ANTIDOTES FOR BENZENE HEXACHLORIDE RESIDUES IN SOILS

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INTRODUCTION

During recent years the development of new and better insecticides has been extremely rapid. Information as to the effect of residues left in the soil is coming more slowly. It has been observed that many of the new products are relatively stable and apparently persist in the soil with little evidence of decomposition for months and even years. The build-up of residues harmful to plants in the soil appears to be a distinct possibility. A method for deactivating such accumulations would be of great value.

One of the most important groups of new organic compounds found to have insecticidal properties are the chlorinated hydrocarbons. Benzene hexachloride, one of this group, has shown great promise in the control of many crop-damaging insects and is especially effective in the control of soil pests such as wireworms and the potato flea beetle. It has the disadvantages of being toxic at fairly low concentrations to some plants and imparts an off-flavor to others.

This study consists of an attempt to determine the effect of temperature, lime, manure, sulfur, magnesium carbonate, and activated charcoal upon the deactivation of harmful soil residues of benzene hexachloride as measured by their toxicity to plants. Limited observations on other factors such as soil texture, leaching, and soil reaction were made.

HISTORICAL

Benzene hexachloride, commonly known as "BHC" is the generally accepted name for the chemical 1.2.3.4.5.6. hexachlorocyclohexane. In England this compound was originally referred to as "666" or "Gammexane". Michael Faraday first made benzene hexachloride in 1825 and in 1833 Mitscherlich described its decomposition by bases to form trichlorobenzene.

Its insecticidal properties were discovered in France in 1941 and independently in England in 1942. Dupire and Roucourt (10) published the earliest report concerning the use of BHC as an insecticide in 1943. This report and subsequent ones did not reach this country until early 1946. Meanwhile late in 1945 Slade (21) published the results of comprehensive studies which had been undertaken in early 1942 in England. O'Kane (17) also claims the independent discovery of insecticidal properties of BHC at the New Hampshire experiment station in 1945.

Properties of Benzene Hexachloride

BHC is prepared by chlorinating benzene in the presence of actinic light. The product consists of five isomers. The gamma isomer, which is present in the crude material to the extent of 10-12%, is the principal ingredient toxic to insects. In the pure state the isomers of BHC are well defined, colorless crystals, relatively insoluble in water, soluble in organic solvents, practically odorless, and having a bitter taste. The original crude reaction product has an unpleasant odor most of which may be removed to give a purer product with 20% gamma isomer.

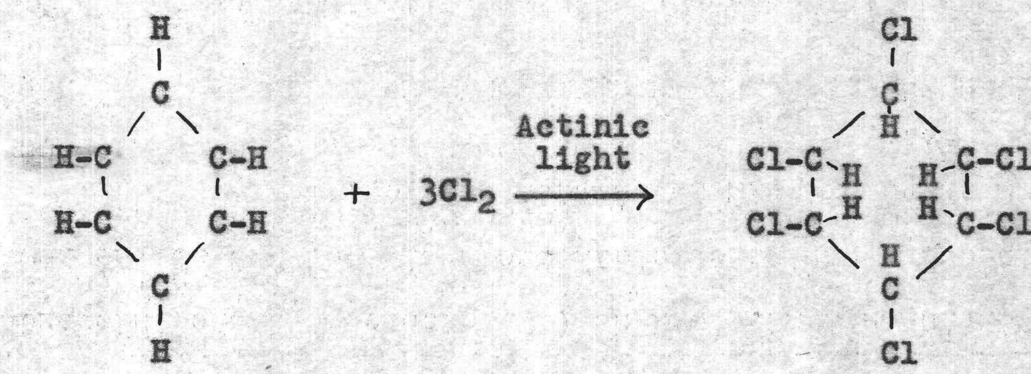


Figure 1. Preparation of BHC

BHC possesses considerable stability and can be exposed to hot water or light for a considerable period. However it decomposes readily in weak alkalies giving a mixture of the isomers of trichlorobenzene (9) and liberating hydrochloric acid. Dehydrochlorination does not take place

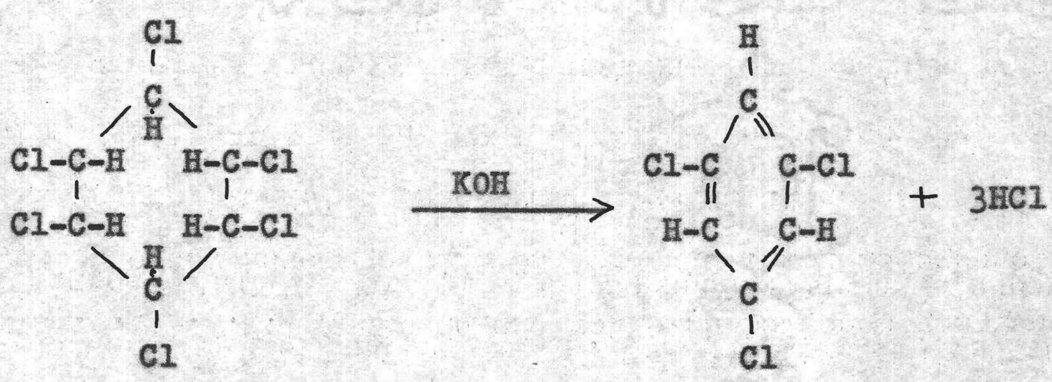


Figure 2. Dehydrochlorination of BHC

with ground limestone (21) or chalk at room temperature, or at 60° F., either dry or in the presence of water, however, it may react with dry lime.

The melting point varies greatly among the isomers, the gamma at 112.5° while the beta isomer melts at over 300°. Benzene hexachloride is insoluble in water, but soluble in certain organic solvents. Solvents used in practice include xylene, carbon tetrachloride, perchloroethylene and decahydronaphthalene. Solutions containing 5% or more of the gamma isomer may be diluted for use with kerosene.

Commercial Forms Available

Commercial material has been available principally in two forms, dusts and wettable powders. Dusts usually have a low per cent gamma isomer content and are used directly on crops or worked into the soil. The wettable powders containing a higher percentage of gamma isomer are mixed with water and used as a vegetative spray. The wettable preparations usually possess a persistant and obnoxious odor.

Agricultural Uses

BHC is more effective against some insects than any other insecticide discovered. In trials against soil insects, such as wireworms and flea beetle, the product has proved outstanding. As little as two pounds of BHC dust per acre gave excellent control of wireworm (7).

Phytotoxicity and Off-Flavors

Numerous reports that BHC is toxic to plants at certain concentrations have appeared in the literature. Morrison et al (15) lists 32 different crops in order of their susceptibility to injury from 27.5 pounds of gamma BHC per acre in the Corvallis area. Stitt and Evanson (22) report that 2 pounds gamma BHC significantly reduced the stand of beets and Swiss chard. Bush beans and turnips also showed a highly significant lower stand count when treated with 2 pounds gamma BHC. "As low as one-half pound treatment of the gamma isomer caused a highly significant reduction of the turnip stands". Several greenhouse and field studies (1, 2, 4, 5, 11, 15, 19) indicate that BHC is toxic to many plants and even within species as to the susceptibility to injury from BHC.

Off-flavors, described as bitter or musty, have been reported imparted to potatoes treated with benzene hexachloride. Greenwood and Tice (8) concluded that "the intensity of the flavor increased with the dosage of insecticide and was detected even at the minimum level tested of 1 pound per acre". Other studies (11, 16, 18) bear out these conclusions. Taste tests for off-flavor depend to a great extent upon the differences between individuals. Rarely are the results of these tests unanimanous. In fact some people (18) cannot note any impairment in flavor from applications up to 10 pounds per acre.

Attempts to reduce the off-flavor has resulted in a more highly purified product called the "pure gamma" benzene hexachloride. Although flavoring less than the technical grades it still imparts an off-flavor in some cases.

A preliminary report (12) indicates that "activated charcoal at the rate of 1 ton per acre removed the off-flavor of potatoes almost completely". Hydrated lime at 5 ton per acre was almost as effective. Liquid lime-sulfur was partially effective while the results from ferric chloride and alcoholic potash were negative. The amounts necessary to off-set the flavor in the potatoes probably make this method impractical for field use.

Persistence in Soils

A few studies have been made regarding the accumulation and persistence of BHC in soils. Smith (22) incorporated BHC into acid and alkaline soils which were exposed to the atmosphere or subjected to controlled leaching. Periodically the soils were chemically analyzed for the insecticide. Results showed that BHC was very stable in the soils, from 80 - 94% being recoverable after 18 months.

Cullinan (5) states that under greenhouse conditions BHC incorporated into the soil proved to be toxic to plants even at fairly low levels for at least four years.

There appears (24) to be no important effect on soil microorganisms even at very high concentration.

Certain soils, for example peat and muck soils, appear to have a buffer effect as far as the off-flavor is concerned. Hopkins et al (11) reports that no off-flavor was detected in potatoes grown in a New Jersey muck having a 500 pound treatment per acre of technical BHC.

In 1949 Powers began greenhouse experiments to determine whether various chemicals would counteract the off-flavor potatoes grown in several different soils. This work was handicapped by lack of analytical methods for measuring BHC in soils and plants.

In July 1950 a continuation of Dr. Powers studies utilizing biological rather than chemical methods for measuring the toxicity of BHC, was undertaken.

EXPERIMENTAL

Description of Soils

Three widely differing soil types were used in this experiment in an effort to observe the influence of soil type upon increasing or decreasing the toxicity of residual BHC. The soils used were (1) Willamette silty clay loam (2) Newberg fine sandy loam, (3) and Labish peat. A brief description of each is as follows:

Willamette silty clay loam - This soil consists of 10-14 inches of light brown friable silty clay loam over a brown moderately compact subsoil. It is found on gently sloping or rolling topography and is well drained. It is derived from sedimentary material and is classed as an "old valley filling" soil.

Newberg fine sandy loam - This is a light textured chocolate-brown soil with a coarser subsoil. Drainage is good to excessive. It is subject to overflow and is classified as a "recent" soil.

Labish Peat - Labish peat is a willow-sedge peat, high in colloidal material. The surface 12-16 inches is black and this rests on a dark-brown fibrous subsoil.

A sample of the surface seven inches was screened, thoroughly mixed, and brought into the greenhouse.

Methods and Materials

The greenhouse method of growing plants in small containers of soil treated with varying amounts of BHC or other chemicals was used in this experiment. Usually two crops were grown, the first one of Bountiful bush beans the second of sunflowers. The green and dry yields were then used as a basis for determining the effect of the various soil treatments upon plant growth.

A one per cent gamma BHC dust was used for the BHC treatments.

The Effect of Temperature

To determine the effect of temperature upon the deactivation of soil residues of BHC the following experiment was set up.

A series of 500 gram samples of Willamette soil was treated at rate of 0, 10, 50 and 100 pounds of BHC per acre and stored at 10° C. for 16 days. A duplicate set was stored for 30 days. Another series was treated similarily but stored at room temperature which fluctuated between 20° and 30° C; a third series was held at 60° C. Four replicates of each treatment were prepared.

After 16 days one half of the soil was taken from storage, placed in No. 2 cans in the greenhouse, and planted to sunflowers. When well established, the plants were thinned to 5 plants per pot and each series randomized.

Notes were taken as to the heights every ten days. After approximately 7 weeks the plants were harvested and the green and dry yields taken. After 30 days the remaining samples were taken from storage and treated similarly.

Results

The results from this experiment are given in Table I. These data indicate that low temperatures may decrease the toxicity of BHC residues to plants for a short time at least. This conclusion is born out by a statistically significant decrease in yield at the high temperature, especially at the higher rates of BHC in the 15 day series without a corresponding decrease for the lower temperatures. The yield data from the 30 day series show that the toxicity was about equal to all incubation temperatures. This could be explained on the basis that the cooler temperatures only temporarily inhibited the toxicity of BHC to the sunflower plants.

Figure 3 shows the variation in heights between treatments at ten day intervals through the growth period for the 16 day series. Throughout, the soil incubated at low temperatures was less toxic to plant growth than those grown at 60° C. A similar growth pattern is shown in Figure 4 for the 30 day series. The only difference is that the 10° gave the least toxicity by harvest time while in the 15 day set the 20-30° range gave slightly better

TABLE I

The Effect of Temperature Upon the Phytotoxicity of Benzene Hexachloride in the Soil

	Incubated 16 days		
	60° C	20-30° C	10° C
BHC-lbs/A	grams	grams	grams
CK	2.98*	2.38	2.45
10#	2.35	2.00	2.18
50#	2.08	2.50	2.53
100#	1.40	2.50	2.50
	Incubated 30 days		
CK	2.38	2.23	1.90
10#	2.50	2.50	2.35
50#	1.80	2.28	2.10
100#	1.53	1.68	1.65

* Each weight is the average dry yield from 4 replicated pots with 5 sunflower plants in each. Least significant difference for the 16 day set = .557

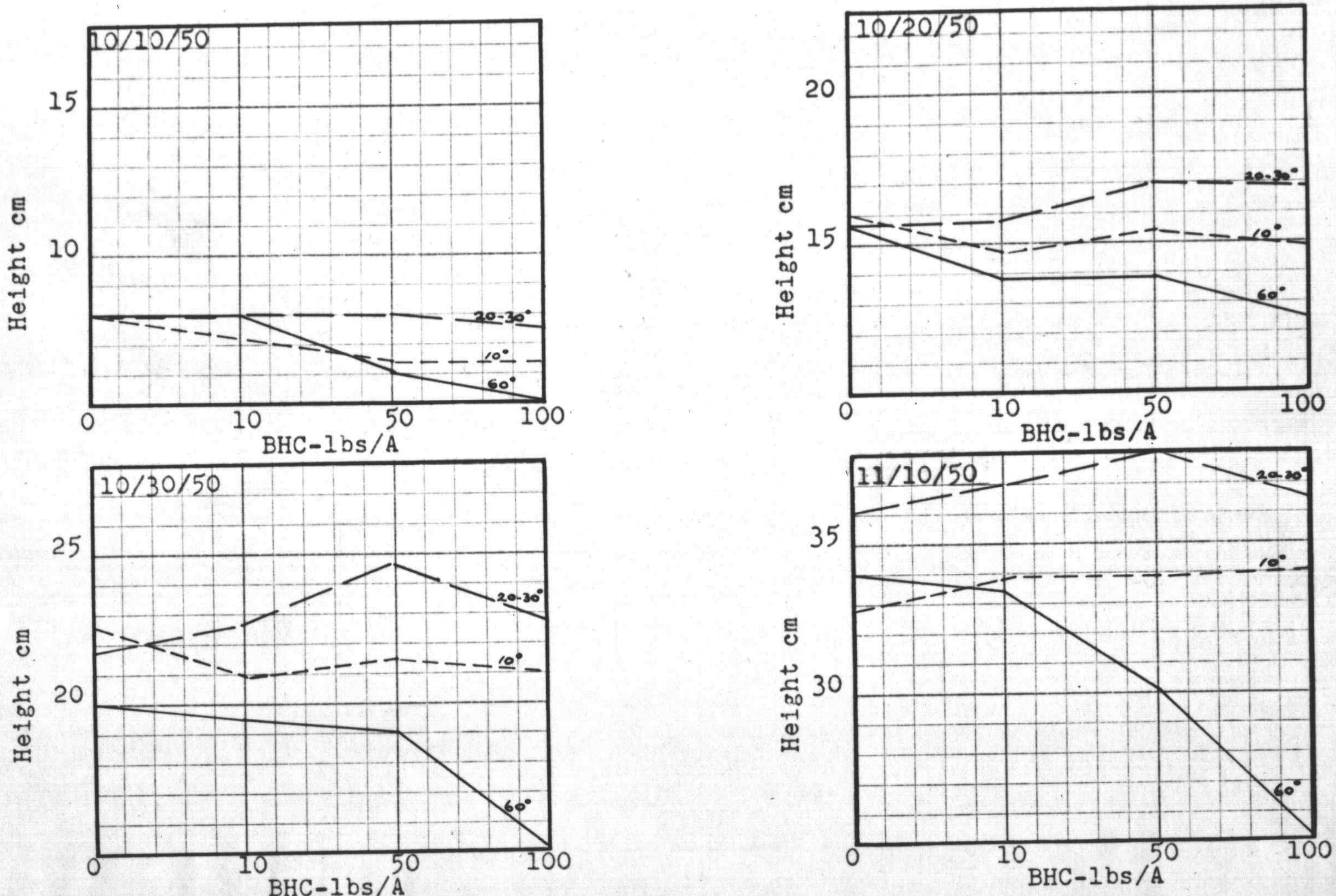


Figure 3. The Effect of Temperature Upon BHC in the Soil as Measured by Plant Growth.

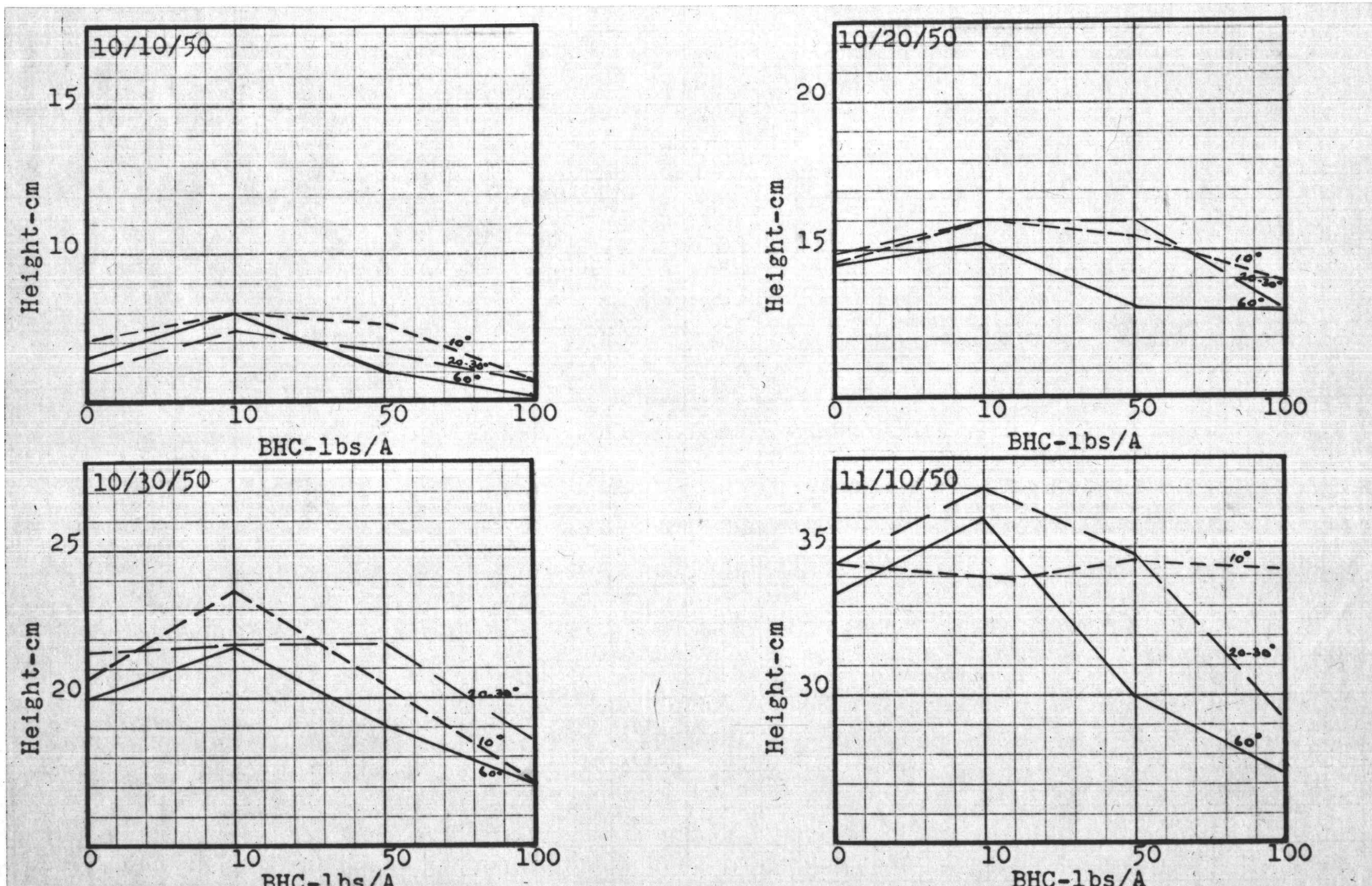


Figure 4 . The Effect of Temperature Upon BHC in the Soil as Measured by Plant Growth.

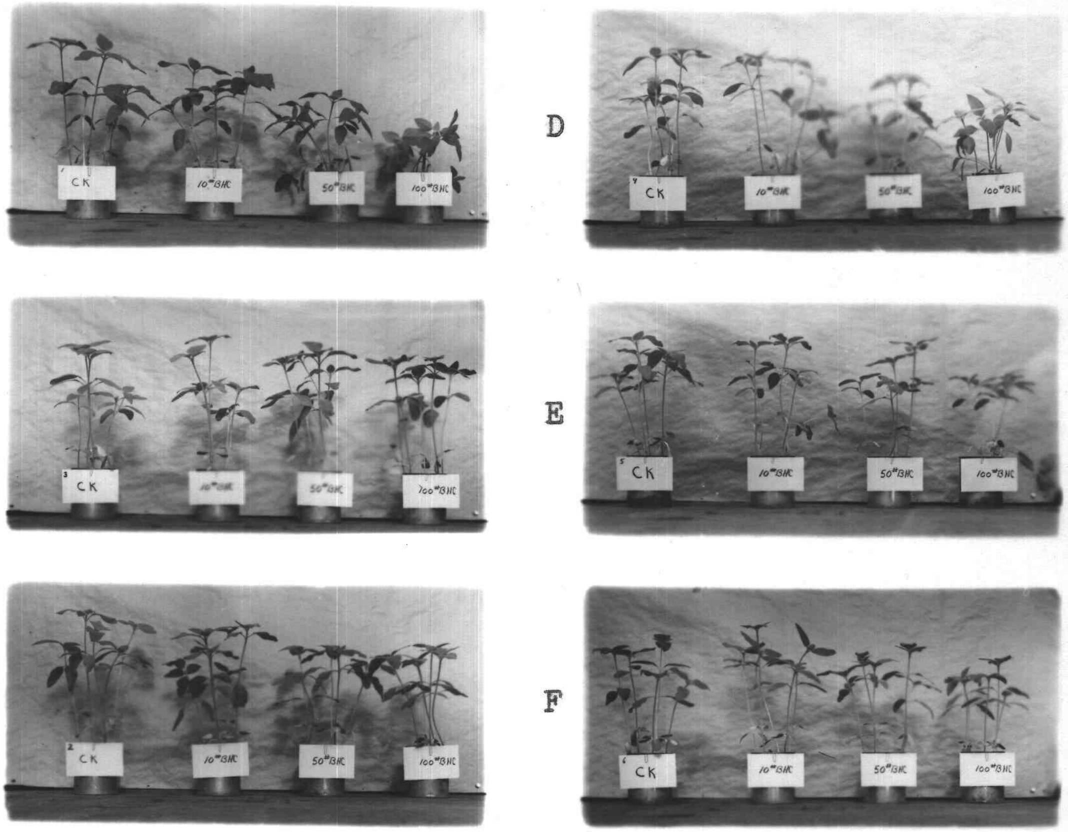
growth.

Further indication that BHC is less toxic at low temperatures may be seen in Figure 5. Here photographs of the plants taken a week before harvest suggest that the low temperature treatments produced better plant growth. Toxicity was particularily severe at the 100 lb. rate of BHC when incubated at 60° C. in both the 10 and 30 day series. Weak, twisted, plants were produced which had to be staked and tied to keep them erect. Other plants receiving 100 lbs BHC per acre but incubated at lower temperatures showed little or no tendency to fall over, and no staking was necessary.

Chemical Antidotes for Toxic Soil Residues

The following experiment was designed to determine whether soil amendments would deactivate toxic residues of BHC in the soil.

Seventeen pound portions of thoroughly mixed and screened Willamette topsoil were placed in two gallon flower pots in the lathhouse. One series was treated as follows: (1) CK - no treatment (2) Lime - 2 T/A (3) Magnesium Carbonate - 2 T/A (4) Manure - 20 T/A (5) Sulfur - 500 lbs/A (6) Activated charcoal - 2.5 grams per pot. A duplicate series was prepared and BHC at the rate of 10 lbs per acre was added to each. Each treatment was replicated four times.



A D

B E

C F

Figure 5. The Effect of Temperature Upon BHC Residues In The Soil. Series A, B, and C, were incubated at 60°, 20-30°, and 10° C., respectively for 16 days. Series D, E, and F were incubated at the same temperatures for 30 days before planting.

After applying the amendments to the soil, beans were planted in each pot. After two weeks the plants were thinned to three per pot. The pots were harvested at the blooming stage and the green and dry yields taken from each. Immediately the pots were replanted to sunflowers for the second crop.

Results

The yield data from the bean crop are summarized in Table II. Significant increases in yields were obtained from lime and manure. Sulfur depressed the yields. All pots receiving BHC showed some decrease in yields. Of these BHC combined with the check, lime, manure and sulfur treatments gave significant reductions in both dry and green yields. Sulfur depressed the yield independently of the BHC. Activated carbon showed more promise in off-setting the toxicity than any of the other material, however, it also showed a tendency to depress growth independently of the BHC.

The yield data from the second crop of sunflowers are given in Table III. Here again the lime and manure, continued to give a significant increase in yields. There was no significant depression in growth from the BHC treatments. In fact in a few cases BHC slightly increased the yields but not significantly. Again activated carbon and sulfur depressed the growth below that of the check.

TABLE II

The Average Dry Yields of Beans With and Without Benzene Hexachloride

Treatment	Yield of Beans (g/pot) Without BHC	With BHC	Increase or Decrease due to BHC grams/pot	%
CK	9.9*	6.8	3.1	-31.3
Lime	10.8	8.6	2.2	-20.4
$MgCO_3$	9.9	8.8	1.1	-11.1
Manure	10.9	8.4	2.5	-22.9
Sulfur	8.3	6.8	1.5	-22.1
Act.Carbon	9.1	8.2	.9	- 9.9

* Average of 4 replications
** Least significant difference = 1.499

There are two possibilities why BHC was not toxic to the second crop. One, it might have become less toxic after two months in the soil or it is possible that sunflowers are more tolerant to BHC residues and thus would not show toxicity to the extent that the first crop of beans did. This will be discussed later.

Generally, there appeared to be no decrease in toxicity from adding these materials at the rates used.

TABLE III

The Average Dry Yield of Sunflowers with and Without Benzene Hexachloride

Treatment	Sunflower dry Weights Without BHC	With BHC	Increase or Decrease due to BHC grams	%
CK	4.8*	5.1	.3**	- 6.3
Lime	5.8	5.3	.5	- 8.6
$MgCO_3$	5.3	5.6	.3	+ 5.7
Manure	6.2	5.1	1.1	-17.8
Sulfur	4.5	4.0	.5	-11.1
Act.Carbon	3.6	4.3	.7	+19.4

* Average weights from 4 replications
** Least significant difference at the 5% level= 0.9510

Soil Reaction

To observe the effect of soil reaction upon the toxicity of BHC, soil samples were taken and the pH determined after harvesting each crop. The various soil amendments gave a range of pH values from 4.7 to 7.4. A comparison of dry yields and the pH values for each treatment is given in Table IV.

Although crop yields correlated very closely with the

TABLE IV

The Relationship of Soil Reaction and BHC Residues in the Soil

Treatment	Beans		Sunflowers	
	Dry yield	pH	Dry yield	pH
CK	9.9*	5.9	4.8	5.7
BHC	6.8	6.1	5.1	5.8
Lime	10.8	7.4	5.8	6.9
Lime - BHC	8.6	7.1	5.3	6.7
$MgCO_3$	9.9	7.3	5.3	7.3
$MgCO_3$ - BHC	8.8	7.3	5.6	7.3
Manure	10.9	6.0	6.2	5.9
Manure - BHC	8.4	6.0	5.1	6.0
Sulfur	8.3	4.7	4.5	4.7
Sulfur - BHC	6.8	4.9	4.0	5.0
Act.Carbon	9.1	5.7	3.6	5.6
Act.Carbon-BHC	8.2	5.8	4.3	5.7

* Average yield from 4 replications.

soil reaction no relationship between BHC toxicity and the pH, in the range from 4.7 to 7.4, was found.

Soil Texture

To determine the effect of soil texture upon increasing or decreasing the toxicity of BHC the following pot study was set up. A series of pots varying the amount of BHC from one to fifty pounds per acre was prepared on three soils, Willamette silty clay loam, Newberg fine sandy loam, and Labish peat. The treatments were 0, 2.5, 5, 10, 50 pounds per acres applications of BHC. Bean and sunflower crops were grown in 500 grams of soil in No. 2 cans in the greenhouse. Considerable difficulty in getting a stand of beans because of a period of cold weather handicapped this experiment. A stand of one plant per pot was all that could be obtained on the Willamette and Labish series. The yields from the Newberg pots were not taken because of an even poorer stand. This was apparently not associated with the BHC treatments since checks and treated pots suffered equally.

The yield data from the Willamette and the Labish appear in Tables V and VI. The green and dry weights do not show toxicity even at the higher applications of BHC. However, stunting of heights was definite in the Willamette series but not on the Labish soils.

Table VII gives the results from the second crop, on these same three soils. Here a good stand of five plants

TABLE V

Average Bean Yields from Increasing Amounts of BHC on Willamette Soil

Treatment	Yields of Beans		
	Green w'ts	Dry w'ts	Height
lbs/A	grams	grams	cm
CK	12.48*	2.19	30.50**
1#	11.07	2.03	29.67
2.5#	13.48	2.63	25.67
5#	12.78	2.15	28.67
10#	11.65	2.02	24.33
25#	12.57	2.18	27.33
50#	11.83	1.87	18.33

* Average of 6 replications
** Least significant difference in heights = 3.69

per pot was obtained. Since there were no obvious differences the heights were not taken. The dry and green yields indicate that there was no toxicity to sunflowers on any of the soils up to fifty pounds per acre. This strongly suggests that BHC residues under greenhouse conditions, are not toxic to sunflower plants three months after treatment.

TABLE VI

Average Bean Yield From Increasing Amounts of BHC on Labish Peat

Treatment	Yields of Beans		
	Green w'ts	Dry w'ts	Height
lbs/A	grams	grams	cm
CK	17.5*	3.75	36.3**
1#	17.2	3.65	38.0
2.5#	17.4	3.59	36.7
5#	16.0	3.32	31.3
10#	16.9	3.78	33.2
25#	15.4	3.55	33.3
50#	17.5	3.92	33.7

* Average of 6 replications
** Least significant difference in heights = 4.67

Leaching

A leaching experiment was designed in the following manner.

Twelve large glazed stoneware crocks were filled with approximately 17 pounds of Willamette topsoil. Eight pots were treated with BHC at the rate of ten pounds per acre. Of these, four were leached with measured amounts of water

TABLE VII

Average Sunflower Yields from 3 Soils Receiving Various BHC Treatments

Treatment	Willamette		Newberg		Labish	
	green wts	dry wts	green wts	dry wts	green wts	dry wts
lbs/A	grams	grams	grams	grams	grams	grams
CK	20.41*	3.83	19.37	4.51	19.95	3.75
1#	19.69	3.80	22.33	4.50	20.33	3.65
2.5#	19.40	3.80	21.97	4.45	20.62	3.58
5#	17.87	3.53	23.73	4.70	21.32	3.32
10#	17.12	3.12	21.27	4.71	20.10	3.78
25#	17.72	3.28	22.33	4.57	19.75	3.55
50#	19.03	3.83	24.62	5.15	21.12	3.92

* Average yields of 6 randomized pots harvested 9-27-50. Treated 6-16-50.

every few days for two weeks. Four pots were left untreated and unleached for checks. Beans were planted, and later thinned to four plants per pot, for the indicator crop. The yields are given in Table VIII.

TABLE VIII

The Effect of Leaching on BHC Residues

Treatment	Green w'ts	Dry w'ts
	gms.	gms.
CK	133.1*	17.4
BHC	145.6	19.1
BHC - Leached	103.3	15.4

* Average yield from four replications.

Toward the middle of the growing period it was obvious that the plants in the leached pots were suffering from a nitrogen deficiency. All pots were immediately watered with a complete nutrient solution, however, the plants in the leached pots did not recover before harvest. This is borne out by the comparatively low yield in those pots. Apparently the ten pounds per acre application of BHC was not high enough to produce toxicity to beans in the greenhouse. This will be discussed in detail in a later section.

Generally speaking, no conclusions concerning the effect of leaching in deactivating toxic residues are possible on the basis of this experiment.

DISCUSSION

The possibility of an accumulation of toxic BHC residues in the soil which would seriously limit plant growth does not appear improbable. Not only soil treatments but also sprays and dusts applied to the aerial portions of orchards and many other crops find their way into the soil, either directly as drippings from the foliage, or with the plant residues returned to the soil at the end of the season. In a few years it is quite possible that a toxic concentration of BHC could build up in the soil.

Removal or deactivation of harmful soil residues could conceivably result from leaching, decomposition by microorganisms, chemical reaction with soil constituents, from fixation or absorption by soil colloids, or possibly from volatilization. When BHC reaches the soil it is probably immediately fixed on the colloidal fraction to be slowly released and absorbed by the plant. That this release is speeded by high temperatures and inhibited by low temperatures is indicated by the temperature study. Increased biological action due to high temperature may also enter into the release of toxic compounds for plant absorption.

That BHC is translocated in the plant is fairly certain from persistant reports of off-flavors in many crops. One way of removing BHC from soils is thus obvious - removal in the harvested portions of the plant. It would be interesting to determine the BHC content of plants grown on BHC plants grown on BHC treated soil. Absorption would have to be quite high for this to be an important method of removal.

The most promising method of deactivating soil residues would appear to be by the use of chemicals. Benzene hexachloride has been reported (21) to decompose in the presence of lime water and other alkaline substances. Neither $CaCO_3$ nor $MgCO_3$ decreased the toxicity in this experiment although both resulted in higher yields. Manure also increased the yield but showed no tendency to counteract the toxicity from the BHC. In parallel experiments, Dr. Powers reports that manure did lessen musty flavor. Sulfur depressed the yields and increased soil acidity but also showed no effect in decreasing toxicity.

It was expected that activated charcoal because of its high absorbent properties might absorb some of the toxic substances. The data from this experiment did not bear out this theory but neither did it refute it. The response may be masked because the activated carbon was found to depress the yield independently of the BHC. This is especially evident in the second crop. The data in table IV show that activated carbon has more tendency to reduce the BHC toxicity than any of the other treatments. More study along this line is needed.

Soil texture, and especially the amount of colloidal material in the soil is an important factor in absorbing BHC. This is illustrated by the high rates of BHC that muck and peat soils are able to absorb without any noticeable plant toxicity. However, between two mineral soils, Willamette silty clay loam, and Newberg fine sandy loam, no difference in toxicity was observed from rates of BHC up to fifty pounds per acre.

There is apparently little relationship between the soil reaction and the toxicity of BHC. Hopes that BHC residues might decompose more readily in alkaline soils are apparently unfounded. This experiment bears out a previous report (22) to that effect.

Because of the insolubility of BHC in water it may be predicted that little will be leached from the soil. The results from leaching in this study were inconclusive. It might be interesting to leach soils with alkaline water or other dissolving substances.

Other factors such as moisture relationships probably should be considered. Examples of apparently conflicting data may be found in this study. In one case 10 pounds per acre BHC was toxic to beans in the lathhouse but not in the greenhouse. The only important variables were (1) a less rigorous climate inside the greenhouse (2) the treated soil in the greenhouse was kept moist for two weeks before planting. In another case 50 pounds per acre of BHC failed to produce toxicity to sunflowers while in the temperature study as low as 10 pounds toxic in some cases. Conditions not altogether clear are responsible for considerable variation in degree of injury from BHC in the soil.

Although insecticidal considerations are based upon the gamma isomer content of BHC it should be remembered that the other isomers undoubtedly affect plant growth. For each pound of gamma isomer added from 2 to 8 pounds of other isomers, depending upon the purity of the product, also reach the soil. One way for the farmer to materially reduce the danger of toxic accumulations is to use the purest form of gamma BHC which is available.

SUMMARY AND CONCLUSIONS

A greenhouse experiment was designed to determine the effect of temperature, lime, manure, sulfur, magnesium carbonate, and activated charcoal upon the deactivation of harmful soil residues of BHC in the soil as measured their toxicity to plants. Limited observations on other factors such as soil texture, leaching, and soil reaction were made.

When incubated at low temperatures for 16 days before planting the toxicity of BHC to sunflower plants was decreased. After 30 days incubation all temperatures produced about equal toxicity.

Lime, manure, sulfur, and magnesium carbonate when applied with BHC gave no indication of counteracting the toxicity. Activated charcoal showed slightly more promise as an antidote but tended to depress growth, at the rate use, independently of BHC.

BHC was more toxic on a mineral soil than on Labish peat. No difference in toxicity was observed between a fine sandy loam and a silty clay loam in the second crop of sunflowers, from rates up to 50 pounds per acre.

No relationship between the toxicity of BHC and the soil reaction in the range from 4.7 to 7.4 was observed.

The results from the leaching experiment were inconclusive.

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