	JOHN FRANCIS VESECKY	for the	M.S.				
	(Name)		(Degree)				
in	FARM CROPS	presented on	July 22, 1968				
	(Major)		(Date)				
Title:	INFLUENCE OF SOIL MOISTURE	ON MOVEMENT OF	TERBACIL IN SOIL				
		1	•				
Abstract approved: Redacted for privacy							
	D:	r. Arnold P& App	pleby				

Previous field research with terbacil (3-<u>tert</u>-butyl-5-chloro-6-methyluracil) has shown that early fall applications to dry soil performed better than did late fall or early winter applications to wet soil. Field and greenhouse studies were conducted to determine the effect of soil moisture content at time of application and the effect of moisture applied after herbicide application on the leaching of terbacil.

A field trial was initiated during the summer in which terbacil was applied to saturated and dry soil and allowed to dry one week before sprinkler irrigation. Terbacil was also applied to saturated and dry soil which was irrigated immediately. Three-and-one-half inches of irrigation water were applied. Of the three test species, rape (<u>Brassica napus</u> L.) was very sensitive, oats (<u>Avena sativa</u> L.) was moderately sensitive, and corn (<u>Zea mays</u> L.) was relatively tolerant to terbacil. The field bioassay showed that terbacil was the least effective when applied to dry soil with a one-week interim before irrigation. Herbicidal activity was noticeably better when terbacil was applied to wet soil or to dry soil if irrigated immediately. Bioassays of core samples taken from the plots indicated that terbacil sprayed on dry soil one week before irrigation was the least subject to leaching. Terbacil applied to dry soil and irrigated immediately appeared to leach the greatest distance.

Leaching studies in the greenhouse were conducted with soil columns consisting of two layers of soil. The bottom nine-inch layer was saturated and the top two-inch layer, to which terbacil was applied, was either saturated or air dry. Ten inches of water were added immediately, three days, and seven days after application of the herbicide. Bioassay tests showed that terbacil leached the greatest distance when water was applied immediately. When water was applied seven days after terbacil applications, slightly greater leaching of terbacil occurred when applied to wet soil than to dry soil. Analysis of the leachates by gas-liquid chromatography showed that less terbacil was leached through columns in which water was applied three days after application compared to those receiving water immediately after application. However, the speed with which water moved through the columns was quite variable.

Analysis of leachates from soil columns in which the entire soil mass was at one moisture level, saturated or air dry, showed that more terbacil leached through the wet soil than the dry soil when ten inches of water was applied. Again, less terbacil was found in the leachates from columns leached three days after terbacil application compared to those leached immediately. Core samples were taken in April from a field trial in which terbacil had been applied in October and December. The October treatments had given excellent control of winter-germinating weeds while the December treatments failed to give satisfactory control at any rate. Bioassays of the core samples showed no marked differences in depth of leaching of the terbacil from the two dates of application. These results would indicate that the large differences in weed control that have consistently been observed between early fall and winter applications of terbacil are not due to differences in depth of leaching.

# INFLUENCE OF SOIL MOISTURE ON MOVEMENT OF TERBACIL IN SOIL

by

JOHN FRANCIS VESECKY

A THESIS

## submitted to

OREGON STATE UNIVERSITY

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

June 1969

APPROVED:

# **Redacted for privacy**

Associate Professor of Agronomy // in charge of major

# **Redacted for privacy**

Head of Department of Farm Crops

# **Redacted for privacy**

Dean of Graduate School

Date thesis is presented \_\_\_\_\_July 22, 1968\_\_\_\_\_

Typed by Judith A. Gariepy

## ACKNOWLEDGEMENTS

I wish to express my appreciation to Dr. Arnold P. Appleby for his guidance and assistance throughout my graduate program and in the preparation of this manuscript.

I am grateful to Dr. Orvid Lee and Mrs. La Rea Johnston for serving on my committee and reviewing the manuscript.

My appreciation is expressed to Dr. Robert L. Zimdahl and Eugene Johnson for their assistance with the chemical analyses.

# TABLE OF CONTENTS

Chapte	<u>r</u>	Page
I.	INTRODUCTION	1
II.	LITERATURE REVIEW	3
	Properties of Terbacil Mobility of Herbicides in Soil Adsorption Adsorption Competition	3 4 7 10
III.	FIELD LEACHING AND GREENHOUSE BIOASSAY STUDY	12
	Methods and Materials Results	12 14
IV.	TIMING TRIAL - GREENHOUSE BIOASSAY	20
	Methods and Materials Results	20 22
V.	GREENHOUSE LEACHING EXPERIMENT I	26
	Methods and Materials Results	26 29
VI.	GREENHOUSE LEACHING EXPERIMENT II	36
	Methods and Materials Results	36 37
VII.	GREENHOUSE LEACHING EXPERIMENT III	39
	Methods and Materials Results	39 40
VIII.	DISCUSSION AND CONCLUSIONS	43
IX.	SUMMARY	46
Х.	BIBLIOGRAPHY	4.8
XI.	APPENDIX	51

# LIST OF TABLES

Table		Page
1.	Chemical and mechanical analysis of soil from experimental area.	12
2.	Cumulative inches of rainfall after October 9 and December 12 applications.	21
3.	Chemical and mechanical analysis of Woodburn silt loam used in soil columns.	27
4.	Chemical and mechanical analysis of sandy loam soil used in soil columns.	39

# LIST OF FIGURES

Figure		Page
1.	Visual evaluations of stand reduction in field bioassay.	17
2.	Dry weights measured as percent of check from bioassay of field core samples, 0.8 lb/acre rate.	18
3.	Dry weights measured as percent of check from bioassay of field core samples, 1.2 lb/acre rate.	19
4.	Dry weights as percent of check of oats foliage harvested from core samples taken from an established peppermint trial. Core samples were taken April 27.	24
5.	Oats grown in core samples taken from established peppermint trial. Samples were taken on April 27.	25
6.	Dry weight of plant material grown in soil columns leached immediately after terbacil application.	31
7.	Dry weight of plant material grown in soil columns leached three days after terbacil application.	32
8.	Dry weight of plant material grown in soil columns dried thoroughly and leached seven days after terbacil application.	33
9.	Dry weight of plant material grown in soil columns covered and leached seven days after application.	34
10.	Terbacil recovered from leachate. Average of two replications.	35
11.	Terbacil recovered from leachate. Average of three observations.	38
12.	Terbacil recovered from leachate. Average of three observations.	42

## LIST OF APPENDIX TABLES

Table		Page
1.	Influence of moisture, both before and after terbacil application, on growth of bioassay plants in the field.	51
2.	Influence of irrigation on the depth of leaching of terbacil applied at two rates to dry and saturated soil. Measured in milligrams of dry plant weight using oats as the bioassay plant.	52
3.	Analysis of variance table for data in Table 2.	54
4.	Dry weight of oat plants grown in core samples taken from WSS, WSW, DSS, and DSW treatments. Measured as percent of check.	55
5.	Milligrams of dry plant material and injury ratings of oat plants grown in core samples taken from an established peppermint trial.	56
6.	Analysis of variance table for data in Table 5.	57
7.	Milligrams of dry plant material harvested from soil columns. Greenhouse Leaching Experiment I.	58
8.	Analysis of variance table for data in Table 7.	61
9.	Micrograms of terbacil recovered from greenhouse leaching experiment I leachate.	62
10.	Analysis of variance table for data in Table 9.	62
11.	Micrograms of terbacil recovered from greenhouse leaching experiment II leachate.	63
12.	Analysis of variance table for data in Table ll.	63
13.	Micrograms of terbacil recovered from greenhouse leaching experiment III leachate.	64

<u>Table</u>

14.	Analysis	of	variance	table	for	data	in		
	Table 13	,							64

Page

INFLUENCE OF SOIL MOISTURE ON MOVEMENT OF TERBACIL IN SOIL

#### I. INTRODUCTION

Today's agriculture demands more information regarding the persistence and disappearance of herbicides from the soil. Such information is vitally important for at least two reasons. First, with the increasing use of soil-applied herbicides such information is essential in determining whether or not a herbicide will remain active in the soil long enough to give satisfactory weed control; and second, phytotoxic residues must disappear rapidly enough to prevent injury to sensitive crops which might follow.

Disappearance of herbicides from the soil can occur in several ways. These are: leaching, microbiological breakdown, volatilization, chemical breakdown, photodecomposition, and plant uptake. In any given situation, the persistence of a herbicide is governed by one or more of these variables.

It has been found that 3-<u>tert</u>-butyl-5-chloro-6-methyluracil (terbacil), a soil-active herbicide, shows promise of selectively controlling a wide spectrum of annual herbaceous broadleaf weeds and weed grasses in a variety of crops. Terbacil is also effective in controlling such perennial weeds as quackgrass (<u>Agropyron repens</u> (L.) Beauv.), nutsedge (<u>Cyperus</u> spp.), and johnsongrass (<u>Sorghum halepense</u> (L.) Pers.).

Under western Oregon conditions terbacil and 5-bromo-3-<u>sec</u>butyl-6-methyluracil (bromacil), a closely related herbicide, have given better weed control when applied in the early fall than when applied in the late fall or winter. Climatic conditions in the Willamette Valley are of a modified-marine type influenced by coastal weather conditions. Annual rainfall averages about 40 inches. About 70 percent of this precipitation occurs during the five months from November through March. Only about five percent of the rainfall occurs during the three summer months. It is speculated that the reduced herbicidal activity from the late fall and early winter applications may have been caused by increased leaching when the herbicides were applied to moist soil and followed by steady rainfall which is common in the Willamette Valley during December and January. Herbicidal activity was good when the herbicides were applied to dry soil or to damp soil which was allowed to dry before the heavy winter rains began.

The objective of this study was to determine the relationship between the soil moisture content at the time of herbicide application and the effect of subsequent moisture applied after application upon the leaching of terbacil.

## II. LITERATURE REVIEW

#### Properties of Terbacil

Terbacil (3-<u>tert</u>-butyl-5-chloro-6-methyluracil) belongs to a family of herbicides known as substituted uracils and is formulated as an 80 percent wettable powder. The structural formula is:



The Technical Data Sheet of E. I. duPont, deNemours and Company (1967) stated that the pure chemical is an odorless white crystalline solid with a melting point of 175-177°C. It is stable in water and common organic solvents at room temperature and is subject to microbial decomposition under moist soil conditions. Its water solubility is 710 ppm at 25°C and its specific gravity is 1.34.

The LD<sub>50</sub> for rats was found to be between 5000 and 7500 mg/kg body weight. Exposing the skin of guinea pigs to terbacil produced no skin irritation or sensitization. Extensive amounts of terbacil produced no clinical signs of toxicity on rabbits through skin absorption.

Terbacil was the first of the substituted uracil family to be used for selective weed control in U.S. crops. DuPont (1967) has found the compound to be highly effective in orchards against hard-to-kill perennial weeds such as johnsongrass, bermudagrass (Cynodon dactylon (L.) Pers.), quackgrass, and nutsedge. Terbacil is

finding wide use in Oregon on peppermint, a crop which will tolerate very high rates without injury.

Terbacil appears to control weeds by interfering with the plant's photosynthetic process. Hilton, <u>et al</u>. (1964) noted that when applied to isolated chloroplasts, all of the substituted uracils studied were strong inhibitors of the Hill reaction. The substituted uracils enter plants through the roots and are translocated to other parts of the plant. Weed control is best when the chemical is applied just before or soon after weed emergence. Rainfall or sprinkler irrigation is required after application to move the herbicide into the surface soil where the weed seeds are germinating. Compared to many other herbicides, the timing of rainfall or irrigation is less critical because terbacil is relatively resistant to volatilization and photodecomposition.

#### Mobility of Herbicides in Soil

The mobility of herbicides in the soil greatly influences herbicidal activity. Three factors usually recognized as having notable influence on herbicide mobility in soils are soil type, organic matter content, and the amount of water that passes through the soil.

Herbicides have been observed to move more readily in light (sandy) soils than in heavier (clay and muck) soils (Burnside, Fenster and Wicks, 1963; Danielson, 1956; Ogle and Warren, 1954; Upchurch and Pierce, 1957, 1958; Dickens and Hiltbold, 1967; and Dubey and Freeman, 1965). Price and Fisher (1967), working with two orchard soils in Delaware, found terbacil to leach to greater depths in a sandy loam soil than in a silt loam soil. Upon bioassay of core samples, it was found that more herbicide moved out of the 0-2 inch layer with higher precipitation. The differences due to precipitation level were greater on the silt loam soil.

Lambert, Porter and Schieferstein (1965), working with a slotted tube test for evaluating the leaching of herbicides, found that soil organic matter greatly influenced movement of the herbicides studied. Rainfall studies by Burnside, Wicks and Fenster (1963) in Nebraska showed that 2,3,6-trichlorobenzoic acid (2,3,6-TBA) leached to greater depths in a clay loam soil at Lincoln than in a loam soil at North Platte and Alliance. Organic matter content of the three soils was 3.0, 2.6, and 2.2 percent, respectively. About twice as much rainfall at Lincoln as at North Platte and Alliance was responsible for greater leaching in the heavier soil. Soil samples from different depths indicated that 2,3,6-TBA leached to four or five feet when rainfall was sufficient.

Upchurch and Pierce (1957) reported a direct relationship between the amount of water applied and the amount of 3-(p-chlorophenyl)-1,ldimethylurea (monuron) leached from the surface two inches of soil. When monuron was applied at 40 lbs/acre, each inch of water removed approximately 5.7 lbs/acre of monuron until 30 lbs/acre, or 75 percent, were removed from the top two inches of soil. Four inches of simulated rainfall applied in eight increments at one-half hour intervals leached eight percent of the 40 lbs/acre application below 24 inches. Fifty-one percent was leached below 24 inches when 12

inches of water was applied in a similar manner. Logan, Odell and Freed (1953) indicated a possible linear relationship between the movement of isopropyl N-phenylcarbamate (IPC) in a sandy loam soil and the amount of simulated rainfall applied. They, like Upchurch and Pierce (1957), suggested a possible relationship between the depth of herbicide penetration and the interval of time allowed for leaching. Friesen (1965) reported deeper penetration of 2-methoxy-3,6dichlorobenzoic acid (dicamba) with increasing amounts of leaching water. According to Ogle and Warren (1954) 16 inches of water removed all toxic proportions of monuron from the surface of a silt clay loam soil. Eight inches of water removed only about 50 percent of the toxic monuron from the surface. Trichloroacetic acid (TCA) and 2,4-dichlorophenoxyacetic acid (2,4-D) followed a similar pattern except TCA required four inches of water and 2,4-D required eight inches of water for complete removal of all toxic material from the surface. Of the three compounds TCA is the most soluble and monuron the least.

Soil moisture content at time of herbicide application greatly influences the leaching of some herbicides. Wiese and Davis (1964), using several herbicides, reported a general tendency for herbicides to move deeper when applied to wet soil than when applied to dry soil. Opposite results were obtained by Sherburne, Freed and Fang (1956) with monuron and Geissbühler, Haselbach and Aebi (1963) with 3-(3,4-dichlorophenyl)-1-methyl,1 butylurea (neburon). They found that as the amount of moisture in the soil at the time of application increased, the depth to which the herbicide leached decreased.

Sherburne, <u>et al</u>. (1956) attributed this result to slower movement of water through the more moist soil. However, Merkle, Bovey and Davis (1967) found that one inch of simulated rainfall moved 4-amino-3,5,6trichloropicolinic acid (picloram) faster through soils which had been pre-moistened to field capacity than through soil which had been air dry. From the work of Upchurch and Pierce (1958) it appeared that soil moisture content had little or no influence on the leaching of monuron from Lakeland sand soil. The higher moisture level (6 percent or field capacity) retained 34 percent of the monuron applied in the 0-2 inch layer, compared to 27 percent for the dry treatment (0.3 percent or air dry).

Work by Hurtt, Meade and Santelmann (1958) suggested that soil type in combination with different moisture levels influences leaching. Isopropyl N-(3-chlorophenyl)carbamate (CIPC) moved deeper in air dry sandy loam soil than in moist sandy loam soil. When clay soil was used, CIPC penetrated deeper in moist soil than in dry.

### Adsorption

At one time it was believed that a compound's water solubility was the key to leachability. Although solubility is important, it has been dismissed by many investigators as an explanation for a relatively low rate of leaching. It is now known that some relatively insoluble compounds, such as monuron, leach more readily than 9,10-dihydro-8a,10a-diazoniaphenanthrene-2A (diquat), which is very soluble in water. Many workers now believe that adsorption of herbicides on the enormous surface area provided by the soil matrix

is more important than water solubility in influencing leachability. Relationship between solubility and leachability is usually restricted to compounds within a class and is frequently due to a relationship between adsorption and solubility within that class of compounds (Upchurch, 1966). Therefore, leachability may be measured better by adsorption than by solubility.

The phenomenon of adsorption has been used by many investigators to explain erratic results in herbicidal activity. Sorption of a compound by a soil colloid is complex because of the attraction and repulsion between the colloid and, in this case, water and the herbicide in solution or in the vapor phase. Bailey and White (1964) refer to adsorption of two general types: physical adsorption and chemical adsorption. Physical adsorption of compounds by soil surfaces is accomplished by van der Waals forces and hydrogen bonding. Chemical adsorption involves the actual formation of ionic bonds between adsorbent and adsorbate.

Generally speaking, physical adsorption results in low bonding strength, while chemical adsorption results in a greater bonding strength. Physical adsorption may give rise to several monolayers, whereas in chemical adsorption only the first layer is chemically bonded to the surface although several more monolayers may be held by hydrogen bonding. Freed, Vernetti and Montgomery (1967) confirmed a correlation between the heat of solubility and the bonding strength of many herbicides. Usually, as the heat of solution increases, so does adsorption. Ethyl N,N-dipropylthiocarbamate (EPTC), with a heat of solution of -3.9 Kcal is 52 percent adsorbed at 3<sup>o</sup>C, whereas

CIPC (heat of solution = +4.9 Kcal) is 71 percent adsorbed at 3°C.

Soil organic matter content appears to be highly and positively correlated with adsorption of many herbicides (Bailey and White, 1964 and Ward and Upchurch, 1965). This positive correlation usually results in reduced herbicidal activity. Upchurch and Mason (1962) found that the amount of herbicide required to cause a 50 percent reduction in dry shoot weight of cotton varied directly with the organic matter content of the soil. Approximately five times as much herbicide was required for a 50 percent kill on soil containing 20 percent organic matter as compared to the amount required for a soil with four percent organic matter.

Herbicides and clay particles have little attraction for each other (Hartley, 1964). However; due to the enormous surface area of clay particles available in the soil, in relation to the low density of herbicide molecules usually present, clays are very important as herbicide adsorbants. Theoretically, a soil consisting entirely of clay particles would have only 0.001 percent or less of the surface occupied by herbicide molecules if the herbicide was applied at a concentration of practical interest, and if all molecules were adsorbed.

Adsorption is a reversible process if the same final distribution between soil and water is obtained without regard to the initial distribution (Hartley, 1964). Reversible adsorption was obtained when samples which had been stored at 0°C were heated to 50°C (Harris and Warren, 1964). Herbicides adsorbed as a result of low temperature were completely released upon heating. Temperature exerts

an indirect influence on adsorption through its effect on solubility. With few exceptions these two factors work together; that is, both lead to decreased adsorption as temperature rises.

# Adsorption Competition

Moisture content of the soil appears to influence both the degree of adsorption and the bioactivity of herbicides. It has been recognized that water and some herbicides may compete for adsorption sites in the soil. Deming (1963) reported that increasing temperature and lowering soil moisture levels retarded the loss of 2-chloro-N,Ndiallylacetamide (CDAA) because more sites were made available for adsorption by increased moisture loss. As soil moisture content and temperature increased, so did CDAA loss. This was assumed to be due to fewer available adsorption sites as well as the direct effect of temperature on the vapor pressure of CDAA causing increased loss. Results similar to Deming's were obtained by Parochetti and Warren (1966) with IPC and Fang, Theisen and Freed (1961) with EPTC. Vapor loss of these compounds was less from air dry soils than from wet soils. The lower vapor loss from the dry soil was credited to greater adsorption of the herbicide molecules by the dry soil surface.

Hance (1965) demonstrated competition between 3-(3,4dichlorophenyl)-1,1-dimethylurea (diuron) and water for adsorption sites. Diuron is a stronger competitor with water at soil organic matter surfaces than at soil mineral surfaces. Therefore, when water is present, organic matter is the most important adsorption site for diuron. Upchurch (1957) found diuron to be more toxic to cotton under wet soil conditions than under dry conditions. This indicated the possibility of more adsorption of diuron by the soil surface under dry conditions. Diuron has a relatively low vapor pressure and may be desorbed in the presence of water, thereby increasing the concentration in soil solution to a toxic level.

Water is a very polar substance and is strongly adsorbed to many surfaces. As moisture level in the soil decreases, fewer water molecules are present to compete with the less polar organic molecules for adsorption sites on the soil surface. If the organic molecules have been adsorbed under low moisture conditions, they may be displaced by water and made biologically active.

#### III. FIELD LEACHING AND GREENHOUSE BIOASSAY STUDY

#### Methods and Materials

A field trial was established July, 1967, to determine the relationship between the soil moisture content at time of application and subsequent overhead irrigation upon the leaching of terbacil. Four main treatments were used: (1) terbacil sprayed on saturated soil and allowed to dry for one week before sprinkling (WSW); (2) terbacil sprayed on dry soil and allowed to dry one week before sprinkling (DSW); (3) terbacil sprayed on saturated soil and sprinkled immediately (WSS); and (4) terbacil sprayed on dry soil and sprinkled immediately (DSS). A split-plot design with four replications was used. Main plots consisted of two moisture levels at time of application--saturated and dry. Rates of 0.8 and 1.2 pounds of active ingredient per acre made up the sub-plots. Main plots were 10 x 13 feet and sub-plots were 5 x 10 feet with three-foot bordered alleys separating sub-plots to facilitate irrigation. The sub-plots were sprayed by means of a back-pack compressed-air plot sprayer.

Table 1. Chemical and mechanical analysis of soil from experimental area.

Soil	CEC	%	% sand	% silt	% clay
pH	me/100g	0.M.	>0.05mm	0.05-0.002mm	<0.002mm
6	17.11	1.74	50.12	32.33	17.55

The trial area had been tilled previously. Four-inch-high dikes were constructed around the sub-plots with disks mounted at an angle on the cultivator shanks of an International Cub tractor. In order to use regular field sprinkler irrigation, it was necessary to make two herbicide applications one week apart.

On July 19 one main plot in each replication was flooded until about four inches of water was standing on the surface. As soon as all water disappeared from the surface, the plots were sprayed. Terbacil was applied also to dry soil on an additional main plot in each replication. No irrigation was applied immediately to these plots. One week later terbacil was applied to two more main plots in each replication using the same procedure as before. Upon completion of spraying, all plots were irrigated by sprinkler. The irrigation water was turned on within 20 minutes after the last herbicide application. A total of three-and-one-half inches of water was applied.

Core samples one inch in diameter and 12 inches deep were taken on September 6 and 7, 1967. Each sample was divided into two-inch segments. Six core samples were removed from each sub-plot in order to obtain enough soil for bioassay purposes. All samples from each depth within each sub-plot were bulked, ground with a soil grinder, and thoroughly mixed before placing into 2.75 x 2.75 x 2.75 inch plastic pots for bioassay in the greenhouse. Because of shortage of greenhouse space all four replications were not planted at the same time. Six oat seeds were planted 0.5 inches deep in each pot. Seedlings were later thinned to five plants per pot. The pots from each replication were randomized in watering trays and watered by sub-irrigation. Pots within a watering tray were rotated every few days to obtain more uniformity. Temperature in the greenhouse ranged from 65 degrees at night to 75-85 degrees during the day with higher temperatures on days with bright sunshine. All oat plants were harvested one month after planting and dry weights were determined.

After the core samples were taken, the borders separating sub-plots were leveled and the entire trial was irrigated with two inches of water since no significant amount of rain had fallen since the trial was established. On September 15, three test species--rape (<u>Brassica napus</u> L.), oats (<u>Avena sativa</u> L.), and corn (<u>Zea mays</u> L.)-were planted across the treatments. Greenhouse persistence studies had shown rape to be sensitive, oats moderately sensitive, and corn to be relatively tolerant to terbacil. The trial was irrigated as needed until the fall rains started. After four weeks, visual observations of stand density were made. Treated plots were compared to check plots which were assigned ratings of zero percent reduction of stand density. Evaluations were made by three persons and average ratings for each treatment were calculated.

## Results

Of the three test species used in the field bioassay, rape was by far the most sensitive to terbacil. All rape plants in all treatments were killed within one week after emergence. Corn plants were much more tolerant and were very slow to show differences between treatments. Oat plants were moderately tolerant to terbacil and

produced the most informative results. Both oats and corn showed the same general trend. Visual evaluation ratings remained relatively constant (8.35 percent maximum range for single rate application) for treatments WSS, WSW, and DSS (Figure 1). Considerably more plant material was noticed on the DSW-treated plots. This may indicate that most of the terbacil was held in the very top layer of soil following treatment DSW. Seeding of these two crops, oats and corn, in the DSWtreated plots may have resulted in the seed being placed in the soil below the zone of maximum terbacil concentration and thereby escaping damage as severe as in the other treatments. Averages of the three original evaluations are in the Appendix, Table 1.

Oat plants were used as the test species in the greenhouse because of its moderate sensitivity. Figures 2 and 3 indicate that when treatments WSW, WSS, and DSS were applied a large amount of the terbacil remained in the top two-inch layer of soil. Less terbacil toxicity was observed in the top two-inch layer following treatment DSW. A relatively large amount of terbacil was also found in the two- to four-inch layer following treatments WSW, WSS, and DSS. Again the DSW treatment showed much less toxicity. Terbacil may have been located at the soil surface and adsorbed to the soil colloids so tightly that irrigation water or plants could not remove it. Terbacil appeared to leach to the greatest depth after the DSS treatment when applied at 1.2 lb/acre.

In several cases a percent of check greater than 100 was noticed, especially at the lower depths. This is more pronounced where the 0.8 lb/acre rate was applied than where 1.2 lb/acre of terbacil

was applied. These results would seem to indicate that there is a stimulatory effect from very minute amounts of terbacil. If this is the case, it appears that the amount of terbacil leached to the lower depths from the 1.2 lb/acre application was usually in large enough amounts to be inhibitory rather than stimulatory with the exception of the DSW treatment.



Spraying Procedure

Figure 1. Visual evaluations of stand reduction in field bioassay.

**∆**\_\_\_\_**∆** 0.8 lb/acre

0---

-O 1.2 lb/acre



Depth of core sample in inches



Depth of core samples in inches

## IV. TIMING TRIAL - GREENHOUSE BIOASSAY

#### Methods and Materials

Considering the data obtained from leaching and bioassay experiments, it was of interest to obtain more information on the movement of terbacil from fall and winter applications in the field. To obtain this information, core samples were taken on April 27, 1968, from a timing trial started in the fall of 1967 on established peppermint. The experiment was arranged as a randomized block design with three replications. Individual plots were 10 x 30 feet. Terbacil was applied at two rates, 0.8 and 1.6 pounds active ingredient per acre, and on two dates, October 9 and December 12. Moisture content of the soil was 26 percent at the time of the October application and 30.5 percent at the December date. Rainfall following both dates of application is recorded in Table 2. October applications have usually resulted in good weed control in contrast to considerably less weed control when terbacil was applied in December. Visual evaluations were made on June 4 for control of winter- and spring-germinated weeds. The October-applied treatments gave 100 percent weed control whereas the December treatments averaged only 53 percent for the low rate and 58 percent for the high rate.

		Days	after appli	cation	
Date	1	2	3	7	30
October 9	0.0	0.0	0.1	0.31	3.69
December 12	0.04	0.04	0.04	0.67	5.53

Table 2. Cumulative inches of rainfall after October 9 and December 12 applications.

Rainfall amounting to 12.13 inches fell between the October application and the December application. A total of 34.69 inches of rain fell between the October application and the date of core sampling. Rainfall between the December application and the date of core sampling was 22.56 inches or about 65 percent as much as between the October date and sampling.

Four core samples three inches in diameter and 12 inches deep were taken at random from each plot. The core sampler was a hydraulic apparatus mounted on a pickup. Cores were divided into segments of 0-0.5, 0.5-1, 1-2, 2-3, 3-6, and 6-12 inches in depth. All samples from each depth within each plot were bulked, sieved through a 0.1875-inch screen and thoroughly mixed. They were then transferred to 2.75 x 2.75 x 2.75 inch plastic pots. Eight oat seeds were planted 0.5 inches deep in each pot on May 1 and thinned to the most uniform six plants one week after emergence. Pots were placed at random within watering trays and watered by sub-irrigation. Pots were rearranged every three or four days to obtain more uniformity. Three weeks after planting, visual observations for injury were made, plants were harvested, and dry weights were recorded.

#### Results

Dry weights of the oat plants are recorded in Table 5 in the Appendix and plotted as percent of check in Figure 4. Figures 4 and 5 show that date and rate of application had essentially no effect on the toxicity of terbacil in the 0-0.5 inch layer of soil. Almost all plants in the 0-0.5 inch layer were dead. A majority of the plants were also dead in the 0.5-1 and 1-2 inch layers following the October application at 1.6 lb/acre and both December treatments. The October 0.8 lb/acre application resulted in fewer dead plants in the 0.5-1 and 1-2 inch soil layers; however, differences were minor.

The 0.8 lb/acre rate applied at different times showed only slight differences in leaching patterns. When 0.8 lb/acre of terbacil was applied in October, it appeared to remain concentrated in the 0-0.5 inch layer and gradually decreased with depth. Terbacil applied at 0.8 lb/acre in December showed more activity in the 1-2 inch layer than did the October application. Herbicidal activity in the 2-3 inch layer was much less from both application dates with no injury observed below three inches.

The 1.6 lb/acre rate reduced the dry plant material at the 2-3 inch level more than did the 0.8 lb/acre rate. Apparently since more terbacil was applied, more was available for leaching. Terbacil applied in October at the 1.6 lb/acre rate caused more injury and reduction of plant weight in the 3-6 inch level than did the December application. The additional leaching, compared to the December application at the 1.6 lb/acre rate, may have been caused partially by the extra 12.13 inches of rainfall received by the October application.





Core samples were taken on April 27. Dry weights as per from core samples taken from an established peppermint trial. foliage harvested





Figure 5. Oats grown in core samples taken from established peppermint trial. Samples were taken on April 27.
### V. GREENHOUSE LEACHING EXPERIMENT I

## Methods and Materials

An experiment was initiated in the greenhouse involving leaching in soil columns to simulate late fall and early winter field conditions. Leachates were collected to determine the percent of applied terbacil which was coming through the column of soil. The study consisted of one rate of application, one quantity of leaching water, two moisture levels in the surface two inches of soil at time of terbacil application, and three dates of terbacil application. Treatments were replicated five times, but because of malfunctioning of a timeclock only three replications could be used. Of the three replications, one was rendered useless in the laboratory so only two were analyzed by gas-liquid chromatography (GLC).

Paper tubes measuring four inches, inside diameter, by 14 inches long with a wall thickness of 0.1875 inches were used. The tubes were split lengthwise on a band saw to produce two halves. The matching halves were taped together to form an intact tube. The tubes were rotated in hot paraffin to receive a thin waterproof coating. Fiberglass screen held in place with rubber bands was used as bottoms. The tubes were filled with air-dry soil (approximately three percent moisture content) which had been sieved through a 0.25-inch screen. Chemical and mechanical analysis of soil used is found in Table 3. A thin-walled metal tube three inches in diameter with a funnel top was constructed to use in filling the tubes. The metal tube was filled with soil and the soil transferred to form a soil column in such a way

as to have a minimum of free-falling soil. Packing of the soil was accomplished by dropping the column from a height of one inch 20 times. This procedure produced columns with approximately nine inches of packed soil. Moisture content of the top two inches of soil was controlled by applying a weighed amount of air-dry soil to the top of each column. The columns had previously been saturated and allowed to drain for 48 hours. One-half inch of 20 mesh white sand was placed between the bottom nine inches of soil and the top two inches to break capillary movement of water from the soil at field capacity in the bottom of the column to the air-dry top layer. To produce field capacity moisture conditions in the top layer of soil, a measured amount of water was added to the soil surface of the desired columns. It was hoped that saturating the lower soil layer would provide more uniform leaching with application of equal amounts of water.

Table 3. Chemical and mechanical analysis of Woodburn silt loam used in soil columns.

Soil	CEC	%	% sand	% silt	% clay
pH	me/100g	0.M.	>0.05mm	0.05-0.002mm	< 0.002mm
5.2	15.3	3.32	13.92	50.15	35.93

Terbacil was sprayed on the surface at the rate of 1.2 pounds active ingredient per acre by means of a greenhouse sprayer calibrated to deliver 85 gallons of water per acre with one pass over the columns. A layer of sand was placed on top of each column to prevent puddling during leaching. Columns were leached immediately after spraying (O-day), three days after spraying (3-day), and seven days after spraying with terbacil. Columns leached seven days after spraying were divided into two groups--seven-day-covered (7-D-C) and seven-day-uncovered (7-D-U). Seven-day-covered columns were covered with plastic bags to prevent evaporation until leaching occurred; columns labeled 7-D-U were placed in the path of circulating air to dry the soil.

Leaching was accomplished by placing the columns on racks in gallon cans on a turntable which rotated at seven r.p.m. under a spray nozzle adjusted to deliver one inch of water per hour. The turntable and spray were regulated by a timeclock to come on for 15 minutes every four hours until a total of 10 inches of water had been applied. Plastic bags with both ends open were placed around each can and tube and fastened at the top with a rubber band to prevent spray water from entering the cans. Leachate from the columns was collected in the cans and drained into glass jars where it was concentrated to about 100 ml before being analyzed for terbacil with GLC.

To provide for uniform extraction, all concentrated leachates were made up to 200 ml with tap water and extracted three times with 30-ml aliquots of benzene. The three combined extracts (90 ml) from each leachate were passed through a filter paper on which anhydrous sodium sulfate granular reagent was held to collect any water which may have been in the extract. Extracts were then concentrated to five ml for analysis of terbacil by GLC.

A model C-100 Dohrmann microcoulometric gas chromatograph equipped with a T-200-S halide sensitive titration cell was used. An

oven temperature of 185°C and a nitrogen gas flow rate of 120 cc per minute with a resistance of 32 ohms gave a retention time of 120 seconds. Twenty microliters of a standard which contained 100 micrograms of technical terbacil per ml was injected. The amount of terbacil present in the unknown samples was determined by comparing integrator strokes of standards with the unknown samples. Three terbacil recovery studies yielded results ranging from 100 to 104 percent with an average of 101.9 percent.

Three days after the last application of water the soil columns were split with a knife where the two halves of the paper tubes were taped together. The halves were then placed in flats for planting. Three rows of oat seeds were planted in each column flush with the soil surface with brush end up. Seeds were 0.5 inches apart in the rows. Dividers cut to fit the column halves were inserted at one-inch intervals throughout the columns to prevent lateral movement of roots. The soil surface was covered with 0.25 inches of sand to prevent rapid drying. Irrigation was from the surface. Dry weight of plants from each depth was determined one month after planting.

# Results

Results of the bioassay showed that the time interval between terbacil application and leaching had some effect on the pattern of leaching. All columns retained enough terbacil in the 0-1 inch layer to effectively kill the oat plants. Columns leached immediately after terbacil application reduced the dry plant material at lower depths more than did columns leached three and seven days after application

(Figures 6-9). This may indicate that some period of time is required for the terbacil to be adsorbed by the soil surface.

Leachates analyzed by GLC are for only two replications and are quite variable. More terbacil was recovered from the leachate of columns where application was to air-dry soil than to wet soil (Figure 10). Total terbacil recovered was higher from leachate of columns which were leached immediately than from any other columns. This same treatment also killed the bioassay plants to the greatest depth in the soil column. This again tends to indicate the need of a waiting period for terbacil to react with the soil in some manner to prevent leaching.



Figure 6. Dry weight of plant material grown in soil columns leached immediately after terbacil application.



Figure 7. Dry weight of plant material grown in soil columns leached three days after terbacil application.



amsigilim

Figure 8. Dry weight of plant material grown in soil columns dried thoroughly and leached seven days after terbacil application.



Figure 9. Dry weight of plant material grown in soil columns covered and leached seven days after application.



• Air dry top layer

A-

A Top layer at field capacity

Treatment

Figure 10. Terbacil recovered from leachate. Average of two replications.

#### VI. GREENHOUSE LEACHING EXPERIMENT II

## Methods and Materials

Certain modifications in technique were made following Experiment I. New tubes were made of four-inch diameter black plastic sewer pipe cut into two-inch lengths. Sections were sealed with lanolin and taped with a waterproof tape to make a tube 14 inches long. Fiberglass screen was used as bottoms. The filling and packing procedure was the same as in the preceding experiment. This produced columns with approximately 12 inches of soil which was saturated after packing. Soil used was from the same lot as used in Experiment I.

Controlling soil moisture content in the top two inches was accomplished by filling 24 two-inch sections of pipe with air-dry soil. Sections were placed on a board in groups of six per board for ease of handling and timing of spraying. Terbacil was applied at the same rate as before. Sections to be placed on columns leached seven days after spraying were sprayed first, the covering procedure was the same as in Experiment I. Four days later the 3-day sections were sprayed and on the seventh day the 0-day sections were sprayed. This staggered spraying procedure made it possible to leach all of the columns simultaneously and to conduct the experiment in a completely randomized manner. To provide top sections with soil at field capacity, half of the sections to be sprayed within each group were saturated two days before spraying. The soil columns which had previously been saturated were cut and all soil above ten inches was removed. Sprayed top sections were then lifted with a metal spatula and placed on the columns making a column of soil 12 inches long. Another two inch section of pipe was taped on top to act as a reservoir for leaching water.

Columns were put on metal racks over gallon cans to collect the leachate which was analyzed as before. Before leaching, a thin layer of sand was applied to the top of each column to prevent puddling. Leaching was accomplished by adding seven inches of water in one-inch increments at eight-hour intervals.

## Results

The terbacil recovery pattern from this group of columns did not agree with that obtained from Experiment I. The first striking difference was that more terbacil leached through 7-D-U columns which had the top two-inch layer of soil at field capacity (Figure 11). It was also observed that much less terbacil was recovered from the 0-day leachates. This could be because the 0-day top sections for these columns were wetter than the other top sections when placed on the columns. All top sections were placed on the columns at the same time to facilitate simultaneous leaching. This may have caused more settling of the top layer of soil in the 0-day field capacity sections, thereby slowing down the flow rate through this top layer and resulting in more retention of terbacil. In general the flow rate of water through this group of columns was very uneven, which may explain the inconsistency in the results obtained from this experiment.



Figure 11. Terbacil recovered from leachate. Average of three observations.

## VII. GREENHOUSE LEACHING EXPERIMENT III

#### Methods and Materials

After completing two experiments in which most of the soil in each column was at field capacity, it was of interest to conduct an experiment in which the entire soil mass within a column was at the same moisture level. It was also suspected that moving the columns after they were saturated resulted in settling of the wet soil. This in turn may have altered the speed with which percolating water moved through the columns.

To overcome these difficulties, several changes in technique were made. Four-inch black plastic sewer pipe cut into 12-inch lengths was used. A two-inch section of pipe was taped to each 12-inch section to make a total length of 14 inches. Fiberglass screen was again used for bottoms. Pipe sections were filled with 3040 grams of air-dry soil and packed as before. Analysis of soil used is found in Table 4. Covering procedure for the seven-day columns was the same as before. This produced soil columns with approximately 12.25 inches of soil. Columns were then placed on racks over gallon cans with plastic bag liners and remained stationary for the duration of the experiment.

Table 4. Chemical and mechanical analysis of sandy loam soil used in soil columns.

Soil	CEC	%	% sand	% silt	% clay
pH	me/100g	0.M.	>0.05mm	0.05-0.002mm	<0.002mm
5.5	16.1	2.53	33.02	40.54	26.44

Terbacil application was staggered as before to enable simultaneous leaching of all columns. Two days before terbacil application, 900 ml of water were applied to three columns for each treatment. This amount of water moistened the entire soil column without running through into the plastic bags. Terbacil was mixed in acetone and applied at the rate of 12.6 lb active ingredient per acre to each column with a pipette. The acetone was allowed to evaporate before covering the 7-D-C columns.

A layer of sand was applied to the top of each column before leaching. Ten inches of water were applied in one-half inch increments at six-hour intervals. Leachate was collected in the plastic bags and analyzed as before.

# Results

Applying terbacil in soil sterilant quantities resulted in a very small percentage being found in the leachate: 0.6 to 8.8 percent (Table 13). This implies that terbacil is relatively resistant to leaching, even with large amounts of water. Approximately three to eleven times as much terbacil leached through columns containing wet soil as through dry soil. The greatest difference was in the 3-day treatment. This difference may have occurred because four-andone-half inches of the ten inches of water applied to the columns was used to wet the soil in the air-dry soil columns. This was not the case with the columns in which the soil was at field capacity. About ten inches of water came through these columns whereas only about fiveand-one-half inches came through the columns in which the soil was air dry. The difference may also have been due to continuous water columns in the wet soil which would aid in downward movement of water. This would not have been the case with the dry soil.

The amount of terbacil recovered from columns in which the soil was air dry was less if three or more days elapsed between terbacil application and leaching than if leached immediately. This indicates that a waiting period of approximately three days or less is needed before applying water to prevent leaching of terbacil when applied to air-dry soil. Applying terbacil to columns of soil at field capacity required a waiting period of seven days to markedly reduce leaching, provided the columns were uncovered and the top soil allowed to dry thoroughly before applying water. This would indicate that if terbacil was applied to wet soil and did not have the opportunity to dry before a rain fell, it would be more subject to leaching than if allowed to dry.



Treatment

Figure 12. Terbacil recovered from leachate. Average of three observations.

#### VIII. DISCUSSION AND CONCLUSIONS

Observations of the performance of terbacil for the past several years have indicated that poor weed control results when terbacil is applied to wet soil to which water is applied soon after spraying. This study did not confirm those observations. Poor weed control was obtained from irrigating the soil one week after spraying dry soil with terbacil. Excellent control resulted by applying terbacil to wet soil with either immediate or delayed irrigation or by applying terbacil to dry soil with immediate irrigation.

Bioassay experiments showed that the terbacil was concentrated mainly in the top two inches of soil when the terbacil was applied to dry soil with delayed irrigation. This would indicate that terbacil was adsorbed at the surface of the soil. Roots of weeds would develop below that level, so plant uptake of the herbicide would be negligible. When applied in this manner, terbacil is ineffective as a herbicide.

It was expected that terbacil applied to wet soil with immediate irrigation would not be adsorbed so readily as terbacil applied to dry soil with delayed irrigation. This experiment showed that a waiting period before application of water is essential for adsorption of terbacil to the soil. The extent of leaching of terbacil was quite small for all treatments. Perhaps if more than 3-4 inches of water were applied, the degree of leaching would increase.

Leaching studies in which terbacil was applied to 12-inch soil columns in soil sterilant quantities (12.6 lb/acre) followed by

10 inches of water showed that the addition of water immediately after terbacil application resulted in the greatest amount (8.8 percent or 1.1 lb/acre) of terbacil in the leachate. This occurred on both wet and dry soil. Three days, or less, were required to markedly reduce the amount of terbacil recovered from the leachates from columns in which terbacil was applied to dry soil. When terbacil was applied to wet soil, a longer interval of time was required before the amount of terbacil in the leachate was markedly reduced. This indicates that terbacil may be competing with water for adsorption sites on the soil colloids under wet soil conditions. This would not be the problem when applied to dry soil, but some period of time seems to be required for adsorption to take place to reduce leaching.

From the experimental data in this thesis it can be concluded that terbacil is relatively resistant to leaching, even when applied to very wet soil and followed immediately with large amounts of water. From this it seems that leaching is not the reason for poor weed control when terbacil is applied in late fall or early winter. Greenhouse bioassays of core samples taken from plots in which terbacil was applied in October and December showed high levels of terbacil in the upper soil levels even in plots which had shown poor weed control in the field. If weeds are present when terbacil is applied in late fall or early winter, the weed roots may continue to grow downward more rapidly than terbacil could leach into the 2-3 inch zone, thereby escaping normally toxic amounts of the herbicide. Other factors such as lower light intensity, lower temperature, and higher humidity may also reduce the degree of weed control obtained with

early winter applications of terbacil. Periodic fall and winter applications of terbacil, with careful observation of weed growth and subsequent analysis of core samples, may provide more information with which to better understand the differing results obtained from early fall and winter applications of terbacil.

## 1X. SUMMARY

Terbacil was sprayed on wet and dry soil and leached with water from sprinkler irrigation immediately and seven days after application. Core samples were taken for bioassay in the greenhouse. Core samples from an experiment where terbacil was applied in October and December were also bioassayed. Leaching studies were conducted in the greenhouse, soil columns were bioassayed, and the leachates were analyzed by gas-liquid chromatography.

Results obtained were:

- 1. Terbacil applied to dry soil in the field one week before sprinkler irrigation showed the least herbicidal activity. Application of terbacil to dry soil followed immediately with irrigation and to wet soil, whether irrigation was immediate or one week after application, resulted in increased herbicidal activity. Bioassay of core samples showed terbacil to leach the least when applied to dry soil one week before irrigation.
- 2. Core samples taken from another field trial in which terbacil was applied to relatively wet soil in October and December showed that date of application had little effect on the depth to which terbacil leached.
- 3. Bioassay of soil columns with two distinct soil layers showed that terbacil leached to a greater extent when leaching water was applied immediately after application. Analysis of the leachates by gas-liquid chromatography

indicated the need of a waiting period between the application of terbacil and the application of leaching water to reduce movement of terbacil.

4. Analysis of leachates from soil columns in which the entire soil mass within a column was at one moisture level resulted in more terbacil coming through columns in which terbacil was applied to wet soil rather than dry. Again the amount of terbacil recovered from the leachates was greatly reduced if application of leaching water was delayed at least three days after terbacil application.

## X. BIBLIOGRAPHY

- Bailey, George W. and Joe L. White. 1964. Soil-pesticide relationships. Review of adsorption and desorption of organic pesticides by soil colloids, with implications concerning pesticide bioactivity. Journal of Agricultural and Food Chemistry 12:324-332.
- Burnside, O. C., C. R. Fenster, and G. A. Wicks. 1963. Dissipation and leaching of monuron, simazine and atrazine in Nebraska soils. Weeds 11:209-213.
- Burnside, O. C., G. A. Wicks, and C. R. Fenster. 1963. The effect of rainfall and soil type on the disappearance of 2,3,6-TBA. Weeds 11:45-47.
- Danielson, L. L. 1956. The crop toxicity period of CMU in a sandy clay loam soil. Weeds 4:255-263.
- Deming, J. M. 1963. Determination of volatility losses of C<sup>14</sup>-CDAA from soil surfaces. Weeds 11:91-95.
- Dickens, Ray and A. E. Hiltbold. 1967. Movement and persistence of methanearsonates in soil. Weeds 15:299-304.
- Dubey, H. D. and J. F. Freeman. 1965. Leaching of linuron and diphenamid in soils. Weeds 13:360-362.
- DuPont, deNemours and Company. 1967. Technical data sheet on terbacil. Wilmington, Delaware. 3 p.
- Fang, S. C., Patricia Theisen, and V. H. Freed. 1961. Effects of water evaporation, temperature, and rates of application on the retention of ethyl-N,N-di-n-propylthiocarbamate in various soils. Weeds 9:569-574.
- Freed, V. H., J. B. Vernetti, and M. L. Montgomery. 1967. The soil behavior of herbicides as influenced by their physical properties. In: Environmental and other factors in the response of plants to herbicides, ed. by V. H. Freed and R. O. Morris. Corvallis. p. 107-128. (Oregon Agricultural Experiment Station. Technical Bulletin 100)
- Friesen, H. A. 1965. The movement and persistence of dicamba in soil. Weeds 13:30-33.
- Geissbühler, H., C. Haselbach, and H. Aebi. 1963. The fate of N'-(4-chlorophenoxy)-phenyl-NN-dimethylurea (C-1983) in soils and plants. I. Adsorption and leaching in different soils. Weed Research 3:140-153.

- Hance, R. J. 1965. The adsorption of urea and some of its derivatives by a variety of soils. Weed Research 5:98-107.
- Harris, C. I. and G. F. Warren. 1964. Adsorption and desorption of herbicides by soil. Weeds 12:120-126.
- Hartley, G. S. 1964. Herbicide behavior in the soil. I. Physical factors and action through the soil. In: The physiology and biochemistry of herbicides, ed. by L. J. Audus. New York, Academic. p. 111-161.
- Hilton, J. L., T. J. Monaco, D. E. Moreland, and W. A. Gentner. 1964. Mode of action of substituted uracil herbicides. Weeds 12:129-131.
- Hurtt, W., J. A. Meade, and P. W. Santelmann. 1958. The effect of various factors on the movement of CIPC in certain soils. Weeds 6:425-431.
- Lambert, S. M., P. E. Porter, and R. H. Schieferstein. 1965. Movement and sorption of chemicals applied to the soil. Weeds 13:185-190.
- Logan, A. V., N. R. Odell, and V. H. Freed. 1953. The use of C<sup>14</sup> in a study of the leaching rate of isopropyl N-phenylcarbamate. Weeds 2:24-26.
- Merkle, M. G., R. W. Bovey, and F. S. Davis. 1967. The fate of picloram in soils. Proceedings of the Southern Weed Conference 20:392.
- Ogle, R. E. and G. F. Warren. 1954. Fate and activity of herbicides in soils. Weeds 3:257-273.
- Parochetti, J. V. and G. F. Warren. 1966. Vapor losses of IPC and CIPC. Weeds 14:281-285.
- Price, Hugh C. and Vernon J. Fisher. 1967. The distribution of terbacil, DuPont herbicide 733, and amizine in two orchard soils under two precipitation levels. (Abstract) Abstracts of the Annual Meeting of the Weed Society of America, 1967, p. 71-72.
- Sherburne, H. R., V. H. Freed, and S. C. Fang. 1956. The use of C<sup>14</sup> carbonyl labeled 3(P-chlorophenyl)-1,1-dimethyl urea in a leaching study. Weeds 4:50-54.
- Upchurch, Robert P. 1957. The influence of soil-moisture content on the response of cotton to herbicides. Weeds 5:112-120.

1966. Behavior of herbicides in soil. Residue Reviews 16:46-85.

- Upchurch, R. P. and D. D. Mason. 1962. The influence of soil organic matter on phytotoxicity of herbicides. Weeds 10:9-14.
- Upchurch, R. P. and W. C. Pierce. 1957. The leaching of monuron from Lakeland sand soil. Part I. The effect of amount, intensity, and frequency of simulated rainfall. Weeds 5:321-330.

1958. The leaching of monuron from Lakeland sand soil. Part II. The effect of soil temperature, organic matter, soil moisture, and amount of herbicide. Weeds 6:24-33.

- Ward, Thomas M. and Robert P. Upchurch. 1965. Role of amide group in adsorption mechanisms. Journal of Agricultural and Food Chemistry 13:334-340.
- Weed killers branch out. 1967. DuPont Agricultural News Letter 34:8-10. Fall.
- Wiese, A. F. and R. G. Davis. 1964. Herbicide movement in soil with various amounts of water. Weeds 12:101-103.

APPENDIX

		<u> </u>	Tox	icity Rat	ing <sup>a</sup>	
	Rate	Rep.	Rep.	Rep.	Rep.	Avg.
Treatment	lb/acre	I	II	III	IV	
		Ra	ine			
			<u>. F - </u>			
WSS	0.8	100	100	100	100	100
	1.2	100	100	100	100	100
WSW	0.8	100	100	100	100	100
	1.2	100	100	100	100	100
DSS	0.8	100	100	100	100	100
	1.2	100	100	100	100	100
DSW	0.8	100	b	100	100	100
	1.2	100	100	100	100	100
		<u>0</u> a	its			
WSS	0.8	81	82.3	76.6	85	81.22
	1.2	92	82.3	85	82.3	85.4
WSW	0.8	76.6	75	77.3	68.3	74.30
	1.2	83.3	85	83.3	88.3	84.98
DSS	0.8	85	88.3	71.6	82.3	81.80
	1.2	89	88.3	85.6	90.6	88.37
DSW	0.8	41.6	Ъ	33.3	30	34.97
	1.2	43.3	33.3	40	53.3	42.73
		Cc	orn			
WSS	0.8	33.3	35	31.6	28.3	32.05
	1.2	38.3	38.3	36.3	48.3	40.30
WSW	0.8	21.6	23.3	26.6	23.3	23.7
	1.2	31.6	25	48.3	35	34.98
DSS	0.8	21.6	40	28.3	21.6	27.88
	1.2	43.3	40	46.6	40	42.48
DSW	0.8	1.6	b	15	1.6	6.07
	1.2	15	11.6	16.6	1.6	11.20

Table	1.	Influence	e of	E moist	ure,	, both be	efore an	d af	Eter	terbacil
	appl	lication,	on	growth	of	bioassay	/ plants	in	the	field.

a Rating scale: 0-100, with 100 equal to complete kill.
 b Observations only used from three replications.

	Rate	Depth in	Rep.	Rep.	Rep.	Rep.	Avg.
Treatment	lb/acre	soil column	Ī	II	III	IV	
		(inches)					
WSS	0.8	0- 2	42	61	68	34	51
		2-4	119	127	213	141	150
		4- 6	323	180	679	513	423
		6-8	372	309	424	283	347
		8-10	556	368	539	504	491
		10-12	255	332	1003	392	<u>495</u>
					Ave	erage	326.1
	1.2	0- 2	66	50	423	42	145
		2-4	62	198	515	78	213
		4-6	112	509	421	257	324
		6- 8	78	335	563	117	273
		8-10	111	120	631	285	287
		10-12	273	251	560	175	315
					Ave	erage	259.5
				Treat	ment Ave	erage	292.8
WSW	0.8	0-2	67	60	215	32	93
		2- 4	174	246	366	74	2 <b>1</b> 5
		4-6	5 <b>0</b> 2	354	581	607	511
		6- 8	555	292	522	376	436
		8-10	591	322	732	563	552
		10-12	423	285	526	373	401
					Ave	erage	368
	1.2	0- 2	70	54	68	32	56
		2-4	81	78	311	200	168
		4- 6	388	312	249	698	412
		6- 8	455	200	341	334	333
		8-10	463	285	378	629	439
		10-12	509	354	456	322	410
					Ave	erage	303
				Treat	ment Ave	erage	335.5
DSS	0.8	0- 2	64	61	54	40	54
200		2-4	86	61	465	82	173
		4 <b>-</b> 6	302	196	454	389	335
		6-8	336	250	465	298	337
		8-10	447	520	451	549	491
		10-12	636	372	799	381	547
		10 12	000	0,2	 Avr	erage	322.8
						460	$an \pm 1d$

Table 2. Influence of irrigation on the depth of leaching of terbacil applied at two rates to dry and saturated soil. Measured in milligrams of dry plant weight using oats as the bioassay plant.

<sup>(</sup>cont'd.)

	Rate	Depth in	Rep.	Rep.	Rep. Rep.	Avg.
Treatment	lb/acre	soil column	I	II	<u> </u>	
		(inches)				_
DSS	1.2	0-2	77	60	65 31	58
		2-4	71	52	529 34	172
		4- 6	71	55	405 43	144
		6-8	89	224	579 73	241
		8-10	400	254	756 75	371
		10-12	382	360	427 137	327
					Average	218.8
				Treat	ment Average	270.8
DSW	0.8	0- 2	80	311	193 43	156
		2- 4	529	281	516 727	513
		4- 6	560	390	629 592	542
		6- 8	611	308	598 537	513
		8-10	537	282	750 578	536
		10-12	491	413	694 605	550
					Average	468.3
	1.2	0- 2	75	113	133 40	90
		2- 4	618	116	634 358	432
		4- 6	499	468	437 604	502
		6- 8	534	368	692 650	561
		8-10	571	350	603 497	505
		10-12	550	348	710 433	510
					Average	433.3
				Treat	ment Average	450.8
Check <sup>a</sup>	0.0	0-2	698	318	572 390	495
		2-4	685	435	565 709	599
		4- 6	479	278	587 841	546
		6- 8	391	289	599 431	428
		8-10	364	318	588 518	447
		10-12	347	316	813 619	524
				Treat	ment Average	506.5
					0	

<sup>a</sup> Check not included in analysis of variance.

Source of variation	df	SS	MS	F
Replication	3	135,552	45,184**	8.963
Treatment	3	92,735	30,912*	6.132
Error(a)	9	45,375	5,041	
Rate	1	22,270	22,270*	5.400
Rate x treatment	3	2,873	958	.232
Error(b)	12	49,499	4,124	
Levels	5	323,126	64,625**	58,273
Levels x treatment	15	43,571	2,904**	2.618
Levels x rate	5	10,236	2,047	1.845
Levels x rate x treatment	15	17,970	1,198	1.080
Error(c)	120	133,159	1,109	
Total	191	876,366		

Table 3. Analysis of variance table for data in Table 2.

\* Significant at the 95% level \*\* Significant at the 99% level

C.V. for treatments = 21%C.V. for rates = 19%C.V. for levels = 10%

Treatment LSD at 0.05 level = 32.81, at 0.01 level = 47.09Rate LSD at 0.05 level = 20.17, at 0.01 level = 28.31Level LSD at 0.05 level = 16.49, at 0.01 level = 21.45

							0.8	3 lb/acr	e					
Treat.	Rep.	0-2	2-4	4-6	6-8	8-10	10-12		0-2	2-4	4-6	6-8	8-10	10-12
WSS	т	6	17	67	95	152	73	DSS	9	13	63	86	123	183
	ΤŤ	19	29	65	107	116	105	000	19	14	70	86	164	118
	III	12	38	115	71	92	123		9	82	77	78	77	98
	IV	9	20	61	66	97	63		10	12	46	69	106	62
	Avg.	12	26	77	85	114	91		12	30	64	80	118	115
WSW	I	10	25	105	142	163	122	DSW	11	77	117	156	148	141
	II	19	57	127	101	101	90		98	64	140	106	89	131
	III	38	65	99	87	124	65		34	91	107	100	128	85
	IV	8	10	72	87	109	60		11	103	70	125	112	98
	Avg.	19	39	101	104	124	84		39	84	109	122	119	114
							1.2	<u>l</u> b/acre						
WSS	I	9	9	23	20	31	79	DSS	11	10	15	23	110	110
	II	16	46	183	116	38	79		19	12	20	77	80	114
	III	7	91	72	94	107	67		11	94	69	97	129	52
	IV	11	11	31	27	55	28		8	5	5	17	15	22
	Avg.	11	39	77	64	58	63		12	30	27	54	84	75
WSW	I	10	12	81	116	127	147	DSW	11	90	104	136	157	158
	II	17	18	112	69	90	112		35	27	168	127	110	110
	III	12	55	42	57	64	56		23	112	74	115	103	87
	IV	8	28	83	78	121	52		10	50	72	151	96	70
	Avg.	12	28	80	80	100	92		20	70	105	132	117	106

Table 4. Dry weight of oat plants grown in core samples taken from WSS, WSW, DSS, and DSW treatments. Measured as percent of check.<sup>a</sup>

<sup>a</sup> Figures are rounded to nearest whole number.

	Depth of								
	soil		Replic	atior	1.S	Inj	ury	Ratin	gs <sup>a</sup>
	column	I	II	111	Avg.	I	II	III	Avg.
Oct. 9 0.8 lb/acre	(inches) 0 - 0.5 0.5- 1 1 - 2 2 - 3 3 - 6 6 -12	42 47 52 71 342 318	46 58 88 287 260 280	66 223 335 212 435 <b>2</b> 93	51 109 158 190 345 297 ₹ 191.6	4 4 3 0 0	4 3 2 0 0 0	3 2 0 0 0 0	3.6 3.0 2.0 1.0 0.0 0.0
Oct. 9 1.6 lb/acre	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	59 50 42 41 55 333	45 49 49 44 65 231	49 42 46 52 141 323	51 47 45 45 87 <u>295</u> 95.0	4 4 4 3 1	4 4 4 2 1	4 4 4 2 0	4.0 4.0 4.0 2.3 0.6
Dec. 12 0.8 lb/acre	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	42 48 44 135 326 271	49 49 53 290 289 384	63 77 77 287 343 396	$51 58 237 319 \overline{x} 178.8$	4 4 2 0 0	4 4 1 0	4 4 3 1 0 0	4.0 4.0 3.6 1.3 0.0 0.0
Dec. 12 1.6 lb/acre	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	42 35 38 49 252 302	50 38 52 56 232 286	48 42 45 128 292 339	$46384577258309\overline{x} 128.8$	4 4 3 0 0	4 4 3 0	4 . 4 2 0 0	4.0 4.0 2.6 0.0 0.0
Check <sup>b</sup>	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	243 263 368 326 357 310	211 326 368 370 320 245	220 247 345 349 349 384	$224 \\ 278 \\ 360 \\ 348 \\ 342 \\ 313 \\ \overline{x} 310.8$	1 0 0 0 0 0	1 0 0 0 0	1 0 0 0 0 0	1.0 0.0 0.0 0.0 0.0 0.0

Table 5. Milligrams of dry plant material and injury ratings of oat plants grown in core samples taken from an established peppermint trial.

<sup>a</sup> Rating scale 0-4 with 4 equal to complete kill, 0 equal to no injury symptoms

<sup>b</sup> Check not included in analysis of variance

39,696 1,953	19,848	11.380
1,953	1 953	
1,700	1 7 7 7	1.119
3,488	1,744	
96.288	96,288*	20.127
9,917	9,917	2.073
19,137	4,784	
720,733	144,146**	63.249
32,208	6,441*	2.826
67,712	13,542**	5.942
29,633	5,926*	2.600
91,187	2,279	
,111,952		
	3,488 96,288 9,917 19,137 720,733 32,208 67,712 29,633 91,187 ,111,952	3,488       1,744         96,288       96,288*         9,917       9,917         19,137       4,784         720,733       144,146**         32,208       6,441*         67,712       13,542**         29,633       5,926*         91,187       2,279         ,111,952

Table 6. Analysis of variance table for data in Table 5.

\* Significant at the 95% level \*\* Significant at the 99% level

C.V. for dates = 28% C.V. for rates = 46% C.V. for levels = 32%

Date LSD at 0.05 level = 42.34, at 0.01 level = 97.66 Rate LSD at 0.05 level = 45.24, at 0.01 level = 75.04 Level LSD at 0.05 level = 39.38, at 0.01 level = 52.70

Depth in				Treatment	- O-day <sup>a</sup>			
soil column	,	Air	dryb			Field ca	pacity <sup>c</sup>	
(inches)	Rep I	Rep II	Rep III	Avg.	Rep I	Rep II	Rep III	Avg.
0- 1	64	66	75	68	76	73	79	76
1 <b>- 2</b>	94	87	94	91	106	152	103	120
2-3	99	124	65	96	110	88	94	97
3- 4	106	207	100	137	107	160	92	119
4- 5	110	255	201	188	104	168	82	118
5-6	161	234	386	260	102	165	272	180
6-7	361	367	399	375	198	1 <b>7</b> 5	305	226
7-8	426	333	662	474	272	167	363	267
8-9	501	384	710	531	382	161	577	373
9 <b>-</b> 10	520	348	712	526	413	103	670	395
10-11	507	355	775	546	476	87	882	481
11-12	529	446	916	630	441	127	979	515
			Average	326.8			Average	247.2
			Ū.			Treatment	Average	287.0
				Treatmen	<mark>t -</mark> 3-day <sup>d</sup>			
0- 1	74	71	72	72	86	73	61	73
1 <b>-</b> 2	80	73	156	103	99	396	82	192
2-3	216	71	172	153	222	213	140	191
3-4	176	298	151	208	149	563	217	309
4 <b>-</b> 5	230	580	181	330	123	454	218	265
5-6	266	578	205	349	304	381	257	314
6 <del>-</del> 7	513	532	182	409	305	453	274	344
7-8	570	566	137	424	360	213	291	288

# Table 7. Milligrams of dry plant material harvested from soil columns. Greenhouse Leaching Experiment I.

58 (cont'd.)

Depth in				Treatment	t - 3-day <sup>d</sup>			
soil column		Air	dry5			Field ca	apacity <sup>c</sup>	
(inches)	Rep I	Rep I1	Rep III	Avg.	Rep I	Rep II	Rep III	Avg.
8- 9	563	564	184	437	364	298	333	331
9-10	616	633	265	504	421	357	295	358
10-11	483	759	315	519	488	424	330	414
11 <b>-</b> 12	488	925	290	567	494	911	277	560
			Average	339.6			Average	303.2
			0			Treatment	t Average	321.4
				Treatmen	<u>t - 7-D-U</u> e			
0- 1	94	83	66	81	79	100	56	78
1- 2	194	222	116	177	147	180	94	140
2 <b>-</b> 3	156	358	161	225	126	77	119	107
3- 4	245	619	299	387	114	80	96	96
4 <b>-</b> 5	308	437	288	344	161	366	178	235
5- 6	378	325	281	328	200	421	189	270
6-7	529	313	217	353	172	459	174	268
7-8	422	207	395	341	231	259	164	218
8-9	420	198	195	271	278	299	199	258
9-10	444	296	132	290	319	410	182	303
10-11	488	337	151	325	314	631	142	362
11-12	528	329	131	329	329	862	170	453
			Average	287.5			Average	232.3
						Treatmen	t Average	260.4

Table	7.	continued
-------	----	-----------

(cont'd.) 🕠

Depth in soil column (inches)	Treatment - 7-D-C <sup>t</sup>								
	Air dryb			Field capacity <sup>C</sup>					
	Rep I	Rep II	Rep III	Avg.	Rep I	Rep II	Rep III	Avg.	
0- 1	83	74	60	72	63	52	74	63	
1 <b>-</b> 2	167	454	219	280	225	157	67	149	
2-3	150	537	305	330	117	172	237	175	
3- 4	116	622	260	332	132	363	305	266	
4- 5	215	638	257	370	218	272	319	269	
5- 6	389	404	320	371	357	338	420	371	
6-7	470	443	333	415	346	501	360	402	
7-8	414	338	248	333	408	670	377	485	
8-9	426	480	268	391	469	834	360	554	
9-10	494	544	210	416	691	910	390	663	
10-11	604	940	310	618	475	961	313	583	
11-12	469	935	315	573	469	835	261	521	
			Average	375.1			Average	375.1	
						Treatment Average			

# Table 7. continued

- <sup>a</sup> Columns were leached 0 days after spraying.
  <sup>b</sup> Top layer of soil was air dry.
  <sup>c</sup> Top layer of soil was at field capacity.
  <sup>d</sup> Columns were leached 3 days after spraying.
  <sup>e</sup> Columns were uncovered and leached 7 days after spraying.
  <sup>f</sup> Columns were covered and leached 7 days after spraying.
| Source of variation       | df  | SS         | MS        | F            |
|---------------------------|-----|------------|-----------|--------------|
| Replication               | 2   | 556.597    | 278.298   | 1.038        |
| Treatment-date            | - 3 | 532,412    | 177.470   | .662         |
| Error(a)                  | 6   | 1,608,376  | 268,062   |              |
| Moisture                  | 1   | 131,541    | 131,541   | 5.747        |
| Moisture x treatment-date | 3   | 61,243     | 20,414    | .891         |
| Error(b)                  | 8   | 183,096    | 22,887    |              |
| Levels                    | 11  | 4,920,963  | 447,360** | 27.582       |
| Levels x treatment-date   | 33  | 633,785    | 192,056** | 11.841       |
| Levels x moisture         | 11  | 63,823     | 5,802     | <b>.</b> 357 |
| Levels x moisture x       |     |            |           |              |
| treatment-date            | 33  | 575,375    | 17,435    | 1.074        |
| Error(c)                  | 176 | 2,854,711  | 16,219    |              |
| Total                     | 287 | 12,121,922 |           |              |

Table 8. Analysis of variance table for data in Table 7.

\* Significant at the 95% level \*\* Significant at the 99% level

Treatment-date LSD at 0.05 level = 211.15, at 0.01 level = 319.98 Moisture LSD at 0.05 level = 41.11, at 0.01 level = 59.81 Level LSD at 0.05 = 72.04, at 0.01 level = 94.69

Moisture				
level <sup>a</sup>	0-day	3-day	7-D-C	7 - D - U
15	1.05	2.0	6.0	- /
AD	425	28	63	76
AD	137	193	249	81
	281	110.5	156	78.5
edb	27.7	10.8	15.4	7.7
FC	421	16	35	44
FC	115	130	74	62
	268	73	54.5	53
ed	26.4	7.1	5.3	5.2
	Moisture level <sup>a</sup> AD AD ed <sup>b</sup> FC FC	Moisture level <sup>a</sup> 0-day       AD     425       AD     137       281     281       ed <sup>b</sup> 27.7       FC     421       FC     115       268     26.4	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

Table 9. Micrograms of terbacil recovered from greenhouse leaching experiment I leachate.

<sup>a</sup> AD indicates the top two inches of soil were air dry, about three percent moisture content.
FC indicates the top two inches of soil were at field capacity moisture level.

 $^{\rm b}$  Expressed as a percent of the total 1014  ${\rm \mu g}$  of terbacil which were applied to each column.

Source of variation	df	SS	MS	F
Replications	1	280.56	280.56	.007
Treatments	3	108,038.19	36,012.73	.089
Error(a)	3	120,177.19	40,059.06	
Moisture	1	7,876.56	7,876.56	5.101
Moisture x treatment	3	4,651.19	1,500.39	1.004
Error(b)	4	6,175.75	1,543.93	
Total	15	247,199.44		

Table 10. Analysis of variance table for data in Table 9.

C.V. for treatments = 58%

C.V. for moisture = 36%

Moisture				
level <sup>a</sup>	0-day	3-day	7-D-C	7-D-U
	07		( )	
AD	37	23	62	16
	34	20	70	27
	47	31	53	45
Avg.	39.3	24.6	61.6	29.3
Avg. % recovered <sup>b</sup>	3.8	2.4	6.0	2.8
FC	10	С	24	125
	33	14	32	85
	12	20	48	41
Avg.	18.3	12.3	34.6	83.6
Avg. % recovered	1.8	1.1	3.4	8.2

Table 11. Micrograms of terbacil recovered from greenhouse leaching experiment II leachate.

<sup>a</sup> AD indicates the top two inches of soil were air dry, about three percent moisture content FC indicates the top two inches of soil were at field capacity moisture level.

- <sup>b</sup> Expressed as a percent of the total 1014 µg of terbacil which were applied to each column.
- <sup>c</sup> The amount of terbacil present was too small to determine.

Table 12. Analysis of variance table for data in Table 11.

Source of variation	df	SS	MS	F
Between treatments Within treatments	7 16	12,027.29 5,095.34	1,718.184* 318.458	5.395
Total	23	17,122.63		

\* Significant at the 95% level

C.V. = 47%

0-day	3-day	7-D-C	7 <b>-</b> D- U
100		0 -	
133	50	35	68
483	83	118	13
310	70	105	14
308.6	67.6	86	31.6
2.8	•6	.8	.3
964	818	386	205
894	1164	901	136
965	344	934	193
941	775.3	740.3	178
8.8	7.3	6.9	1.7
	133 483 310 308.6 2.8 964 894 965 941 8.8	133     50       483     83       310     70       308.6     67.6       2.8     .6       964     818       894     1164       965     344       941     775.3       8.8     7.3	133     50     35       133     50     35       483     83     118       310     70     105       308.6     67.6     86       2.8     .6     .8       964     818     386       894     1164     901       965     344     934       941     775.3     740.3       8.8     7.3     6.9

Table 13. Micrograms of terbacil recovered from greenhouse leaching experiment III leachate.

<sup>a</sup> AD indicates soil throughout the entire column was air dry, about three percent moisture content.
FC indicates the soil throughout the entire column was at field capacity moisture level.

<sup>b</sup> Expressed as a percent of the total 10,647 µg of terbacil which were applied to each column.

Table 14. Analysis of variance table for data in Table 13.

Source of variation	df	SS	MS	F
Between treatments Within treatments	7 16	2,853,250.50 601,607.34	407,607.21** 37,600.458	10.84
Total	23	3,454,857.84		

\*\* Significant at the 99% level

C.V. = 49.6%

 $LSD_{.05} = 335.3$