

Relation of Herbicidal Activity to the Amide Moiety of *N*-Substituted Alpha-Chloroacetamides

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The herbicidal activities of a variety of *N*-substituted α -chloroacetamides are reported. The limits of activity have been clearly defined with respect to the amide moiety. As a class, the α -chloroacetamides, as pre-emergence herbicides, possess outstanding effectiveness and selectivity for the control of annual grasses in pre-emergence application.

A STUDY OF THE PHYTOTOXICITY OF *N*-substituted α -chloroacetamides has not previously been reported. A few α -chloroacetanilides (5) have shown a low order of activity in root elongation tests. Fungicidal activity (1, 2) has been reported for only *N*-substituted α -bromo- (6), α,α -dibromo- (3), and α,α -dichloroacetamides (4), *N*-substituted α -bromopropionamides (7), and a wide variety of *N*-substituted α -haloacetanilides (7). A study of the phytotoxic properties of some of these compounds and, in particular, of the *N*-substituted α -chloroacetamides has therefore been undertaken.

As a class the *N*-substituted α -chloroacetamides possess outstanding effectiveness and selectivity for the control of annual grasses. Their activity on germinating grass seeds is comparable to 2,4-D in the control of broadleaf weeds. *N,N*-Diallyl- α -chloroacetamide is as effective a pre-emergence herbicide on most weedy seeds as 2,4-D is on susceptible broadleaf plant seeds under the same conditions at any reasonable rate of application, such as 1 pound per acre.

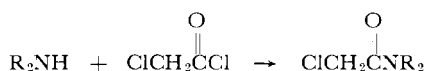
A point of primary interest is the herbicidal potency of these new chemicals. As little as 1 part in 40,000,000 (0.025 p.p.m.) of certain *N*-substituted α -chloroacetamides is required for 80% growth inhibition of rye grass seeds. Comparable results are obtained with isopropyl *N*-(3-chlorophenyl) carbamate (CIPC) in this same test. In more practical terms, it appears that rates of 3 to 6 pounds per acre will adequately control most annual grasses in the field.

The correlation of structural configuration of the α -chloroacetamides with pre-emergence, grass-specific herbicidal properties is the subject of this paper.

Methods of Preparation

The *N*-substituted α -chloroacetamides were prepared by the addition of chloroacetyl chloride to a mixture consisting of the primary or secondary amine, 20% sodium hydroxide, and an inert

solvent such as ethylene dichloride below 0° C. The chloroacetamides were recovered from the solvent layer and purified by distillation or recrystallization from an appropriate solvent. The yields were usually greater than 75%.



Many modifications of this general method have been used, owing to the nature of the amine. The amines were for the most part commercially available. The primary amines, when required, were prepared from potassium phthalimide or hexamethylenetetramine and the corresponding halide, while the secondary amines were prepared by the calcium cyanamide method, by alkylation of the primary amine, or from ammonia and the halide. The methods of preparation and chemical properties of these compounds will be published separately.

Method of Testing

The results discussed here are based on greenhouse screening studies. The screening evaluation methods for pre-emergence herbicidal action are simple. The chemical to be tested is applied in spray form to soil seeded to representative grass and broadleaf plants. A 13 × 9 × 2 inch aluminum pan, perforated on the bottom, is level-filled with standard soil preparation to a depth of 3/8 inch from the pan top. Twenty seeds each of three weedy grasses—wild oat, cheat grass, and rye—and five corn seeds are scattered randomly over one third of the soil surface area. Twenty seeds of five or six representative broadleaf plants—buckwheat, mustard, red clover, beet, cotton, and cucumber—are scattered on the remaining two thirds of the soil area. The seeds are covered with soil to the pan top. The planted pans are placed in an exhaust hood and sprayed with 30 ml. of an aqueous solution containing 1% of a 20-20-20 water-soluble fertilizer (Monsanto, Folium) and

0.1% octamethyl pyrophosphoramidate. The pan is next sprayed with 30 ml. of an organic solvent (usually acetone) containing the chemical to be tested. The spray volume is kept constant, and the rate per acre of active material is varied as desired.

The prepared pan is next placed in an aluminum greenhouse bench in 0.5 inch of water and allowed to take up water from below until two thirds of the top surface shows evidence of moisture. The pan is removed and placed in the greenhouse on a moist soil bench. The seeds germinate and emerge in 3 to 4 days, and observations on the degree of activity are made on the 10th day after planting.

The herbicidal ratings are determined for each grass species according to the following scale.

% Germination and Emergence	Herbicidal Rating
0-25	3
26-50	2
51-75	1
76-100	0

The herbicidal ratings reported in the tables are achieved by a summation of the herbicidal ratings for each weedy grass species. Any rating from 9 to 0 is possible, with a maximum score of 9, assuming three ratings for each grass species. A simple arbitrary scale was used to arrive at a summation herbicidal rating for the three weedy grasses used in the tests.

Summation of Individual Grass Ratings	Summation Herbicidal Rating
9	4
7-8	3
5-6	2
3-4	1
1-2	0

Results

Data on the herbicidal activity of a variety of α -chloroacetamides are given in

Tables I, II, and III. The compounds listed in Table I are divided into sections in order to illustrate structural similarities. The relation of structural isomers possessing a three-carbon chain is shown in Table II and the herbicidal activity of compounds containing more than one chloroacetamido group is presented in Table III.

Herbicidal ratings of individual grass and broadleaf test plants for one of the α -chloroacetamides, *N,N*-diallyl- α -chloroacetamide, are given in Table IV. In Table V, plant species are categorized according to their tolerance to these herbicides.

In most instances, only one rate of application is given for each compound. However, upper and lower limits of activity have been determined. For example, a compound may be given a 0 rating at 5 pounds per acre and will be considered inactive at this rate, yet the compound may show a 4 rating at 10 pounds per acre. The optimum rate may be between 5 and 10 pounds and at this rate the compound would be highly active.

Discussion

The relationship between structural configuration and activity is illuminating, in that the limits of activity appear to be clearly established. With respect to the amide moiety, an α -chloroacetamide must carry no more than one hydrogen on the nitrogen atom for activity. Table I, A, illustrates the effect of saturated straight-chain substituents. The data indicate a very large increase in activity with a single *N*-methyl substituent. The activity increases gradually to a peak with the propyl derivative, then decreases relatively rapidly to a low order of activity with *N*-amyl and above. When two straight-chain alkyl groups (Table I, B) are attached to the nitrogen atom of α -chloroacetamide, activity is enhanced. The activity increase from the parent compound, chloroacetamide, to the dimethyl derivative is enormous. Reduced rates of application indicate a general increase in activity from methyl to propyl. Activity drops off rapidly as in the case of the monosubstitution products. It appears that a three-carbon chain achieves some necessary requirement for maximum activity. In general, these compounds are more active than the corresponding monosubstitution products.

Monosubstitution with branched alkyl groups, however, shows greater activity than the corresponding *N*-disubstituted derivatives, as illustrated in Table I, C and D. This activity may be due to the apparent importance of the three-carbon straight-chain effect as depicted in Table II. Thus, the propyl and allyl derivatives (3 at 5) are

Table I. Herbicidal Activity of *N*-Substituted α -Chloroacetamides

$$\begin{array}{c} \text{R} \quad \text{O} \\ \diagdown \quad | \\ \text{N} \text{---} \text{C} \text{---} \text{CH}_2\text{Cl} \\ \diagup \\ \text{R}' \end{array}$$

	R	R'	Herbicidal Rating ^a	Pounds/Acre		
A.	Hydrogen	Hydrogen	0	15		
		Methyl	3	15		
		Ethyl	4(1)	10(5)		
		Propyl	3	5		
		Butyl	2(4)	10(25)		
		Amyl	1	10		
		Tetradecyl	1	25		
B.	Hydrogen	Hydrogen	0(0)	5(15)		
		Methyl	4	5		
		Ethyl	4	5		
		Propyl	4	5		
		Butyl	2	5		
		Amyl	0	5		
C.	Hydrogen	Isopropyl	0(2)	5(10)		
		Isobutyl	4	5		
		<i>sec</i> -Butyl	3	5		
		<i>tert</i> -Butyl	0(2)	15(25)		
		Isoamyl	3	5		
D.	Isopropyl	Isopropyl	0	5		
		Isobutyl	0	5		
		<i>sec</i> -Butyl	0	5		
		Isoamyl	1	5		
		2-Ethylhexyl	0	5		
E.	Hydrogen	Propyl	3	5		
		Allyl	3	5		
		Propyl	4	5		
		Allyl	4(4)	5(1)		
		Propargyl	4	5		
		Hydrogen	4	5		
		Isobutyl	1(3)	5(25)		
		Methyl	0	5		
		Methyl	0(3)	5(25)		
F.	Hydrogen	2-Chloroethyl	3	5		
		2-Chloropropyl	4	5		
		3-Chloropropyl	3	5		
		2,3-Dichloropropyl	2	5		
		2-Chloroallyl	1(3)	5(15)		
		3-Chloroallyl	2(3)	5(15)		
		2,3-Dichloroallyl	2(4)	5(25)		
		3-Methoxypropyl	2	5		
		3-Isopropoxypropyl	4	5		
		G.	2-Chloroethyl	2-Chloroethyl	1(0)	25(5)
2-Chloropropyl	4(0)			25(5)		
2,3-Dichloropropyl	2			5		
2-Chloroallyl	2(1)			15(5)		
3-Chloroallyl	3(1)			15(5)		
H.	Ethyl	Butyl	2	25		
		2-Chloroallyl	2	10		
		Ethyl	2	5		
		Butyl	2	5		
		Allyl	2	2.5		
I.	Hydrogen	Furfuryl	3	5		
		Tetrahydrofurfuryl	4	2.5		
		2-Thienyl	4	5		
		3-Oxapentamethylene	4	2.5		
		Tetramethylene	4	5		
J.	Pentamethylene	1-Methylpentamethylene	4	5		
		2-Methylpentamethylene	4	2.5		
		3-Methylpentamethylene	4	2.5		
		1,3-Dimethylpentamethylene	4	2.5		
		1,5-Dimethylpentamethylene	3	2.5		
		1-Methyl-4-ethylpentamethylene	4	2.5		
		1,3,5-Trimethylpentamethylene	4	2.5		
		K.	Hydrogen	Cyclohexyl	4	5
Methyl	2			5		
Ethyl	1			5		
Allyl	3			5		
2-Chloroallyl	3			5		
3-Chloro-2-butenyl	0(2)			5(25)		
Tetrahydrofurfuryl	2			25		
Cyclohexyl	0			25		

Table I. Herbicidal Activity of *N*-Substituted α -Chloroacetamides (Continued)

$$\begin{array}{c} \text{R} \\ \diagdown \\ \text{N}=\text{C} \\ \diagup \\ \text{R}' \end{array} \text{C}(\text{O})\text{CH}_2\text{Cl}$$

	R	R'	Herbicidal Rating ^a	Pounds/Acre	
L.	Hydrogen	Phenyl	0(3)	15(25)	
	Methyl	Phenyl	2	5	
	Ethyl	Phenyl	2	5	
	Propyl	Phenyl	3	5	
	2-Chloroallyl	Phenyl	1(4)	5(25)	
	Butyl	Phenyl	2	5	
	Hydrogen	4-Chlorophenyl	0	25	
	Ethyl	4-Chlorophenyl	3	5	
	M.	Hydrogen	Benzyl	3	5
			2,4-Dichlorobenzyl	2	25
		3,4-Dichlorobenzyl	1	25	
		2,4,6-Trichlorobenzyl	0	25	
Benzyl		Benzyl	0	25	
Hydrogen		2-Phenylethyl	2	15	

^a Figures in parentheses under Herbicidal Rating represent the rating of the corresponding concentration shown parenthetically in the last column under pounds/acre.

considerably more active than the ethyl homolog (1 at 5). The isopropyl group, which is considerably less active than its isomer, is in effect a two-carbon chain with a methyl group in a branched position, and its activity very closely corresponds to that obtained with the ethyl group. The *tert*-butyl group (0 at 5), which is also structurally related to the isopropyl or ethyl radicals, imparts the same degree of activity to the chloro-

acetamide. Both the isobutyl (4 at 5) and *sec*-butyl (3 at 5) radicals, however, possess a three-carbon chain bearing a methyl substituent and their activities are comparable to the propyl amide. Those compounds possessing normal alkyl groups of four and five carbon atoms (*N*-butyl and *N*-amyl) are appreciably less active than the branched-chain isomers (isoamyl, 3 at 5). Steric factors are probably not important in this series, since the isopropyl and *sec*-butyl groups are almost equivalent, sterically. However, the low order of activity of those compounds in which both alkyl groups possess branching (Table I, D) may be due to steric factors. Steric hindrance may be important if the assumption is made that the activity of the α -chloroacetamides is in some way related to the accessibility of the nitrogen atom. This may be a fallacious argument.

In the three-carbon chain, activity is maintained or enhanced when unsaturation is present. This is shown in Table I, E, which contains some of the most active compounds tested. The methallyl derivative is somewhat less active than the isobutyl.

Table I, F, indicates that the *N*-substituent can carry a variety of functional groups without appreciable loss of activity. It is interesting to note that *N*-(3-methoxypropyl)- and *N*-(3-isopropoxypropyl)- α -chloroacetamides possess outstanding activity. The length of the

alkyl group attached to the nitrogen atom can exceed three carbon atoms, and activity can be maintained, if a hetero atom is included in the chain. The activity of the *N,N*-disubstituted chloroacetamides, derived from those entities indicated in Table I, F, is markedly reduced (Table I, G). These compounds behave in a manner similar to the *N*-disubstituted, branched-chain, aliphatic chloroacetamides. Steric factors may be important in these compounds.

A number of the unsymmetrical *N*-substituted α -chloroacetamides are active compounds (Table I, H). The activity appears, in general, to be a result of the additive effects of the individual substituents. The reduced action of *N*-(2-chloroallyl)-*N*-isopropyl- α -chloroacetamide (2 at 10) illustrates the nullifying effect of branched chains (isopropyl) combined with the substituted straight-chain entities. This low order of activity is comparable to the *N*-disubstituted derivatives listed in Table I, D and G.

Among the most active groups of α -chloroacetamides are those in which the *N*-substituent is heterocyclic. Thus in Table I, I, the furfuryl, tetrahydrofurfuryl, and 2-thenyl- α -chloroacetamides are highly active at 2.5 to 5 pounds per acre. The amide nitrogen atom may become part of the alicyclic ring as in the morpholine and pyrrolidine derivatives. The piperidine nucleus also appears active. The number and position of alkyl groups present in the piperidine nucleus produce no large differences in activity (Table I, J).

The activity of *N*-cyclohexyl- α -chloroacetamide (Table I, K) is not increased when a second group is attached to the nitrogen atom. Activity decreases in *N*-substitution after allyl and chloroallyl. This may be the optimum action of the three-carbon chain asserting itself.

In the aromatic series, activity drops off considerably; a phenyl or substituted phenyl attached to the nitrogen atom of chloroacetamide gives α -chloroacetanilides which are practically devoid of pre-emergence activity. Regardless of the position or number of substituents (Cl, NO₂, CH₃O, and alkyl), activity to any great extent is not obtained. When, however, an alkyl group is attached to the nitrogen atom of the α -chloroacetanilide, activity is greatly enhanced and can be made to approach that of the aliphatic compounds (Table I, L).

Table II. Efficacy of Three-Carbon Straight Chain

(As illustrated by structural isomerism)

$$\begin{array}{c} \text{H} \\ \diagdown \\ \text{N}=\text{C} \\ \diagup \\ \text{R} \end{array} \text{C}(\text{O})\text{CH}_2\text{Cl}$$

R	Structural Formula	Herbicidal Rating at 5 lb./Acre
Ethyl	C—C—	1
Isopropyl	C—C—	0
	 C	
<i>tert</i> -Butyl	C	0
	C—C— C	
Propyl	C—C—C—	3
Allyl	C—C=C—	3
Isobutyl	C—C—C—	4
	 C	
<i>sec</i> -Butyl	C—C—C	3
	 C	
Isoamyl	C—C—C—C—	3
	 C	
Butyl	C—C—C—C—	1
Amyl	C—C—C—C—C	0

Table III. Effect of Polychloroacetyl Groups

Compound	Herbicidal Ratings	Pounds/Acre
ClCH ₂ CONHCH ₂ NHCOCH ₂ Cl	0	25
ClCH ₂ CONHCH ₂ CH ₂ NHCOCH ₂ Cl	1	25
ClCH ₂ CONHCH ₂ CH ₂ CH ₂ NHCOCH ₂ Cl	3	25
(ClCH ₂ CONHCH ₂ CH ₂) ₂ NCOCH ₂ Cl	0	25
(ClCH ₂ CONHCH ₂ CH ₂ CH ₂) ₂ NCOCH ₂ Cl	1	25

In Table I, M, *N*-benzyl- α -chloroacetamide is active at 5 pounds per acre. Activity is not maintained, however, in the other derivatives. The 2-phenylethylamide is considerably less active than the benzyl homolog.

Increasing the number of chloroacetamido groups in a molecule does not lead to more active compounds (Table III). For example, chloroacetylation of diamines such as ethylenediamine and trimethylenediamine gives rise to bis- α -chloroacetamides which are almost devoid of activity at 25 pounds per acre.

Physiological Effects

The data presented in the tables correlating structure and activity are comparisons of emergence prevention of the seedlings of three test grasses. The evaluation is one of degree rather than descriptive of a certain visual response.

Definition of the exact physiological effect of the α -chloroacetamides on the germination and growth of grass seedling must await intensive biochemical studies of various intercellular metabolic processes. The activity per quantity of chemical is great enough to indicate enzymatic interference or an antimetabolite action at some critical point in the growth process. Germination and growth of rye grass in aerated solutions are 80% inhibited at a concentration of 0.025 p.p.m.

The germination of grass seeds is completely inhibited at varying threshold concentrations of chemical, depending upon the grass species. At lower chemical concentrations the grass seeds germinate and the coleoptile grows to varying lengths inversely proportional to the rate of chemical applied. In this range the coleoptile or leaf sheath stops growing. At just the right concentrations the true leaf will rupture the side of the coleoptile and emerge. The plant may or may not survive.

Many of the dicotyledonous plant families become increasingly susceptible to the α -chloroacetamides when the concentration is raised above the threshold toxicity level for grasses. At rates of 25 pounds per acre and above an increasing number of broadleaf plant families succumb. Fifty pounds per acre result in almost complete germination inhibition of all seeds tested.

The action of these new compounds on members of the mustard family (*Cruciferae*) is in sharp contrast to the action of the chlorinated phenoxyacetic acid herbicides. Most members of the mustard family are very susceptible to phenoxyacetic acid derivatives. All members tested are very resistant to the action of the α -chloroacetamides. Buckwheat and members of the buckwheat family (*Polygonaceae*) are susceptible to the carbamate-type herbicides. Buckwheat is relatively resistant to the α -chloroacetamides.

Table IV. Herbicidal Ratings^a at Various Application Rates

(*N,N*-diallyl- α -chloroacetamide)

Plants Tested	Pounds per Acre				
	50	25	15	5	1
Wild oat	3	3	3	3	3
Rye grass	3	3	3	3	3
Cheat grass	3	3	3	3	3
Corn	..	1	0	0	0
Buckwheat	0	0	0	0	0
Radish	0	1	0	0	0
Red clover	3	3	3	2	1
Sugar beet	3	3	1	1	1
Cucumber	3	3	2	2	0
Cotton	0	0	0	0	0

^a See discussion on herbicidal ratings.

Table IV presents a graphic picture of increasing plant spectrum susceptibility with increasing application rate. It also points out the apparent large safety factor for certain crops. These results are based on a relatively short-time observation (2 weeks).

The effective action against grasses is evidenced by the consistent control from 50 down to 1 pound per acre. The resistance of buckwheat, radish, cotton, and corn is outstanding at almost all rates. The susceptibility of cucumber, sugar beets, and red clover increases with increasing concentration. Some injury is evident at relatively low rates.

Table V represents a classification of various plants into (1) susceptible, (2) moderately resistant, and (3) resistant categories. The herbicidal ratings for the individual plant species were obtained from greenhouse and field tests with *N,N*-diallyl- α -chloroacetamide.

In general, the heterocyclic derivatives such as the morpholine and methylpiperidine isomers are least specific—i. e., would have the lowest selectivity quotient. The saturated aliphatic derivatives appear to have the highest selectivity quotient—i. e., good grass control with a minimum of toxicity to any broadleaf species. A variety of crops exhibited an amazing tolerance to these chemicals in extensive greenhouse and field tests. The α -chloroacetamides caused no visible crop injury or reduction in yield of the major cultivated agronomic and horticultural crops when applied at 12 pounds per acre. This is two to four times the recommended rate for satisfactory weed control.

Perhaps the most outstanding attribute of this class of chemicals is their use as pre-emergence herbicides to remove annual grasses such as crab grass, foxtail, cheat grass, and wild oats from corn without injury to corn. Weed control was equal or superior to that obtained with commercially available compounds, without apparent stunting of the crop or reduction of final yields. These compounds have adequately removed annual grasses in pre-emergence application from such field crops as soybeans, cotton, tobacco, peanuts, and flax,

and from horticultural crops such as onions, snap beans, lima beans, sweet corn, carrots, broccoli, spinach, turnips, radishes, and table beets.

The α -chloroacetamides are classed generally as grass-specific, but they are not confined solely to grass control. Many common broadleaf weeds such as pigweed, carpet weed, and prostrate spurge are controlled well to very well in pre-emergence application. Good to excellent grass control has been achieved with several derivatives when used as a contact spray on weedy annual grasses.

Table V. Plant Tolerance to *N,N*-Diallyl- α -chloroacetamide

Plant Specie	Herbicidal Rating	Pounds/Acre
Susceptible Species		
Rye grass	3	5
Giant foxtail	3	5
Green foxtail	3	5
Yellow foxtail	3	5
Golden millet	3	4
Cheat grass	3	5
Downy brome	3	5
Rice	3	5
Rough pigweed	3	5
Green amaranth	3	5
Crab grass	3	5
Cucumber	2	5
Purslane	3	4
Barnyard grass	3	6
Moderately Resistant Species		
Table beets	1	5
Sugar beets	1	5
Wheat	2	5
Oats	2	5
Lamb's-quarters	1	5
Kinghead	1	5
Spotted smartweed	1	5
Flax	1	8
Resistant Species		
Sorghum	0	8
Sweet corn	0	12
Field corn	0	12
Sugar cane	0	10
Cotton	0	10
Soybeans	0	8
Onions	0	5
Peas	0	8
Asparagus	0	8
Potatoes	0	8
Radish	0	12
Spinach	0	8
Peanuts	0	8
Barley	0	8

Conclusions

N-Mono- and disubstituted α -chloroacetamides exhibit outstanding activity as grass-specific, pre-emergence herbicides.

A three-carbon chain fulfills some requirement for maximum activity.

N-Disubstitution appears superior to *N*-monosubstitution with a straight-chain aliphatic group.

Branched-chain aliphatic groups show less activity in the *N*-disubstituted chloroacetamides than in the *N*-monosubstituted compounds.

A number of heterocyclic derivatives are among the most active compounds tested.

The aromatic compounds are almost

inactive unless an aliphatic substituent is attached to the nitrogen atom.

Acknowledgment

The valuable assistance of John M. Deming and Clyde L. Wilson in conducting extensive greenhouse and field studies is gratefully acknowledged. Discussions with Ernest G. Jaworski were of immeasurable value.

The help of D. B. Mackenzie and J. H. Nolan in preparing the herbicidal tests is gratefully acknowledged.

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Received for review October 3, 1955. Accepted February 11, 1956. Division of Agricultural and Food Chemistry, 127th Meeting, ACS, Cincinnati, Ohio, March-April 1955, and North Central Weed Control Conference, Fargo, N. D., December 1954.

PESTICIDE RESIDUES

Determination of *O*-(3-Chloro-4-nitrophenyl)-*O*,*O*-dimethyl Phosphorothioate (Chlorthion) Residues in Cottonseed

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An analytical method described for the determination of chlorthion residues in cottonseed is spectrophotometrically sensitive to 0.02 p.p.m. in 200 grams of cottonseed. The isolative procedures include extraction, liquid-liquid partition, and chromatography. Cottonseed from a planting treated with dosages of chlorthion probably greater than that expected in commercial treatment was found to contain no residues when a visual comparative method sensitive to 0.01 p.p.m. was used.

THE USEFULNESS OF *O*-(3-chloro-4-nitrophenyl)-*O*,*O*-dimethyl phosphorothioate (chlorthion) for the control of several cotton pests (3) requires an investigation into the possible presence of residues of this material, as the expressed oil of cottonseed may be converted to edible oils and the cottonseed cake may be used as livestock feed.

Cottonseed obtained from a plot, planted and treated with chlorthion especially for this investigation, was mechanically ginned, delinted with concentrated sulfuric acid, ground, and extracted with pentane in the apparatus depicted in Figure 1. Chlorthion was separated from coextracted cottonseed oil by partition between pentane and acetonitrile. Pigments and other interfering materials were then removed from the acetonitrile solution of chlorthion by chromatography through activated alumina. Finally, the colored compound was developed with chlorthion (6) in a manner analogous to that with parathion according to Averell and Norris (7).

In Figure 2 are shown the spectral curves for chlorthion recovered from fortified cottonseed and for a typical control blank. For chlorthion, the ab-

sorption maximum is at 545 $m\mu$; 5-cm. cells were used. Control blanks are faintly yellow with absorption at 545 $m\mu$

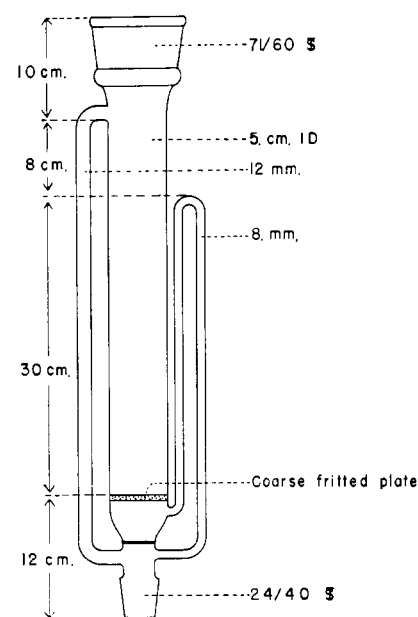


Figure 1. Apparatus for continuous extraction of cottonseed

equivalent to 3 to 4 γ of chlorthion (0.02 p.p.m.). This blank is nearly independent of the quantity of cottonseed control sample used, as illustrated in Table I. In this investigation, however, the control samples were equal in weight to the treated.

In Figure 3 are shown the absorbance-concentration plots for pure chlorthion and for chlorthion recovered from fortified cottonseed. Four micrograms of chlorthion from 200 grams of fortified cottonseed in a final volume of colored solution of 50 ml. were easily detected with the naked eye and possessed a transmittancy reading of 90% when 5-cm. cells were used. In visual comparison with Nessler tubes filled to 25-cm. depth, 0.01 p.p.m. could be detected. There was no indication of the presence of chlorthion in the treated cottonseed.

Field Treatment

A small planting of cotton, variety Acala 4-42, on Citrus Experiment Station property was used for this chlorthion residue test. The cotton was planted in five small blocks, each eight