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THE PERSISTENCE OF DEPOSITS OF DDT, BHC AND DIELDRIN  
ON MUD SURFACES IN WESTERN SOKOTO<sup>1</sup>

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## 1. INTRODUCTION

The disappearance and inactivation of insecticide deposits from mud surfaces is a well known phenomenon in malaria control by residual spraying. Hadaway and Barlow<sup>(1)</sup> have attributed the rapid disappearance from these surfaces to adsorption. This renders the insecticides unavailable to alighting insects. Since the adsorption process occurs from the vapour phase, an insecticide having a high vapour pressure will be more rapidly adsorbed than one with a low vapour pressure. Thus BHC will be removed more rapidly than either DDT or dieldrin. Since the vapour pressure usually increases with increase of temperature, this factor will also speed up the adsorption of insecticides. However it has been maintained that, whereas complete loss of biological activity occurs with DDT and dieldrin, the more volatile BHC is desorbed from the absorbent surface and reactivates it.

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<sup>1</sup> The work described in this paper forms a part of the programme of research carried out in connexion with the Western Sokoto Malaria Control Pilot Project in Northern Nigeria and is in the nature of an interim report. The conclusions stated are, therefore, tentative and may be modified by the results of subsequent investigations.

## 2. METHODS

The residual surface deposits of the three insecticides were sampled by carefully scraping away a representative area of the wall surface aiming to remove as little as possible of the underlying wall. It was not found possible to remove less than 0.3-0.4 mm of the wall in depth except on very smooth surfaces.

After extraction with suitable solvents the insecticide content of the scraping was determined chemically. The techniques described below are standard methods modified to suit tropical conditions. DDT was determined colorimetrically using the method of Schechter <sup>(2)</sup>; BHC by a sodio-reduction total chlorine technique in n-hexane solution; dieldrin by a semi-micro Stepanow method followed by turbidimetric estimation of chloride <sup>(3)</sup>.

### 2.1 Estimation of DDT

After extraction with acetone the DDT was determined colorimetrically using a modification of the method by Schechter as described by Barlow (loc. cit.)

A standard curve was obtained using known amounts of technical DDT. Aliquots of a solution containing 4 mg of DDT in 100 ml acetone were pipetted into a series of test tubes to give a range of DDT concentrations from 0 to 200 micrograms. After removal of the solvent on the water bath, 1 ml of a nitrating mixture, containing equal volumes of fuming nitric acid, S.G. 1.52, and concentrated sulphuric acid, S.G. 1.84, was added. The test tubes were placed in boiling water for 20 minutes and, after cooling, 4 ml of cold distilled water was added. The contents of the tubes were poured into separating funnels and the tubes washed out with small amounts of distilled water to a total volume of 10 ml. 8 ml of AnalaR Benzene was added and after shaking for one minute, the aqueous layer was run off and discarded. Approximately 6 ml of the benzene extract was transferred to a dry stoppered tube and 0.2 g of anhydrous sodium carbonate added. Finally 4 ml of the benzene extract was transferred to a 12 ml ground glass stopped pyrex tube. 6 ml of a solution of 5% sodium methoxide in dry methyl alcohol was added down the side of the tube. After thoroughly mixing the blue colour was allowed to develop for exactly 60 seconds and the absorption read using an absorptiometer with a filter giving a maximum transmission at 5,800 Å.

In order to check the linear nature of the standard curve, two aliquots of the unknown DDT solution were taken, one of which contained exactly twice as much DDT as the other. In this way the linearity of the calibration curve was constantly under check and erroneous results, due to variable voltages, local conditions etc., were avoided.

The sodium methoxide solution was filtered with suction through a No. 4 porosity sintered glass funnel and stored in a refrigerator to avoid decomposition.

In tropical temperatures and conditions it was found that the blue colour developed its maximum intensity one minute after mixing the reagents; it then faded rapidly and had vanished after 10 minutes.

## 2.2 Estimation of BHC

BHC was determined conveniently by a total chlorine sodio-reduction technique.

In all sodio-reduction determinations it is advisable to purify the sodium to avoid the error due to the presence of variable amounts of chloride unevenly distributed through the sample.

This has been performed by carefully melting a quantity of sodium under liquid paraffin in a pyrex test tube. The 'dross' from the molten sodium was brought to the surface by gentle stirring. The clean sodium was withdrawn through a heated pipette and released under a separate quantity of liquid paraffin. The sodium was produced in a convenient form for use by remelting and drawing up into a heated 4-5 mm bore glass tube. After cooling and solidifying the sodium was extruded as required.

A solution of the BHC in n-hexane was evaporated to approximately 50 ml and, when relatively cool, 1.5 g of purified sodium, cut into small pieces, and 5 ml isopropyl alcohol were added. After refluxing for 30 minutes, excess sodium metal was removed by adding 20 ml of distilled water cautiously down the condenser. When no more sodium was visible (after 30-40 minutes) a further 20 ml of water was added. The solution was neutralized by 50% nitric acid and then adding 2.0 ml in excess. The contents of the flask were transferred to a separating funnel and the flask washed out with two successive portions of 20 ml of distilled water. After shaking vigorously the layers were allowed to settle and the aqueous portion run off into a conical flask.

The hexane layer was washed twice with 20 ml portions of water and these washings added to the contents of the conical flask.

The chloride ion in the solution was determined by Volhard titration using either 0.1 or 0.2 N silver nitrate and 0.2 or 0.4 N potassium thiocyanate.

One millilitre of 0.1 N silver nitrate is equivalent to 4.85 mg of BHC.

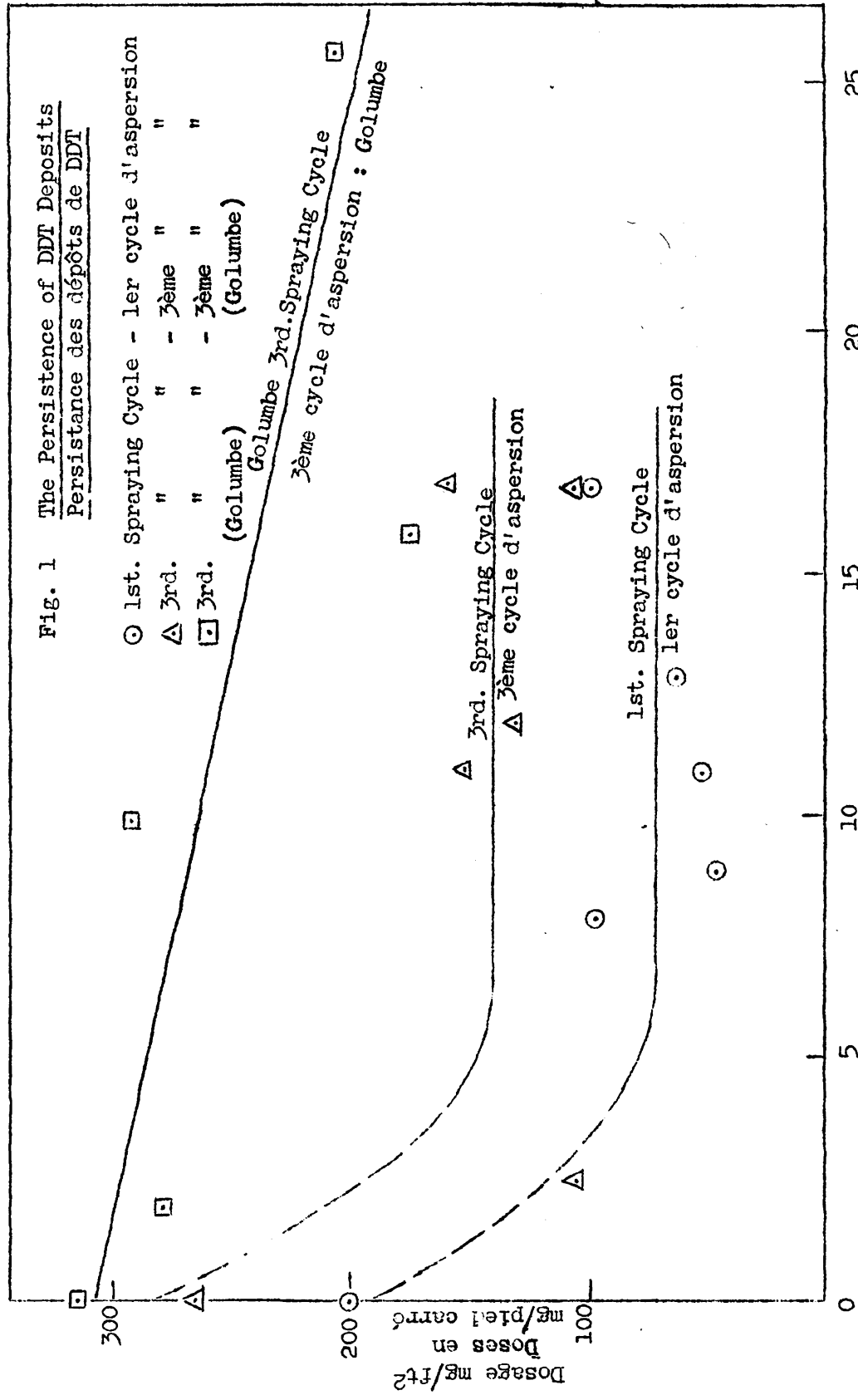
### 2.3 Estimation of dieldrin

A semi-micro Stepanow determination has been found convenient for the estimation of dieldrin residues.

An acetone or benzene extract of the dieldrin was evaporated just to dryness on the water bath in a small round bottom flask. 4 ml of dry iso-propyl alcohol and 0.3 g of purified sodium cut into small pieces were added to the residue. The reactants were gently refluxed for 30 minutes and then 2 ml of 1 : 1 aqueous iso-propyl alcohol was added. In high humidities it is important not to heat too strongly otherwise the sodium disappears before reacting with the dieldrin; a spirit lamp was successfully used at this stage. The heating was discontinued when all the residual sodium had disappeared and 5 ml of water was added down the condenser. Phenolphthalein was added and the solution neutralized with 1 : 2 nitric acid. The mixture was cooled in a refrigerator to below 10°C and then transferred to a separatory funnel. The flask was rinsed out with small amounts of cold water to a total volume of 25 ml. The solution was extracted with 10 ml of cold ether and after shaking, the aqueous layer was run off into a 100 ml conical flask. The ether layer was washed with 10 ml of cold water and the aqueous extracts combined. 5 drops of 20 volume hydrogen peroxide were added followed by 20% w/v potassium hydroxide until alkaline. The solution was boiled until no turbidity was visible, cooled, acidified with 1 : 2 nitric acid, and made up to 50 ml in a volumetric flask.

The chloride in the resulting solution was determined turbidimetrically by comparison with solutions of accurately known chloride content. This was conveniently performed by using an absorptiometer as a nephelometer with a filter giving a maximum transmission at 4,300 Å. The turbidities could also be compared visually.

Fig. 1 The Persistence of DDT Deposits  
 Persistence des dépôts de DDT



Weeks after Spraying - Nombre de semaines après l'aspersion



A standard solution of N/10 000 potassium chloride (1 ml = 0.0063 mg dieldrin) was prepared. 0, 5, 10, 15 and 20 ml aliquots of this solution were placed in 50 ml flasks. Suitable aliquots of the unknown chloride solutions, to contain a chloride equivalent of 5 to 20 ml N/10 000 potassium chloride, were taken. To each solution was added 5 ml 1:4 nitric acid, 2 ml iso-propyl alcohol and water up to approximately 30 ml. 1 ml of 0.1 N silver nitrate was added and after shaking the flasks were allowed to stand in darkness for one hour. The turbidities were rapidly measured in the absorptiometer. A calibration curve was drawn of chloride content against absorptiometer reading. For each series of unknown solutions a new series of standards were prepared, both standards and unknowns being determined at the same time.

Blank determinations were carried out on the reagents. The dieldrin content of the reagents was never found to be greater than 0.2 mg dieldrin for 300 mg sodium.

The reliability of the method is improved by taking two different aliquots of the unknown chloride solution, both within the range of the calibration curve, as was described for DDT. The linear nature of the calibration curve is thus checked at each measurement.

### 3. RESULTS

The persistence of DDT deposits on the wall surfaces in several localities are illustrated in Fig. 1. The variable dosages sprayed by the different spray teams (some typical values are shown in Table 2) make conclusions difficult.

Table 1

Persistence of DDT deposits during the first spraying-cycle  
(dosage 200 mg/ft<sup>2</sup> of technical DDT)

Age of deposit (weeks)	DDT in mg/ft <sup>2</sup>
2	277
8	96
9	43
11	50
13	64
17	102

These results are quoted from the "First Annual Report 1954-1955 of the Malaria Control Pilot Project in Western Sokoto".

Table 2

Dosages of DDT 75% water-dispersible powders sprayed during the third spraying cycle (scheduled dosage 267 mg/ft<sup>2</sup> of technical DDT)

Town	Dosage in mg/ft <sup>2</sup>						Average
	1	2	3	4	5	6	
(a)	196	295	173	286	-	-	238
(b)	339	291	319	-	-	-	316
(c)	348	229	470	309	370	372	344

The wide variations in the amount of residual DDT deposits shown in Table 3 are due to these initial uneven dosages. The results show that the superimposition of the deposits in the course of the six-monthly spraying programme enhances the residual life of succeeding deposits. Fifteen weeks after the third spraying cycle approximately twice as much DDT remains as in the first cycle. However it must be borne in mind that the dosage during the first spraying was 200 mg/ft<sup>2</sup>, and in the second and third sprayings, 267 mg/ft<sup>2</sup>, of technical DDT. The curved portions of the graphs are tentatively drawn with reference to Fig. 2.

Table 3

Persistence of DDT deposits during the third spraying cycle  
(dosage 267 mg/ft<sup>2</sup> of technical DDT)

Age of deposit (weeks)	Town	Surface deposit of DDT (0.0 to 0.30 mm) mg/ft <sup>2</sup>							
		Samples						Standard deviation	Average
		1	2	3	4	5	6		
2	(b)	304	220	328	176	332	320	156	280
4	(a)	72	116	102	124	116	88	20	103
10	(b)	198	320	344	266	240	378	157	294
11	(d)	144	102	174	188	210	90	46	152
12	(a)	258	38	216	54	104	126	88	133
16	(b)	232	204	84	166	182	-	54	174
17	(d)	144	126	76	137	-	42	43	105
17	(e)	320	80	179	141	85	168	89	162
26	(b)	256	200	160	248	160	236	43	210

Designation of towns, (a) Kardi, (b) Golumbe, (d) Bassabra, (e) Nufawa.

Golumbe, village (b) in the tables, shows a longer residual life than the average. There is no appreciable loss of DDT from the outermost surface and the deposit is still visible after six months. Work on this phenomenon is still proceeding but it is noticeable that the town is constructed with a lighter coloured mud, possibly indicating a lower iron content, than anywhere else in the area.\*

\* Samples of building soils from the Birnin-Kebbi area were analysed through the courtesy of Colonial Geological Surveys Imperial Institute, London. The results obtained were as follows:

	"Readily available" iron oxide Fe <sub>2</sub> O <sub>3</sub>	Total iron oxide Fe <sub>2</sub> O <sub>3</sub>
Sample A	0.7%	1.5%
Sample B	1.6%	3.0%

For the purpose of correlating these results with those of other workers, the first column of results, obtained by the method of C. D. Jeffries, is relevant.

Table 4 shows the distribution of DDT within the wall surface at the completion of the second and third six-monthly spraying cycles. The increase in the residual life of the deposit is clearly shown. The dosage remaining in the outermost layer at the end of the third cycle is over 2<sup>1</sup>/<sub>2</sub> times that found at the end of the previous spraying. In this case the surface appears to be approaching saturation as far as adsorption of the DDT is concerned.

Table 4  
Distribution of DDT within wall surfaces 26 weeks  
after spraying (at Columbe)

(a) At completion of second spraying cycle

Depth of wall sampled (mms)	DDT in mg/ft <sup>2</sup> at depths shown						
	Samples						Average
	1	2	3	4	5	6	
Surface - 0.4	164	56	44	48	108	64	81
" 0.4 - 0.8	120	60	48	60	104	-	78
" 0.8 - 1.2	68	32	28	44	76	-	50

(b) At completion of third spraying cycle

Depth of wall sampled (mms)	DDT in mg/ft <sup>2</sup> at depths shown						
	Samples						Average
	1	2	3	4	5	6	
Surface - 0.4	256	200	160	248	160	236	210
" 0.4 - 0.8	136	52	109	157	100	-	111
" 0.8 - 1.2	78	40	80	56	61	-	63
" 1.2 - 1.6	56	33	84	40	50	-	53

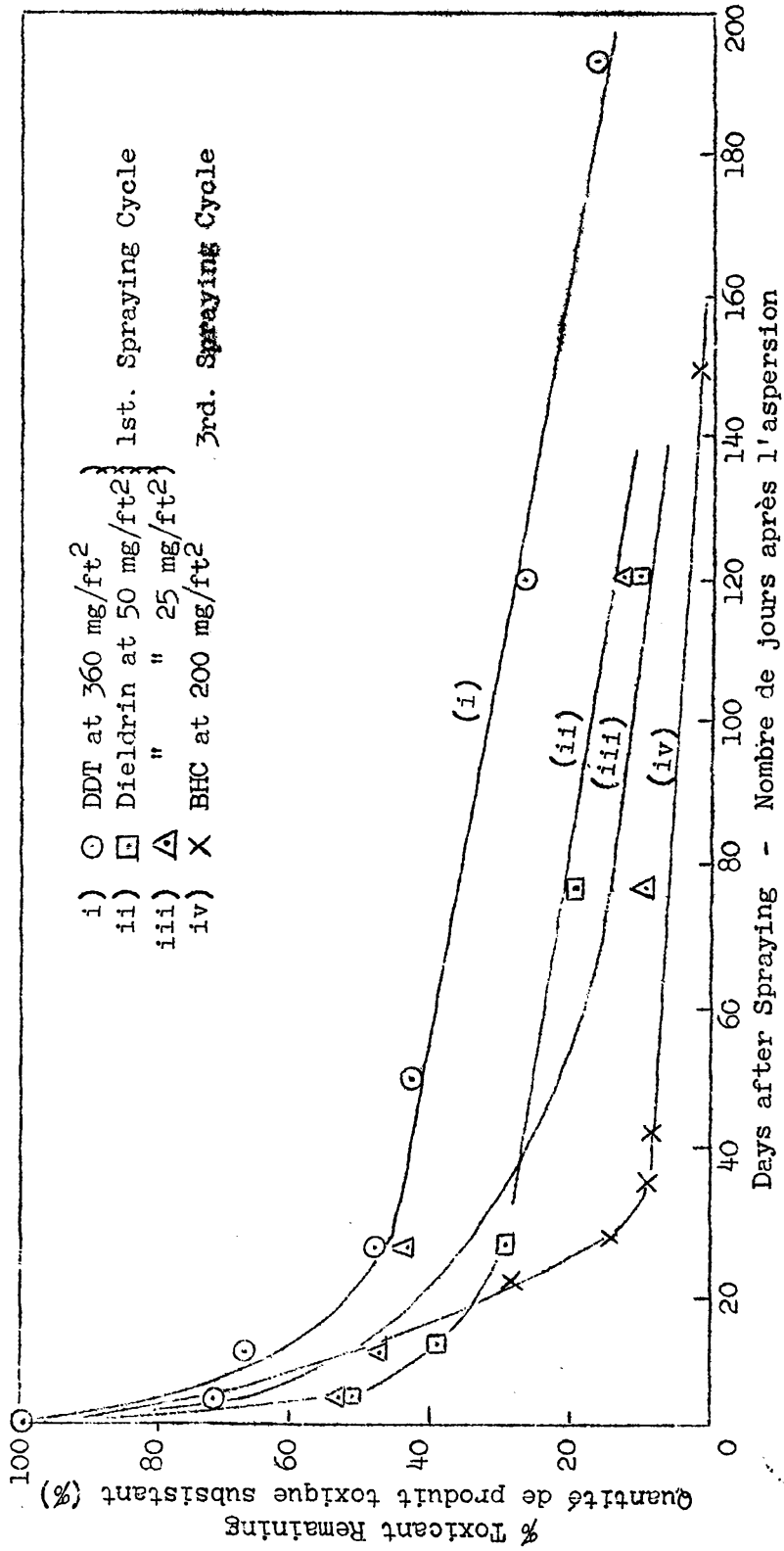
A comparison of the residual life of DDT and dieldrin dosages, the latter at both 25 and 50 mg/ft<sup>2</sup>, have been determined under conditions allowing for no outside

Note

The differences between layers of the same spraying cycle are only significant in part of this table.

Fig. 2 Persistence of Surface Deposits of DDT, Dieldrin and BHC on mud walls in Western Sokoto

Fig. 2 Persistance des dépôts superficiels de DDT, de dieldrine et d'HCH sur des murs de terre, dans le Sokoto occidental



i) ○ DDT à raison de 360 mg/pied carré  
 ii) □ Dieldrine à raison de 50 mg/pied carré } 1er cycle d'aspersion  
 iii) △ Dieldrine à raison de 25 mg/pied carré }  
 iv) X HCH à raison de 200 mg/pied carré } 3ème cycle d'aspersion



interference by the accidental removal of the deposits by cattle or other local hazards. Three identical mud huts were built conforming in every way to local building practice and these were sprayed with the standard dosage of DDT and with 25 and 50 mg/ft<sup>2</sup> of dieldrin respectively. The results are shown in Table 5 and are graphically illustrated in Fig. 2.

Table 5

Persistence of surface deposits of DDT and dieldrin  
(25 mg/ft<sup>2</sup> and 50 mg/ft<sup>2</sup>) on similar mud surfaces after the first  
spraying

Age of deposit (days)	DDT		Dieldrin 25 mg		Dieldrin 50 mg	
	Dosage mg/ft <sup>2</sup>	% dosage remaining	Dosage mg/ft <sup>2</sup>	% dosage remaining	Dosage mg/ft <sup>2</sup>	% dosage remaining
0	366	100	31.2	100	46.4	100
4	266	72.6	16.8	54.0	24.8	53.5
11	246	67.1	15.2	48.7	18.4	39.7
26	172	47.0	13.6	43.6	14.0	30.2
50	157	42.9	-	-	-	-
76	-	-	2.8	9.8	9.6	20.7
120	101	27.6	4.0	12.8	5.2	11.2
194	59	16.1	-	-	-	-

The amounts of BHC remaining on the walls during the third spraying cycle are shown in Table 6.

Table 6

Persistence of surface deposits of BHC during the third spraying cycle (dosage 25 mg/ft<sup>2</sup> gamma isomer)

Age of deposit (weeks)	Town	Surface deposit of BHC (0.0 to 0.3 mm) mg/ft <sup>2</sup>								
		Samples							Average	% remaining
		1	2	3	4	5	6	7		
3	(j)	153.1	16.2	10.6	49.7	-	-	-	57.4	29.4
4	(k)	26.0	16.4	72.8	7.3	7.3	29.8	-	26.6	13.6
4	(j)	25.8	24.4	37.5	21.8	10.9	-	-	24.1	12.4
5	(j)	14.9	37.9	18.2	12.6	8.9	10.2	-	17.1	9.0
5	(l)	9.3	18.2	10.2	17.8	13.4	14.2	-	13.8	7.1
6	(m)	17.3	27.5	19.5	14.7	18.2	18.4	-	19.2	9.7
21	(j)	7.6	5.6	3.9	2.1	1.1	9.2	2.8	4.6	2.4

Designation of towns, (j) Jega, (k) Sadam-Jega, (l) Danwarai, (m) Aliero.

Note

The results are given in mg/ft<sup>2</sup> of total BHC. The BHC formulation contained 6.5% of gamma isomer.

Fig.2 shows that all three insecticides are removed rapidly from the surface during the first two weeks after application as water-dispersible powders. After two weeks 60% of the initial DDT dosage, 50% of the BHC and 46% and 40% respectively of the 25 and 50 mg/ft<sup>2</sup> dieldrin dosages remain within 0.3 mm of the wall surface. In the cases of DDT and dieldrin this initial rapid loss is followed by a more gradual decline until, after 5 weeks, 45% of the DDT, 29% of the 25 mg and 27% of the 50 mg/ft<sup>2</sup> dieldrin deposits remain. The BHC deposit on the contrary continues to show rapid deterioration and after 5 weeks only 9% of the initial dosage remains. The rate of loss from then is approximately linear and at the end of 15 weeks 32% of the DDT, 12% of the 25 mg and 18% of the 50 mg dieldrin deposits are found in the surface layer.

BHC deposits have not been found to show any increase in residual life through the superimposition of succeeding six-monthly dosages as demonstrated for DDT.

Table 7 showing the persistence of the 25 mg dieldrin dosage during the first spraying cycle, confirms the results indicated in Fig. 2.

Table 7  
Persistence of surface deposits of dieldrin during the first spraying cycle (dosage 25 mg/ft<sup>2</sup>)

Age of deposit (weeks)	Town	Surface deposit of dieldrin in mg/ft <sup>2</sup>						Average
		Samples						
		1	2	3	4	5		
12	(f)	5.0	1.2	1.5	4.1	r	3.0	
20	(g)	0.4	1.6	3.1	2.7	6.5	2.4	

Note

These samples were analysed by the kind co-operation of "The Colonial Products Laboratory", London.

The residual life of the 50 mg dieldrin dosage, sprayed in an area after two cycles at 25 mg/ft<sup>2</sup>, is shown in Table 8. There is approximately 20% of the initial deposit remaining between 16 and 18 weeks after application.

Table 8  
Persistence of surface deposits of dieldrin during the third spraying cycle (dosage 50 mg/ft<sup>2</sup>)

Age of deposit (weeks)	Town	Surface deposit of dieldrin in mg/ft <sup>2</sup>								Average
		Samples								
		1	2	3	4	5	6	7	8	
16	(f)	15.2	9.2	11.2	8.4	8.8	9.2	11.2	-	10.5
18	(h)	13.2	4.8	12.0	11.7	7.6	8.8	10.4	13.2	10.2

Designation of towns for Tables 7 and 8: (f) Ambursa, (g) Janzomo and (h) Gwandangaji.

Table 9 showing the dosages of the dieldrin water-dispersible powder sprayed during third cycle, indicates that the scheduled 50 mg/ft<sup>2</sup> dosage was exceeded and therefore the small enhancement of the 50 mg deposit by superimposition as shown by Table 8 may not be real.

Table 9  
Dosages of dieldrin 50% water-dispersible powders sprayed  
during the third spraying cycle (50 mg/ft<sup>2</sup>)

Town	Dosage mg/ft <sup>2</sup>						Average
	1	2	3	4	5	6	
(i)	79.2	70.4	41.8	49.0	-	-	60.1
(h)	84.4	64.9	83.4	48.8	48.8	62.2	65.4

Designation of towns (h) Gwandangaji, (i) Kalgo.

The weathering of DDT and dieldrin dosages is illustrated by Table 10. Table 10(i) shows the effect of exposure to non-shade temperatures on the outside walls of the three huts. The higher wall temperatures cause increased adsorption of both the 25 and 50 mg dieldrin dosages as would be expected. At the end of 25 days there is a 50% difference between the residual life of the dosage on the outer walls and that on the inner. No comparable results are available for DDT. In Table 10 (ii) the deposits were subjected to a heavy storm 2 days after spraying which washed away an appreciable quantity of the outermost mud surface. The larger percentages of the dieldrin dosages remaining may be due to the more rapid adsorption of the dieldrin during the first two days after application than occurs with DDT. The action of the rain would remove most of the less rapidly adsorbed DDT deposit whilst a considerable proportion of the dieldrin dosage would be in the sorbed condition and so protected from the rain.

Table 10

Effect of direct exposure of deposits of DDT and dieldrin to atmospheric conditions (on the outer walls of mud dwellings)

(i) Shielded from rain action but not sun

Age of deposit (days)	Dieldrin 25 mg/ft <sup>2</sup>			Dieldrin 50 mg/ft <sup>2</sup>		
	Dosage mg/ft <sup>2</sup>	Residual dosage %	Inside dosage %	Dosage mg/ft <sup>2</sup>	Residual dosage %	Inside dosage %
0	36.1	100	100	53.7	100	100
4	14.8	41.0	54.0	23.6	43.9	53.5
11	16.0	44.3	48.7	14.0	26.1	39.7
25	8.0	22.1	43.6	8.0	14.9	30.2

(ii) Deposit rained on two days after spraying

Age of deposit (days)	DDT		Dieldrin 25 mg		Dieldrin 50 mg	
	Dosage mg/ft <sup>2</sup>	Residual dosage %	Dosage mg/ft <sup>2</sup>	Residual dosage %	Dosage mg/ft <sup>2</sup>	Residual dosage %
0	360	100	36.1	100	53.7	100
4	50	13.9	8.9	24.7	20.7	38.6
11	39	10.8	7.1	19.7	9.9	18.4
25	24	6.7	3.3	9.1	9.4	17.5

The inadequacy of sampling surface deposits of insecticides on absorbent surfaces for evidence of residual life by scraping has been pointed out by Barlow (4).

The insecticide recovered in this way may not be biologically available due to it either being in crevices in the wall or else in the sorbed condition inside the wall surface. Barlow (loc. cit.) has suggested the sampling technique whereby strips of adhesive tape (cellotape) are pressed firmly to the surface to be sampled.

This method has been tried and has been found to give satisfactory results. A comparison of the amount of DDT recovered by the two sampling methods in the same huts is shown in Table 11. The recovery by the tape is 30% of that obtained by scraping. This gives a more reliable indication of the deposit actually available for the direct contact with the mosquito.

After 16 weeks  $55 \text{ mg/ft}^2$  of the DDT was found on the surface by the tape method as compared with  $174 \text{ mg/ft}^2$  by scraping.

Table 11  
Comparison of sampling surface deposits of DDT by  
(i) scraping and (ii) adhesive tape (cellotape)

Method of sampling	DDT in $\text{mg/ft}^2$ (technical DDT)						
	1	2	3	4	5	6	Average
(i) Scraping	84	232	204	166	182	-	174
(ii) Tape	20	119	30	69	50	37	55
(ii) as % of (i)	24	51	15	24	42	28	31

#### 4. SUMMARY

1. A series of investigations are described of the persistence on mud walls of the three insecticides used in the Western Sokoto Malaria Control Pilot Project.
2. Samples removed from the walls of sprayed houses, mainly by mechanical scraping, were subjected, after extraction, to chemical estimation, using the following methods:
  - DDT : colorimetric determination by Schechter's method, as modified by Barlow.
  - BHC : total chlorine sodio-reduction technique in n-hexane solution.
  - Dieldrin : semi-micro Stepanow method followed by turbidimetric estimation of chloride.

3. In all three insecticides the initial loss in the first two weeks after spraying was found to amount to between 40 and 60%.
4. DDT showed a more gradual rate of decline after the first two weeks. Wide variations in residual deposits of DDT were found to be due partly to uneven original dosage. In one case a particular type of mud, probably of low iron content, led to higher residues than elsewhere. The distribution in depth of DDT on the walls suggested that after three cycles of spraying the surface layers approach saturation with the toxicant.
5. The loss of BHC from the surface continues at a high rate even after the first two weeks, so that by the end of the fifth week 90% of the original deposit is lost. Saturation by superimposition of successive deposits, as in DDT does not take place with BHC.
6. Dieldrin residues decline more slowly, following the pattern of DDT, after the initial period of high loss. Where weathering takes place the deposits of dieldrin may be more persistent than those of DDT, due to a greater proportion of dieldrin being in the sorbed condition, and so protected from erosion.
7. The method of sampling by mechanical scraping was compared with Barlow's method, using cellophane adhesive tape. The approximate amounts of the toxicant removed by the latter method average about  $\frac{1}{3}$  of those obtained by scraping.

#### 5. REFERENCES

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