

Pathways of Loss of Dieldrin from Soils Under Field Conditions

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Dieldrin was applied to a depth of 7.5 cm to two small watersheds, one watershed in each of 2 years, and the soils, crops, runoff water, sediments, and overlying air were periodically assayed for dieldrin content over a 2- to 4-year period. Soil analyses were highly variable and no statistically significant decrease in pesticide content with time could be shown. Dieldrin in the runoff water was, at most, only 0.07% of the original dosage in the first season, with the largest losses occurring in the first 2 months after application. Highest dieldrin concentration in the water was 20 ppb soon after application, and was always less than 2 ppb in the second year.

Where erosion occurred, losses of dieldrin in the sediments were appreciable, reaching 2.2% of the application. Maize plants accumulated 0.03% of the application, largely in the leaves. Most of the dieldrin loss was by volatilization; within one season horizontal filter traps collected 2.9% of the pesticide applied to the underlying soil. In summary, the main conclusions are that the major pathways of dieldrin loss were by volatilization and sediment transport, but that the amounts of dieldrin in the runoff water and the crop were significant. All losses can be appreciably reduced by improved management practices.

Ample evidence exists that use of the organochlorine insecticides for the past 20 odd years has produced ubiquitous, low-level residues in the biosphere that are of grave concern to ecologists and conservationists, especially with regard to long-term effects on nontarget organisms (Stickel, 1968). The pesticides are released to the environment primarily from pesticide manufacturing operations and from use of the products in control of human disease vectors, soil and crop pests, and household insects. The relative contribution of each of these sources to the total pesticide burden in the environment, or even to individual components of the environment, is not known, yet such knowledge is needed if the chemicals are to be used with minimum impact on nontarget species. The objective of the investigation described here was to examine the rate at which dieldrin was lost to the surrounding environment after application and mixing into a field soil subjected to normal agricultural management.

EXPERIMENTAL

General Description of the Experiments. The field work was conducted at the North Appalachian Experimental Watershed, a Soil and Water Conservation Research Division station located at Coshocton, Ohio. In May 1966 a small watershed was treated with dieldrin immediately before maize (*Zea mays*) was planted. In May 1968 a similar treatment was made on a second watershed, again before maize planting. From 1966 through 1969 posttreatment samples of soil, crops, runoff water, and sediments from both watersheds were analyzed for dieldrin residues at the U.S. Soils Laboratory at Beltsville, Md. In 1968, the dieldrin residues adsorbed on fiberglass filters suspended above the newly treated field were measured to evaluate the magnitude of the loss of dieldrin by volatilization to the air.

In addition to the dieldrin analyses, the amounts of runoff water during and after rainfall were measured automatically on each watershed as part of the regular hydrologic studies on the fields, and the amount of sediment carried off was measured in a Coshocton-type runoff sampler (Parsons, 1955). The amount of sediment retained in the flumes at the foot of the watersheds after each rainfall was determined by direct field observation.

Watershed Treatments. The more important characteristics of the watersheds, which were described in detail by McGuinness *et al.* (1960), are summarized in Table I. The soil on both watersheds was Muskingum silt loam. Normal management practice on both watersheds included a 4-year crop rotation, as shown in the table.

The soils were fertilized, plowed, and disked about 1 month before the dieldrin application. In 1968, the soil was also cultipacked to deliberately increase the likelihood of runoff. Dieldrin applications were made on May 20, 1966, and May 2, 1968, as a uniform spray of aqueous emulsion from a 20-ft truck boom at a rate of 5.6 kg/hectare (5 lb/acre) of active HEOD (1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-*endo,exo*-5,8-dimethanonaphthalene). The pesticide was disked into the soil to a depth of 7.5 cm (3 in.) immediately after application.

Sampling Program. Soil samples were taken to a depth of 17.7 cm (7 in.) within 1 day of dieldrin application, after harvest about 5 months later, and during each succeeding spring and fall through 1969. For sampling purposes, the larger watershed (1966 treatment) was divided into eight subplots, and the smaller (1968 treatment) into five subplots, as shown in Figure 1. Twelve randomly spaced holes were dug in each subplot, and a vertical slice of soil was taken from the side of each with a spade. Each slice was then cut horizontally to retain the sample to the 17.7-cm depth. The 12 slices were then composited to give the bulk sample for the subplot. In 1969, a refined procedure was substituted in an attempt to reduce the variability of the results. In this procedure, 75 cores, each 21 mm in diameter, were taken to a depth of 17.7 cm on each subplot, and again combined to give a single bulk sample.

In each runoff occurrence, samples of runoff water were taken with special automatic sampling equipment installed in the flume at the foot of the watershed (Harrold *et al.*, 1967). In this equipment, operation of which was automatically triggered by the appearance of water in the flume, a stream of runoff water was pumped from a sampling wand in the flume into a refrigerated chamber containing 12 1-gal jugs, which were filled according to a preset time sequence. In a typical sequence, gallon samples were collected at 3-min intervals during the early part of the runoff over the peak flow, and at 6- to 12-min intervals during the latter part.

Whenever suspended solids or sediments appeared in the water samples, they were separated by continuous centrifuga-

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Table I. Physical Characteristics and Crops on Experimental Watersheds, Coshocton, Ohio

	Year of Dieldrin Treatment	
	1966	1968
Watershed No.	128	106
Area, hectares	1.09	0.68
Average slope, %	13.6	14.3
Crop in		
1966	Maize	(Meadow)
67	Wheat	(Meadow)
68	Meadow	Maize
69	Meadow	Wheat

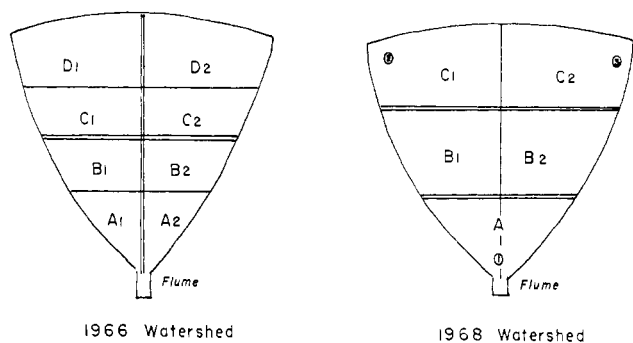


Figure 1. Location of subplots in the treated watersheds. Subplot combinations for maize sampling are indicated by double lines; location of filter traps indicated by numbers within circles

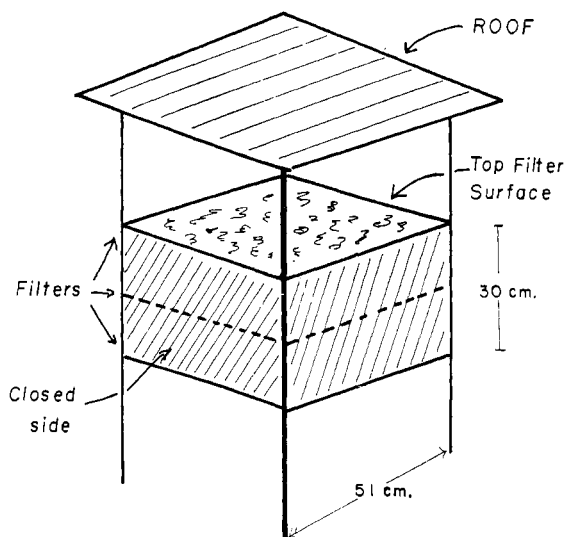


Figure 2. Filter trap for measurement of dieldrin in air

tion, frozen, and analyzed separately. Sediments remaining in the flume at the end of each rainfall were sampled by hand and also frozen for later analysis. The flume was cleared of solid residues between rainfalls.

Crops were sampled from combined subplots, as indicated in Figure 1. Ten whole maize plants were taken at maturity from each of the combined subplots and separated into leaves, stalks, and ears. The 10 separates were then composited into bulk samples of plant parts from each combined subplot and frozen for storage before analysis. Kernels were separated from cobs in the analytical laboratory.

Air sampling was conducted in 1968. Three filter traps,

which provided a mineral surface on which dieldrin could condense, were placed within the boundaries of the freshly treated watershed. The traps were placed at the three points shown in Figure 1, chosen so that one was always on the leeward side of the treated area. Each trap, the design of which is illustrated in Figure 2, consisted of a set of three glass fiber furnace filters placed horizontally within a wooden box at heights of 30, 45, and 60 cm above the soil surface. The bottom and top filters were left open to the air, and the entire assembly was protected from rain by a roof. All filters were replaced biweekly throughout the growing season and analyzed individually for dieldrin content.

Analytical Methods. **SOILS.** The moisture in a 10- to 20-g sample of the soil passing a 4-mesh sieve was adjusted to 1 to 10 bars moisture tension with use of a pressure membrane, and the prepared sample was extracted in a Soxhlet apparatus for 3 hr with 3:1 hexane:isopropyl alcohol. The extract was passed through a short column of powdered alumina to reduce emulsions, and the column eluate was washed with water to remove the isopropyl alcohol. The residual hexane was diluted to 250 ml and a 2- to 8- μ l aliquot was injected into a gas chromatograph. The glass glc column, 2 mm \times 165 cm, was packed with a 1:1 mixture of 10% DC-200 and 15% QF-1 on Gas Chrom Q. Temperatures of injection port, column oven, and ^{63}Ni electron capture detector were 235°, 220°, and 275° C, respectively. The flow rate of the carrier gas, 95:5 argon:methane, was 70 ml per min. Under these conditions, the retention time of dieldrin was approximately 7 min. The method gave 92-95% recovery of dieldrin from fortified soils, for which correction was made in the reported results.

WATERS. A 1-l. sample was extracted by 1-min successive shaking in three separatory funnels, each containing 33 ml of 3:1 hexane:ethyl ether. The residual organic phases in the funnels were combined and concentrated to 10 ml. The concentrate was cleaned up, when necessary, on a charcoal-celite column, with elution with 15:85 ethyl ether:hexane, and the eluate was further concentrated to about 1 ml. A 2- to 8- μ l aliquot of the final concentrate was injected into a gas chromatograph, and the dieldrin content determined under the same conditions described above for soils. The method gave essentially quantitative recovery of dieldrin from fortified waters stored in glass bottles.

SEDIMENTS. The frozen sample was thawed, placed on a filter paper in a Buchner funnel, and brought to a moderate moisture level by application of vacuum. The moist sediment was removed from the filter as a cake, crushed, and subjected to extraction and analysis as described for soils.

PLANT MATERIAL. The bulk sample of each of the corn plant parts was divided mechanically into small pieces and the required analytical sample (25 g, except 15 g for leaves) was obtained by quartering. The sample was blended with 2:1 acetonitrile:water for 3 min, then filtered through glass fiber paper. The residue was extracted in a Soxhlet apparatus for 12 hr with 1:1 chloroform:methanol, and the extract was passed through a short Florisil column for preliminary clean-up. The column eluate was partitioned into hexane and combined with the original acetonitrile:water filtrate, which had also been partitioned into hexane. The combined hexane fractions were cleaned up by Florisil column chromatography with 15:85 ethyl ether:hexane as eluant. The eluate was concentrated to 10 ml and the dieldrin content was determined by gas chromatography, as described above for soils. Extracts of corn kernels, because of high fat content, required an additional clean-up prior to glc analysis. The concentrated Florisil column eluate was placed on a saponification column

consisting of two layers of magnesia and one of potassium hydroxide interlayered with anhydrous sodium sulfate. The column was eluted with 15:85 benzene:hexane, and the eluate was concentrated.

Discovery of possible errors in dieldrin analysis of corn leaves introduced by plant pigments led to a modification in the analytical procedures for the leaves obtained in 1968. *In lieu* of column chromatography, the extract was cleaned up by treatment with hydrogen peroxide before glc analysis (Glotfelty and Caro, 1970). A refinement was also made in analysis of the kernels obtained in 1968. *In lieu* of column saponification, an alkaline precolumn was inserted at the head of the column within the gas chromatograph to destroy fats and oils in the extracts (Miller and Wells, 1969).

AIR FILTERS. The entire furnace filter was placed in a 1-qt jar and the pesticide was extracted with hexane by tumbling for 3 hr. A 2- to 3- μ l aliquot of the hexane was injected directly into the gas chromatograph and the dieldrin content determined.

RESULTS

Soils. The results of the analyses of the soil samples taken up to 41 months after application are summarized in Figure 3. Comparison of data from the subplots permitted calculation of the degree of variability of the dieldrin content of the samples. The standard deviations, indicated by the lines associated with the points in Figure 3, show that this is large, and it is not possible to show statistically that there was any loss of dieldrin from the soil of the 1966 treatment over 41 months. The results from individual subplots at different times showed the same amount of random variation. The data in Figure 3 also show that the variability did not decrease with time, nor with modification in sampling techniques.

Replicate analyses of portions of each sample showed that analytical variability was very low compared to that between samples, suggesting that the pesticide was irregularly distributed in the soil and that the sampling techniques failed to give representative samples. The disk harrow has, in fact, been shown to give nonuniform pesticide distribution in the soil in both vertical and horizontal directions (Read *et al.*,

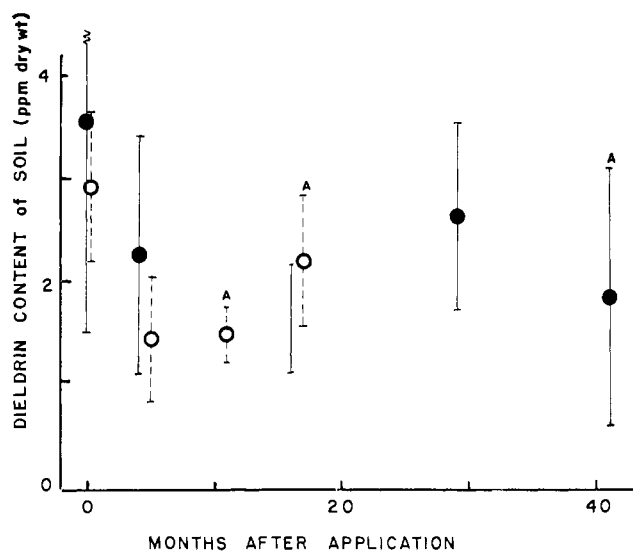


Figure 3. Measured dieldrin contents of soils of treated watersheds. Points and lines indicate mean and standard deviation of each set of subplot analyses. Solid lines for watershed treated in 1966; dashed lines for watershed treated in 1968. Samplings marked A were conducted using revised core sampling technique

1968). Accurate measurement of the pesticide content of field soils, which is the only satisfactory basis for estimating the persistence of these compounds, presents a considerable problem. Often the quantity of a pesticide in a soil "increases" between samplings taken 3 to 5 years apart (Nash and Woolson, 1967). The results obtained in the present study further illustrate the magnitude of this difficulty. The source of the variability is being investigated in more detail in continuing work in this laboratory.

Runoff. The measurements of the amounts of dieldrin carried in the runoff water are summarized in Tables II and III. The total water and dieldrin fluxes for each runoff event were calculated from the water flow and pesticide concentration data using a computer program. The ranges of dieldrin concentration are those found in the 1-gal samples collected auto-

Table II. Runoff-Producing Rainfalls and Dieldrin Losses in the Runoff, 26-Month Period, Watershed Treated in 1966

Days After Pesticide Application	Amount of Runoff, l.	Duration of Runoff, Min	Maximum Runoff Rate, l. per Min	Dieldrin Concentration Range, μ g/l.	Total Dieldrin in Runoff mg
1966					
55	602	50	49	2.4-3.9	1.9
204	3948	510	12	2.7-3.0	11.1
1967					
295	62590	6905	158	0.8-2.7	81.0
300	38340	1800	103	1.2-4.1	98.9
303	8985	428	158	2.2-3.0	24.1
304	3751	178	103	1.0-1.4	5.5
318	20350	528	143	1.9-2.3	42.9
338	3078	150	143	1.3-1.5	5.3
358	6608	160	444	1.3-1.7	9.5
1968					
631	129200	7430	190	0.7-1.0	107.4
694	3221	516	69	1.2	3.9
750	8825	606	103	1.1	9.9
808	26830	82	2167	0.4-1.6	27.2
Totals	316328				428.6

Table III. Runoff-Producing Rainfalls and Dieldrin Losses in the Runoff, 8-Month Period, Watershed Treated in 1968

Days After Pesticide Application	Amount of Runoff, l.	Duration of Runoff, Min	Maximum Runoff Rate, l. per Min	Dieldrin Concentration Range, $\mu\text{g/l.}$	Total Dieldrin in Runoff, mg
13	51560	275	11730	17.0-20.0	1204.0
17	438	21	78	6.6	2.9
18	1754	30	373	4.3-6.7	9.3
19	516	25	89	9.7	5.0
21	21550	1399	198	5.3-8.0	143.0
24	37700	2048	329	3.1-5.0	181.9
29	3090	130	289	7.2	22.3
39	996	60	78	10.9-14.8	13.4
39	9740	121	912	8.0-11.2	86.1
82	54350	75	4620	3.9-6.5	581.4
85	2140	127	138	3.5-4.6	8.6
89	1143	32	125	2.3-2.8	2.9
120	1474	98	138	5.1-5.3	7.7
237	22260	1980	59	1.9-3.2	91.1
Totals	208711				2359.6

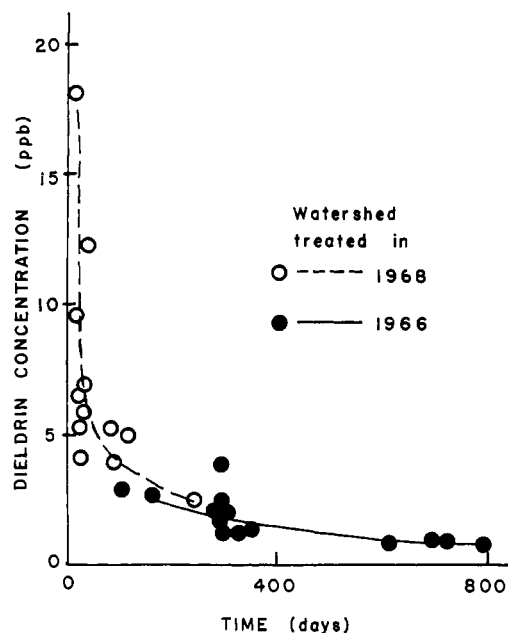


Figure 4. Changes in dieldrin concentrations in runoff water with time after application

matically. Where a single figure is given, only one sample was analyzed.

Only two small runoff events occurred in the summer of 1966; therefore, no data were obtained on the amounts of dieldrin that could be lost in runoff soon after application. Conditions in 1968 were much more favorable in this respect. The contrast between the two sets of data is exemplified in the total quantity of dieldrin removed from the two watersheds, the loss from the 1968 experiment in the first year being 5.5 times that from the 3 years of the 1966 experiment. One-half the 1968 loss took place in a single event 13 days after pesticide application. These differences reflect natural conditions and illustrate the importance of the time at which runoff takes place. It should be noted that the amount of dieldrin lost in the runoff from the 1968 watershed, which had been pretreated to make soil conditions favorable for runoff, was nevertheless only a small fraction, 0.07%, of the total applied.

The highest concentration of dieldrin in the runoff water, 20 ppb, occurred during the event of May 15, 1968. The concentrations in subsequent storms varied widely in a manner that cannot be explained in terms of the other data available. No relationships can be found between concentration and volume of water, maximum flow rate, or duration of runoff, nor is there any continuous decrease in concentration with time. The variations in concentration probably reflect a number of natural variables such as soil moisture content and surface structure, which fluctuate with the climatic conditions before the rainfall, and effective area of the watershed from which the runoff came, which fluctuates with the severity of the rainfall.

A more consistent pattern is evident when the data from both watersheds are combined to examine long-term effects. This procedure is justifiable, since the watersheds were similar in contour, shape, and cropping sequence. The average concentrations from events on both watersheds are plotted in Figure 4 as a function of time. It is evident that the high concentrations are confined to the period within about 50-80 days after application, after which the concentrations are always less than 5 ppb, falling to less than 2 ppb in the second year. Although these changes may reflect changes in the degree of adsorption of the dieldrin by the soil, the condition and exposure of the soil surface are likely to be more important factors. The critical period of 50 days corresponds fairly closely to that in which the soil was bare or only partly covered by the growing crop, and was directly exposed to the stirring and mixing caused by rainfall impact. The 1967 data in Table II represent runoff from soil protected by winter wheat undersown with alfalfa and grass, and concentrations remain low. The 1968 results in the same table represent runoff from grass meadow, where the water is flowing through vegetation rather than over bare soil. In general, the amount of dieldrin lost in solution in the runoff water is a very small fraction of the quantity applied, amounting to 0.007% in 26 months after the 1966 application, and 0.07% in the first season of the 1968 experiment. The largest losses are most likely when runoff takes place within about 2 months after application, before a dense cover crop is established. Although no simple relationship between concentration and water volume is evident, the latter appears to be the dominant factor controlling the amount of pesticide lost within each runoff occurrence, so that highest runoff is usually associated with largest loss. Good conservation practices that tend to reduce runoff will therefore reduce environmental contamination from this particular source.

Sediments and Silts. It has long been known that under the proper conditions heavy rains can carry away significant amounts of organochlorine insecticides with the surface soil from treated fields (Stickel, 1968), but quantitative information has been lacking. The data obtained in the present study (Table IV) permit estimation of the quantities of pesticide transported in this manner under field conditions in which no attempt was made to minimize erosion.

Throughout the study no measurable sediment was lost from the watershed treated in 1966. In contrast, six of the 14 runoff events in 1968 resulted in loss of sediment from the watershed treated that year. The concentrations of pesticide carried in these sediments were about three orders of magnitude higher than in the associated runoff water. The dieldrin in the solids totaled 77 g for the entire year, representing 2.2% of the pesticide applied to the field. This is about 30 times that in the associated water. The dieldrin concentration in the flume deposits decreased sharply and regularly in succes-

Table IV. Dieldrin in Solids Lost in 1968 From Newly Treated Watershed

Rainfall Date	Days After Pesticide Application	Type of Solid ^a	Dieldrin Content, $\mu\text{g/g}$ Dry Weight	Total Solids Weight, kg	Total Dieldrin in Solids, g
May 15	13	SS	14.2	3202	45.47
		FD	8.6	1784	15.34
May 19	17	SS	8.2	249	1.49
		FD	...		
May 20	18	SS	7.4	249	1.49
		FD	4.8		
May 21	19	SS	6.1	485	1.94
		FD	4.2		
June 11	39	SS	6.7	485	1.94
		FD	2.6		
July 14	82	SS	5.0 ^d	2505	12.53
		FD	1.6	272	0.44
Totals				8497	77.21

^a SS = sediment and silt collected automatically in runoff water jugs; FD = sediment deposited on floor of flume after rainfall. ^b Estimated average 6.0 $\mu\text{g/g}$. ^c Estimated average 4.0 $\mu\text{g/g}$. ^d Estimated.

sive events (Figure 5). This decrease suggests that the solid washoff was not from the entire watershed, but from the lower part close to the flume, which was denuded of topsoil in the earlier events so that in the later events the sediments were diluted with original subsoil containing little or no dieldrin.

Although the amount of pesticide lost on the sediments in 1968 was among the largest of any of the pathways examined in this study, this pathway is also the most easily controlled. The use of well-known soil management practices to control erosion will also curb or eliminate the concomitant pesticide loss.

Plant Materials. Dieldrin contents in the maize plants grown on both treated watersheds are presented in Table V. Although the maize was grown in separate years, results in the two fields show excellent agreement. Pesticide concentrations were highest in the leaves, which were exposed to the air, probably because vapor-phase adsorption is the principal route of accumulation of soil insecticides by plants (Barrows *et al.*, 1969). Dieldrin in the ears was generally at or below the sensitivity of the analytical method. An estimate of the total dry matter production based upon the measured maize yields (632 and 640 kg per hectare in 1966 and 1968, respectively) showed that the entire crop accumulated approximately 1.60 g of dieldrin per hectare, equivalent to 0.03% of the original dosage.

Air. Significant amounts of dieldrin were found on the furnace filters exposed to the air above the 1968-treated watershed (Table VI). The total trapped over the season, 12.41 mg, represented 2.9% of the dosage applied to the soil directly under the filters. The filter experiment was not begun until 4 weeks after application, so that the dieldrin in the air during that period could not be included in the total. In any event, the amounts trapped should be considered as minimum values because the trapping efficiency of the filters was not known. The large amounts of dieldrin trapped by the relatively crude filters prompted a more refined experiment on another watershed in 1969 in which sophisticated air sampling equipment was used. Preliminary analysis of results of this work indi-

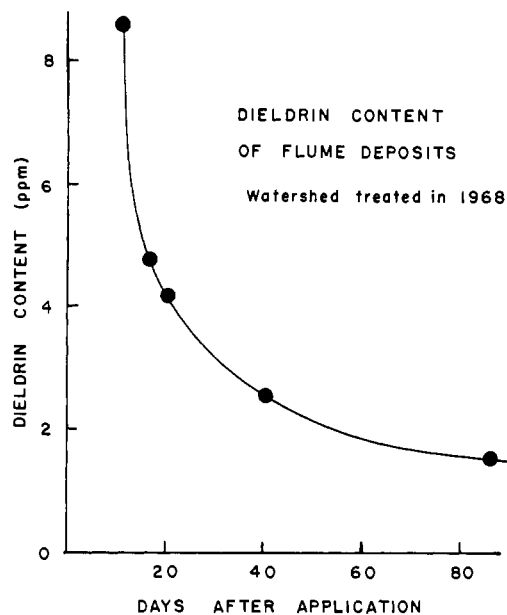


Figure 5. Changes in dieldrin content of flume deposits with time after application

Table V. Dieldrin in Maize Plants Grown on Two Treated Watersheds

Plant Part	Dieldrin in Plant Part, $\mu\text{g/g}$ Dry Weight				Average
	Maize Grown on Watershed Treated in 1966				
	Subplot ^a				
	A1-B1	A2-B2	C1-D1	C2-D2	
Stalks	0.09	0.20	0.05	0.08	0.10
Leaves	0.88	0.83	0.64	0.58	0.75
Cobs	0.01	0.01	<0.01	<0.01	0.01
Kernels	0.03	0.02	0.02	0.02	0.02
	Maize Grown on Watershed Treated in 1968				
	Subplot ^a			Average	
	A	B1-B2	C1-C2		
Stalks	0.13	0.09	0.24	0.15	
Leaves	0.68	0.59	0.86	0.71	
Cobs	<0.01	<0.01	<0.01	<0.01	
Kernels	<0.01	<0.01	<0.01	<0.01	

^a See Figure 1 for subplot locations.

Table VI. Dieldrin Trapped on Fiberglass Filters on Watershed Treated in 1968

Exposure Time, Weeks After Pesticide Application	Dieldrin Trapped, mg			Total for 2-Week Period
	At Location ^a			
	1	2	3	
4-6	0.83	0.70	1.01	2.54
6-8	0.45	1.06	1.01	2.52
8-10	0.24	0.67	0.61	1.52
10-12	0.22	0.71	0.59	1.52
12-14	0.16	0.75	0.46	1.37
14-16	0.11	0.47	0.43	1.01
16-18	0.09	0.43	0.27	0.79
18-20	0.10	0.35	0.17	0.62
20-22	0.04	0.34	0.14	0.52
Total	2.24	5.48	4.69	12.41

^a See Figure 1 for location sites.

cates that the glass fiber filters were relatively efficient traps for dieldrin, but the absolute relationship between the amounts of dieldrin retained on the traps and the pesticide vapor density in the air to which the traps were exposed cannot be estimated.

The quantities of dieldrin trapped decreased regularly throughout the year, suggesting that the concentration of pesticide in the surface layer of soil is the controlling factor. Exposure of new surfaces of treated soil by cultivation has, in fact, been shown to reduce the residence time of the pesticide in the soil (Lichtenstein and Schulz, 1961).

Less dieldrin was retained on the trap at location 1 than on those at the other two locations because the surface soil under the trap was removed by erosion during the rainstorms in late May. This observation further supports the supposition that the pesticide-bearing sediments removed from the treated field in the runoff water originated mainly from the soil directly above the flume.

DISCUSSION

The measurements show that with the soil management practices employed dieldrin was lost from the soil mainly by volatilization to the air and by sediment transport. Lesser amounts were lost in runoff water and accumulated by the maize crop grown on the treated soil. In environmental terms, volatilization is the most serious problem, since it will always take place where the dieldrin is applied to or mixed in surface soil. The loss by erosion transport is much more sporadic and can be controlled by conservation practices. It should be remembered that although the loss by erosion was significant in 1968, no dieldrin was lost from the 1966 watershed by this mechanism during the three succeeding years. The deposition of dieldrin-bearing sediment in streambeds will provide a continuing source of the pesticide to the water flowing over it. Where the eroded material is mixed with dieldrin-free sediments, the concentrations will be greatly reduced because the pesticide will become redistributed over the whole material and be held by a greater number of strong adsorption sites. Analyses of the stream waters draining the land area in which the experiments were conducted did not

reveal any measurable concentrations of dieldrin at any time during the study.

In terms of direct entry into biological food chains and toxicity to nontarget organisms, the amounts present in the runoff water and crop are serious (Tarzwell and Henderson, 1957; Harris and Sans, 1969). The extent of this hazard can, however, probably be greatly reduced by better management practices. For example, the amounts carried in runoff and eroding topsoil and the amounts released to the air are likely to be much reduced by banding the pesticide in the soil or covering it with a layer of pesticide-free soil.

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