
12		-		-	30/12	Endosulfan* Fenvalerate

** Frame spray to edges of numbers only.*

Table 2.15 shows similar estimations for the other detected insecticides. Tolerance levels for endosulphan residues in grain sorghum appear not to be established but for wheat and barley are 0.1 mg/kg and for maize is 0.2 mg/kg. From this it appears that pesticide residues in sorghum grain resulting from airspray to cotton pose no hazard to man or livestock.

- Vegetables

The analytical results for vegetables show most insecticide residues to be below the limits of detection. Endosulphan residues detected in peppers and okra sampled in mid season are well below the tolerance levels established by FAO or other countries for similar vegetables (tomato, cucumber, aubergines) at 0.5 to 2.0 mg/kg. Again these appear to cause no hazard to consumers.

TABLE 2.15 ESTIMATED INTAKE OF PESTICIDE RESIDUES IN SORGHUM GRAIN BY A TYPICAL SUDANESE WORKER

	Detected Residue PPM	No. Samples	Estimated Intake (as total[1]) mg/day	Estimated Intake (by bodyweight[2]) mg/kg
Amitraz	0.14	3	0.084	0.0014
Endosulphan	0.06	3	0.036	0.0006
Dimethoate	0.08	3	0.048	0.0008
Triazophos	0.02	2	0.016	0.0003
Cypermethrin	0.07	1	0.042	0.0007
Fenvalerate	0.04	1	0.024	0.0004

Note: [1] *Assumed average daily consumption as 600 g sorghum as dough (kisra; asida; etc).*
[2] *Assumed weight of worker 60 kg.*

- Grain eating birds

A possible hazard to migrant and indigenous grain-eating birds exists, including species classed as pests. The degree of hazard is difficult to assess as data on No Effect Levels (NEL) for the comounds under consideration could not be obtained, and in fact does not exist in sufficient detail to forecast toxicity for the range of species which may be involved. If the data available for quail and pheasant are considered there appears to be a low level of hazard. Assuming quail weigh 250 g and eat 50 g sorghum grain per day, and taking the highest residue figures, they would consume about 0.17 mg triazophos or 0.015 mg quinalphos. The LD50 values are 1.1 mg and 7.5 mg. While there is no hazard with quinalphos there seems to be a slight possibility that triazophos might accumulate to toxic levels where the quail feeds repetitively for a week or more. This depends on the rate of breakdown in the bird. Where a 1 kg pheasant is assumed to eat 200 g grain per day it would consume 0.024 mg dimethoate or 0.7 mg endosulphan. The LD50 values here are 15 mg and 620 mg, far in excess of the amount consumed, with both chemicals being rapidly metabolised. In general the degree of hazard to bird life seems very low but the possibility of adverse effects on reproduction should be borne in mind.

LD50 data (Ref. Pesticide Manual)

Amitraz	(Quail)	1800 mg/kg diet
Dimethoate	(Pheasant)	15 mg/kg
Endosulphan	(Pheasant)	620-1000 mg/kg
Quinalphos	(Quail)	150 mg/kg diet
Triazophos	(Quail)	42-27.1 mg/kg

2.4 INSECTICIDE RESIDUE IN WATER

2.4.1 General

The principle source of water for human consumption on the Gezira is now from boreholes, reported to average 30-40 m deep, but workers will not hesitate to drink from canals when working in the fields. On other production areas water from canals is rather more frequently drunk. In order to assess the level of contamination likely to be encountered water from a canal passing a small village, and from a household standpipe in the village was sampled for analysis.

At Geiger in the south White Nile production area the pesticide store is sited badly with high probability of contamination of a minor canal passing 10 m from old leaking drums, from which water for domestic purposes is drawn 20 m and 200 m downstream. This was considered the most seriously hazardous storage site encountered, and a water sample was drawn from the canal to check contamination.

2.4.2 Methodology

Water samples were drawn directly into new liquor bottles thoroughly washed out with iso-propyl alcohol. The bottles were kept in an efficient domestic refrigerator until they could be transported to UK for analysis in insulated cool boxes, except for sample W9, from White Nile which was frozen until four days before despatch. One sample, W4, was broken in transit. Unfortunately it was not possible to arrange for the samples to be extracted by collaborators in Sudan since the required solvents could not be obtained in time, and as a result the numbers of samples and hence sites had to be severely reduced.

2.4.3 Results and Hazard Assessment

Results of analyses are summarised in Table 2.16, with full data in Appendix D. The canal sampled, Abu Galil, was in Block 20. Table 2.14 details the chemicals applied over the season.

No aldicarb was used in Messallamia Group area in 1985, and none has ever been used in the White Nile cotton area. A routine analysis was however carried out with no residues being detected. The question of aldicarb residues in village water sources is further discussed in Main Report Annex B1, giving results of the survey carried out by the Consortium for International Crop Protection in which they conclude that the chance of aldicarb reaching village water sources near to fields in which aldicarb has been used is remote.

Over the three month period sampled, mid to late season, no detectable residues were found in tap water, except endosulphan at 0.01 ppb in one sample. There seems to be little chance of insecticide residues leaching into the groundwater to cause environmental contamination nor any hazard from use of borehole water by man or livestock.

The highest levels of residues reported from canal water would also not be hazardous to man. Where a 60 kg man is assumed to drink 5 l water per day his intake of insecticide would fall well below the Acceptable Daily Intake levels set for drinking water as follows:

	A.D.I.	For 60 kg man	Actual Intake
Endosulphan	0.008 mg/kg	0.48 mg/man	0.049 mg
Chlorpyrifos	0.01 mg/kg	0.60 mg/man	0.015 mg
Dimethoate	0.02 mg/kg	1.20 mg/man	0.001 mg
Thiometon	0.003 mg/kg	0.18 mg/man	0.005 mg
Profenofos	None Established	-	0.006 mg

TABLE 2.16 RESIDUES OF INSECTICIDE IN WATER USED FOR HUMAN CONSUMPTION - ppb

	Tap - Abu Galil, Gezira				Canal - Abu Galil, Gezira				Geiger, W.Nile	Limit of Detection
	1985		1986		1985		1986		1986	
	3/12	18/12[1]	5/2	26/2	3/12	18/12	5/2	26/2		
Insecticide										
Aldicarb[2]	-	-	ND	ND	-	-	ND	ND	ND	0.005 ppm
Amitraz	ND	-	ND	ND	ND	ND	ND	ND	ND	0.05 ppb
Endosulphan	0.01	-	ND	ND	2.82	9.71	ND	0.01	ND	0.01 ppb
Chlorpyriphos	ND	-	ND	ND	0.97	3.02	ND	0.02	ND	0.02 ppb
Dimethoate	ND	-	ND	ND	0.12	0.26	ND	ND	0.15	0.10 ppb
Profenofos	ND	-	ND	ND	1.13	0.19	ND	ND	ND	0.05 ppb
Quinalphos	ND	-	ND	ND	0.35	0.11	ND	ND	ND	0.01 ppb
Thiometon	ND	-	ND	ND	0.08	0.96	ND	0.08	ND	0.01 ppb
Triazophos	ND	-	ND	ND	0.32	0.14	ND	ND	ND	0.05 ppb
Cypermethrin	ND	-	ND	ND	ND	ND	ND	ND	0.13	0.05 ppb
Deltamethrin	ND	-	ND	ND	ND	ND	ND	ND	ND	0.02 ppb
Fenvalerate	ND	-	ND	ND	ND	ND	ND	ND	ND	0.05 ppb

Notes: [1] *Sample bottle broken in transit.*

[2] *Analyses expressed as parts per million.*

2.5 OVERALL HAZARD ASSESSMENT - WAD SHANNAN VILLAGE, BLOCK 7 SOUTH GROUP

In their proposal the Consultants planned to select two Blocks, in the north and south areas of the Gezira, in which an in-depth hazard assessment would be carried out. Problems in coordination of the airspray monitoring studies with other project activities and with commercial application problems made this impossible in the north, but it was satisfactorily undertaken in South Group.

The village of Wad Shannan was selected since it is small and situated halfway along a cotton number so that the spray aircraft overflew the village each pass. While spray application was cut off each time hazard from drift and direct contamination was inveitably higher. The Numbers adjacent to cotton on the south, downwind side were fallow, which allowed easy drift assessment. Finally there was a dispensary near by with an exceptionally able and well known medical assistant who organised a village blood cholinesterase monitoring programme.

Overall hazard assessment is based on the following studies:

(i) Villagers Blood Cholinesterase Monitoring - 153 tests

(ii) Downwind Insecticide Drift - two studies

(ii) Soil Sample Residue Analysis - aldicarb

(iv) Soil Sample Residue Analysis - general

(v) Vegetable Residue Analysis - general

(vi) Sorghum Grain Residue Analysis - general

Sorghum head and other vegetable samples were obtained in neighbouring blocks, but unfortunately no water samples were taken in the area, due to limitations on numbers of analyses possible. Results on sections (ii) to (vi) have been discussed in detail in the main report and other parts of this volume.

Table 2.17 summarises the data on blood tests. Both the Indirect Contact group, which comprised farmers and field workers, and the No Contact group showed sharp declines in the percentage of normal (100 per cent) test results as compared to the average for all tests in the Southern Gezira area for the same groups. This was no doubt due to the location of the village. However since this drop was only to 87.5 per cent and 75 per cent of normal levels there is no cause for concern regarding the general health of the villagers and farmers of Wad Shannan . None of the villagers returned levels below 75 per cent of normal. These results substantiate the conclusions drawn in Sections 2.2, 2.3 and 2.4 of this volume, and in Chapter 8 of the main report, that air-spraying as practised at present in the Sudan Gezira poses a low level of hazard to villagers, the general public and the general environment, with those reservations already stated regarding the repetition use of endosulphan.

TABLE 2.17 SUMMARY BLOOD CHOLINESTERASE TESTS - WAD SHANNAN

Numbers of Persons Tested

Date 1985	Percentage of Normal Blood Cholinesterase Level								
	100	87.5	75	62.5	50	37.5	25	12.5	Total
Indirect Contact									
17.11	3	4	4	-	-	-	-	-	11
24.11	9	12	2	-	1	-	-	-	24
1.12	7	11	2	-	-	-	-	-	20
8.12	3	4	5	-	-	-	-	-	12
15.12	1	3	1	-	-	-	-	-	5
22.12	9	11	10	6	3	-	-	-	39
	32	**45**	**24**	**6**	**4**	-	-	-	**111**
% Total Tests	29	40	22	5	4	-	-	-	100
Average % S. Gezira	44	30	21	4	1	-	-	-	100
No Contact									
1.12	3	2	6	-	-	-	-	-	11
8.12	1	2	3	-	-	-	-	-	6
15.12	3	8	4	-	-	-	-	-	15
22.12	-	5	5	-	-	-	-	-	10
	7	**17**	**18**	-	-	-	-	-	**42**
% Total Tests	17	40	45	-	-	-	-	-	100
Average % S. Gezira	47	34	19	-	-	-	-	-	100

Note: The data for Wad Shannan are included in Table AD.2 in the Annex to main report, from which Gezira South average % figures are taken.

CHAPTER 3

THE APPLICATION OF INTEGRATED PEST MANAGEMENT (IPM) TECHNIQUES TO SUDAN COTTON

3.1 BACKGROUND

The SGB Crop Protection Department have proposed that an integrated pest management strategy should be already implemented as the basis for control of cotton pests by 1990. In fact some components of such a system have been practised for many years, for example the close season for cotton on the Gezira and introduction of hairy Acala varieties having jassid resistance (but also being susceptible to whitefly). However application of chemicals remains the major method of control at present. Experience over the last 10 years shows that, unless there is a change in the fundamental approach to pest control, cotton production will cease to be economic in the Sudan. This has been emphasised by many visiting experts and an FAO/UNEP project to set up a basis for introduction has been operating for about six years in collaboration with ARC, Wad Medani. Field scale implementation of IPM will require some fundamental changes by SGB field staff, not only in practice but in the attitude and approach to pest control. It is therefore useful to define the IPM concept and assess what changes its introduction may impose on SGB and other Corporations.

3.2 THE CONCEPT

The concept of integrated pest control envisages the use of all possible control methods to reduce insect pest damage to a level that is economically acceptable. This is opposed to reliance on schedules of insecticide sprays as the principle way of control. Integrated control includes the use of resistant or tolerant varieties, the manipulation of sowing dates, the adoption of agronomic practices that avoid excessively verdant growth, biological control - both natural and artificial, (the use of viruses, fungi and bacteria), and that most basic practise of cotton sanitation, the close season. The concept accepts that the use of insecticides will be an integral part of any control systems, provided that their use is as and when pest populations rise to levels requiring application despite other control measures. Thus the timing of insecticide application is critical and dependant on a knowledge of that population of a particular pest which will result in yield loss or quality reduction, if not controlled. As noted in Chapter 7 of the main report this population level may be termed the economic threshold if sufficiently detailed knowledge is available. Alternatively, and more practically, it may be termed the 'action level' or 'action threshold' (Sterling and Lincoln 1977, Main Report, Ref. 32) because by definition of 'economic threshold' a great number of variables have to be taken into account and the threshold will vary from season to season. The complexity may be illustrated by noting some of the variables that have been taken into account by Sterling and Lincoln (1977). These apply to most pest control situations, but here *Heliothis armigera* is specifically considered. The variables include:

1. Variation of climate and its effect on the development of the cotton plant and on *Heliothis* and other pest populations

2. Cotton variety

3. Economic factors that may fluctuate annually:
   - sale price of cotton
   - cost of application of insecticides
   - cost of alternative control measures
   - other production costs

4. Cropping practises

5. Consideration of measures to delay onset of insecticide resistance in the *Heliothis* population

6. Influence of insecticide spraying on potential biological control organisms and on potential secondary pest development

7. Variation in potential yield associated with variable soil fertility

8. Fluctuation of *Heliothis* adult populations and their movement between host crops. (This may be monitored by use of pheromone traps)

9. Spatial distribution of larvae on the plant related to growth stage, i.e. which part of plant is chosen for sampling, and to non-random oviposition (clumping or aggregation)

10. The relationship between boll-loss caused by *Heliothis* and other factors such as boll-rot, *Fusarium*, nematodes and other insect pests

11. The efficiency of the sampling system. If this is inadequate the whole concept collapses since data on populations is unreliable

Some of the factors are already taken into consideration by SGB (Main Report, Appendix I, Ref. 6).

The scope for application of IPM has been discussed by the Consultants in an earlier project in 1983 (Main Report, Appendix I, Ref. 7) with regard to whitefly, as well as by a number of other authorities, and warrant no further discussion here.

3.3 IMPLEMENTATION BY SGB

3.3.1 Scouting

Timing of applications is critical to IPM. The first step has been taken already in that 'Marginal, Partial or Selective spraying is recommended' and this has lead to reduction of spraying, particularly early and late in the season as compared to blanket spraying of whole Numbers when the spray threshold is exceeded. The expression of the potential of beneficial insects to aid pest control can only be realised by delaying early season applications as long as possible. Delaying early application can only be instituted where (i) a good knowledge of the pest populations and (ii) the early season spray action thresholds is obtained. The second of these is the responsibility of research. The first will require general upgrading of the scouting system as has been discussed in Chapter 7 of the main report. An efficient, scheme-wide, uniform scouting system is critical to introduction of IPM. It will involve SGB in increased expenditure on recruiting permanent scouts, training, transportation and possibly communications.

3.3.2 Agronomic Practises

A number of these have great influence on development of whitefly, notably application of nitrogen, length of the planting season, thinning and uniformity of irrigation. The most critical is the cut-off date for irrigation. These have all been discussed in the main report and thus need no elaboration here, but their acceptance, notably of earlier cessation of irrigation, will require a major extension exercise to educate farmers, and even of management staff at all levels.

3.3.3 Resistant Varieties

Varieties resistant to whitefly are being bred in Sudan and the first, Sudac K, has been released. It has not been popular with farmers (Dr H. Khalifa, Pers. Com.) because of the lower yield when compared to Acala varieties. This implies that prices in relation to variety and quality, as expressed by reduced stickiness, will have to be reviewed. Changes in pricing policy by the Cotton Production Corporation will be needed. Again a major farmer education programme would seem necessary, which will need to include appropriate changes in cultural practices.

3.3.4 Biological Control - Beneficial Insects

If the potential of parasites and predators are to be realised the policy on insecticide selection may have to be altered, as well as delaying early season spraying to the economically justifiable maximum. The present policy requires that the same mixture may not be applied more than once to any Block during one season. This is designed to postpone development of resistance in the insect population.

Recent experience is that certain insecticides and mixtures are particularly potent against beneficials. The Decis/dimethoate ULV mixture is suspect while other insecticides need to be screened for potency. If one or more chemicals or modes of application cause little disruption of beneficials then these should be preferred and their use repetitively will need consideration. Examples are:

- selective chemicals e.g. pirimicarb for aphids
- short residual effect with stomach action
- soil or seed treatment with systemics
- strip or spot spraying

Full introduction of each of these would require changes, or more rigorous implementation, of SGB policies.

Assessment of potential for biological control can only be properly investigated by allocation of a substantial block of cotton to be left unsprayed and observed over several seasons. (Ref. Main Report, Section 8.3.5).

CHAPTER 4

RESEARCH REQUIRED

It is not within the Terms of Reference of this project to comment on research needs but at informal discussions with the Acting Director of the Agricultural Research Council at Wad Medani it was suggested comments could be useful. Subsequently the 1986 draft programme for cotton research was passed to the Consultants. The following suggestions are made on the basis of information and experience acquired over the 1985-86 season by the Consultants.

4.1 ACTION THRESHOLDS

The need for the present thresholds to be reviewed and researched has been discussed fully in the Main Report. The outline of a major research project, 'Establishment of the Economic Thresholds for the Major Cotton Pests', is included in the ARC programme as small scale replicated field trials. If the selected levels of infestation can be maintained in the plots this will provide valuable information. It is suggested that large scale field trials should also be undertaken in which treatment with insecticides would vary according to pest population levels. Two series of trials could be carried out, Early and Late season. In the early season (September to early November) interferance by jassid with *Heliothis* could be diminished by use of a selective systemic, while in the late season trial similar use of pirimicarb would reduce interferance by aphid on the whitefly damage. Standard Treatments would follow or precede the trial period.

4.2 SCOUTING SYSTEMS

The system used in Sudan has been discussed in detail in the main report and recommendations have been made to improve the system and results obtained. Anticipating the introduction of IPM systems and the greater need for precise pest counts it is suggested that sequential scouting using charts based on in-depth knowledge of population dynamics and pest-related damage. Development of these plans would take several years in absence of data. There should however be much valuable data in SGB crop protection department dating over many years and research personnel could process these data to prepare initial sampling plans.

It is appropriate here to again emphasise the importance of uniform scouting procedures with specially trained teams being used for any pesticide performance assessments as well as for formal trials.

4.3 INSECTICIDE SCREENING IN THE LABORATORY

The introduction of routine insecticide screening is recommended as being critical to assessment of insecticides within an IPM programme. Bioassay of insecticides helps to determine:

- the most effective compounds
- possible onset of insecticide resistance and cross resistance
- the quality of carryover stocks of insecticide
- which chemicals have least effect on beneficial insects

The immediate result of this screening would be to cut down the number of new chemicals or formulations going through to large scale field trials, thereby releasing staff for

more important work and cutting costs. A further important aspect is that screening continues throughout the year, thereby permitting more data to be accumulated. One advantage of using laboratory reared insects is that a standard population is used for all basic assessment and that conditions can be carefully controlled.

4.4 BENEFICIAL INSECTS

Three coleopterous predators were identified in the field studies as being of potential value for biological control. These are *Scymnus marginalis, Coccinella rufresceus* and *Cydonia vicina,* and they represented 61 per cent of the predators collected in the sweep net sampling. It is recommended that investigation be undertaken into their efficacy as predators and if this proves satisfactory the possibility of artificial rearing and mass release should be examined.

More general recommendations for research are given in Section 8.3.5 of the main report.

4.5 GROUND APPLICATION

Certain areas of cotton can never be sprayed by air, e.g. adjacent to power lines and telephone cables. Air spray contractors are supposed to spray these from the ground but this is seldom carried out. Control of whitefly by airspraying is clearly often poor and sometimes totally ineffective. The use of ground application methods should be re-examined. This has become a practical proposition now that tractors using very low pressure tyres have been developed together with spray equipment that applies insecticides spraying upwards from near the ground to give good under surface cover to the leaves. It is recommended that these possibilities be investigated for better whitefly control.

4.6 RATES OF APPLICATIONS AGAINST WHITEFLY

In view of the difficulties of controlling whitefly adults with residual insecticides and the tolerance of whitefly nymphs to all insecticides, the possibility of controlling mobile adults seems worth examination, since some newer chemicals have a considerable "knock-down" effect. These can be applied as ULV oil formulations drift sprayed across 200-300 m swaths, permitting low overall rates of application. Daily spraying at these low rates aimed at killing the adults as they emerge should lead to rapid population reductions by eliminating egg laying. Daily spraying using 200 or 300 m swath widths should more or less equate with weekly spraying at 25 m swaths. It is suggested this possibility be investigated to ascertain how long daily spraying would have to continue to reduce populations to an acceptable level, and how long it takes before spraying has to start again. It is emphasised that rates of application could be reduced to a few per cent of present levels and that similar methods have been successfully tried in other crops.

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APPENDIX A

RESULTS OF GLC ANALYSES OF INSECTICIDES ON ACRYLIC WOOL COLLECTORS

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RESULTS OF GLC ANALYSES OF INSECTICIDES ON ACRYLIC WOOL COLLECTORS

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DEPARTMENT OF ANALYTICAL CHEMISTRY

CERTIFICATE OF ANALYSIS

The Determination of Concentrations of Insecticides in Acrylic Wool Collectors

HTS/2

5th March 1986

Registered Office: Woolley Road, Alconbury, Huntingdon, Cambs. PE18 6ES Registered in England No. 1815730

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HRC Project Number: HTS/2.

Date of commission: This work was commissioned by Mr. C. R. Whetnall of Hunting Technical Services Ltd on 12th November 1985.

Samples: Samples were received between 22nd October 1985 and 17th February 1986 in a good condition and stored at -20°C prior to analysis.

Study: Residue analysis.

Method: The samples were analysed by soxhlet extraction for 2 hours with dichloromethane. Extracts were concentrated using Kuderna-Danish evaporator containing 1 ml of toluene as keeper. Extracts were made up to volume (2.0 ml) with 2,2,4-trimethylpentane and retained for quantitation by gas-liquid chromatography using conditions detailed in Appendix 1.

Results: The results obtained, uncorrected for control or procedural recovery values, are shown in Tables 1-19. Procedural recovery values, corrected for appropriate control residues, are shown in Tables 20-31.

Results are reported as micrograms insecticide found per metre length of acrylic wool (µg/m).

Endosulphan is reported as the total of alpha and beta isomers.

Procedural recoveries were carried out using commerically available acrylic wool, previously cleaned by soxhlet extraction with both acetone and dichloromethane. On each occasion a 3.0 m length of acrylic wool was spiked with a 1.0 ml solution of appropriate insecticide mixture. The solvent was allowed to evaporate and the sample extracted and analysed concurrently with the field treated samples.

As control samples supplied from Sudan were contaminated by the compounds sprayed, results from HRC control samples are also reported.

Typical analytical chromatograms are shown in Figures 1-12.

Chemistry Laboratory Notebook Reference: 2421/86/06

TABLE 1

Concentrations of Dimethoate and Fenvalerate found in acrylic wool collectors

Study 11

HRC Reference	Sample Reference	Dimethoate (µg/m)	Fenvalerate (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/7599	R_3.0 5 m length	1032.2	548.7
85/7600	R_3.20 5 m length	133.5	140.2
85/7601	R_3.50 5 m length	50.9	75.4
85/7602	R_3.100 5 m length	25.4	51.3
85/7603	R_3.200 5 m length	14.9	37.8
85/7604	R_3.300 5 m length	11.4	39.3
85/7605	R_3.400 5 m length	6.2	20.5
85/7606	R_3.600 4 m length	12.9	36.6

TABLE 2

Concentrations of Chlorpyrifos and Cypermethrin found in acrylic wool collectors

Study 12

HRC Reference	Sample Reference	Chlorpyrifos (µg/m)	Cypermethrin (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/7590	6NH.0 5 m length	414.7	58.7
85/7591	6NH.20 5 m length	353.6	52.5
85/7592	6NH.50 5 m length	126.7	34.6
85/7593	6NH.100 5 m length	79.0	17.0
85/7594	6NH.200 5 m length	30.5	13.0
85/7595	6NH.300 5 m length	9.3	4.2
85/7596	6NH.400 5 m length	2.9	1.8
85/7597	6NH.600 5 m length	3.1	2.8
85/7598	6NH.700 5 m length	2.0	1.6

TABLE 3

Concentrations of Dimethoate and Cypermethrin found in acrylic wool collectors

Study 1 (Replicate 1)

HRC Reference	Sample Reference	Dimethoate (µg/m)	Cypermethrin (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8620	Control HTS 5 m length	1.04	0.18
85/8621	R_1G_1.25 2 m length	465.4	154.5
85/8622	R_1G_1.25 3 m length	429.3	144.9
85/8623	R_1G_1.50 2 m length	397.8	134.1
85/8624	R_1G_1.50 3 m length	352.6	146.7
85/8625	R_1G_1.100 2 m length	216.6	69.1
85/8626	R_1G_1.100 2 m length Sample 2	221.6	81.7
85/8627	R_1G_1.100 3 m length Sample 2	208.2	79.6
85/8628	R_1G_1.100 3 m length	214.2	68.4
85/8629	R_1G_1.250 2 m length	111.2	36.7
85/8630	R_1G_1.250 3 m length	92.5	31.0
85/8631	R_1G_1.250 2 m length	62.2	15.4
85/8632	R_1G_1.250 3 m length	55.4	15.7

TABLE 4

Concentrations of Dimethoate and Cypermethrin found in acrylic wool collectors

Study 1 (Replicate 2)

HRC Reference	Sample Reference	Dimethoate (µg/m)	Cypermethrin (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8620	Control HTS 5 m length	1.04	0.18
85/8633	R_2G_1.25 2 m length	358.8	167.2
85/8634	R_2G_1.25 3 m length	457.9	196.5
85/8635	R_2G_1.50 2 m length	317.8	149.6
85/8636	R_2G_1.50 3 m length	712.9	211.9
85/8637	R_2G_1.100 2 m length	560.6	167.6
85/8638	R_2G_1.100 3 m length	359.9	116.4
85/8639	R_2G_1.250 2 m length	238.8	78.6
85/8640	R_2G_1.250 3 m length	214.2	68.9

TABLE 5

Concentrations of Amitraz and Endosulphan found in acrylic wool collectors

Study 2 (Replicate 1)

HRC Reference	Sample Reference	Amitraz (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8641	G_2^2A.25 3 m length	452.3	817.8
85/8642	G_2^2A.50 3 m length	408.6	679.9
85/8643	G_2^2A.100 3 m length	517.0	826.6
85/8644	G_2^2A.200 3 m length	173.0	271.0
85/8645	G_2^2A.400 3 m length	44.8	70.2

TABLE 6

Concentrations of Amitraz and Endosulphan found in acrylic wool collectors

Study 2 (Replicate 2)

HRC Reference	Sample Reference	Amitraz (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8646	G_2^2B.25 3 m length	744.6	1373.0
85/8647	G_2^2B.50 3 m length	876.6	1516.8
85/8648	G_2^2B.100 3 m length	673.4	1085.7
85/8649	G_2^2B.200 3 m length	239.2	366.5
85/8650	G_2^2B.400 3 m length	73.0	107.0

TABLE 7

Concentrations of Decamethrin found in acrylic wool collectors

Study 3

HRC Reference	Sample Reference	Decamethrin (µg/m)
86/0464	Control HRC	<0.05
85/8651	Control HTS 3 m length	<0.05
85/8652	3G.25 3 m length	4.1
85/8653	3G.50 3 m length	2.9
85/8654	3G.100 3 m length	2.1
85/8655	3G.250 3 m length	0.33
85/8656	3G.500 3 m length	<0.05

TABLE 8

Concentrations of Dimethoate and Alphamethrin found in acrylic wool collectors

Study 4

HRC Reference	Sample Reference	Dimethoate (µg/m)	Alphamethrin (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8657	Control HTS 3 m length	341.2	29.4
85/8658	4G.25 3 m length	355.6	31.4
85/8659	4G.50 3 m length	146.9	23.7
85/8660	4G.100 3 m length	127.6	18.4
85/8661	4G.250 3 m length	34.3	6.4
85/8662	4G.500 3 m length	17.4	2.41

TABLE 9

Concentrations of Dicrotophos and Endosulphan found in acrylic wool collectors

Study 18

HRC Reference	Sample Reference	Dicrotophos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8663	Control HTS 3 m length	2.8	14.7
85/8664	5R.25 3 m length	157.3	949.6
85/8665	5R.50 3 m length	157.3	909.8
85/8666	5R.100 3 m length	143.2	658.5
85/8667	5R.250 3 m length	79.5	366.5
85/8668	5R.500 3 m length	42.7	142.6

TABLE 10

Concentrations of Triazophos and Endosulphan found in acrylic wool collectors

Study 5

HRC Reference	Sample Reference	Triazophos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8669	Control HTS 3 m length	14.8	10.1
85/8670	6G.25 3 m length	585.5	784.1
85/8671	6G.50 3 m length	547.4	719.2
85/8672	6G.100 3 m length	289.8	371.1
85/8673	6G.250 3 m length	179.1	102.1
85/8674	6G.500 3 m length	127.3	124.4

TABLE 11

Concentrations of Quinalphos and Endosulphan found in acrylic wool collectors

Study 6

HRC Reference	Sample Reference	Quinalphos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/8675	Control HTS 3 m length	25.2	62.2
85/8676	7G.25 3 m length	439.9	675.5
85/8677	7G.50 3 m length	299.2	444.4
85/8678	7G.100 3 m length	405.4	572.4
85/8679	7G.250 3 m length	134.2	205.0
85/8680	7G.500 3 m legnth	53.2	78.1

TABLE 12

Concentrations of Thiometon and Endosulphan found in acrylic wool collectors

Study 7

HRC Reference	Sample Reference	Thiometon (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9142	Control HTS 3 m length	5.7	55.1
85/9143	8G.25 3 m length	35.4	647.9
85/9144	8G.50 3 m length	30.4	522.2
85/9145	8G.100 3 m length	36.9	549.2
85/9146	8G.250 3 m length	34.5	384.4
85/9147	8G.500 3 m length	45.3	364.0

TABLE 13

Concentrations of Chlorpyrifos and Endolsuphan found in acrylic wool collectors

Study 8

HRC Reference	Sample Reference	Chlorpyrifos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9148	Control HTS 3 m length	0.44	0.60
85/9149	9G.25 3 m length	321.1	366.6
85/9150	9G.50 3 m length	552.8	632.1
85/9151	9G.100 3 m length	373.9	423.5
85/9152	9G.250 3 m length	74.0	85.1
85/9153	9G.500 3 m length	7.2	15.2

TABLE 14

Concentrations of Dicrotophos and Endosulphan found in acrylic wool collectors

Study 15

HRC Reference	Sample Reference	Dicrotophos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9449	R9.000 5 m length	145.3	544.6
85/9450	R9.005 5 m length	92.9	389.4
85/9451	R9.020 5 m length	101.3	305.8
85/9452	R9.050 6 m length	40.0	168.8
85/9453	R9.100 5 m length	16.0	110.1
85/9454	R9.200 5 m length	28.2	729.2
85/9455	R9.300 5 m length	31.7	-*
85/9456	R9.400 5 m length	20.3	463.0
85/9457	R9.500 5 m length	16.7	364.0
85/9458	R9.600 5 m length	10.9	291.5

* Sample too dirty to chromatograph and resolve peak of interest

TABLE 15

Concentrations of Amitraz and Endosulphan found in acrylic wool collectors

Study 19

HRC Reference	Sample Reference	Amitraz (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9516	Control HTS 3 m length	0.79	0.64
85/9517	1K.0 3 m length	538.9	787.3
85/9519	1K.25 3 m length	473.6	612.3
85/9518	1K.50 3 m length	300.4	371.5
85/9520	1K.100 3 m length	121.9	168.6
85/9522	1K.250 3 m length	64.2	94.4
85/9521	1K.500 3 m length	42.2	65.2

TABLE 16

Concentrations of Triazophos and Endosulphan found in acrylic wool collectors

Study 16

HRC Reference	Sample Reference	Triazophos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9509	Control HTS 0.68 m length	42.3	1.70
85/9510	NH10.005 3 m length	494.4	810.3
85/9511	NH10.025 3 m length	599.3	927.0
85/9512	NH10.050 3 m length	382.7	604.9
85/9513	NH10.100 3 m length	200.1	318.7
85/9514	NH10.250 3 m length	143.1	247.6
85/0515	NH10.500 3 m length	70.0	137.2

TABLE 17

Concentrations of Triazophos and Endosulphan found in acrylic wool collectors

Study 16

HRC Reference	Sample Reference	Triazophos (µg/m)	Endosulphan (µg/m)
86/0464	Control HRC	<0.05	<0.05
85/9523/ 1	NH10 0.5 m length	674.7	1151.4
85/9523/ 2	NH10 0.5 m length	1413.7	3031.1
85/9523/ 3	NH10 0.5 m length	1150.2	2110.9
85/9523/ 4	NH10 0.5 m length	970.3	1575.7
85/9523/ 5	NH10 0.5 m length	1981.0	3684.3
85/9523/ 6	NH10 0.5 m length	342.9	617.0
85/9523/ 7	NH10 0.5 m length	673.0	1184.2
85/9523/ 8	NH10 0.5 m length	2063.0	3684.3
85/9523/ 9	NH10 0.5 m length	352.8	598.7
85/9523/10	NH10 0.5 m length	1411.0	2364.6
85/9523/11	NH10 0.5 m length	1969.2	3438.2
85/9523/12	NH10 0.5 m length	1474.0	2374.6
85/9523/13	NH10 0.5 m length	1129.8	1508.2
85/9523/14	NH10 0.5 m length	1957.5	2911.3
85/9523/15	NH10 0.5 m length	1426.4	2283.1
85/9523/16	NH10 0.5 m length	1667.5	2995.5
85/9523/17	NH10 0.5 m length	521.7	858.5
85/9523/18	NH10 0.5 m length	489.9	817.0
85/9523/19	NH10 0.5 m length	1808.4	2586.3
85/9523/20	NH10 0.5 m length	2372.1	4262.4

TABLE 18

Concentrations of Thiometon found in acrylic wool collectors

Study 20

HRC Reference	Sample Reference	Thiometon (µg/m)
86/0464	Control HRC	<0.05
86/0975	Control HTS 3 m length	0.76
86/0976	Kosti 2.5 3 m length	0.14
86/0977	Kosti 2.25 3 m length	0.17
86/0978	Kosti 2.50 3 m length	0.12
86/0979	Kosti 2.100 3 m length	0.07
86/0980	Kosti 2.250 3 m length	<0.05
86/0981	Kosti 2.500 3 m length	<0.05

TABLE 20

Procedural Recovery Data

Alphamethrin in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	219.24	108
86/0464	1315.44	103

TABLE 21

Procedural Recovery Data

Amitraz in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	191.07	114
86/0464	382.14	75
86/0464	955.35	94

TABLE 22

Procedural Recovery Data

Chlorpyrifos in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	200	90
86/0464	200	100
86/0464	600	102
86/0464	1000	101
86/0464	2000	98

TABLE 23

Proceudral Recovery Data

Cypermethrin in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	220	86
86/0464	220	98
86/0464	440	96
86/0464	660	99
86/0464	1100	95
86/0464	2200	99

TABLE 24

Procedural Recovery Data

Decamethrin in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	11.68	102
86/0464	233.64	89

TABLE 25

Procedural Recovery Data

Dicrotophos in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	210.8	100
86/0464	632.4	85
86/0464	1054.0	87

TABLE 26

Procedural Recovery Data

Dimethoate in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	203.94	96
86/0464	203.94	101
86/0464	407.88	96
86/0464	1019.7	102

TABLE 27

Procedural Recovery Data

Endosulphan in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	9.65	106
86/0464	19.3	97
86/0464	193	96
86/0464	193	104
86/0464	193	105
86/0464	193	111
86/0464	386	71
86/0464	386	85
86/0464	579	87
86/0464	579	88
86/0464	579	107
86/0464	579	107
86/0464	772	96
86/0464	772	102
86/0464	965	81
86/0464	965	90
86/0464	965	102
86/0464	965	105

TABLE 29

Procedural Recovery Data

Quinalphos in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	210.87	90
86/0464	843.48	106

TABLE 28

Procedural Recovery Data

Fenvalerate in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	200.76	104
86/0464	1003.8	102

TABLE 30

Procedural Recovery Data

Thiometon in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	222.13	77
86/0464	222.13	93
86/0464	1110.65	82
86/0464	1110.65	93
86/0464	2221.3	85

TABLE 31

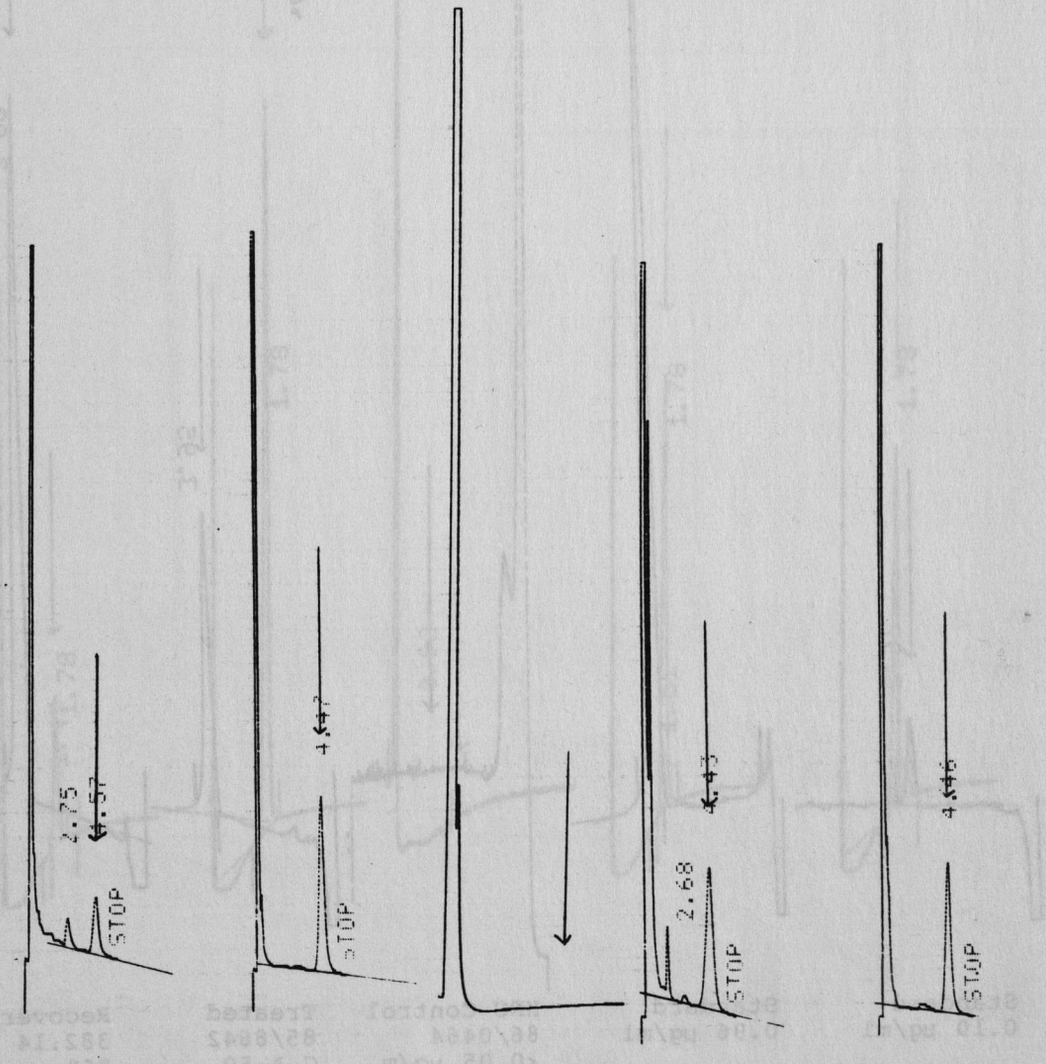
Procedural Recovery Data

Triazophos in acrylic wool

HRC Reference	Level of fortification, (µg)	Recovery (%)
86/0464	20.02	96
86/0464	20.02	103
86/0464	200.2	99
86/0464	200.2	105
86/0464	600.6	96
86/0464	600.6	99

FIGURE 1

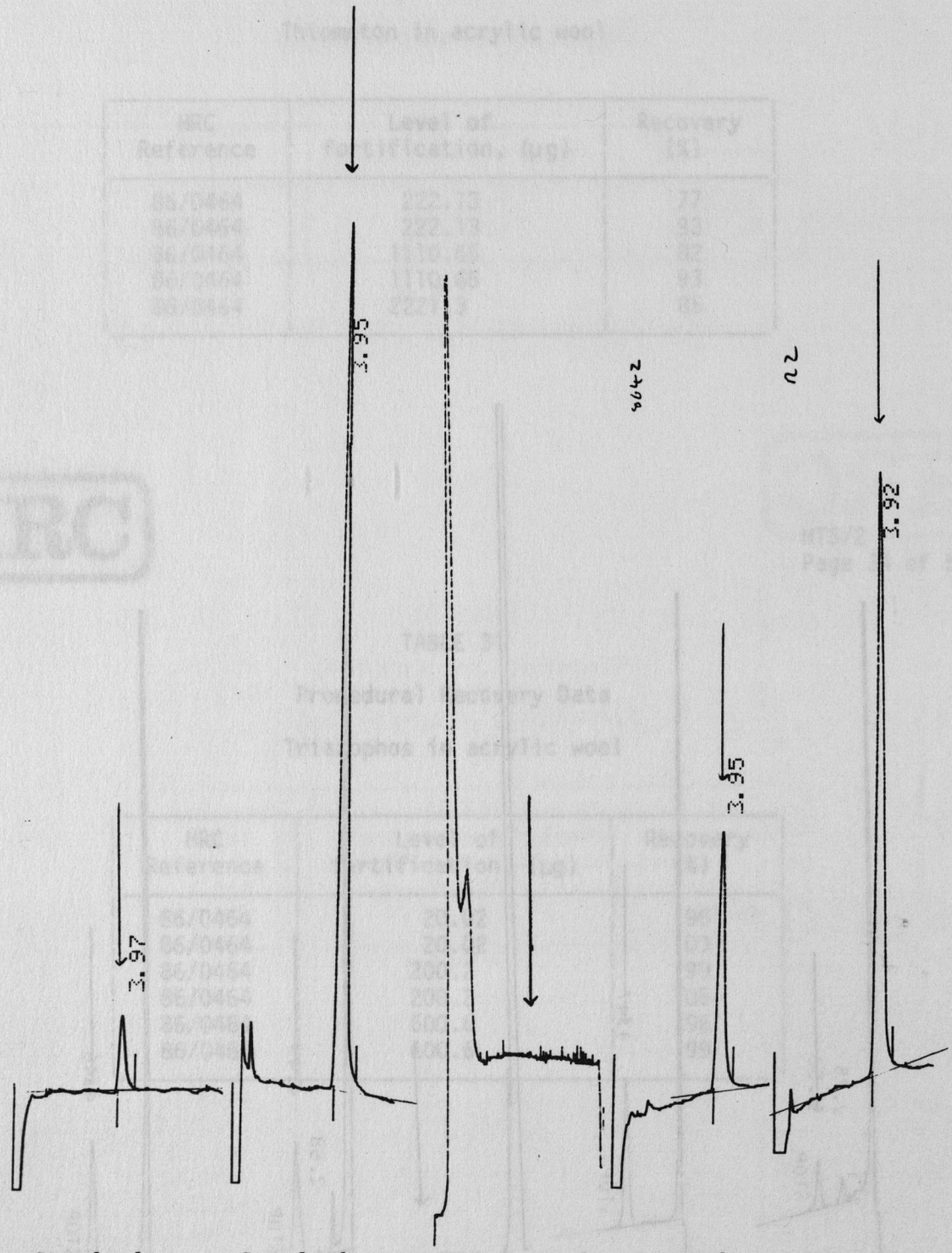
Typical analytical chromatography : Alphamethrin in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.02 µg/ml	0.07 µg/ml	86/0464	85/8660	219.24 µg
		<0.05 µg/m	4G.100	108%
			18.4 µg/m	

FIGURE 2

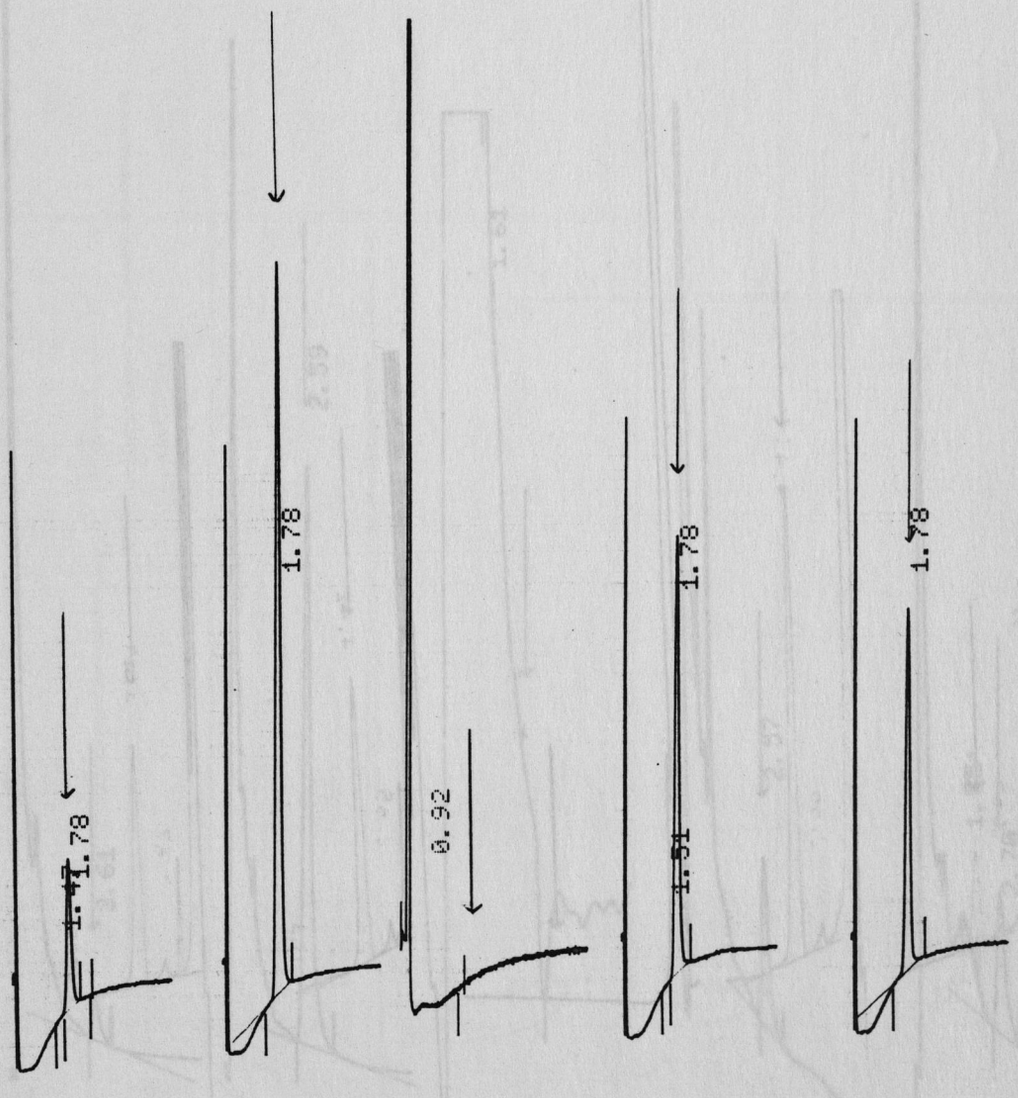
Typical analytical chromatography : Amitraz in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.10 µg/ml	0.96 µg/ml	86/0464	85/8642	382.14 µg
		<0.05 µg/m	G_2A.50	75%
			408.6 µg/m	

FIGURE 3

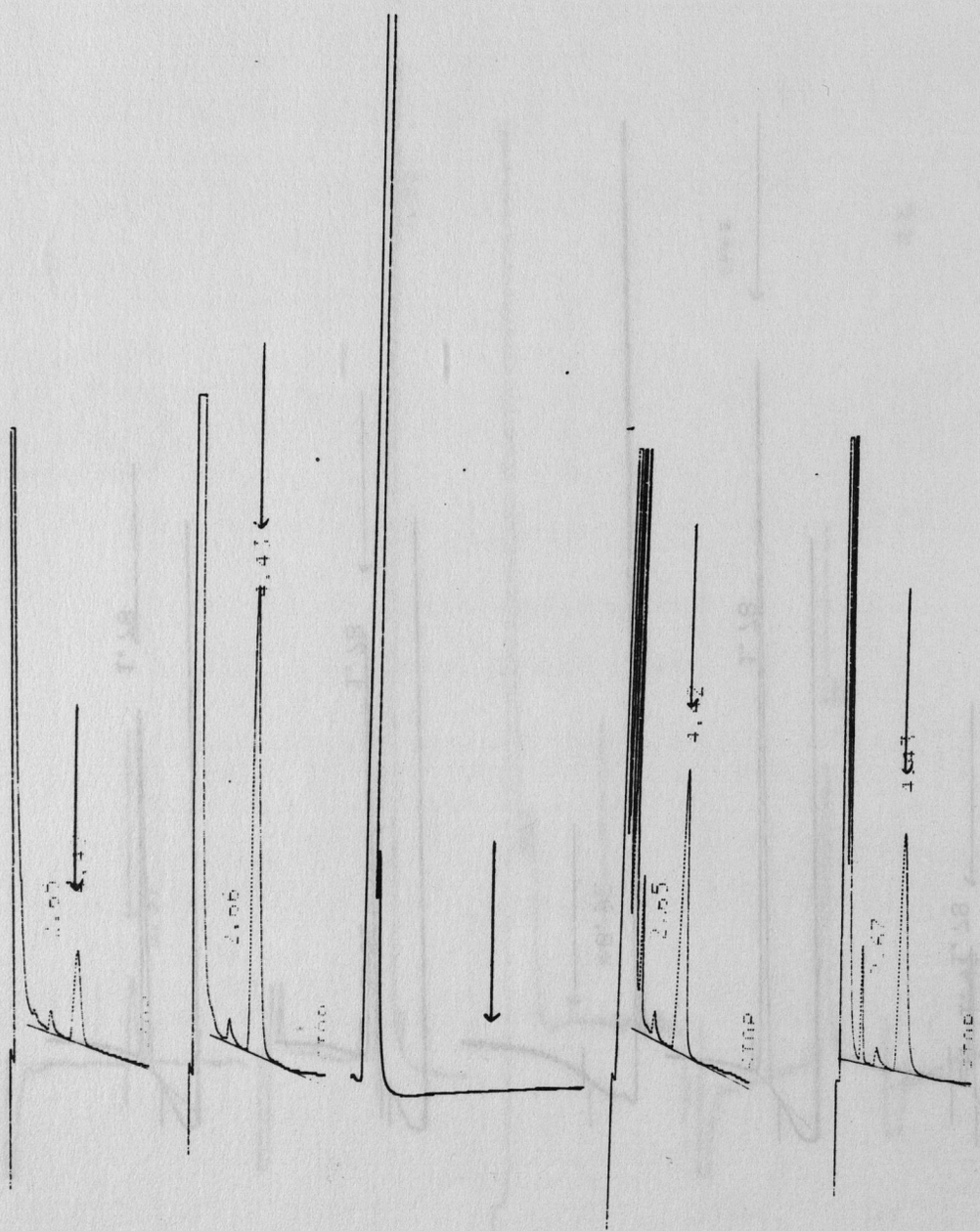
Typical analytical chromatography : Chlorpyrifos in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.2 µg/ml	1.0 µg/ml	86/0464	85/9149	1000 µg/ml
		<0.05 µg/m	9G.25	101%
			321.1 µg/m	

FIGURE 4

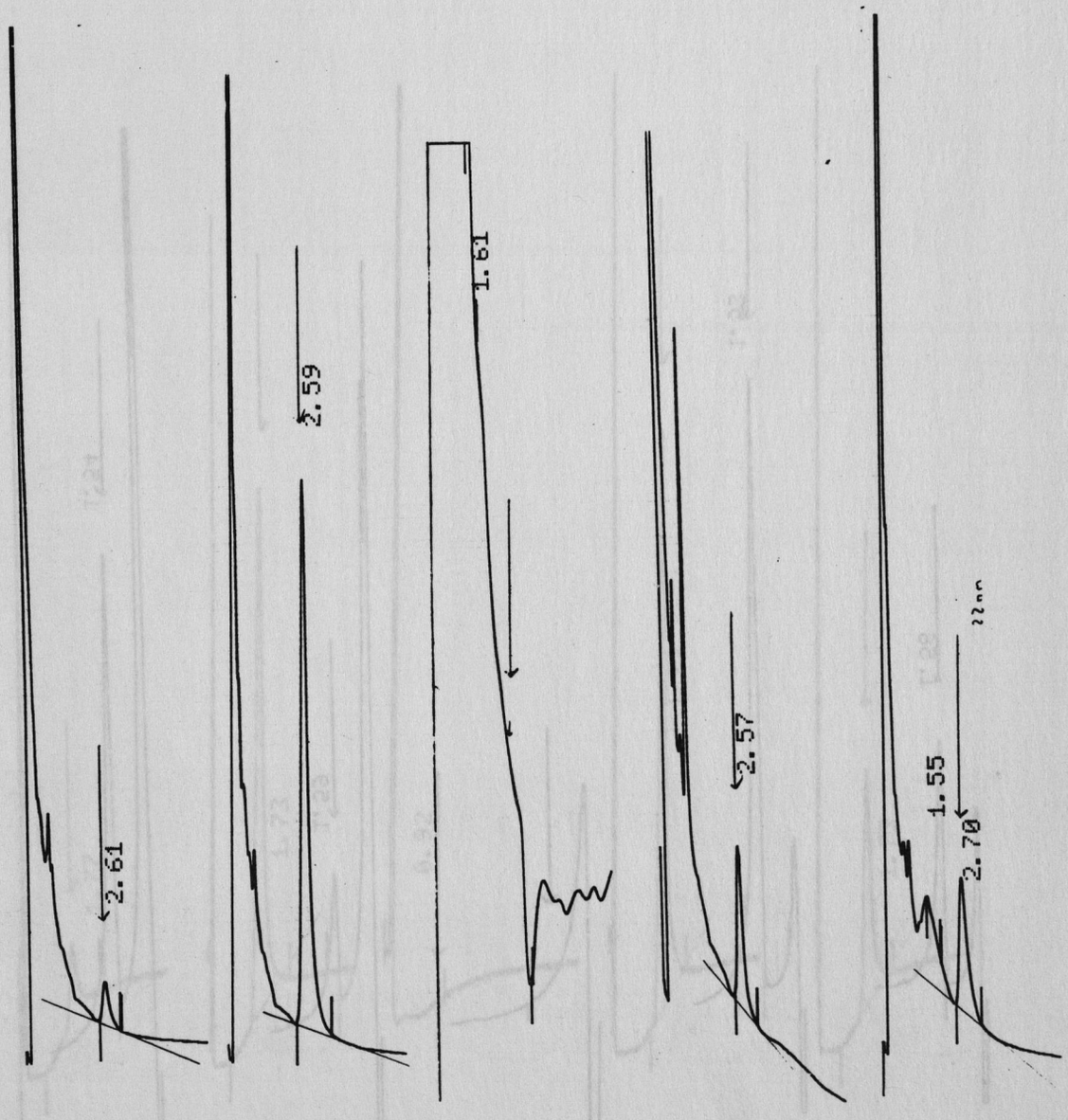
Typical analytical chromatography : Cypermethrin in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.06 µg/ml	0.28 µg/ml	86/0464	85/7591	2200 µg
		<Q.05 µg/m	6NH.20	99%
			52.5 µg/m	

FIGURE 5

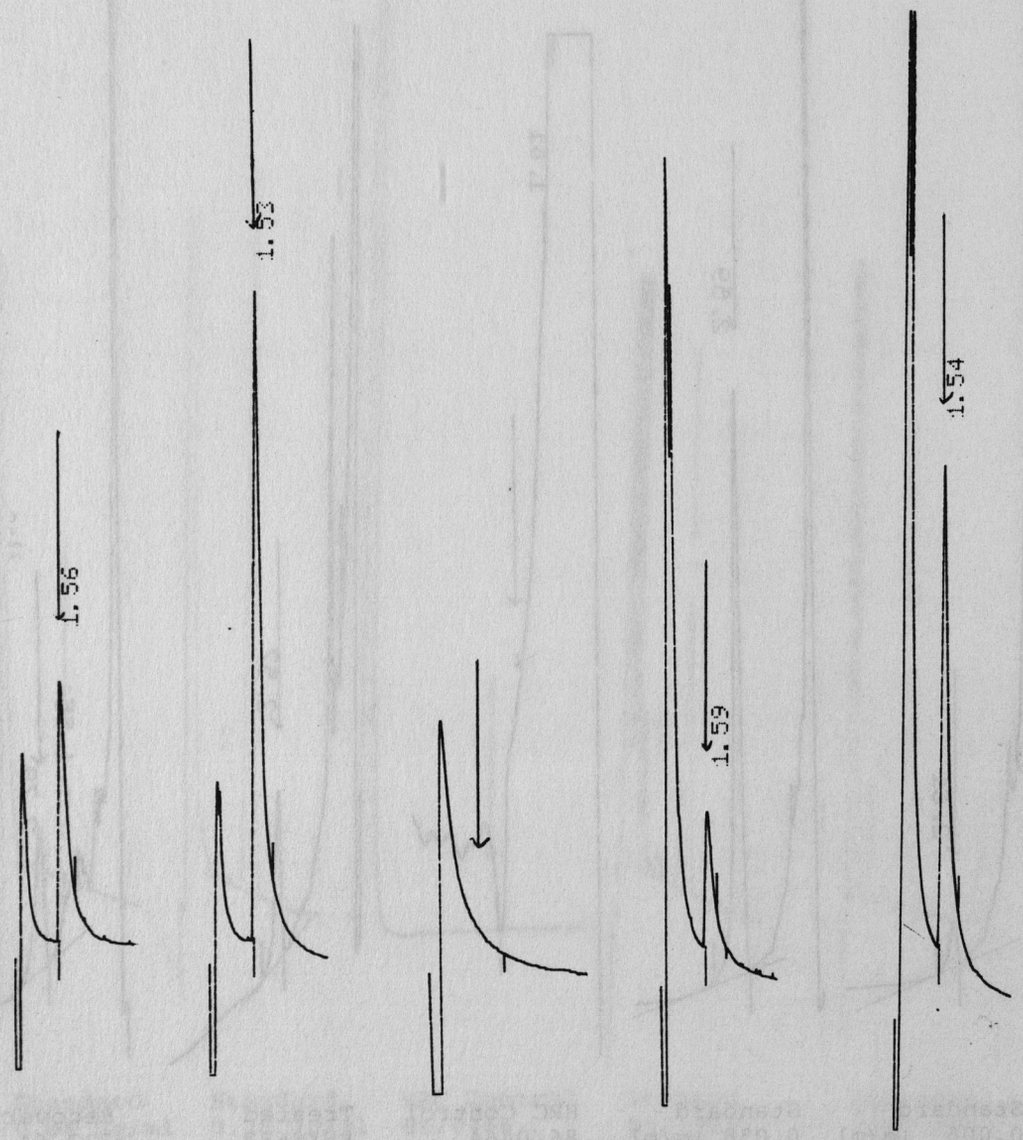
Typical analytical chromatography : Decamethrin in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.006 µg/ml	0.058 µg/ml	86/0464	85/8652	233.64 µg
		<0.05 µg/m	3G.25	89%
			4.1 µg/m	

FIGURE 6

Typical analytical chromatography : Dicrotophos in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.42 µg/ml	0.84 µg/ml	86/0464	85/8668	632.4 µg
		<0.05 µg/m	5R.500	85%
			42.7 µg/m	

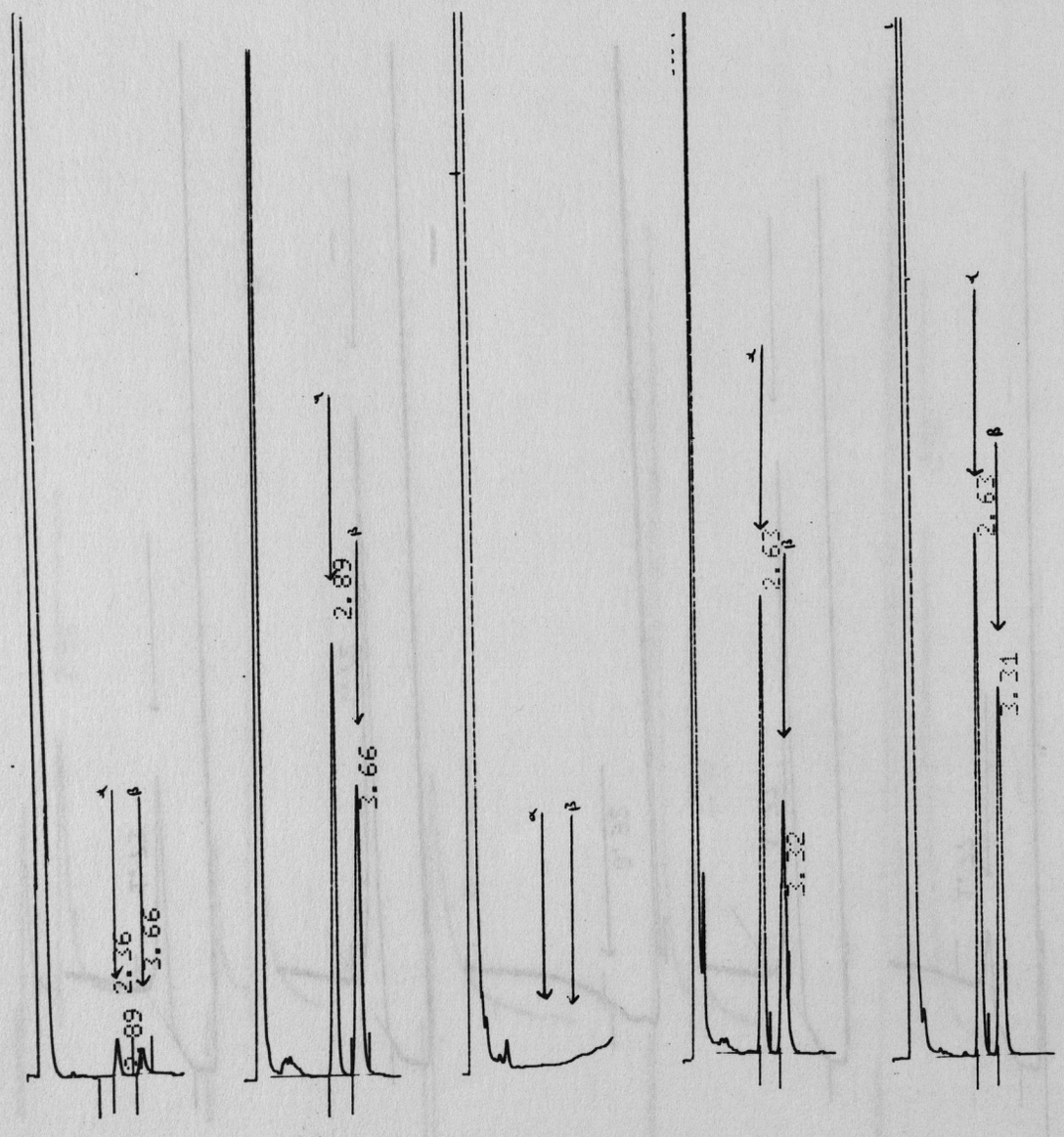
FIGURE 7

Typical analytical chromatography : Dimethoate in acrylic wool

Standard	Standard	HRC Control	Treated	Recovery
0.20 µg/ml	1.02 µg/ml	86/0464	85/7600	1019.7 µg
		<0.05 µg/m	R_3.20	102%
			133.5 µg/m	

FIGURE 8

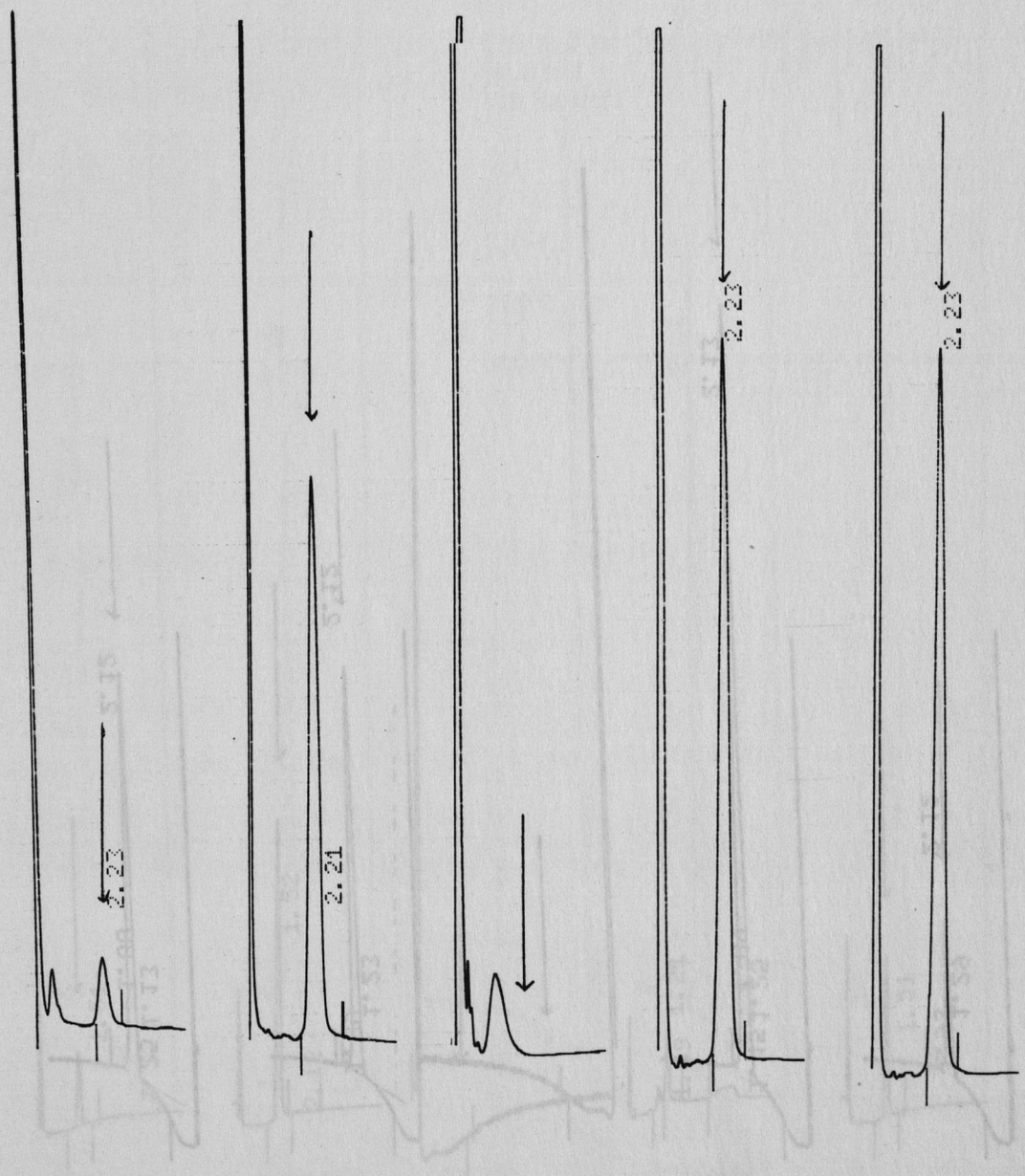
Typical analytical chromatography : Endosulphan in acrylic wool



Standard	Standard	HRC Control	HTS Control	Recovery
0.005 µg/ml	0.05 µg/ml	86/0464	85/8669	9.65 µg
		<0.05 µg/m	10.1 µg/m	106%

FIGURE 9

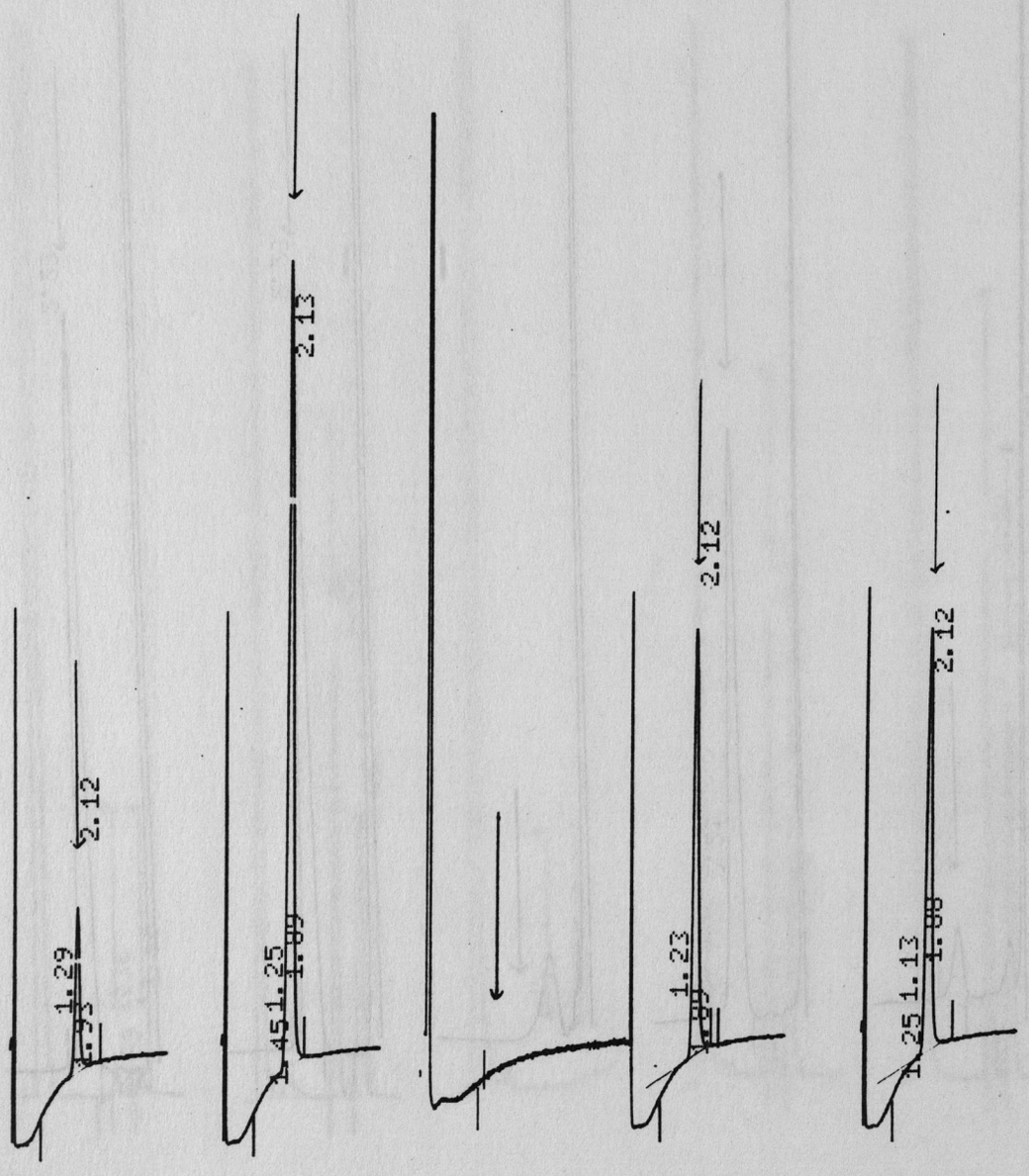
Typical analytical chromatography : Fenvalerate in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.05 µg/ml	0.4 µg/ml	86/0464	85/7605	1008.8 µg
		<0.05 µg/m	R_3.400	102%
			20.5 µg/m	

FIGURE 10

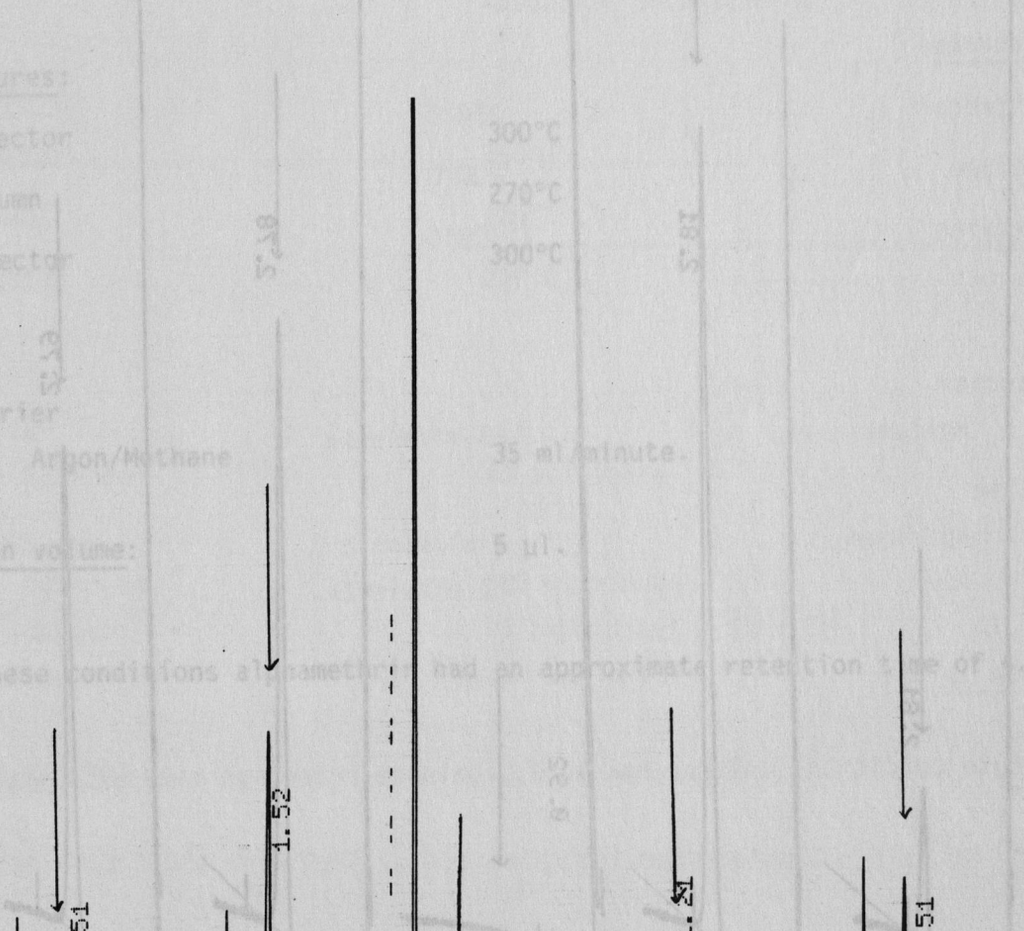
Typical analytical chromatography : Quinalphos in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.21 µg/ml	1.06 µg/ml	86/0464	85/8677	843.48 µg
		<0.05 µg/m	7G.50	106%
			299.2 µg/m	

FIGURE 11

Typical analytical chromatography : Thiometon in acrylic wool



Standard	Standard	HRC Control	Treated	Recovery
0.22 µg/ml	0.89 µg/ml	86/0464	86/0976	2221.3 µg
		<0.05 µg/m	Kosti 2.5	85%
			0.14 µg/m	

FIGURE 12

Typical analytical chromatography : Triazophos in acrylic wool

Standard 0.2 µg/ml	Standard 1.0 µg/ml	HRC Control 86/0464 <0.05 µg/m	Treated 85/9510 NH10.005 494.4 µg	Recovery 600.6 µg 99%

APPENDIX 1/1

Gas chromatographic conditions used for the determination of Alphamethrin

Instrument: Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.

Column: 2 m x 2 mm i.d. glass packed with 1.5% SP 2250 + 1.95% SP 2401 on Supelcon (100-120 mesh).

Temperatures:

Injector	300°C
Column	270°C
Detector	300°C

Gases:

Carrier

Argon/Methane 35 ml/minute.

Injection volume: 5 µl.

Under these conditions alphamethrin had an approximate retention time of 4.5 minutes.

APPENDIX 1/2

Gas chromatographic conditions used for the determination of Amitraz

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a nitrogen phosphorous thermionic specific detector and HP 7671A automatic sampler.
Column:	1.8 m x 2 mm i.d. glass packed with 5% OV 210 on Diatomite CLQ (100-120 mesh).
Temperatures:	
Injector	250°C
Column	200°C
Detector	300°C
Gases:	
Carrier	
Helium	35 ml/minute
Flame	
Hydrogen	3 ml/minute
Air	60 ml/minute.
Injection volume:	5 µl.

Under these conditions amitraz had an approximate retention time of 4 minutes.

APPENDIX 1/3

Gas chromatographic conditions used for the determination of Chlorpyrifos

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a flame photometric detector, in the phosphorus mode, and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 2½% Apiezon L on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	250°C
Column	205°C
Detector	250°C
Gases:	
Carrier	
Nitrogen	40 ml/minute
Flame	
Hydrogen	200 ml/minute
Oxygen	15 ml/minute
Air	50 ml/minute.
Injection volume:	10 µl.

Under these conditions chlorpyrifos had an approximate retention time of 1.8 minutes.

APPENDIX 1/4

Gas chromatographic conditions used for the determination of Cypermethrin

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	2 m x 2 mm i.d. glass packed with 1.5% SP 2250 + 1.95% SP 2401 on Supelcon (100-120 mesh).
Temperatures:	
Injector	300°C
Column	270°C
Detector	300°C
Gases:	
Carrier	
Argon/Methane	35 ml/minute.
Injection volume:	5 µl.

Under these conditions cypermethrin had an approximate retention time of 4.4 minutes.

APPENDIX 1/5

Gas chromatographic conditions used for the determination of Decamethrin

Instrument: Hewlett Packard 5890 gas chromatograph fitted with a ^{63}Ni electron capture detector and a 7672A automatic sampler.

Column: 1.5 m x 3 mm i.d. glass packed with 1% SE 30 on Diatomite CLQ (80-100 mesh).

Temperatures:

Injector	300°C
Column	260°C
Detector	300°C

Gases:

Carrier

Nitrogen 60 ml/minute.

Injection volume: 3 µl.

Under these conditions decamethrin had an approximate retention time of 2.6 minutes.

APPENDIX 1/6

Gas chromatographic conditions used for the determination of Dicrotophos

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a nitrogen phosphorous thermionic specific detector and HP 7671A automatic sampler.
Column:	1.4 m x 2 mm i.d. glass packed with 5% OV 210 on Diatomite CLQ (100-120 mesh).
Temperatures:	
Injector	250°C
Column	210°C
Detector	300°C
Gases:	
Carrier	
Helium	35 ml/minute
Flame	
Hydrogen	3 ml/minute
Air	60 ml/minute
Injection volume:	5 µl.

Under these conditions dicrotophos had an approximate retention time of 1.5 minutes.

APPENDIX 1/7

Gas chromatographic conditions used for the determination of Dimethoate

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a flame photometric detector, in the phosphorous mode, and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 2½% Apiezon L and Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	250°C
Column	180°C
Detector	250°C
Gases:	
Carrier	
Nitrogen	40 ml/minute.
Flame	
Hydrogen	200 ml/minute
Oxygen	15 ml/minute
Air	50 ml/minute.
Injection volume:	10 µl.

Under these conditions dimethoate had an approximate retention time of 1.7 minutes.

APPENDIX 1/8

Gas chromatographic conditions used for the determination of Endosulphan

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	1.8 x 2 mm i.d. glass packed with 3% OV 101 on Chromosorb W-AW-DMCS (100-120 mesh).
Temperatures:	
Injector	250°C
Column	230°C
Detector	300°C
Gases:	
Carrier	
Argon/methane	30 ml/minute
INjection volume:	5 µl.

Under these conditions α endosulphan had an approximate retention time of 2.6 minutes, and β endosulphan had an approximate retention time of 3.3 minutes.

APPENDIX 1/9

Gas chromatographic conditions used for the determination of Fenvalerate

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	0.5 m x 4 mm i.d. glass packed with 3% SF 96 + 3% Apiezon M on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	270°C
Detector	300°C
Gases:	
Carrier	
Argon/Methane	30 ml/minute
Injection volume:	5 µl.

Under these conditions fenvalerate had an approximate retention time of 2.2 minutes.

APPENDIX 1/10

Gas chromatographic conditions used for the determination of Quinalphos

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a flame photometric detector, in the phosphrous mode, and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 2½% Apiezon L on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	250°C
Column	210°C
Detector	250°C
Gases:	
Carrier	
Nitrogen	40 ml/minute
Flame	
Hydrogen	200 ml/minute
Oxygen	15 ml/minute
Air	50 ml/minute
Injection volume:	10 µl.

Under these conditions quinalphos had an approximate retention time of 2.1 minutes.

APPENDIX 1/11

Gas chromatographic conditions used for the determination of Thiometon

Instrument: Hewlett Packard 5710A gas chromatograph fitted with a flame photometric detector, in the phosphorus mode, and HP 7671A automatic sampler.

Column: 1 m x 4 mm i.d. glass packed with 2½% Apiezon L on Diatomite CLQ (80-100 mesh).

Temperatures:

Injector	250°C
Column	205°C
Detector	250°C

Gases:

Carrier	
Nitrogen	30 ml/minute
Flame	
Hydrogen	200 ml/minute
Oxygen	15 ml/minute
Air	50 ml/minute

Injection volume: 10 µl.

Under these conditions thiometon had an approximate retention time of 1.4 minutes.

APPENDIX 1/12

Gas chromatographic conditions used for the determination of Triazophos

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a flame photometric detector, in the phosphorus mode, and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 2½% Apiezon L on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	250°C
Column	220°C
Detector	250°C
Gases:	
Carrier	
Nitrogen	40 ml/minute
Flame	
Hydrogen	200 ml/minute
Oxygen	15 ml/minute
Air	50 ml/minute
Injection volume:	10 µl

Under these conditions triazophos had an approximate retention time of 2.7 minutes.

APPENDIX B

RESULTS OF GLC ANALYSES OF INSECTICIDE SOIL RESIDUES

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DEPARTMENT OF ANALYTICAL CHEMISTRY

CERTIFICATE OF ANALYSIS

The Determination of Concentrations of Insecticides

in Soil

HTS 4

23rd April 1986 81

HRC

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HRC Project number: HTS 4.

Date of commission: This work was commissioned by Mr. C.R. Whetnall of Hunting Technical Services Ltd on 12th November 1985.

Samples: Samples were received between December 1985 and March 1986 in a good condition and stored at -20°C prior to analysis.

Study: Residue analysis.

Method:

1. Aldicarb

The samples were analysed by shaking with 90% v/v acetone in water for 3 hours. An aliquot was evaporated to the aqueous layer, toluene and peracetic acid added and the mixture agitated for 30 minutes to oxidise the aldicarb to aldicarb sulphone. The mixture was neutralised with 10% aqueous sodium hydrogen carbonate solution, washed with hexane and the aqueous phase extracted with chloroform. The chloroform extract was dried using anhydrous sodium sulphate, added to a Florisil clean-up column and eluted with 5% v/v acetone in diethyl ether and 25% v/v acetone in diethyl ether. The latter fraction was collected, evaporated to dryness and taken up in acetone for analysis by GLC using the conditions detailed in Appendix 1a.

2. Amitraz, endosulphan, organophosphorus compounds and pyrethroids

A bulk extract was prepared by moisturising samples with water and shaking with chloroform for 2 hours. An aliquot was then evaporated to dryness and the residue taken up in 1 ; 1 toluene ; 2,2,4-trimethylpentane for quantitation by GLC.

a) Amitraz was quantified using an OV-101 column and a nitrogen phosphrous thermionic specific detector, using conditions detailed in Appendix 1b.

b) Endosulphan was quantified using an OV-210 column and electron capture detector, using conditions detailed in Appendix 1c.

c) Organophosphorus compounds were quantified using an Apiezon L column and flame photometric detector, in the phosphorus mode, using conditions detailed in Appendix 1d.

d) Pyrethroids were quantified using an OV-101 column and electron capture detector, using conditions detailed in Appendix 1e.

Results:

The results obtained, uncorrected for control or procedural recovery values are shown in Tables 1-5. Endosulphan is reported as the total of α and β isomers. Procedural recovery values, corrected for appropriate control residues, are shown in Tables 6-17.

Typical analytical chromatograms are shown in Figures 1-5.

Chemistry Laboratory Notebook Reference: 2421/86/06

SOIL SAMPLES DESCRIPTION - CODE NOS.

	B37 Turabi				B7 Wad Shannan				B9 Seed Farm				B7 Aldicarb	
Date	10/12/85		5/2/86		30/11/85		4/2/86		21/11/85		9/2/86		15/10/85	4/2/86
Rep	A	B	C	D	A	B	C	D	A	B	C	D		
Depth														
0 - 3 cm	39	-	55	58	19	22	45	48	13	16	61	64	12	51
3 - 10 cm	40	-	56	59	20	23	46	49	14	17	62	65	11	52
10 - 20 cm	41	-	57	60	21	24	47	50	15	18	63	66	10	53

Controls	25	0 - 3 cm	Untreated Bush
	26	3 - 20 cm	Untreated Bush
	27	0 - 20 cm	Untreated rough grazing
	42	0 - 3 cm	Untreated Fallow
	43	3 - 10 cm	Untreated Fallow
	44	10 - 20 cm	Untreated Fallow

Samples 1 to 9 were for aldicarb residues in numbers treated in 1982, 1983, 1984 while under cotton. Each field sampled at three depths.

The HRL results are summarised in this report in Table 2.8 as means of replicates for the sampling dates A + B and C + D.

TABLE 1

Concentrations of Aldicarb found in soil

HRC Reference	Sample Description	Aldicarb (ppm)
85/8681*	Sample 1 10-20 cm	<0.02
85/8682*	Control Sample 2 0- 3 cm	<0.02
85/8683*	Control Sample 3 3-10 cm	<0.02
85/8684*	Sample 4 0- 3 cm	<0.02
85/8685*	Sample 5 0- 3 cm	<0.02
85/8686*	Control Sample 6 10-20 cm	<0.02
85/8687*	Sample 7 10-20 cm	<0.02
85/8688*	Sample 8 3-10 cm	<0.02
85/8689*	Sample 9 3-10 cm	<0.02
85/8690*	Sample 10 10-20 cm	0.04+
85/8691*	Sample 11 3-10 cm	0.03+
85/8692*	Sample 12 0- 3 cm	0.15+
85/9160*	Control Sample 25 0- 3 cm	<0.02
85/9161*	Control Sample 26 3-20 cm	<0.02
85/9434*	Control Sample 27	<0.02
86/1467*	4.2.86 Sample 51 0- 3 cm	0.04
86/1468*	4.2.86 Sample 52 3-10 cm	<0.02
86/1469*	4.2.86 Sample 53 10-20 cm	<0.02
86/1470	5.2.86 Sample 55 0- 3 cm C	<0.02
86/1471	5.2.86 Sample 56 3-10 cm C	<0.02
86/1472	5.2.86 Sample 57 10-20 cm C	<0.02
86/1473	5.2.86 Sample 58 0- 3 cm D	<0.02
86/1474	5.2.86 Sample 59 3-10 cm D	<0.02
86/1475	5.2.86 Sample 60 10-20 cm D	<0.02
86/1476	9.2.86 Sample 61 0- 3 cm C	<0.02
86/1477	9.2.86 Sample 62 3-10 cm C	<0.02
86/1478	9.2.86 Sample 63 10-20 cm C	<0.02
86/1479	9.2.86 Sample 64 0- 3 cm D	<0.02
86/1480	9.2.86 Sample 65 3-10 cm D	<0.02
86/1481	9.2.86 Sample 66 10-20 cm D	<0.02

* Previously reported in Certificate of Analysis HTS/1 dated 27.3.86
\+ Average value of two determinations

TABLE 2

Concentrations of Amitraz found in soil

HRC Reference	Sample Description	Amitraz (ppm)
85/9154	Sample 13 0- 3 cm A	<0.05
85/9155	Sample 14 3-10 cm A	<0.05
85/9156	Sample 15 10-20 cm A	<0.05
85/9157	Sample 16 0- 3 cm B	<0.05
85/9158	Sample 17 3-10 cm B	<0.05
85/9159	Sample 18 10-20 cm B	<0.05
85/9160	Control Sample 25 0- 3 cm	<0.05
85/9161	Control Sample 26 3-20 cm	<0.05
86/1476	9.2.86 Sample 61 0- 3 cm C	<0.05
86/1477	9.2.86 Sample 62 3-10 cm C	<0.05
86/1478	9.2.86 Sample 63 10-20 cm C	<0.05
86/1479	9.2.86 Sample 64 0- 3 cm D	<0.05
86/1480	9.2.86 Sample 65 3-10 cm D	<0.05
86/1481	9.2.86 Sample 66 10-20 cm D	<0.05

TABLE 3

Concentrations of Endosulphan* found in soil

HRC Reference	Sample Description	Endosulphan (ppm)
85/9154	Sample 13 0- 3 cm A	3.23
85/9155	Sample 14 3-10 cm A	0.17
85/9156	Sample 15 10-20 cm A	0.03
85/9157	Sample 16 0- 3 cm B	1.54
85/9158	Sample 17 3-10 cm B	0.07
85/9159	Sample 18 10-20 cm B	0.05
85/9160	Control Sample 25 0- 3 cm	<0.01
85/9161	Control Sample 26 3-20 cm	0.03
85/9434	Control Sample 27	0.24
85/9435	30.11.85 Sample 19 0- 3 cm A	0.32
85/9436	30.11.85 Sample 20 3-10 cm A	0.03
85/9437	30.11.85 Sample 21 10-20 cm A	0.03
85/9438	30.11.85 Sample 22 0- 3 cm B	0.06
85/9439	30.11.85 Sample 23 3-10 cm B	0.02
85/9440	30.11.85 Sample 24 10-20 cm B	0.05
85/9441	10.12.85 Sample 34 0- 3 cm	<0.01
85/9442	10.12.85 Sample 40 3-10 cm	<0.01
85/9443	10.12.85 Sample 41 10-20 cm	<0.01
85/9444	Control Sample 42 0- 3 cm	<0.01
85/9445	Control Sample 43 3-10 cm	<0.01
85/9446	Control Sample 44 10-20 cm	<0.01
86/1461	4.2.86 Sample 45 0- 3 cm C	0.05
86/1462	4.2.86 Sample 46 3-10 cm C	0.02
86/1463	4.2.86 Sample 47 10-20 cm C	<0.01
86/1464	4.2.86 Sample 48 0- 3 cm D	0.07
86/1465	4.2.86 Sample 49 3-10 cm D	<0.01
86/1466	4.2.86 Sample 50 10-20 cm D	<0.01
86/1470	5.2.86 Sample 55 0- 3 cm C	0.30
86/1471	5.2.86 Sample 56 3-10 cm C	0.07
86/1472	5.2.86 Sample 57 10-20 cm C	0.06
86/1473	5.2.86 Sample 58 0- 3 cm D	0.49
86/1474	5.2.86 Sample 59 3-10 cm D	0.02
86/1475	5.2.86 Sample 60 10-20 cm D	<0.01
86/1476	9.2.86 Sample 61 0- 3 cm C	0.40
86/1477	9.2.86 Sample 62 3-10 cm C	0.02
86/1478	9.2.86 Sample 63 10-20 cm C	<0.01
86/1479	9.2.86 Sample 64 0- 3 cm D	0.19
86/1480	9.2.86 Sample 65 3-10 cm D	0.02
86/1481	9.2.86 Sample 66 10-20 cm D	<0.01

* Total of α and β isomers

TABLE 4

Concentrations of Organophosphorus compounds in soil

HRC Reference	Sample Description	Chlorpyriphos (ppm)	Dimethoate (ppm)	Profenphos (ppm)	Quinalphos (ppm)	Thiometon (ppm)	Triazophos (ppm)
85/9160	Control Sample 25 0- 3 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9161	Control Sample 26 3-20 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9434+	Control Sample 27	0.08	<0.1	<0.04	<0.01	<0.01	<0.04
85/9154+	Sample 13 0- 3 cm A	<0.02	<0.1	<0.04	<0.01	0.51	<0.04
85/9155+	Sample 14 3-10 cm A	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9156	Sample 15 10-20 cm A	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9157+	Sample 16 0- 3 cm B	<0.02	<0.1	<0.04	<0.01	0.14	<0.04
85/9158	Sample 17 3-10 cm B	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9159	Sample 18 10-20 cm B	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9435+	Sample 19 0- 3 cm A	0.07	<0.1	<0.04	<0.01	0.89	0.04
85/9436	Sample 20 3-10 cm A	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9437	Sample 21 10-20 cm A	0.02	<0.1	<0.04	<0.01	0.80	<0.04
85/9438	Sample 22 0- 3 cm B	0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9439	Sample 23 3-10 cm B	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9440	Sample 24 10-20 cm B	0.03	<0.1	<0.04	<0.01	<0.01	<0.04
85/9441	Sample 39 0- 3 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9442	Sample 40 3-10 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9443	Sample 41 10-20 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9444	Control Sample 42 0- 3 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9445	Control Sample 43 3-10 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
85/9446	Control Sample 44 10-20 cm	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04

+ These samples had peaks at the approximate retention time of Chlorfenvinphos, but these residues were not determined

TABLE 4

(continued)

HRC Reference	Sample Description	Chlorpyriphos (ppm)	Dimethoate (ppm)	Profenphos (ppm)	Quinalphos (ppm)	Thiometon (ppm)	Triazophos (ppm)
86/1461	Sample 45 0- 3 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1462	Sample 46 3-10 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1463	Sample 47 10-20 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1464	Sample 48 0- 3 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1465	Sample 49 3-10 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1466	Sample 50 10-20 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1470+	Sample 55 0- 3 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1471	Sample 56 3-10 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1472	Sample 57 10-20 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1473+	Sample 58 0- 3 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1474	Sample 59 3-10 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1475	Sample 60 10-20 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1476	Sample 61 0- 3 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1477	Sample 62 3-10 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1478	Sample 63 10-20 cm C	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1479	Sample 64 0- 3 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1480	Sample 65 3-10 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04
86/1481	Sample 66 10-20 cm D	<0.02	<0.1	<0.04	<0.01	<0.01	<0.04

+ These samples had peaks at the approximate retention time of Chlorfenvinphos, but these residues were not determined

TABLE 5

Concentrations of Pyrethroids in soil

HRC Reference	Sample Description	Cypermethrin (ppm)	Decamethrin (ppm)	Fenvalerate (ppm)
85/9160	Control Sample 25 0- 3 cm	<0.02	<0.02	<0.05
85/9161	Control Sample 26 3-20 cm	<0.02	<0.02	<0.05
85/9434	Control Sample 27	<0.02	<0.02	<0.05
85/9435	Sample 19 0- 3 cm A	<0.02	<0.02	<0.05
85/9436	Sample 20 3-10 cm A	<0.02	<0.02	<0.05
85/9437	Sample 21 10-20 cm A	<0.02	<0.02	<0.05
85/9438	Sample 22 0- 3 cm B	<0.02	<0.02	<0.05
85/9439	Sample 23 3-10 cm B	<0.02	<0.02	<0.05
85/9440	Sample 24 10-20 cm B	<0.02	<0.02	<0.05
85/9441	Sample 39 0- 3 cm	<0.02	<0.02	<0.05
85/9442	Sample 40 3-10 cm	<0.02	<0.02	<0.05
85/9443	Sample 41 10-20 cm	<0.02	<0.02	<0.05
85/9444	Control Sample 42 0- 3 cm	<0.02	<0.02	<0.05
85/9445	Control Sample 43 3-10 cm	<0.02	<0.02	<0.05
85/9446	Control Sample 44 10-20 cm	<0.02	<0.02	<0.05
86/1461	Sample 45 0- 3 cm C	<0.02	<0.02	<0.05
86/1462	Sample 46 3-10 cm C	<0.02	<0.02	<0.05
86/1463	Sample 47 10-20 cm C	<0.02	<0.02	<0.05
86/1464	Sample 48 0- 3 cm D	<0.02	<0.02	<0.05
86/1465	Sample 49 3-10 cm D	<0.02	<0.02	<0.05
86/1466	Sample 50 10-20 cm D	<0.02	<0.02	<0.05
86/1470	Sample 55 0- 3 cm C	<0.02	<0.02	0.06
86/1471	Sample 56 3-10 cm C	<0.02	<0.02	<0.05
86/1472	Sample 57 10-20 cm C	<0.02	<0.02	<0.05
86/1473	Sample 58 0- 3 cm D	<0.02	<0.02	<0.05
86/1474	Sample 59 3-10 cm D	<0.02	<0.02	<0.05
86/1475	Sample 60 10-20 cm D	<0.02	<0.02	<0.05

TABLE 6

Procedural Recovery Data

Aldicarb in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/8686	0.066	80*
85/8686	0.166	77*
85/9160	0.166	70
85/9161	0.166	69*
85/9160	0.333	75
85/8686	0.499	79*
85/9160	0.833	68
85/9161	0.833	70*

* Previously reported in Certificate of Analysis dated 27.3.86

TABLE 7

Proceudral Recovery Data

Amitraz in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9160	0.10	93
85/9160	0.48	97
85/9161	0.96	109

TABLE 8

Procedural Recovery Data

Chlorpyriphos in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.10	71
85/9160	0.10	96
85/9160	0.20	94
85/9434	0.50	84
85/9160	0.50	93
85/9161	0.50	101
85/9161	1.00	102
85/9161	2.00	93

TABLE 9

Procedural Recovery Data

Cypermethrin in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.11	94
85/9160	0.22	98
85/9434	0.55	107
85/9160	0.55	106
85/9434	1.10	103
85/9161	1.10	100
85/9161	2.20	100

TABLE 10

Procedural Recovery Data

Decamethrin in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.12	90
85/9160	0.23	102
85/9434	0.58	109
85/9160	0.58	109
85/9434	1.17	104
85/9161	1.17	101
85/9161	2.35	101

TABLE 11

Procedural Recovery Data

Dimethoate in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9160	0.10	94
85/9160	0.20	96
85/9160	0.51	88
85/9161	0.51	98
85/9161	1.02	90
85/9161	2.04	85

TABLE 12

Procedural Recovery Data

Endosulphan in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)	
		alpha isomer	beta isomer
85/9434	0.10	77	82
85/9160	0.19	91	107
85/9434	0.48	109	96
85/9160	0.48	103	103
85/9434	0.97	62	86
85/9161	0.97	88	99
85/9161	1.93	90	101

TABLE 13

Procedural Recovery Data

Fenvalerate in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.10	88
85/9160	0.20	99
85/9434	0.50	110
85/9160	0.50	110
85/9434	1.00	100
85/9161	1.00	101
85/9161	2.00	103

TABLE 14

Procedural Recovery Data

Profenphos in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.10	79
85/9160	0.10	94
85/9160	0.19	90
85/9434	0.47	106
85/9160	0.47	99
85/9161	0.47	112
85/9161	0.95	89
85/9161	1.89	118

TABLE 15

Procedural Recovery Data

Quinalphos in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.11	83
85/9160	0.11	96
85/9160	0.21	92
85/9434	0.53	91
85/9160	0.53	91
85/9161	0.53	102
85/9161	1.05	98
85/9161	2.11	88

TABLE 16

Procedural Recovery Data

Thiometon in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9160	0.11	79
85/9160	0.22	85
85/9160	0.56	77
85/9161	1.11	89
85/9161	2.22	82

TABLE 17

Procedural Recovery Data

Triazophos in soil

HRC Reference	Level of fortification (ppm)	Recovery (%)
85/9434	0.10	82
85/9160	0.10	93
85/9160	0.20	81
85/9434	0.50	110
85/9160	0.50	100
85/9161	0.50	113
85/9161	1.00	125
85/9161	2.00	74

FIGURE 1

Typical analytical chromatography of Aldicarb sulphone in soil

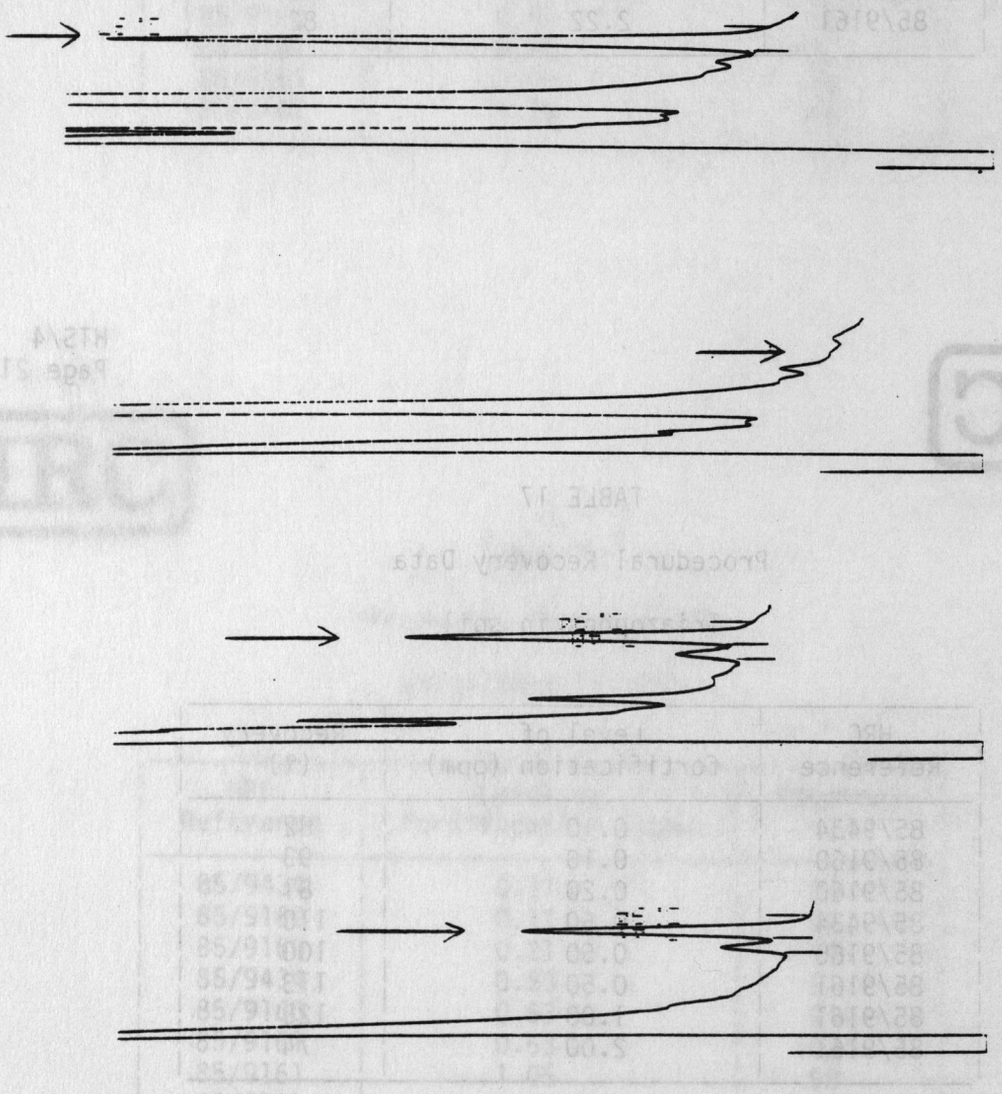


FIGURE 2

Typical analytical chromatography : Amitraz in soil

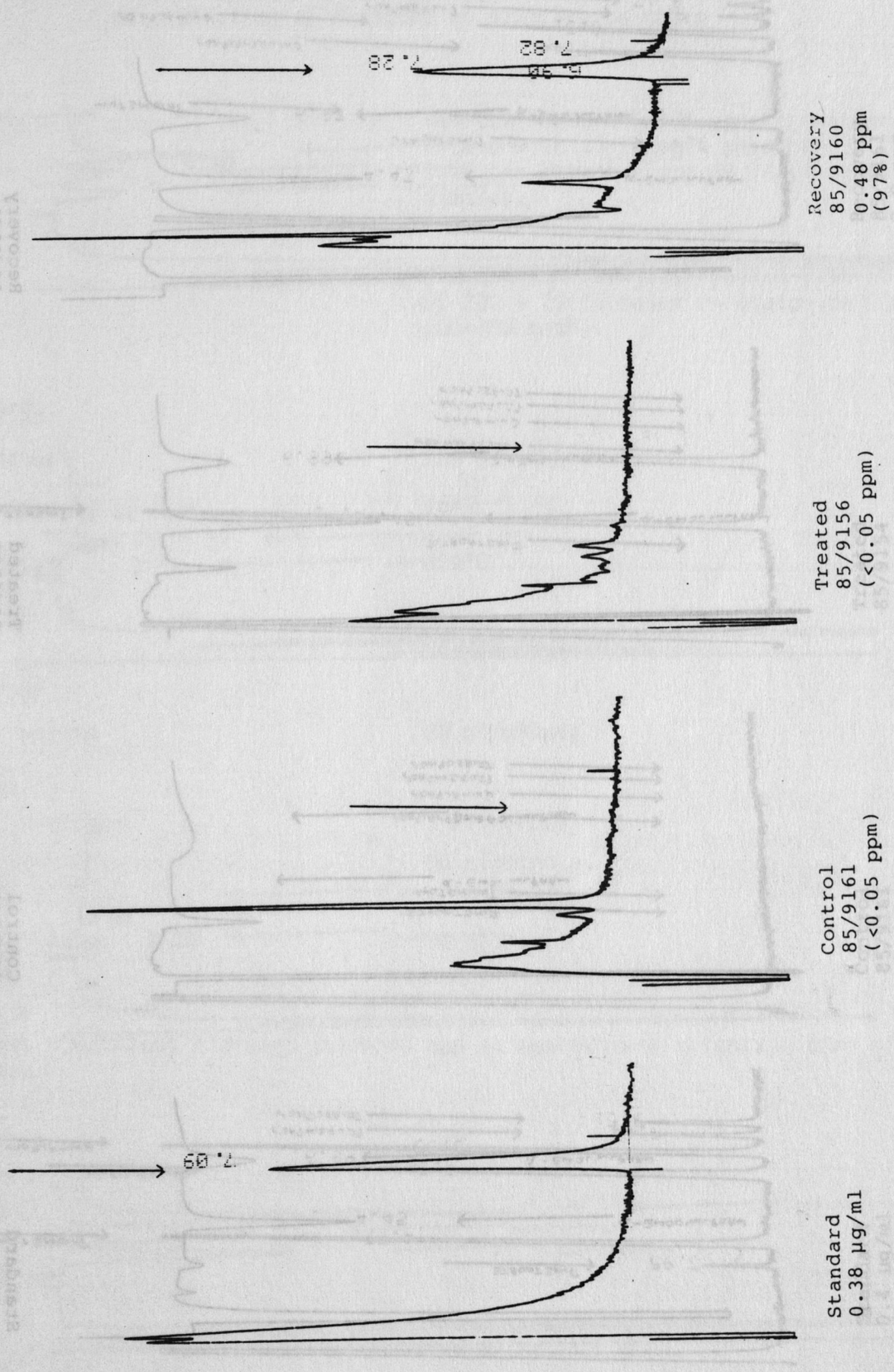


FIGURE 3

Typical analytical chromatography : Endosulphan in soil

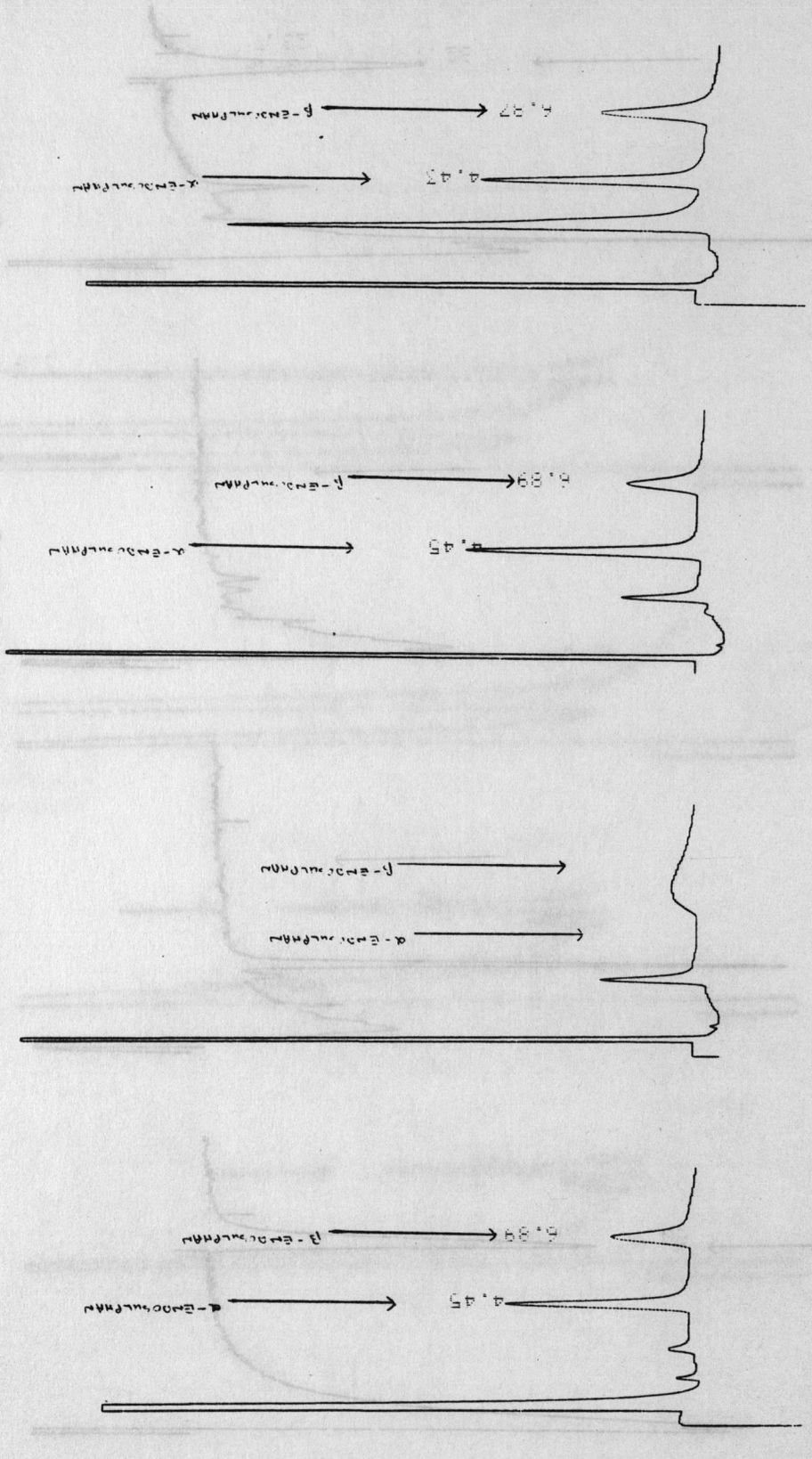


FIGURE 4

Typical analytical chromatography : Organophosphorus compounds in soil

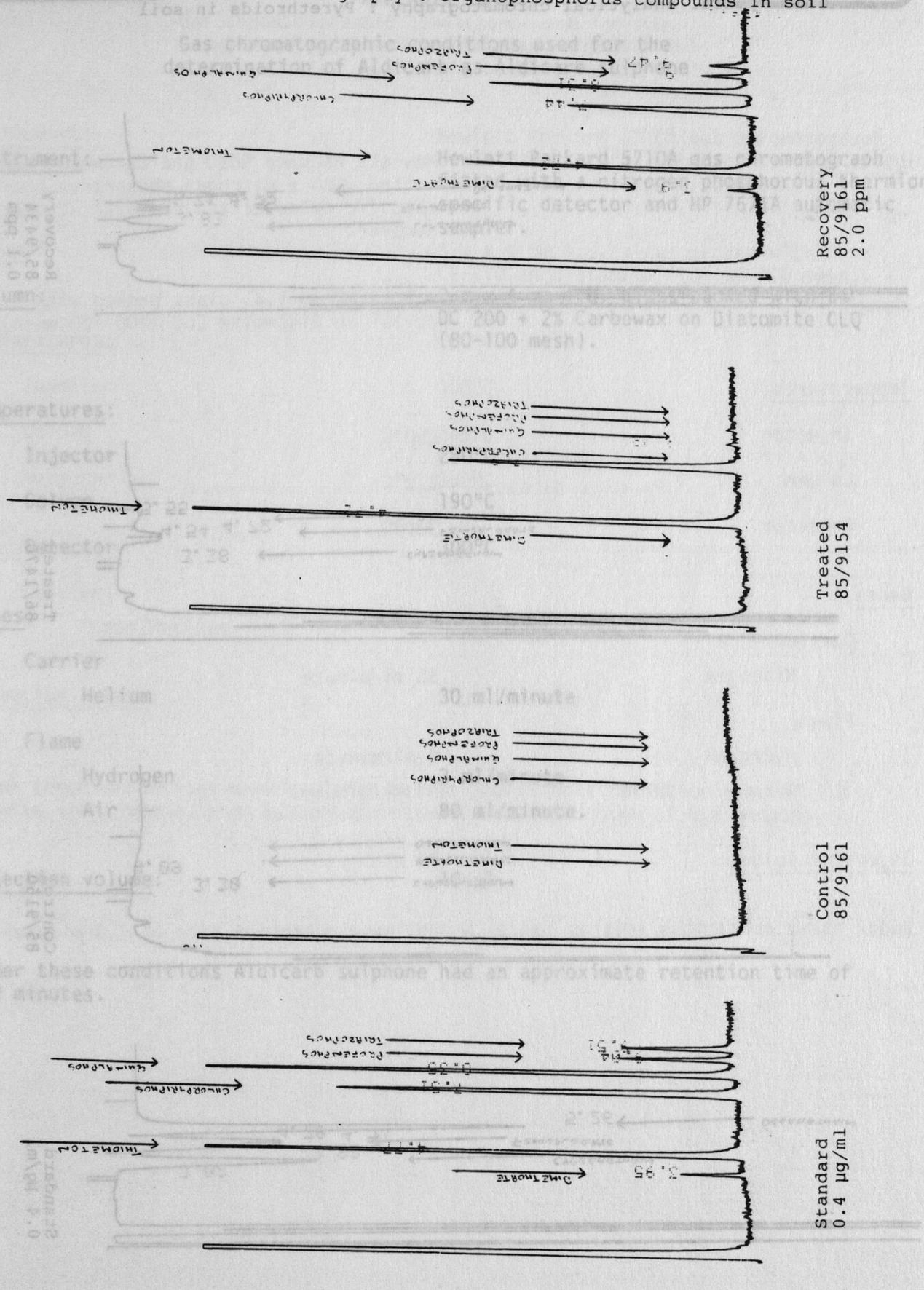
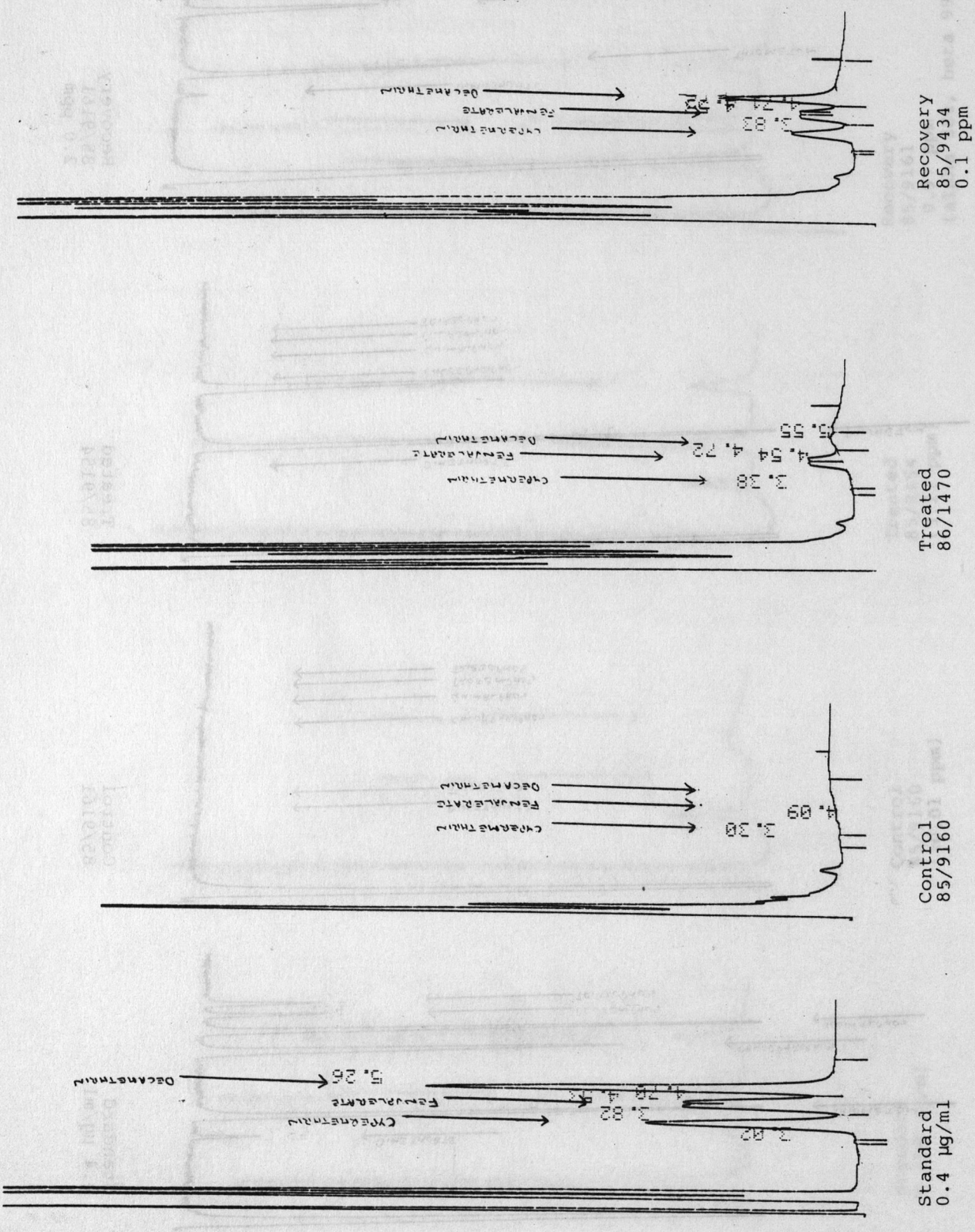


FIGURE 5

Typical analytical chromatography : Pyrethroids in soil



APPENDIX 1a

Gas chromatographic conditions used for the determination of Aldicarb as Aldicarb sulphone

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a nitrogen phosphorous thermionic specific detector and HP 7671A automatic sampler.
Column:	2 m x 4 mm i.d. glass packed with 5% DC 200 + 2% Carbowax on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	250°C
Column	190°C
Detector	300°C
Gases:	
Carrier	
Helium	30 ml/minute
Flame	
Hydrogen	3 ml/minute
Air	80 ml/minute.
Injection volume:	10 µl.

Under these conditions Aldicarb sulphone had an approximate retention time of 3.9 minutes.

APPENDIX 1b

Gas chromatographic conditions used for the determination of Amitraz in soil

Instrument:	Hewlett Packard 5890 gas chromatograph fitted with a nitrogen phosphorus thermionic specific detector and HP 7672A automatic sampler.
Column:	1.8 m x 2 mm i.d. glass packed with 5% OV-101 on Diatomite CLQ (100-120 mesh).
Temperatures:	
Injector	250°C
Column	210°C
Detector	300°C
Gases:	
Carrier	
Nitrogen	35 ml/minute
Flame	
Hydrogen	5 ml/minute
Air	70 ml/minute.
Injection volume:	10 µl.

Under these conditions Amitraz had an approximate retention time of 7.2 minutes.

APPENDIX 1c

Gas chromatographic conditions used for the determination of Endosulphan in soil

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	2 m x 4 mm i.d. glass packed with 5% OV-210 on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	245°C
Detector	350°C
Gases:	
Carrier	
Argon/Methane	40 ml/minute.
Injection volume:	5 µl.

Under these conditions α-endosulphan had an approximate retention time of 4.3 minutes and β-endosulphan had an approximate retention time of 6.6 minutes

APPENDIX 1d

Gas chromatographic conditions used for the determination of organophosphorus compounds (chlorpyriphos, dimethoate, profenphos, quinalphos, thiometon, triazophos) in soil

Instrument:	Varian Vista 6000 fitted with a flame photometric detector in the phosphorus mode and an autosampler.
Column:	1 m x 2 mm i.d. glass packed with 2½% Apiezon L on Gas Chrom Q (100-120 mesh).
Temperatures:	
Injector	225°C
Column	130°C (1 min) $\xrightarrow{25°/min}$ 200°C $\xrightarrow{3°/min}$ 210°C $\xrightarrow{50°/min}$ 270°C (6 min)
Detector	250°C
Gases:	
Carrier	
Helium	30 ml/minute
Flame	
Hydrogen	140 ml/minute
Air	170 ml/minute.
Injection volume:	8 µl.

Under these conditions the organophosphorus compounds had the following approximate retention times:

Chlorpyriphos	7.7 minutes
Dimethoate	4.8 minutes
Profenphos	9.2 minutes
Quinalphos	8.5 minutes
Thiometon	4.7 minutes
Triazophos	4.1 minutes

APPENDIX 1e

Gas chromatographic conditions used for the determination of pyrethroids (cypermethrin, decamethrin and fenvalerate) in soil)

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 1% OV-101 on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	215°C (2 mins) ——— 8°C/min ⟶ 240°C (4 min)
Detector	300°C
Gases:	
Carrier	
Argon/Methane	60 ml/minute.
Injection volume:	5 µl.

Under these conditions cypermethrin, decamethrin and fenvalerate had approximate retention times of 3.8 minutes, 5.3 minutes and 4.6 minutes respectively.

APPENDIX C

RESULTS OF GLC ANALYSES OF INSECTICIDE RESIDUES IN FOODSTUFFS

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DEPARTMENT OF ANALYTICAL CHEMISTRY

CERTIFICATE OF ANALYSIS

The Determination of Concentrations of Insecticides

in Food

HTS 5

23rd May 1986

HRC

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HRC Project number: HTS 5.

Date of commission: This work was commissioned by Mr. C. R. Whetnall of Hunting Technical Services Ltd on 12th November 1985.

Samples: Samples were received between December 1985 and March 1986 in a good condition and stored at -20°C prior to analysis.

Study: Residue analysis.

Method: 1. Aldicarb in Sorghum grains

The samples were analysed by soxhlet extraction with acetone for 2½ hours and the extract evaporated to dryness. The residue was taken up in hexane and partitioned with acetonitrile and the acetonitrile then evaporated to dryness. Water and peracetic acid were added to oxidise the aldicarb to aldicarb sulphone, the acidic solution neutralised with sodium hydrogen carbonate and the aqueous phase extracted with chloroform. The chloroform extract was dried using anhydrous sodium sulphate, added to a Florisil clean-up column and eluted with 5% v/v acetone in diethyl ether and 25% v/v acetone in diethyl ether. The latter fraction was collected, evaporated to dryness and taken up in acetone for analysis by GLC using the conditions detailed in Appendix 1a.

2. i). Amitraz, endosulphan, organophosphorus compounds and pyrethroids in Sorghum grains

The samples were analysed by soxhlet extraction with ethyl acetate for 3 hours and the extract evaporated to dryness. The residue was taken up in hexane, partitioned with acetonitrile and evaporated to dryness. The residue was then dissolved in 1 : 1 toluene : 2,2,4-trimethyl pentane for analysis by GLC.

ii) Amitraz, endosulphan, organophosphorus compounds and pyrethroids in bird peppers, okra, cucumber and aubergines

The samples were macerated with ethyl acetate, the filtered extract evaporated to dryness and residue taken up in hexane. The hexane was partitioned with acetonitrile, the acetonitrile evaporated to dryness and residue dissolved in 1 : 1 toluene : 2,2,4-trimethyl pentane for analysis by GLC.

a) Amitraz was quantified using an OV-101 column and a nitrogen phosphorus thermionic specific detector, using conditions detailed in Appendix 1b.

b) Endosulphan was quantified using an OV-210 column and an electron capture detector, using conditions detailed in Appendix 1c.

c) Organophosphorus compounds were quantified using an Apiezon L column and flame photometric detector in the phosphorus mode, using conditions detailed in Appendix 1d.

d) Pyrethroids were quantified using an OV-101 column and electron capture detector, using conditions detailed in Appendix 1c.

Results:

The results obtained, uncorrected for control or procedural recovery values are shown in Tables 1-5. Endosulphan is reported as the total of α and β isomers. Procedural recovery values, corrected for appropriate control residues are shown in Tables 6-19.

Typical analytical chromatograms are shown in Figures 1-5.

Chemistry Laboratory Notebook Reference: 2421/86/06

HRC

TABLE 1

Concentrations of Aldicarb in food

Food type	HRC Reference	Sample Description	Aldicarb (ppm)
Sorghum grain	85/9162	Control Sample 28	<0.02
Sorghum grain	85/9164	Contaminated Sample 30	<0.02
Sorghum grain	86/1489	Contaminated Sample 35	<0.02

HRC

TABLE 2

Concentrations of Amitraz in food

Food type	HRC Reference	Sample Description	Amitraz (ppm)
Sorghum grain	85/9162	Control Sample 28	0.08
	85/9163	Contaminated Sample 29	0.20
	85/9164	Contaminated Sample 30	26.25
	86/1489	Contaminated Sample 35	0.17
	86/1490	Contaminated Sample 36	0.05
	86/1491	Contaminated Sample 38	0.19
Bird peppers	85/9165	Treated Sample 31 BK 20 3/12/85	0.14
Okra	85/9166	Contaminated Sample 33	<0.05
	85/9167	Control Sample 34	0.05
	86/1493	Contaminated Sample 54	<0.05
Cucumber	85/9459	Contaminated Sample 32 BK 9 29/11/85	<0.05
Aubergine	86/1492	Contaminated Sample 37	<0.05

HRC

TABLE 3

Concentrations of Endosulphan* in foods

Food type	HRC Reference	Sample Description	Endosulphan (ppm)
Sorghum grain	85/9162	Control Sample 28	0.07
	85/9163	Contaminated Sample 29	0.20
	85/9164	Contaminated Sample 30	3.55
	86/1489	Contaminated Sample 35	0.11
	86/1490	Contaminated Sample 36	<0.01
	86/1491	Contaminated Sample 38	0.07
Bird peppers	85/9165	Treated Sample 31 BK 20 3/12/85	0.25
Okra	85/9166	Contaminated Sample 33	0.12
	85/9167	Control Sample 34	0.02
	86/1493	Contaminated Sample 54	<0.01
Cucumber	85/9459	Contaminated Sample 32 BK 9 29/11/85	<0.01
Aubergine	86/1492	Contaminated Sample 37	<0.01

* Total of α and β isomers

TABLE 4

Concentrations of organophosphorus compounds in food

HRC Reference	Sample Description	Chlorpyriphos (ppm)	Chlorfenvinphos (ppm)	Dimethoate (ppm)	Fenitrothion (ppm)	Profenphos (ppm)	Quinalphos (ppm)	Thiometon (ppm)	Triazophos (ppm)
Sorghum grain									
85/9162	Control Spl 28	<0.01	<0.01	0.12	<0.01	<0.01	<0.005	<0.005	<0.01
85/9163	Contaminated Spl 29	<0.01	<0.01	0.04	<0.01	<0.01	0.05	<0.005	0.02
85/9164	Contaminated Spl 30	1.40	0.16	0.11	<0.01	<0.01	0.30	0.03	3.51
86/1489	Contaminated Spl 35	<0.01	<0.01	0.11	<0.01	<0.01	<0.005	<0.005	0.02
86/1490	Contaminated Spl 36	<0.01	<0.01	0.07	<0.01	<0.01	<0.005	<0.005	<0.01
86/1491	Contaminated Spl 38	<0.01	<0.01	0.06	<0.01	<0.01	<0.005	<0.005	0.02
Bird peppers									
85/9165	Treated Spl 31	0.02	<0.01	<0.02	<0.01	0.06	<0.005	<0.005	0.02
Okra									
85/9166	Contaminated Spl 33	<0.01	<0.01	<0.02	<0.01	<0.01	<0.005	<0.005	<0.01
85/9167	Control Spl 34	<0.01	<0.01	<0.02	<0.01	<0.01	<0.005	<0.005	0.01
86/1493	Contaminated Spl 54	<0.01	<0.01	<0.02	<0.01	<0.01	<0.005	<0.005	<0.01
Cucumber									
85/9459	Contaminated Spl 32	<0.01	<0.01	<0.02	<0.01	<0.01	<0.005	<0.005	<0.01
Aubergine									
86/1492	Contaminated Spl 37	<0.01	<0.01	<0.02	<0.01	<0.01	<0.005	<0.005	<0.01

Note: An unidentified phosphorus containing peak was observed in sample extracts at retention time of 3.02 minutes. This peak has not been quantified

TABLE 5

Concentrations of Pyrethroids in food

Food type	HRC Reference	Sample Description	Cypermethrin (ppm)	Decamethrin (ppm)	Fenvalerate (ppm)
Sorghum grain	85/9162	Control Sample 28	<0.02	<0.02	0.02
	85/9163	Contaminated Sample 29	<0.02	<0.02	<0.02
	85/9164	Contaminated Sample 30	0.03	0.04	<0.02
	86/1489	Contaminated Sample 35	0.07	<0.02	<0.02
	86/1490	Contaminated Sample 36	<0.02	<0.02	<0.02
	86/1491	Contaminated Sample 38	<0.02	<0.02	0.04
Bird peppers	85/9165	Treated Sample 31 BK 20 3/12/85	<0.02	<0.02	<0.02
Okra	85/9166	Contaminated Sample 33	<0.02	<0.02	<0.02
	85/9167	Control Sample 34	<0.02	<0.02	<0.02
	86/1493	Contaminated Sample 54	<0.02	0.03	<0.02
Cucumber	85/9459	Contaminated Sample 32 BK 9 29/11/85	<0.02	<0.02	<0.02
Aubergine	86/1492	Contaminated Sample 37	<0.02	<0.02	<0.02

HRC

TABLE 6

Procedural Recovery Data

Aldicarb in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9164	0.20	83
	85/9162	0.25	94

HRC

TABLE 7

Procedural Recovery Data

Amitraz in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.97	68
	85/9162	4.83	69
Bird peppers	85/9165	0.97	89
	85/9165	4.83	124
Okra	85/9167	0.97	80
	85/9167	4.83	88
Cucumber	85/9459	0.97	75
	85/9459	4.83	88
Aubergine	86/1492	0.97	84
	86/1492	4.83	74

HRC

TABLE 8

Procedural Recovery Data

Chlorfenvinphos in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	86
	85/9162	0.51	86
Bird peppers	85/9165	0.10	110
	85/9165	0.51	110
Okra	85/9167	0.10	115
	85/9167	0.51	111
Cucumber	85/9459	0.10	115
	85/9459	0.51	106
Aubergine	86/1492	0.10	97
	86/1492	0.51	93

HRC

TABLE 9

Procedural Recovery Data

Chlorpyriphos in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	78
	85/9162	0.50	77
Bird peppers	85/9165	0.10	83
	85/9165	0.50	92
Okra	85/9167	0.10	94
	85/9167	0.50	95
Cucumber	85/9459	0.10	93
	85/9459	0.50	92
Aubergine	86/1492	0.10	90
	86/1492	0.50	88

HRC

TABLE 10

Procedural Recovery Data

Cypermethrin in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.11	95
	85/9162	0.55	93
Bird peppers	85/9165	0.11	107
	85/9165	0.55	106
Okra	85/9167	0.11	110
	85/9167	0.55	103
Cucumber	85/9459	0.11	110
	85/9459	0.55	110
Aubergine	86/1492	0.11	88
	86/1492	0.55	101

HRC

TABLE 11

Procedural Recovery Data

Decamethrin in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.12	86
	85/9162	0.58	94
Bird peppers	85/9165	0.12	138
	85/9165	0.58	113
Okra	85/9167	0.12	117
	85/9167	0.58	107
Cucumber	85/9459	0.12	123
	85/9459	0.58	115
Aubergine	86/1492	0.12	94
	86/1492	0.58	108

TABLE 12

Procedural Recovery Data

Dimethoate in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	24*
	85/9162	0.51	74
Bird peppers	85/9165	0.10	85
	85/9165	0.51	98
Okra	85/9167	0.10	88
	85/9167	0.51	93
Cucumber	85/9459	0.10	64
	85/9459	0.51	98
Aubergine	86/1492	0.10	90
	86/1492	0.51	91

* Significnat residue (0.12 ppm) present in control sample

TABLE 13

Procedural Recovery Data

Endosulphan in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)	
			alpha isomer	beta isomer
Sorghum grain	85/9162	0.10	83	126
	85/9162	0.48	71	92
	85/9162	1.93	75	82
Bird peppers	85/9165	0.10	59	69
	85/9165	0.48	82	109
Okra	85/9167	0.10	80	93
	85/9167	0.48	83	104
Cucumber	85/9459	0.10	71	104
	85/9459	0.48	80	104
Aubergine	86/1492	0.10	67	91
	86/1492	0.48	74	88

TABLE 14

Procedural Recovery Data

Fenitrothion in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.11	82
	85/9162	0.54	84
Bird peppers	85/9165	0.11	105
	85/9165	0.54	105
Okra	85/9167	0.11	103
	85/9167	0.54	105
Cucumber	85/9459	0.11	105
	85/9459	0.54	103
Aubergine	86/1492	0.11	97
	86/1492	0.54	94

TABLE 15

Procedural Recovery Data

Fenvalerate in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	92
	85/9162	0.50	94
Bird peppers	85/9165	0.10	162
	85/9165	0.50	120
Okra	85/9167	0.10	133
	85/9167	0.50	107
Cucumber	85/9459	0.10	141
	85/9459	0.50	119
Aubergine	86/1492	0.10	95
	86/1492	0.50	115

HRC

TABLE 16

Procedural Recovery Data

Profenphos in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	96
	85/9162	0.47	96
Bird peppers	85/9165	0.10	119
	85/9165	0.47	113
Okra	85/9167	0.10	131
	85/9167	0.47	132
Cucumber	85/9459	0.10	118
	85/9459	0.47	116
Aubergine	86/1492	0.10	109
	86/1492	0.47	105

HRC

TABLE 17

Procedural Recovery Data

Quinalphos in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.11	77
	85/9162	0.53	81
Bird peppers	85/9165	0.11	94
	85/9165	0.53	97
Okra	85/9167	0.11	99
	85/9167	0.53	102
Cucumber	85/9459	0.11	98
	85/9459	0.53	100
Aubergine	86/1492	0.11	90
	86/1492	0.53	90

HRC

TABLE 18

Procedural Recovery Data

Thiometon in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.11	33
	85/9162	0.56	49
Bird peppers	85/9165	0.11	39
	85/9165	0.56	40
Okra	85/9167	0.11	46
	85/9167	0.56	66
Cucumber	85/9459	0.11	47
	85/9459	0.56	72
Aubergine	86/1492	0.11	57
	86/1492	0.56	44

Thiometon unstable and has obviously decomposed during analysis

HRC

TABLE 19

Procedural Recovery Data

Triazophos in food

Food type	HRC Reference	Level of fortification (ppm)	Recovery (%)
Sorghum grain	85/9162	0.10	100
	85/9162	0.50	97
Bird peppers	85/9165	0.10	115
	85/9165	0.50	111
Okra	85/9167	0.10	119
	85/9167	0.50	121
Cucumber	85/9459	0.10	115
	85/9459	0.50	116
Aubergine	86/1492	0.10	101
	86/1492	0.50	97

FIGURE 1

Typical analytical chromatography : Aldicarb in Sorghum grains

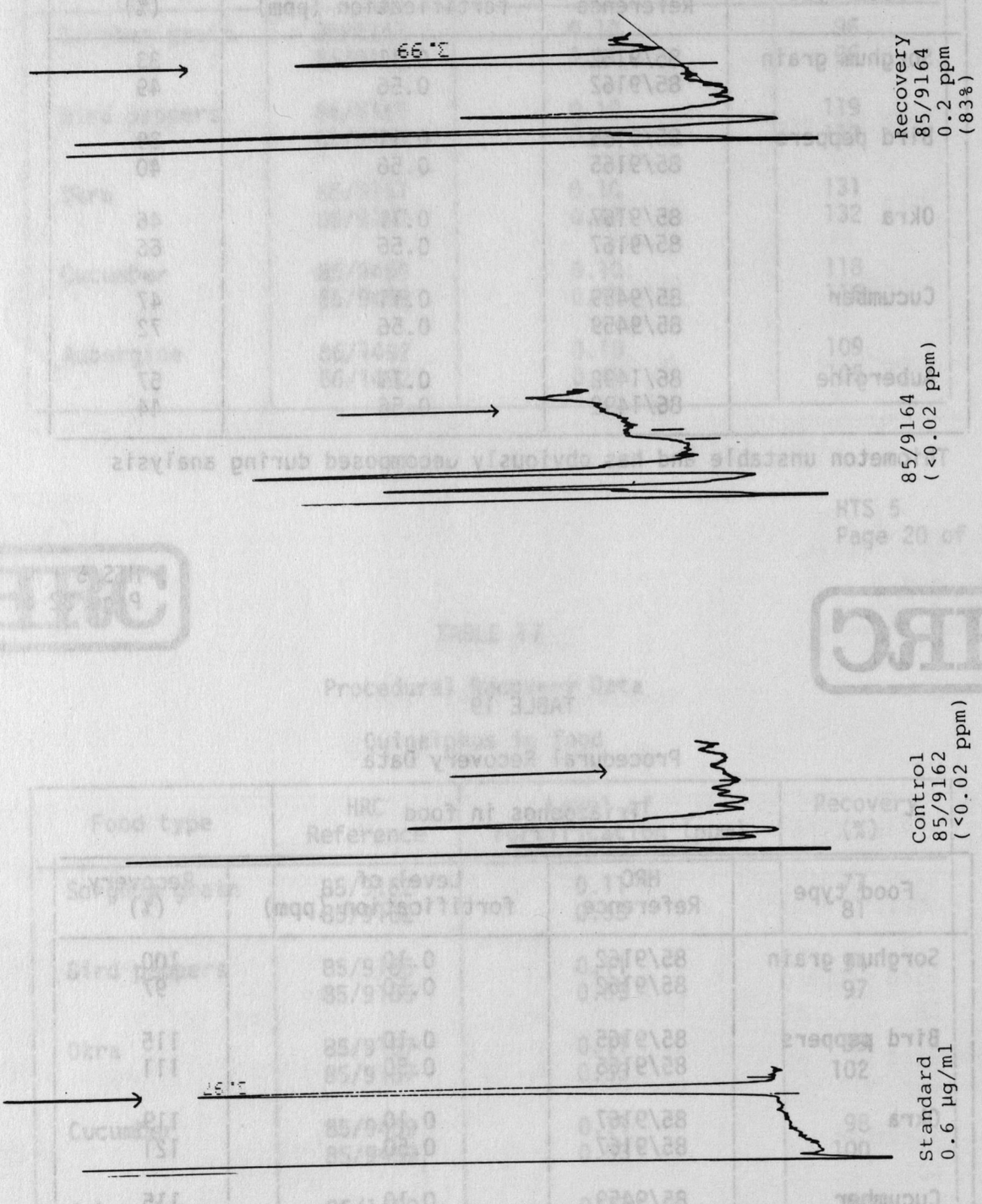


FIGURE 2

Typical analytical chromatography : Amitraz in food

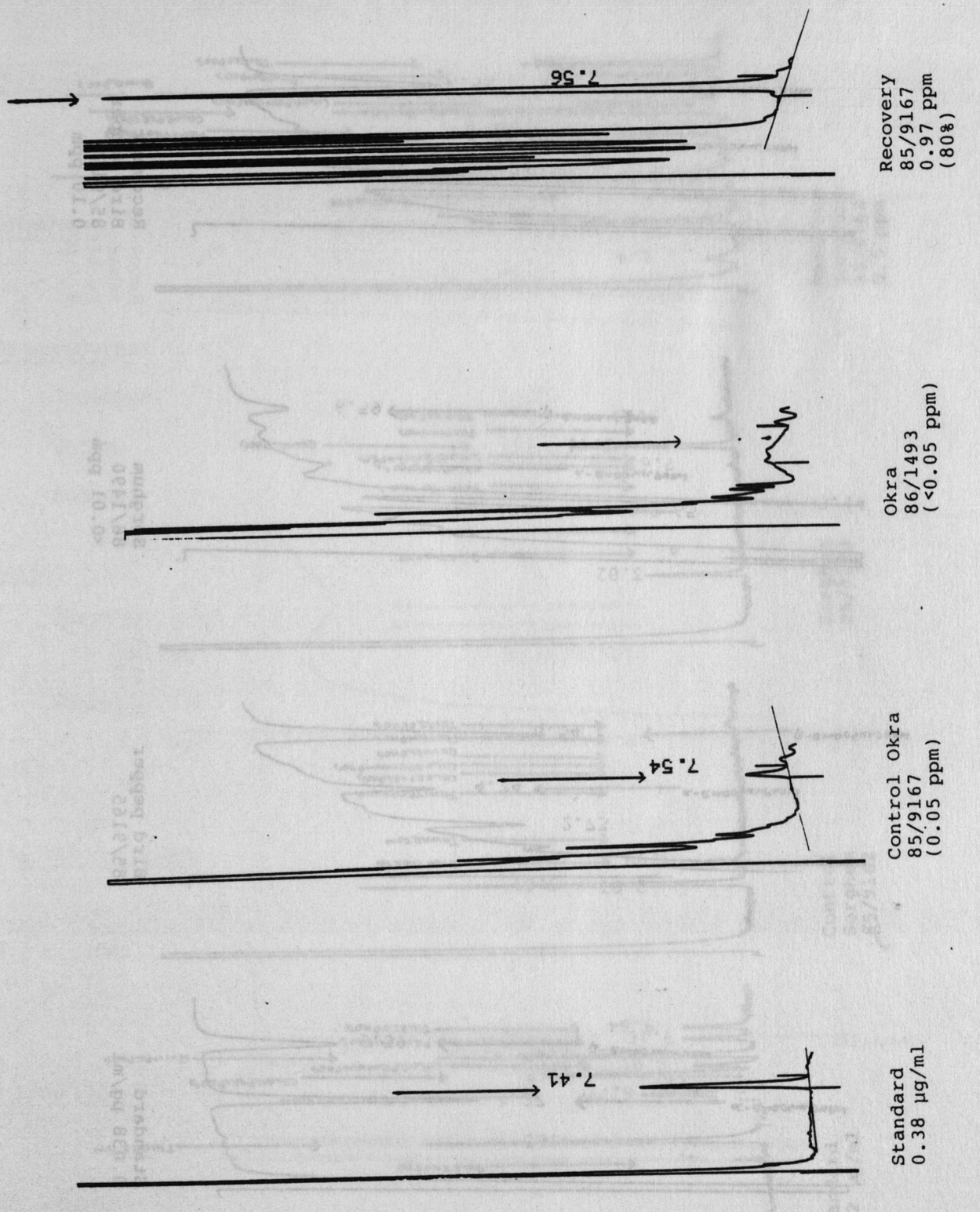


FIGURE 3

Typical analytical chromatography : Endosulphan in foods

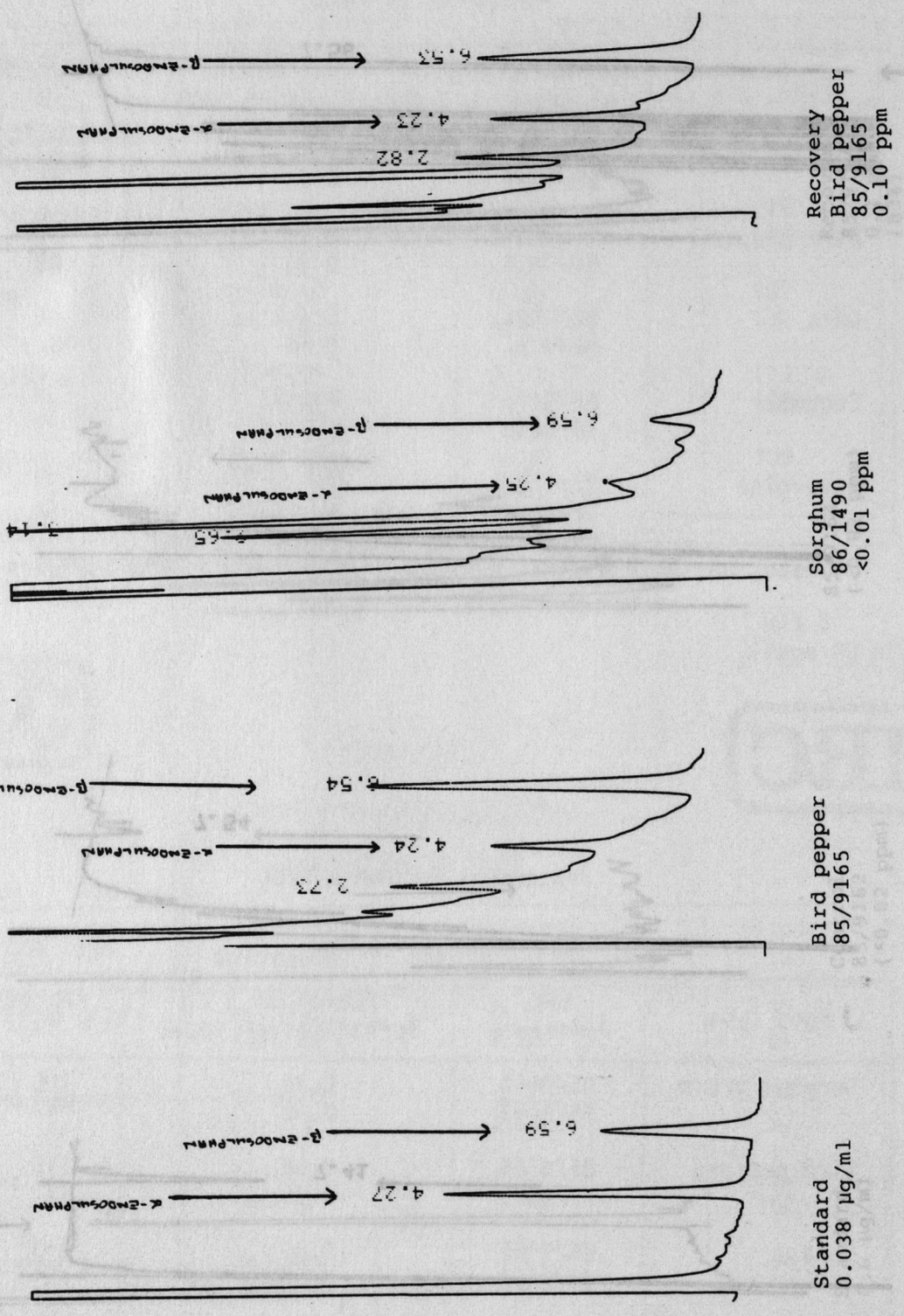


FIGURE 4

Typical analytical chromatography : Organophosphorus compounds in food

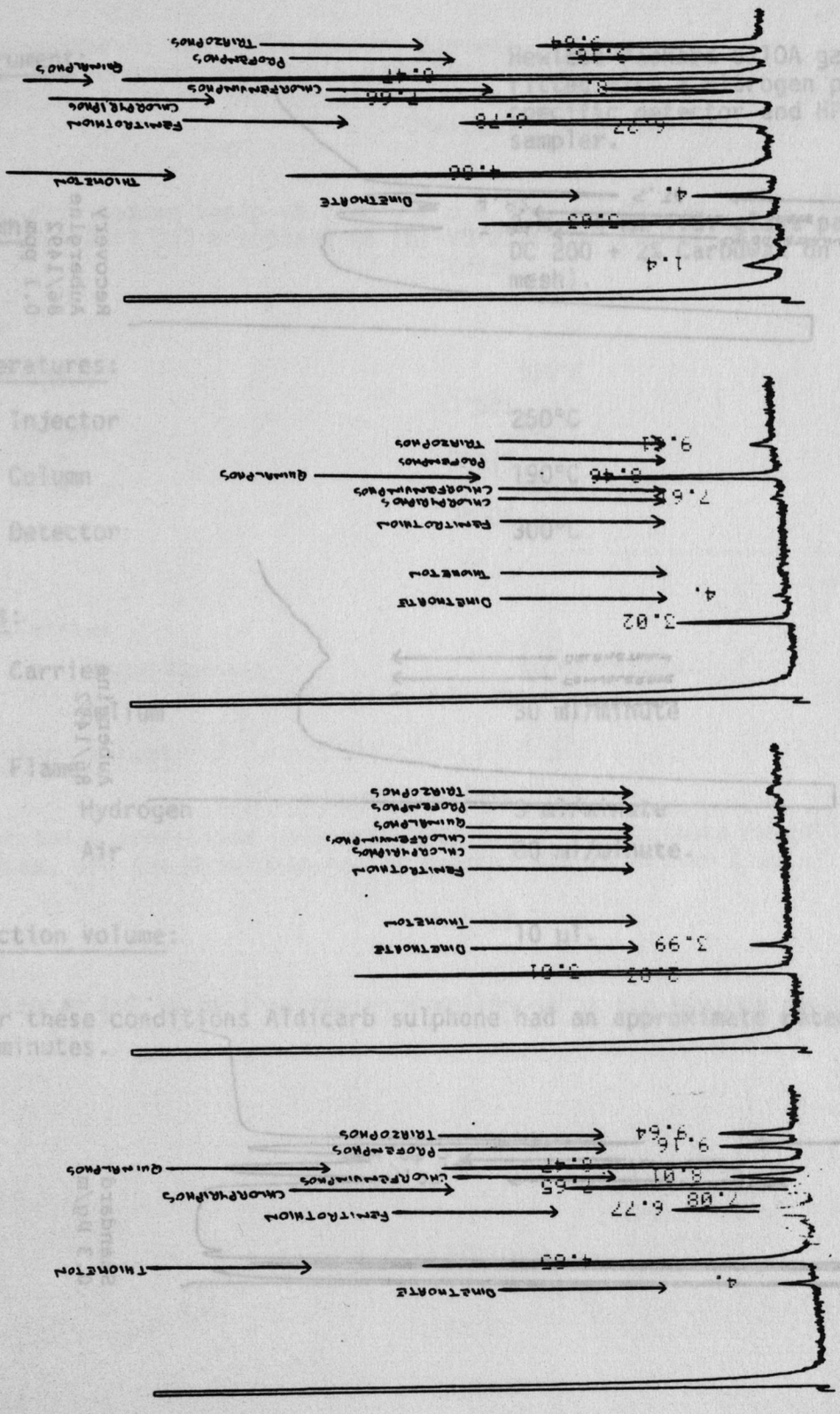
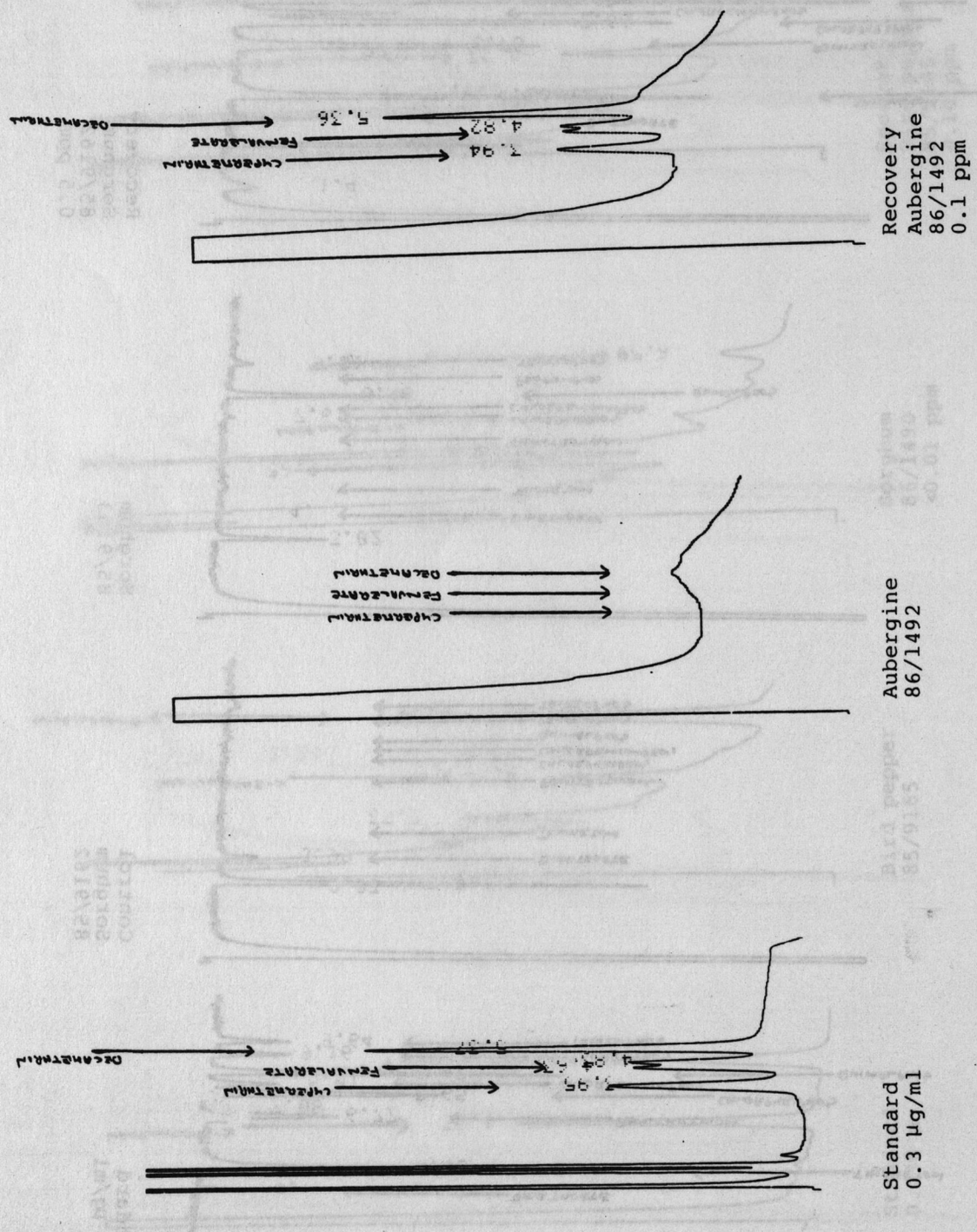


FIGURE 5

Typical analytical chromatography : Pyrethroids in food



HRC

APPENDIX 1a

Gas chromatographic conditions used for the determination of Aldicarb as Aldicarb sulphone

Instrument: Hewlett Packard 5710A gas chromatograph fitted with a nitrogen phosphorous thermionic specific detector and HP 7671A automatic sampler.

Column: 2 m x 4 mm i.d. glass packed with 5% DC 200 + 2% Carbowax on DCLQ (80-100 mesh).

Temperatures:

Injector	250°C
Column	190°C
Detector	300°C

Gases:

Carrier	
Helium	30 ml/minute
Flame	
Hydrogen	3 ml/minute
Air	80 ml/minute.

Injection volume: 10 µl.

Under these conditions Aldicarb sulphone had an approximate retention time of 3.9 minutes.

HRC

APPENDIX 1b

Gas chromatographic conditions used for the determination of amitraz in food

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a nitrogen phosphorus thermionic specific detector and HP 7671A automatic sampler.
Column:	1.8 m x 2 mm i.d. glass packed with 5% OV-101 on Diatomite CLQ (100-120 mesh).
Temperatures:	
Injector	250°C
Column	210°C
Detector	300°C
Gases:	
Carrier	
Nitrogen	35 ml/minute
Flame	
Hydrogen	3 ml/minute
Air	60 ml/minute.
Injection volume:	10 µl.

Under these conditions Amitraz had an approximate retention time of 7.2 minutes.

HRC

APPENDIX 1c

Gas chromatographic conditions used for the determination of endosulphan in food

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	2 m x 4 mm i.d. glass packed with 5% OV-210 on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	245°C
Detector	350°C
Gases:	
Carrier	
Argon/Methane	40 ml/minute.
Injection volume:	5 µl.

Under these conditions α-endosulphan had an approximate retention time of 4.3 minutes, and β-endosulphan had an approximate retention time of 6.6 minutes.

HRC

APPENDIX 1d

Gas chromatographic conditions used for the determination of organophosphorus compounds (chlorpyriphos, chlorfenvinphos, dimethoate, fenitrothion, profenphos, quinalphos, thiometon, triazophos) in foo

Instrument: Varian Vista 6000 fitted with a flame photometric detector in the phosphorus mode and an autosampler.

Column: 2 m x 2 mm i.d. glass packed with 2½% Apiezon L in Gas Chrom Q (100-120 mesh).

Temperatures:

Injector	225°C
Column	130°C(1 min) —25°/min→ 200°C —3°/min→ 210°C —50°/min→ 270°C(6 min)
Detector	250°C

Gases:

Carrier	
Helium	30 ml/minute
Flame	
Hydrogen	140 ml/minute
Air	170 ml/minute.

Injection volume: 8 µl.

Under these conditions the organophosphorus compounds had the following approximate retention times:

Chlorpyriphos	7.7 minutes
Chlorfenvinphos	8.1 minutes
Dimethoate	4.1 minutes
Fenitrothion	6.8 minutes
Profenphos	9.2 minutes
Quinalphos	8.5 minutes
Thiometon	4.9 minutes
Triazophos	9.7 minutes

HRC

APPENDIX 1e

Gas chromatographic conditions used for the determination of pyrethroids (cypermethrin, decamethrin and fenvalerate) in food

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 1% OV-101 on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	215°C(2 min) $\xrightarrow{8°C/min}$ 240°C(4 min)
Detector	300°C
Gases:	
Carrier	
Argon/Methane	60 ml/minute.
Injection volume:	5 µl.

Under these conditions cypermethrin, decamethrin and fenvalerate had approximate retention times of 3.8 minutes, 5.3 minutes and 4.6 minutes respectively.

APPENDIX D

RESULTS OF GLC ANALYSES OF INSECTICIDE RESIDUES IN WATER

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DEPARTMENT OF ANALYTICAL CHEMISTRY

CERTIFICATE OF ANALYSIS

The Determination of Concentrations of Insecticides

in Water

HTS 3

3rd June 1986

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HRC Project number: HTS 3.

Date of commission: This work was commissioned by Mr. C. R. Whetnall of Hunting Technical Services Ltd on 12th November 1985.

Samples: Samples were received bewteen December 1985 and March 1986 in a good condition and extracted immediately on receipt. Extracts were stored at -20°C prior to quantitation by GLC. Sample W4 was lost in transit prior to receipt by HRC.

Method:

1. Aldicarb

The samples were analysed by shaking with peracetic acid for 2 minutes to oxidise aldicarb to aldicarb sulphone. The mixture was neutralised with 10% aqueous sodium hydrogen carbonate solution and the combined aqueous solution extracted with chloroform. The chloroform was dried through anhydrous sodium sulphate and added to a Florisil clean up column. The aldicarb sulphone was eluted with 5% v/v acetone in diethyl ether and 25% v/v acetone in diethyl ether, the latter fraction being collected, evaporated to dryness and taken up in acetone for analysis by GLC.

Aldicarb sulphone was quantified with a nitrogen phosphorus thermionic detector using the conditions detailed in Appendix 1a.

2. Amitraz, decamethrin, endosulphan and organophosphorus compounds

The samples were analysed by liquid partition from water into dichloromethane. the extract was evaporated to dryness and taken up in 1 : 1 toluene : 2,2,4-trimethylpentane for analysis by GLC.

a) Amitraz was quantified on an OV-101 column with a nitrogen phosphorus thermionic specific detector using the conditions detailed in Appendix 1b.

b) Decamethrin, cypermethrin and fenvalerate were quantified on an OV-101 column with an electron capture detector using the conditions detailed in Appendix 1e.

c) Endosulphan was quantified on a capillary column with an electron capture detector using the conditions detailed in Appendix 1c.

d) Organophosphorus compounds were quantified on an Apiezon L column with a flame photometric detector in the phosphorus mode using conditions detailed in Appendix 1d.

Results:

The results obtained, uncorrected for control or procedural recovery values, are shown in Tables 1-5. Procedural recovery values, corrected for appropriate control residues, are shown in Tables 6-17.

Endosulphan is shown as the total of α and β isomers.

Typical analytical chromatograms are shown in Figures 1-5.

HRC tap water was used as control sample, with no detectable residues being present.

Procedural recoveries were also performed on HRC tap water. Organophosphorus compounds (6 components) were determined simultaneously by GLC. The pyrethroids (3 components) were also determined by GLC. Results are reported as parts per billion (ppb) equivalent to micrograms/litre except for aldicarb, which is reported as parts per million (ppm).

Chemistry Laboratory Notebook Reference: 2421/86/06

TABLE 1

Concentrations of Aldicarb found in water

HRC Reference	Sample Description	Aldicarb (ppm)
85/9168*	Sample W1 Canal water 3.12.85	-
85/9169*	Sample W2 Tap water 3.12.85	-
85/9447*	Sample W3 Canal water 18.12.85	-
86/1484	Sample W5 Canal water (Gezira)	<0.005
86/1485	Sample W6 Tap water (Gezira)	<0.005
86/1486	Sample W7 Canal water (Gezira)	<0.005
86/1487	Sample W8 Tap water (Gezira)	<0.005
86/1488	Sample W9 Canal water (El Geiger)	<0.005

* Insufficient sample for analysis to be performed

TABLE 2

Concentrations of Amitraz found in water

HRC Reference	Sample Description	Amitraz (ppb)
85/9168	Sample W1 Canal water 3.12.85	<0.05
85/9169	Sample W2 Tap water 3.12.85	<0.05
85/9447	Sample W3 Canal water 18.12.85	<0.05
86/1484	Sample W5 Canal water (Gezira)	<0.05
86/1485	Sample W6 Tap water (Gezira)	<0.05
86/1486	Sample W7 Canal water (Gezira)	<0.05
86/1487	Sample W8 Tap water (Gezira)	<0.05
86/1488	Sample W9 Canal water (El Geiger)	<0.05

TABLE 3

Concentrations of Endosulphan found in water

HRC Reference	Sample Description	Endosulphan (ppb)
85/9168	Sample W1 Canal water 3.12.85	2.82
85/9169	Sample W2 Tap water 3.12.85	0.01
85/9447	Sample W3 Canal water 18.12.85	9.71
86/1484	Sample W5 Canal water (Gezira)	<0.01
85/1485	Sample W6 Tap water (Gezira)	<0.01
85/1486	Sample W7 Canal water (Gezira)	0.01
85/1487	Sample W8 Tap water (Gezira)	<0.01
85/1488	Sample W9 Canal water (El Geiger)	<0.01

TABLE 4

Concentrations of Organophosphorus compounds found in water

HRC Reference	Sample Description	Chlorpyriphos (ppb)	Dimethoate (ppb)	Profenphos (ppb)	Quinalphos (ppb)	Thiometon (ppb)	Triazophos (ppb)
85/9168	Sample W1 Canal water 3.12.85	0.97	0.12	1.13	0.35	0.08	0.32
85/9169	Sample W2 Tap water 3.12.85	<0.02	<0.10	<0.05	<0.01	<0.01	<0.05
85/9447	Sample W3 Canal water 18.12.85	3.02	0.26	0.19	0.11	0.96	0.14
86/1484	Sample W5 Canal water (Gezira)	<0.02	<0.10	<0.05	<0.01	<0.01	<0.05
86/1485	Sample W6 Tap water (Gezira)	<0.02	<0.10	<0.05	<0.01	<0.01	<0.05
86/1486	Sample W7 Canal water (Gezira)	0.02	<0.10	<0.05	<0.01	0.08	<0.05
86/1487	Sample W8 Tap water (Gezira)	<0.02	<0.10	<0.05	<0.01	<0.01	<0.05
86/1488	Sample W9 Canal water (El Geiger)	<0.02	0.15	<0.05	<0.01	<0.01	<0.05

TABLE 5

Concentrations of Pyrethroid compounds found in water

HRC Reference	Sample Description	Cypermethrin (ppb)	Decamethrin (ppb)	Fenvalerate (ppb)
85/9168	Sample W1 Canal water 3.12.85	<0.05	<0.02	<0.05
85/9169	Sample W2 Tap water 3.12.85	<0.05	<0.02	<0.05
85/9447	Sample W3 Canal water 18.12.85	<0.05	<0.02	<0.05
86/1484	Sample W5 Canal water (Gezira)	<0.05	<0.02	<0.05
86/1485	Sample W6 Tap water (Gezira)	<0.05	<0.02	<0.05
86/1486	Sample W7 Canal water (Gezira)	<0.05	<0.02	<0.05
86/1487	Sample W8 Tap water (Gezira)	<0.05	<0.02	<0.05
86/1488	Sample W9 Canal water (El Geiger)	0.13	<0.02	<0.05

TABLE 6

Procedural Recovery Data

Aldicarb in water

HRC Reference	Level of fortification (ppm)	Recovery (%)
HRC Water	0.25	76
HRC Water	0.50	73

TABLE 7

Procedural Recovery Data

Amitraz in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.48	85
HRC Water	1.91	111

TABLE 8

Procedural Recovery Data

Chlorpyriphos in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.5	119
HRC Water	4.0	76

TABLE 9

Procedural Recovery Data

Cypermethrin in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.55	77
HRC Water	2.2	78

TABLE 10

Procedural Recovery Data

Decamethrin in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.58	78
HRC Water	2.34	74

TABLE 11

Procedural Recovery Data

Dimethoate in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.51	147
HRC Water	4.08	74

TABLE 12

Procedural Recovery Data

Endosulphan in water

HRC Reference	Level of fortification (ppb)	α-endosulphan Recovery (%)	β-endosulphan Recovery (%)
HRC Water	0.48	91	95
HRC Water	1.93	69	74

TABLE 13

Procedural Recovery Data

Fenvalerate in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.50	75
HRC Water	2.01	75

TABLE 14

Procedural Recovery Data

Profenphos in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.47	121
HRC Water	3.78	80

TABLE 15

Procedural Recovery Data

Quinalphos in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.53	120
HRC Water	4.22	76

TABLE 16

Procedural Recovery Data

Thiometon in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.56	95
HRC Water	4.44	76

TABLE 17

Procedural Recovery Data

Triazophos in water

HRC Reference	Level of fortification (ppb)	Recovery (%)
HRC Water	0.5	116
HRC Water	4.0	72

FIGURE 1

Typical analytical chromatography : Aldicarb in water

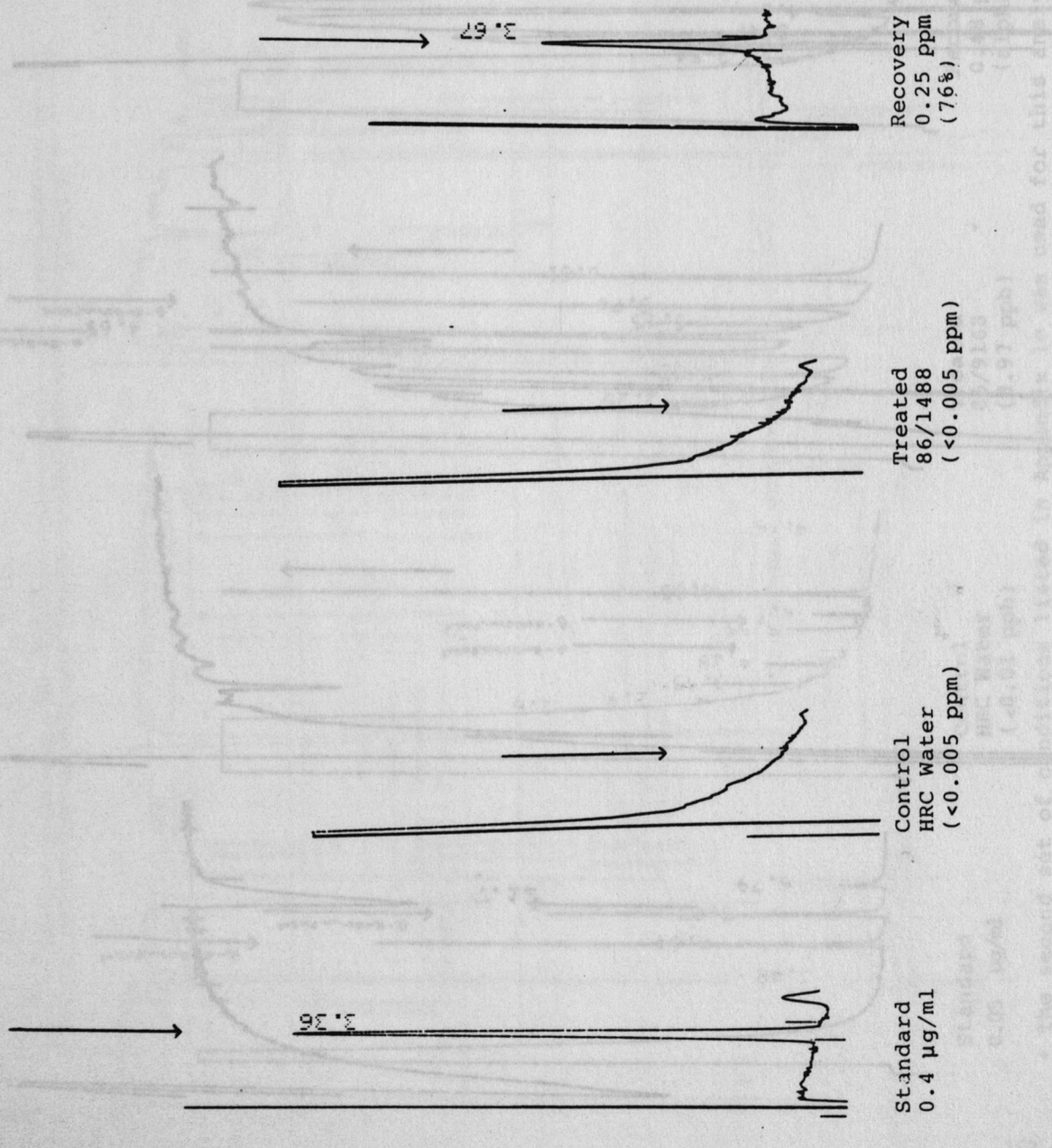


FIGURE 2

Typical analytical chromatography : Amitraz in water

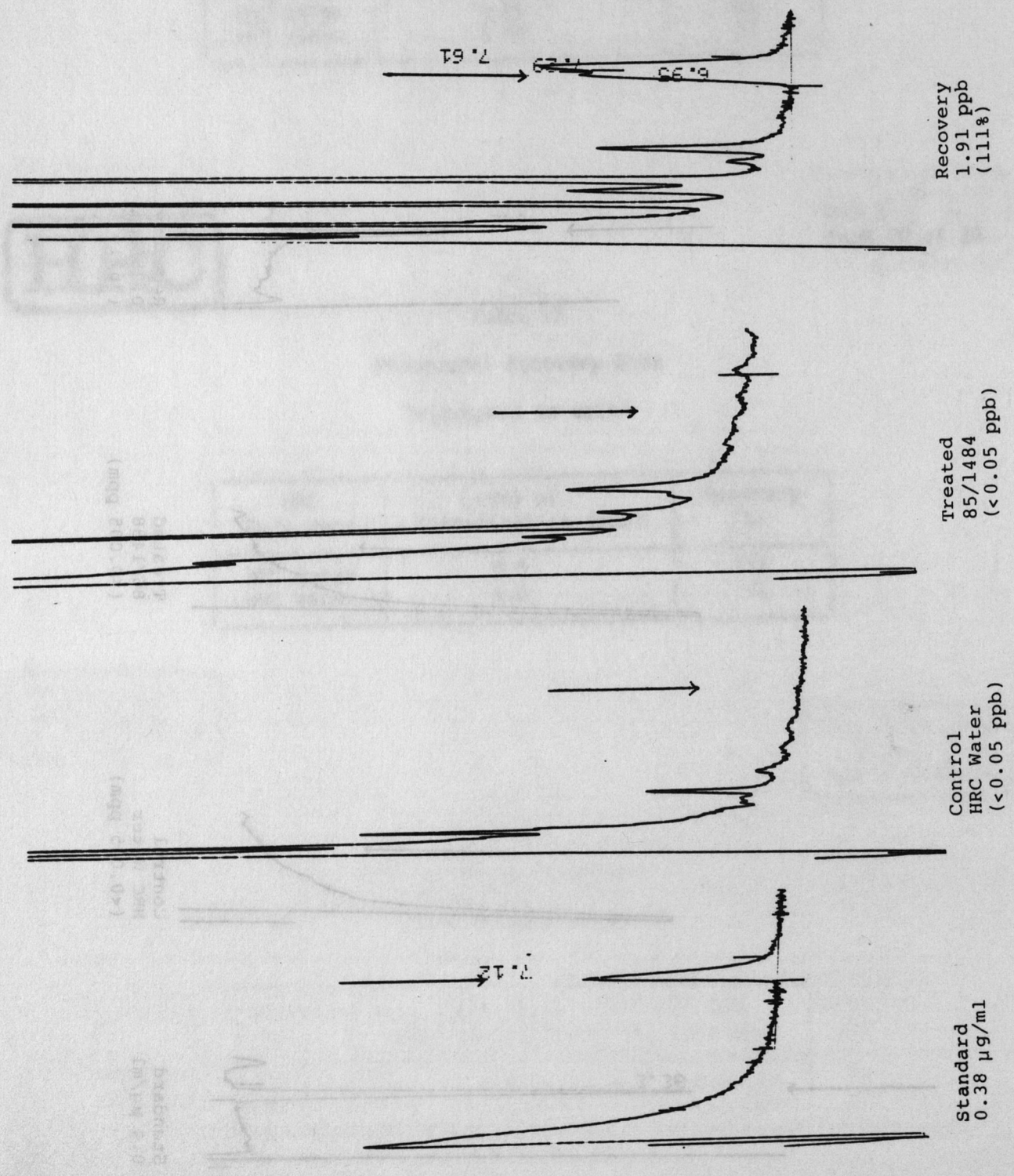
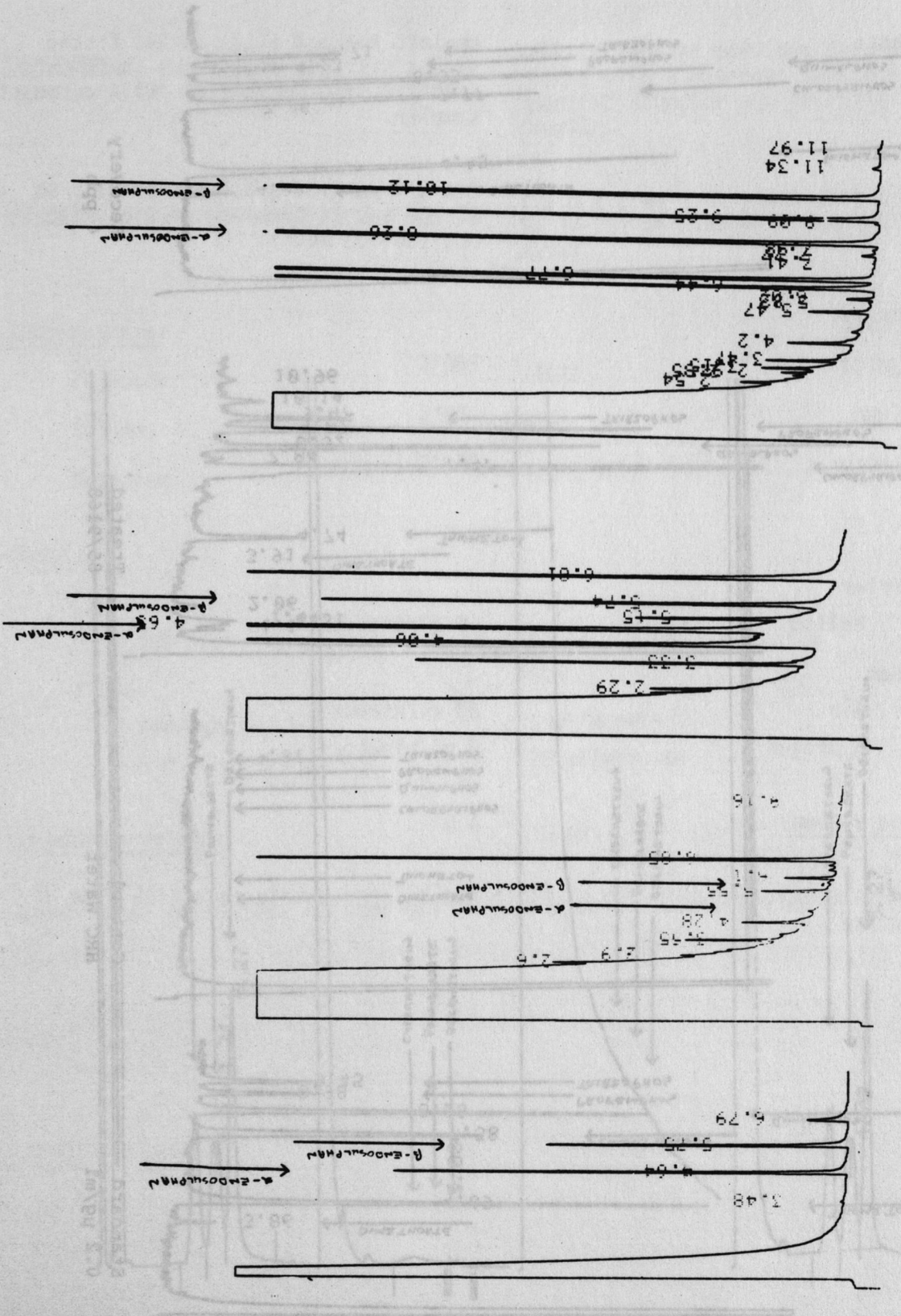


FIGURE 3

Typical analytical chromatography : Endosulphan in water



+ The second set of conditions listed in Appendix lc was used for this analysis

FIGURE 4

Typical analytical chromatography : Organophosphorus compounds in water

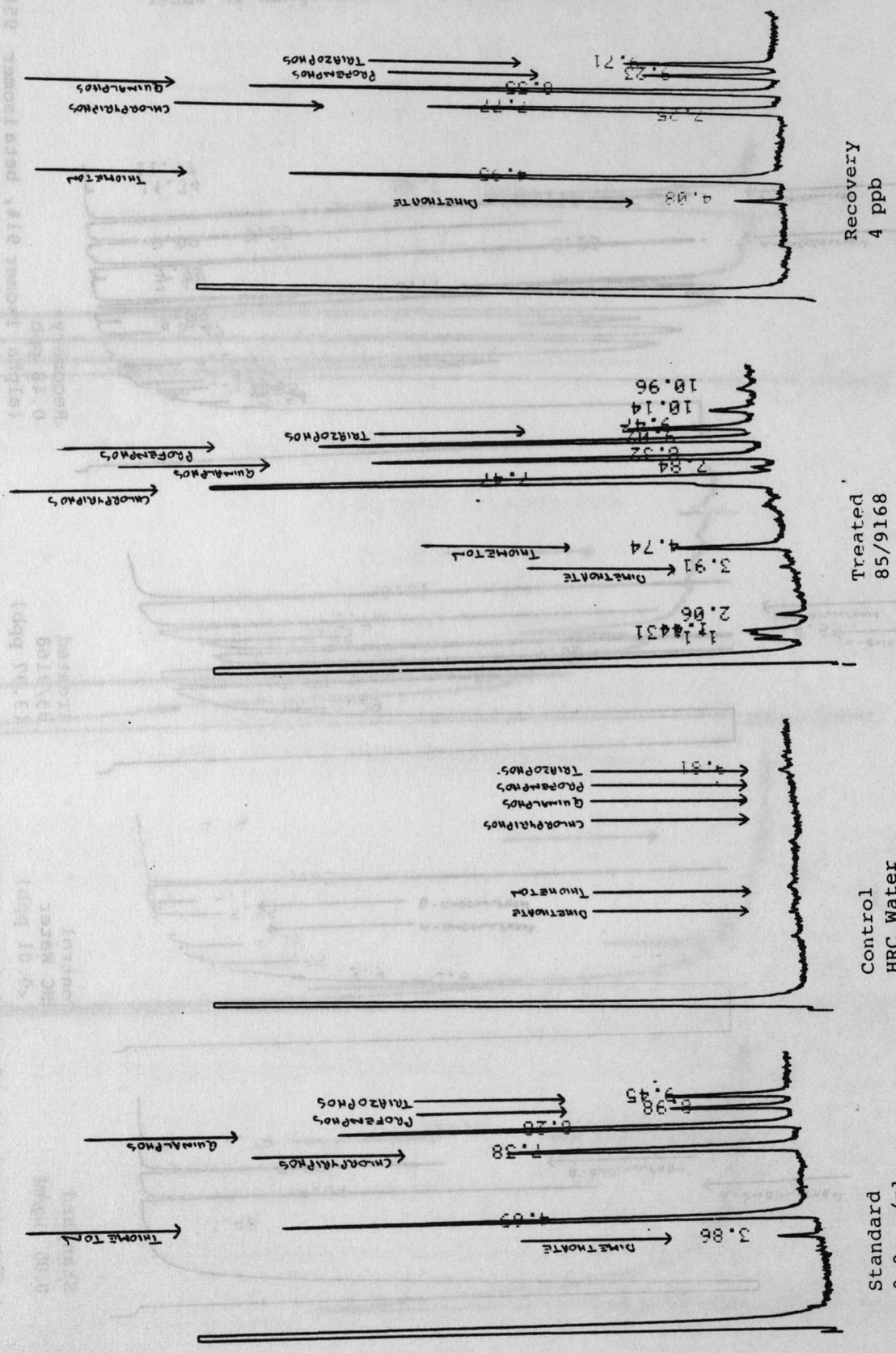
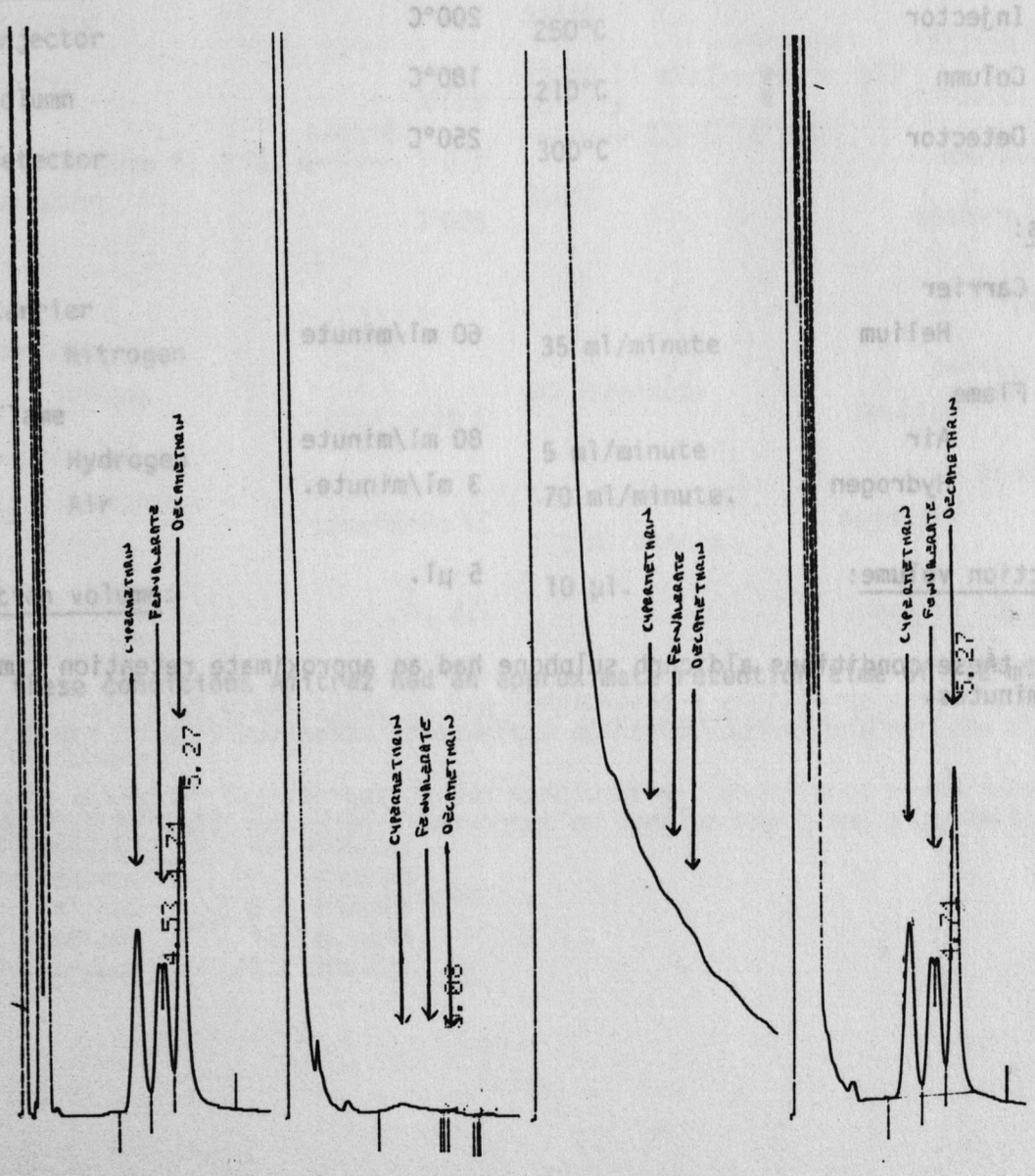


FIGURE 5

Typical analytical chromatography : Pyrethroids in water



Standard	Control	Treated	Recovery
0.35 µg/ml	HRC Water	85/9447	0.58 ppb

APPENDIX 1a

Gas chromatographic conditions used for the determination of aldicarb in water

Instrument:	Hewlett Packard 5710A series fitted with a nitrogen phosphorus thermionic specific detector and HP 7671A automatic sampler.
Column:	2 m x 4 mm i.d. glass packed with 5% DC-200 and 2% Carbowax on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	200°C
Column	180°C
Detector	250°C
Gases:	
Carrier	
Helium	60 ml/minute
Flame	
Air	80 ml/minute
Hydrogen	3 ml/minute.
Injection volume:	5 µl.

Under these conditions aldicarb sulphone had an approximate retention time of 3.1 minutes.

APPENDIX 1b

Gas chromatographic conditions used for the determination of amitraz in water

Instrument:	Hewlett Packard 5890 gas chromatograph fitted with a nitrogen phosphorus thermionic specific detector and HP 7671A automatic sampler.
Column:	1.8 m x 2 mm i.d. glass packed with 5% OV-101 on Diatomite CLQ (100-120 mesh).
Temperatures:	
Injector	250°C
Column	210°C
Detector	300°C
Gases:	
Carrier	
Nitrogen	35 ml/minute
Flame	
Hydrogen	5 ml/minute
Air	70 ml/minute.
Injection volume:	10 µl.

Under these conditions Amitraz had an approximate retention time of 7.2 minutes.

APPENDIX 1c

Gas chromatographic conditions used for the determination of endosulphan in water

Instrument:		Varian Vista 6000 series fitted with a ^{63}Ni electron capture detector and a Varian 8000 autosampler.
Column:	1.	12 m x 0.22 mm i.d. BP 5 bonded phase capillary column (0.25 µm film)
	2.	12 m x 0.32 mm i.d. BP 5 bonded phase capillary column (0.50 µm film)
Temperatures:		
Injector		250°C
Column		170°C $\xrightarrow{5°/min}$ 250°C (7 min)
Detector		250°C
Gases:		
Carrier		
Helium		3 ml/minute
Make up		
Nitrogen		27 ml/minute
Injection volume:		1.5 µl.

1. Under these conditions α-endosulphan had an approximate retention time of 4.6 minutes and β-endosulphan an approximate retention time of 5.8 minutes.

2. Under these conditions α-endosulphan had an approximate retention time of 8.2 minutes and β-endosulphan an approximate retention time of 10.1 minutes.

APPENDIX 1d

Gas chromatographic conditions used for the determination of organophosphorus compounds (chlorpyriphos, dimethoate, profenphos, quinalphos, thiometon, triazophos) in water

Instrument:	Varian Vista 6000 fitted with a flame photometric detector in the phosphorus mode and a Varian 8000 autosampler.
Column:	2 m x 2 mm i.d. glass packed with 2½% Apiezon L on Gas Chrom Q (100-120 mesh).
Temperatures:	
Injector	225°C
Column	130°C (1 min) —25°/min→ 200°C —8°/min→ 210°C —50°/min→ 270°C (6 min)
Detector	250°C
Gases:	
Carrier	
Helium	30 ml/minute
Flame	
Hydrogen	140 ml/minute
Air	170 ml/minute.
Injection volume:	8 µl

Under these conditions the organophosphorus compounds had the following approximate retention times:

Chlorpyriphos	7.7 minutes
Dimethoate	4.1 minutes
Profenphos	9.2 minutes
Quinalphos	8.5 minutes
Thiometon	4.9 minutes
Triazophos	9.7 minutes

APPENDIX 1e

Gas chromatographic conditions used for the determination of pyrethroids in water

Instrument:	Hewlett Packard 5710A gas chromatograph fitted with a ^{63}Ni electron capture detector and HP 7671A automatic sampler.
Column:	1 m x 4 mm i.d. glass packed with 1% OV-101 on Diatomite CLQ (80-100 mesh).
Temperatures:	
Injector	300°C
Column	215°C (2 mins) ⟶ 8°C/min ⟶ 240°C (4 mins)
Detector	300°C
Gases:	
Carrier	
Argon/Methane	60 ml/minute.
Injection volume:	5 µl.

Under these conditions the pyrethroid compounds had the following approximate retention times:

Cypermethrin	3.8 minutes
Decamethrin	5.3 minutes
Fenvalerate	4.6 minutes