

1 THE EFFECT OF IRRADIATION WITH ULTRAVIOLET LIGHT ON
2 CHLORINATED HYDROCARBON INSECTICIDES: A REVIEW

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6
7 INTRODUCTION

8 Pesticide chemists have recognized since the 1940's that
9 ultraviolet light was deleterious to the insecticidal properties of
10 certain compounds. However, it has been only within the last 15 years
11 that a number of photodecomposition products of the chlorinated hydro-
12 carbon insecticides have been isolated and identified. The degradation
13 of insecticides by ultraviolet light may result in products which are
14 more or less toxic than the parent compounds. This implies that
15 photodecomposition of insecticides may in certain cases increase
16 residual toxicities. Thus, this subject should be of vital concern to
17 all persons involved in the use of insecticidal chemicals. This paper
18 summarizes a number of published reports concerning the photodecomposi-
19 tion of chlorinated hydrocarbon insecticides.

20 PHOTODEGRADATION OF DDT AND ITS ANALOGS

21
22 Much of the work on the ultraviolet degradation of DDT (2,2-Bis-
23 (p-chlorophenyl)-1,1,1-trichloroethane) (Compound I, Figure I) was
24 done between 1940 and 1950. Fleck and Haller (1945), active workers
25 during this time, first reported that DDT was apparently relatively

1 stable to ultraviolet rays. They found that irradiation of a thin
2 film of DDT for 35 hours caused the melting point of the resulting
3 material to be lowered by only 2°C. This would imply that the material
4 remaining in the film was relatively pure DDT since small amounts of
5 an impurity generally depress melting points considerably. They also
6 reported that no change was observed in DDT after exposure to sunlight
7 for over a year in an alcoholic solution. Lindquist et al (1946)
8 showed, however, that the effectiveness of DDT against houseflies was
9 reduced by exposure to ultraviolet light in the laboratory or sunlight.
10 They also concluded that DDT is more rapidly decomposed in certain
11 organic solutions than as a solid deposit. Wichmann et al (1946)
12 reported that neither DDT nor DDE (4,4'-dichlorodiphenyldichloro-
13 ethylene) (Compound II, Figure I) were decomposed when irradiated in
14 their crystalline state. In a benzene solution, however, 50% of the
15 DDT was degraded in a few hours, and DDE was degraded even more
16 rapidly. Wichmann's group also isolated p,p'-dichlorobenzophenone
17 (Compound III, Figure I) as its 2,4-dinitrophenylhydrazone derivative
18 from the irradiation products of DDT. The production of this phenone
19 was later confirmed by Fleck (1948). Wichmann et al reported that the
20 isolated ketone was apparently stable to ultraviolet light. They
21 concluded from volatility and exposure tests, that the ultraviolet
22 decomposition products of DDT are more volatile than the parent com-
23 pound and are removed nearly as fast as they are formed under normal
24 field conditions. This theory was further substantiated in a two-year
25 study by Fahey and Rusk (1947). The hypothesis of Wichmann et al may

1 explain why several investigators (Lindquist et al, 1946; Vendramini,
2 1947; Sazonov and Andreev, 1949; Gunther, 1945; Srivastava et al, 1955
3 Blackith, 1952; and Hadaway and Barlow, 1949) found decreased
4 effectiveness of DDT following exposure to artificial ultraviolet
5 light or sunlight. If the degradation products are volatilized very
6 quickly, then the data of these workers fail to reveal the possible
7 toxicity of the degradation products themselves.

8 Other possible degradation compounds of irradiated DDT are:
9 2,3-dichloro-1,1,1,1-tetrakis-(p-chlorophenyl)-2-butene (Fleck, 1949)
10 (Compound IV, Figure I), and DDE (Roburn, 1963). From existing data
11 it is possible to derive a scheme for the ultraviolet degradation of
12 DDT as shown in Figure I. Reaction (A) is the result of Fleck's
13 investigations (1949) and according to that author requires the
14 presence of a solvent which acts as a chlorine acceptor. Reaction (B)
15 has been observed by Wichmann et al (1946) and Fleck (1946), while
16 reactions (C), (D), and (E) have been reported by Roburn (1963).
17 Harrison et al (1967) reported the formation of DDD via Route (F).
18 Gas chromatographic patterns of the degradation products of DDT
19 irradiated in hexane indicate that Route (G) may also exist (Banks
20 and Bills, 1968).

21 Although the proposed scheme may not be considered complete or
22 conclusive, it does show the complexity of the ultraviolet degradation
23 of DDT.

24 Other workers who have observed ultraviolet light induced
25 degradation of DDT, but have not identified breakdown products or

1 studied toxicity effects, are Li and Bradley (1967) and Mitchell
2 (1961).

3 Fleck (1949) reported that DDD (2,2,-Bis-(p-chlorophenyl)-1,1-
4 dichloroethane) (Compound V, Figure I) was not altered by ultraviolet
5 light. However, since that time Mitchell (1961), Roburn (1963) and
6 Banks and Bills (1968) have shown that DDD is degraded at a rate much
7 slower than DDT or DDE. The fast rate of degradation for DDE first
8 observed by Wichmann et al (1946) has also been observed by Mitchell
9 (1961) and Banks and Bills (1968).

10 It should be pointed out that most investigators who have
11 studied the effects of ultraviolet light on DDT and its analogs, have
12 not carefully described the amounts of energy nor the wavelengths
13 produced by the light sources used in their experiments. Variations
14 in the ultraviolet light sources could easily account for deviations
15 in results that have been observed by different laboratories. Also,
16 as has been reported by Sazonov and Andreev (1949), the amount of
17 degradation of DDT by sunlight is decreased by haze, dust and water
18 vapor present in the air. Obviously, the intensity of sunlight itself
19 will vary with the geographic location and the date.

20 PHOTODEGRADATION OF DIELDRIN, ALDRIN, ENDRIN AND ISODRIN

21
22 In his extensive investigation of the effect of ultraviolet
23 light on pesticide chemicals, Mitchell (1961) reported on the photo-
24 decomposition of the aldrin (1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-
25 hexahydro-1,4-endo-exo-5,8-dimethanonaphthalene) (Compound I, Figure

1 II) family of insecticides. Upon irradiation he found that aldrin and
 2 dieldrin (1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-
 3 octahydro-1,4-endo-exo-5,8-dimethanonaphthalene) were extensively
 4 decomposed, and endrin (1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,
 5 7,8,8a-octahydro-1,4-endo-exo-5,8-dimethanonaphthalene) (Compound I,
 6 Figure IV) was substantially degraded. In the same year Bird et al
 7 (1961) presented evidence to show that isodrin (1,2,3,4,10,10-
 8 hexachloro-1,4,4a,5,8,8a-hexahydro-1,4,5,8-endo-endo-dimethanonaphthalene)
 9 was converted to a cage compound (Figure III) when irradiated with a
 10 mercury arc.

11 Dieldrin: Roburn (1963) reported the presence of an unknown compound
 12 on grass which had been treated with dieldrin and exposed to sunlight.
 13 He showed that the same compound could be produced by the ultraviolet
 14 irradiation of a thin film of dieldrin on a glass plate. By irradi-
 15 ating solutions of dieldrin or thin layers of dieldrin on filter paper
 16 at a wavelength of 2537 Å, Robinson et al (1966) obtained a degradation
 17 product which exhibited chromatographic properties similar to those of
 18 the derivative observed by Roburn. Robinson and co-workers tentatively
 19 concluded that the structure of the conversion product was a hexacyclo
 20 isomer (10-oxa-3,6-exo-4,5,13,13-hexachlorohexacyclo(6.3.1.1^{3,6}.1^{9,11}
 21 .0^{2,7}.0^{5,12})tridecane) as shown in Figure II, Compound III. In a
 22 report which appeared at the same time as that of Robinson et al, the
 23 identity of this isomer was confirmed by Rosen et al (1966). Rosen
 24 and co-workers also confirmed Roburn's (1963) observation that the
 25 same photodecomposition product was formed upon exposure of dieldrin

1 to sunlight or artificial irradiation at a wavelength of 2537 Å.
2 Harrison et al (1967) reported on field irradiation and laboratory
3 irradiation of dieldrin. When dieldrin was applied in the field in
4 solutions of 0.2%, four conversion products were detected by gas
5 liquid chromatography (GLC); three of the products were found to have
6 GLC retention times identical to those of compounds produced by the
7 ultraviolet irradiation of pure dieldrin at 2540 Å wavelength in the
8 laboratory. Following irradiation in the field or in the laboratory,
9 the product present in the largest amount behaved similarly on silicone
10 GLC columns to the hexacyclo isomer reported by Roburn and identified
11 by Robinson and Rosen. Harrison proposed that this primary product
12 was Compound I of Figure V which could exist in several isomeric
13 forms. This derivative is the same as the one reported by Rosen and
14 Robinson except for a shift of the epoxide linkage.

15 When a dilute solution of dieldrin in hexane was irradiated at
16 a wavelength of 2537 Å, Henderson and Crosby (1967) reported that
17 dieldrin was converted to a single degradation product which differed
18 from the hexacyclo compound previously reported. They identified this
19 product as a pentachloro derivative of dieldrin (Compound II, Figure
20 V). When dieldrin was exposed to sunlight, they found it was con-
21 verted only to the hexacyclo derivative. Conversely, when dieldrin
22 treated leaves were exposed to ultraviolet light of 2537 Å wavelength
23 in the laboratory, there was considerable conversion to the pentachloro
24 derivative. To simulate field conditions of irradiation in the
25 presence of a hydrocarbon solvent, a hexane solution of dieldrin was

1 irradiated with light of varying wavelengths between 2500 and 3000 Å.
2 Under these conditions, the hexacyclo compound was not formed, and the
3 pentachloro derivative was produced only at wavelengths less than
4 2600 Å. These results seemingly contradict those of Robinson et al
5 (1966), Rosen et al (1966) and Harrison et al (1967). Henderson and
6 Crosby explained this diversity by indicating that the hexacyclo
7 compound had always been obtained under conditions of high concentra-
8 tion which they assumed would favor the intramolecular reaction
9 between carbon 2 and the closely adjacent hydrogens of the methylene
10 bridge. They also concluded that in dilute solutions of a solvent,
11 such as hexane, which can act as a hydrogen donor, the replacement by
12 hydrogen of one chlorine atom bonded to one of the carbon atoms
13 involved in the carbon-to-carbon double bond appears to predominate.
14 Banks and Bills (1968) added support to the latter conclusion when
15 they irradiated hexane solutions of dieldrin ranging in concentration
16 from 0.2 to 10.0 ppm and found a single degradation product which had
17 GLC characteristics very similar to those of the pentachloro derivative
18 described by Henderson and Crosby (1967). It is doubtful that all of
19 the parameters influencing the production of either derivative are well
20 enough known at this time to permit a complete explanation of the
21 various observations.

22 There is evidence to show that the photodecomposition products
23 of dieldrin are more toxic than the parent compound. Rosen et al (1966)
24 tested the hexacyclo derivative against adult houseflies, Musca
25 domestica, and larval mosquitoes, Aedes aegypti. They found that the

1 derivative was approximately two times more toxic to these species
2 than dieldrin. In addition, the derivative produced a much faster
3 toxic response in houseflies. In a later study, Rosen and Sutherland
4 (1967) reported that the toxicity of the degradation compound against
5 Musca domestica was not as great as first reported, but that it was
6 faster acting than dieldrin. They also showed evidence that the
7 compound was two to four times more toxic to male mice than dieldrin.
8 Harrison et al (1967) reported that the isomer they had isolated
9 (Compound I, Figure V) exhibited mammalian toxicity similar to that of
10 dieldrin. Henderson and Crosby (1967) found the pentachloro derivative
11 to be only 60% as effective as dieldrin against Musca domestica, but
12 about five times more toxic to mice.

13 Robinson et al (1966) has conducted a survey of the occurrence
14 of the hexacyclo derivative of dieldrin under natural conditions. He
15 concluded that the compound occurs in very small amounts, if at all, in
16 the environment. None of the isomer was found in the pooled body fat
17 of ten people.

18 Aldrin: Mitchell (1961) found that aldrin on paper chromatograms was
19 converted to three compounds when exposed to ultraviolet light. From
20 GLC data, Roburn (1963) later reported that thin films of aldrin on
21 glass plates were converted chiefly to dieldrin plus a smaller amount
22 of an unknown substance. In field trials, Harrison et al (1967) also
23 found a conversion of aldrin to dieldrin and one unknown substance.
24 The unknown compound had the same GLC retention times as the
25

1 photodecomposition product of dieldrin which he isolated. Rosen and
2 Sutherland (1967) confirmed these findings and isolated a rearranged
3 aldrin isomer (Compound IV, Figure II). They exposed aldrin, in
4 carbon tetrachloride, to sunlight and identified the four compounds
5 shown in Figure II. Furthermore, they noted a large amount of an
6 unidentified polymeric material. In addition to the scheme shown in
7 Figure II, the authors theorized that the aldrin isomer (Compound IV,
8 Figure II) might be converted to the dieldrin hexacyclo isomer (Com-
9 pound III, Figure II). However, they did not observe such a reaction
10 when the aldrin isomer was exposed to ultraviolet light in the
11 laboratory. Henderson and Crosby (1967) obtained a pentachloro
12 derivative (Compound III, Figure V) when a hexane solution of aldrin
13 was irradiated at a wavelength of 2537 Å. The production of this
14 compound is completely analogous to the production of the pentachloro
15 derivative of dieldrin. It occurs only when the wavelength of the
16 light is below 2600 Å and the parent compound is present in a dilute
17 solution of a hydrogen donor solvent. Banks and Bills (1968) obtained
18 similar results based on GLC data.

19 Rosen and Sutherland (1967) found the rearranged aldrin
20 degradation product to be approximately two times as toxic as aldrin
21 against Musca domestica in 24 hours exposures. In 48 hour exposure
22 tests, the two compounds were nearly equal in toxicity. This suggests
23 that the aldrin isomer is faster acting in houseflies rather than
24 being more toxic. These authors also reported that the polymeric
25 material obtained by exposing aldrin in carbon tetrachloride to

1 sunlight was only about 2.5% as toxic to houseflies as aldrin.

2 Endrin: Mitchell (1961, 1962) showed that endrin was degraded upon
3 irradiation with ultraviolet light to yield at least two new compounds.
4 He also found that these degradation compounds were different than the
5 products obtained when dieldrin was irradiated. From a study of the
6 decomposition of endrin during GLC analysis, Phillips et al (1962)
7 presented a scheme for its heat induced degradation (Figure IV). When
8 endrin was irradiated with ultraviolet light, Rosen et al (1966)
9 reported its conversion to two compounds, an aldehyde and a ketone
10 (Compounds II and III, Figure IV). These degradation products are the
11 same as those formed in Routes A and C of the scheme of Phillips et al
12 (Figure IV). Harrison et al (1967) also has reported formation of the
13 aldehyde and ketone under field conditions.

14 From existing evidence, the decomposition products of endrin
15 appear to have little, if any, toxicity. Phillips et al (1962) tested
16 the aldehyde against eight insects and found it to be non-toxic.
17 Against male mice the compound had an acute LD₅₀ greater than 500 mg
18 per kilogram. Rosen et al (1966) reported that both the aldehyde and
19 ketone were non-toxic to adult houseflies and larval mosquitoes.

20 PHOTODEGRADATION OF BHC AND OTHER CHLORINATED HYDROCARBON INSECTICIDES

21
22 Although 14 stereoisomers of BHC (1,2,3,4,5,6-hexachlorocyclo-
23 hexane) are known, only lindane, the gamma isomer, (Figure VI) displays
24 an appreciable degree of insecticidal activity. There seems to be
25 good evidence that none of the isomers of BHC which have been

1 investigated are degraded by ultraviolet light. These results could
2 be expected since the molecule has no unsaturated bonds to absorb
3 light of wavelengths of 2500 Å and above. Blackith (1952) irradiated
4 the pure gamma isomer of BHC on filter paper and found no decrease in
5 its insecticidal capacity. When Klosa (1953) irradiated the alpha,
6 beta, gamma, and delta isomers of BHC with ultraviolet light at
7 elevated temperatures and in the presence of heavy metal catalysts,
8 he observed neither degradation nor conversion of one isomer to
9 another. Neither Mitchell (1961) nor Roburn (1963) observed any
10 degradation when solid deposits of all four of the above BHC isomers
11 were irradiated. Since then, Banks and Bills (1968a) have observed no
12 decomposition of the pure gamma isomer when irradiated in hexane
13 solution.

14 The photodecomposition of other chlorinated hydrocarbon
15 insecticides has not received as much attention as those previously
16 discussed. Although a few investigators have noted that other com-
17 pounds are degraded, there have been no reports of identification of
18 degradation products or of the changes in toxicity upon irradiation.

19 When pesticides were irradiated on paper chromatograms, Mitchell
20 (1961) observed extensive decomposition of methoxychlor (2,2-Bis-
21 (p-methoxyphenol)-1,1,1-trichloroethane), heptachlor (1,4,5,6,7,8,8-
22 heptachloro-2,3-epoxy-2,3,3a,4,7,7a-hexahydro-4,7-methanoindene) and
23 heptachlor epoxide (1,4,5,6,7,8,7-heptachloro-2,3-epoxy-2,3,3a,4,7,7a-
24 hexahydro-4,7-methanoindene). Banks and Bills (1968) also observed
25 degradation of heptachlor and heptachlor epoxide when these compounds

1 were irradiated in hexane solution.

2 POSSIBLE USES OF ULTRAVIOLET DEGRADATION

3
4 Although the photodecomposition of insecticides under field
5 conditions may be detrimental to insecticidal activity, some reports
6 have dealt with the possible usefulness of ultraviolet degradation.
7 Mitchell (1962) found that dieldrin and endrin could not be readily
8 separated by paper chromatography. However, by exposing the spotted
9 chromatograms to a germicidal ultraviolet lamp and chromatographing
10 the degradation products, the two compounds could be easily distinguished.
11 The method was applicable for the two pesticides alone or in mixtures.
12 Banks and Bills (1968) have presented a GLC method for confirming
13 residue identities. The technique involves comparison of standards
14 and unknowns before and after irradiation with ultraviolet light.
15 They found that some of the chlorinated hydrocarbon insecticides
16 yield characteristic degradation patterns that can be observed by
17 electron-capture GLC. These authors also determined the p-values
18 (Beroza and Bowman, 1965) of the major degradation products.

19 Li and Bradley (1967) have described a method for removing
20 insecticides from milk by decomposing them with ultraviolet light.
21 When they irradiated thin films of milk or milk fat flowing over a
22 surface cooler, they found substantial decreases in the parent com-
23 pounds. However, they did not consider the possible toxicities of
24 degradation products that were formed.

25

SUMMARY

1
2 Substantial evidence has been accumulated to demonstrate that
3 many of the insecticides classed as chlorinated hydrocarbons are
4 degraded by ultraviolet light. Degradation of certain compounds by
5 sunlight in the field and by artificial ultraviolet light in the
6 laboratory has been observed. Much of the work, however, has been
7 carried out under ill-defined experimental conditions which have led
8 to confusion concerning the products of such photochemical reactions.
9 Future workers should strive to record more carefully such parameters
10 as wavelength of light, intensity of light, irradiation time, and
11 concentration of insecticide being irradiated.

12 The photochemical degradation of insecticides under field
13 conditions is significant for several reasons. The products of
14 degradation may be more or less toxic than the original compound;
15 degradation products are frequently more volatile and are more easily
16 removed from the site of application; and degradation products of
17 toxicological significance may go undetected during an analysis for
18 the parent compound.

19 It now seems reasonably conclusive that a solvent such as
20 hexane can participate in the photochemical reaction of compounds such
21 as dieldrin and aldrin under certain laboratory conditions. In such
22 cases, the solvent apparently supplies the hydrogen atom which replaces
23 the chlorine atom at the double bond position of the insecticide.
24 Because sunlight provides insufficient irradiation of the wavelengths
25 required for this reaction, there is reason to doubt that the reaction

1 is significant under field conditions.

2 Irradiation of certain insecticides in solution in the
3 laboratory, however, may prove to be a very useful tool for identifi-
4 cation purposes. Irradiation in solution can be controlled more
5 precisely and is thus more readily reproducible than irradiation of
6 crystals or films. A number of chlorinated hydrocarbon insecticides
7 have been shown to yield characteristic degradation patterns under
8 controlled conditions. Such degradation patterns provide a means of
9 identifying certain insecticides at low concentrations for which
10 other identification methods are not applicable.

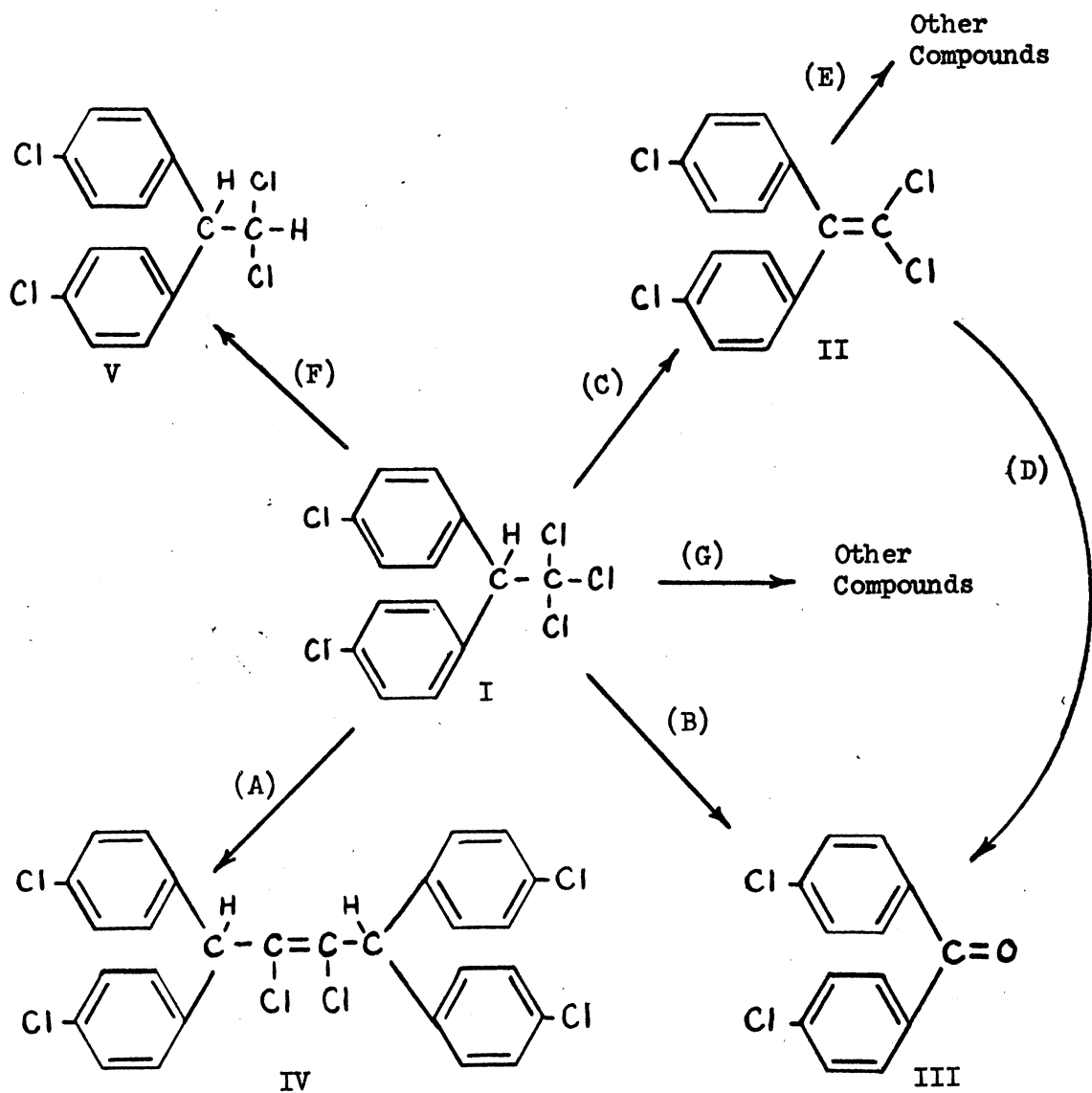
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I = DDT

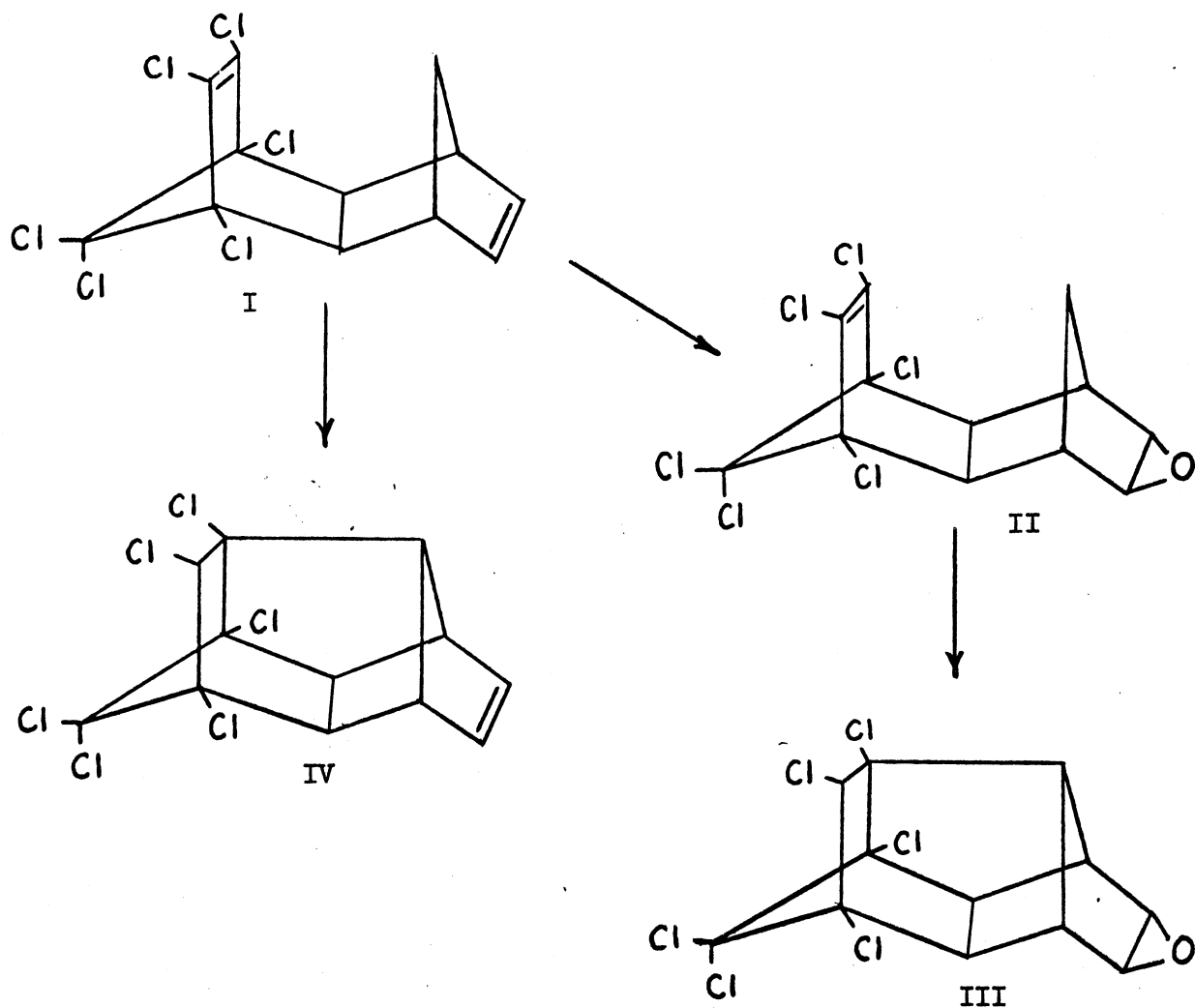
II = DDE

III = 4,4'-p-dichlorobenzophenone

IV = 2,3-dichloro-1,1,4,4-tetrakis-(p-chlorophenyl)-2-butene

V = DDD

FIGURE I. Degradation of DDT by Ultraviolet Light



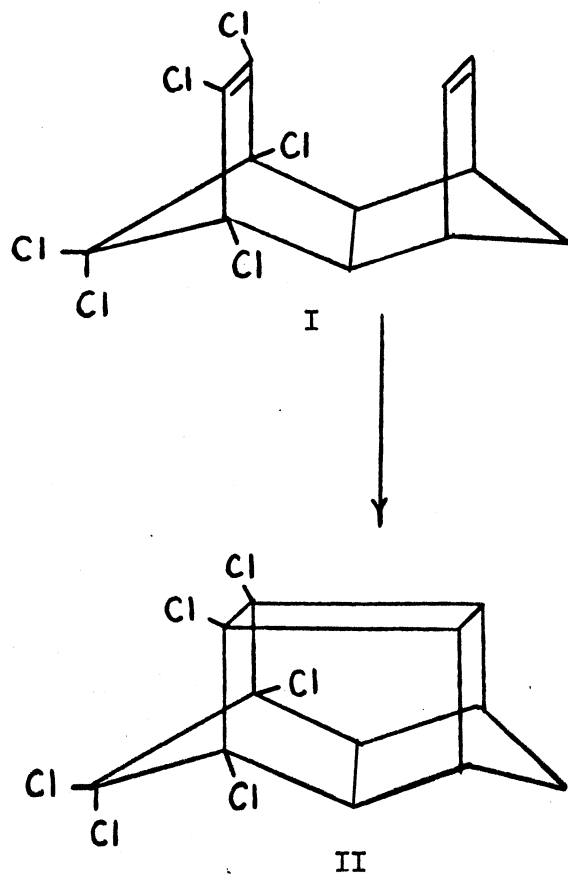
I = Aldrin

II = Dieldrin

III = Hexacyclo dieldrin isomer

IV = Hexacyclo aldrin isomer

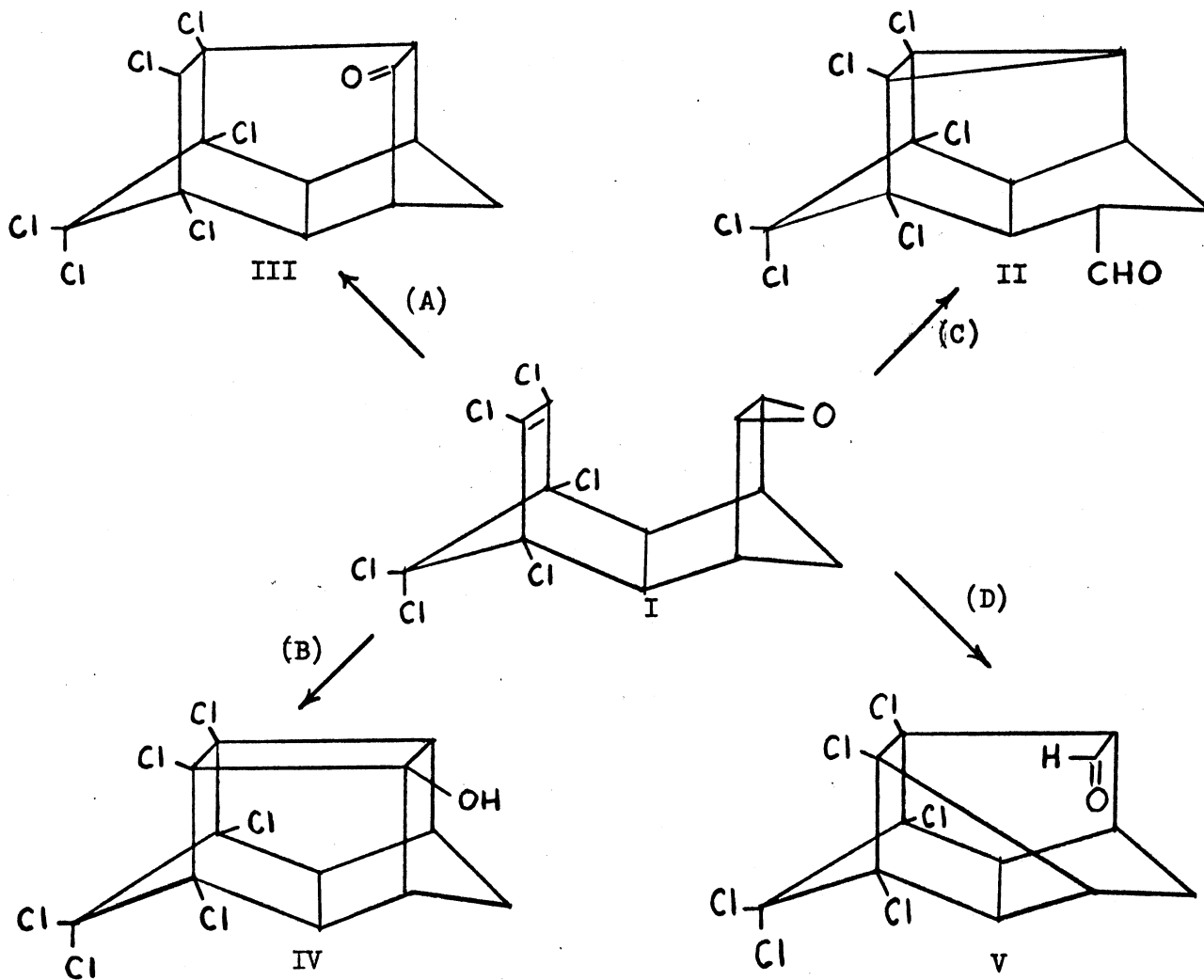
FIGURE II. Degradation of Aldrin by Ultraviolet Light



I = Isodrin

II = Cage derivative of isodrin

FIGURE III. Degradation of Isodrin by Ultraviolet Light



I = Endrin

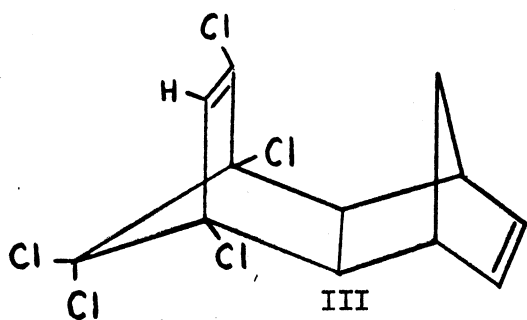
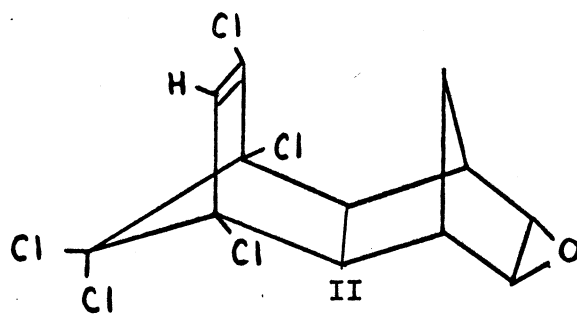
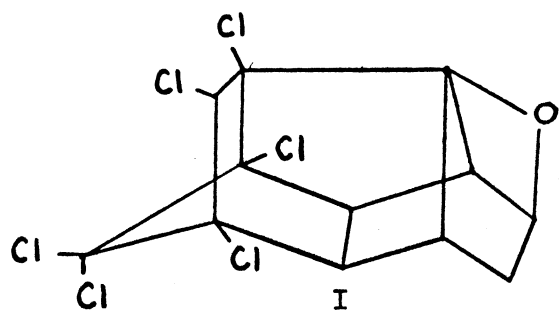
II = Aldehyde derivative of endrin obtained by heat or photochemical degradation.

III = Ketone derivative of endrin obtained by thermal or photochemical degradation.

IV = Alcohol derivative of endrin obtained by thermal degradation.

V = Aldehyde derivative of endrin obtained by thermal degradation.

FIGURE IV. Thermal and Photochemical Degradation of Endrin



I = Rearranged product of dieldrin

II = Pentachloro derivative of dieldrin

III = Pentachloro derivative of aldrin

FIGURE V. Photodecomposition Products of Dieldrin and Aldrin

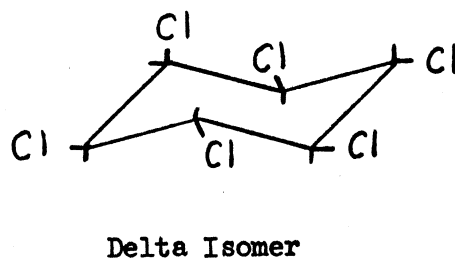
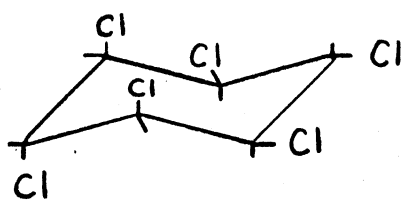
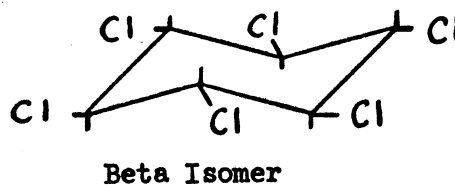
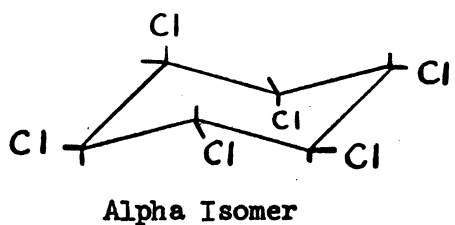


FIGURE VI. Some Isomeric Structures of BHC
(1,2,3,4,5,6-hexachlorocyclohexane) Which Have Been Found to be Stable
to Ultraviolet Light