

MOVEMENT OF FLUOMETURON AND PROMETRYN
ACROSS AND THROUGH THE SOIL

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under row-crop conditions, from the standpoint of runoff and leaching.

The objectives of this research were:

- (1) Evaluate the magnitude of herbicide runoff losses,
- (2) Determine herbicide concentrations in the water and sediment portion of the runoff,
- (3) Evaluate the effect of different environmental factors on the movement of the herbicides across the soil surface and through the soil profile, and
- (4) Study the dissipation and movement of the herbicides during the growing season.

CHAPTER II

REVIEW OF LITERATURE

Pesticide Runoff

The movement of a pesticide by surface runoff is important from the standpoint of environmental pollution, effects on non-target species, and loss of the material from the target site. Questions that need to be answered in this regard include: 1. Is the pesticide transported mainly in the liquid or sediment phases of the runoff? 2. What is the magnitude of these losses? 3. and What conditions influence these losses?

Pesticide losses due to runoff have generally been reported as being small. Edwards and Glass (13) applied 11.2 and 22.4 kg/ha 1,4,5,-T and methoxychlor respectively, and monitored the runoff for 14 months. Runoff in this period removed 0.05% of the applied 2,4,5-T and 0.0004% of the applied methoxychlor. The bulk of the removal occurred in the first four months. Caro and Taylor (7) applied dieldrin to small watersheds and recovered only 0.07 percent of the original dosage in the first season's runoff, with the largest losses occurring in the first two months. The highest concentration in the water was 20 ppb. However, where erosion occurred, losses of dieldrin in the sediment was as high as 2.2% of that applied. Sheets, Bradley, and Jackson (28) applied trifluralin at 1.12 kg/ha and incorporated it to a depth of 4". Samples were collected after each rain that produced

significant runoff. Less than 1% of the total applied was recovered in the runoff over a period of 5-8 months after application. Sediment filtered out of the water contained over 84% of the trifluralin detected in the runoff. Haan (15) used small plots in the greenhouse to study the movement of aldrin, dieldrin, and DDT. Concentrations of the pesticides in the water was only 1 to 70 ppb. However concentrations of the pesticides on the eroded soil were on the order of 10 to 30 ppm. Based on the volumes of water and sediments leaving the plots, more than twice the pesticide was carried by the sediment compared to the water. When 13.4 kg/ha of DDT were applied to cotton by Bradley, Sheets, and Jackson (6), less than 3% of the applied DDT was recovered from surface runoff collected over a 6-month period. Of the DDT recovered, 96% was associated with the sediment. Toxaphene was also used in the study, and less than 1% of the total applied toxaphene was recovered in the runoff. Of that recovered, about 75% was associated with the sediment. Less DDT was recovered in the runoff from plots to which DDT was applied in combination with toxaphene than where DDT was applied alone, even though the same amount of DDT was applied to both plots. It was suggested that the oily toxaphene formulation might have increased the adsorption of DDT to the plant foliage.

Several researchers have studied picloram, a highly mobile herbicide. Trichell et al. (31) reported average picloram losses of approximately 3%. The amount of picloram lost varied with rate of application, but the percentage lost was the same. The slope of the plot and the movement of runoff over untreated soil influenced the amount of picloram detected. Movement over untreated soil reduced the

amount of picloram runoff collected. In a more recent rangeland study, Scifres et al. (27) applied picloram at 0.28 kg/ha and irrigated 10 days later. Runoff water contained 17 ppb picloram. Irrigation or rainfall 20, 30, or 45 days after treatment resulted in less than 1 ppb in the runoff water. Baur, Bovey and Merkle (4) sampled runoff water over a 2 year period from an 8 ha site that had been treated with 1.12 kg/ha of picloram in the spring of 1969 and 70. Runoff water was sampled adjacent to the plots in two creek systems up to 2 Km from the plots. Runoff water from rainfall starting within two days after spraying had residue levels ranging from 26.2 to 89.7 ppb in samples taken adjacent to the treated plots. Samples taken adjacent to the plots declined to less than 10 ppb by 10-12 weeks after application. Eight days after application, water sampled 1.2 Km from the plots contained less than 1 ppb of picloram. Davis, Ingebo, and Pase (12) reported the picloram content of stream water to be 0.37 ppm on the seventh day after treatment following 2.53 inches of rainfall. Picloram was applied to the water shed as pellets at a 9.3 lb a.e/a rate. The results indicate some contamination of the stream water occurred as a result of the soil application of a high rate of picloram.

Little research has been reported on herbicide losses under row cropping conditions. In some early work, White et al. (36) studied atrazine on fallow land (6.5% slope). The herbicide was surface applied at 3.36 kg/ha, and simulated rainfall was used to produce runoff and erosion. The results indicate that atrazine is transported in small but significant amounts in washoff. A simulated rainfall of 2.5"/hr, 95 hr after application, resulted in a loss of 7.3% of the applied atrazine. Losses from a 0.5" storm were 75% less or 0.06 kg/ha.

Greater losses occurred when rainfall was applied immediately after herbicide application. The highest concentration of atrazine was highest in the soil fraction compared to the water. However, most of the atrazine transported was associated with the water fraction because of greater amounts of water lost compared to the soil. In some more recent work, Hall, Pawlus and Higgins (16) studied atrazine losses when applied preemergence to corn. The field plots were 1.8 X 22 m in size on a 14% slope, and were fitted with a catchment device. Atrazine was applied and the plots were seeded to corn (Zea mays L.). Average losses for all rates in runoff water and sediment equaled 2.4% and 0.16% of the total applied, respectively. At the recommended rate of 2.2 kg/ha, composite losses were 2.5% or 0.05 kg/ha. The concentrations in the runoff water after the first rainfall ranged from 0.39 to 4.68 ppm from the lowest (0.6 kg/ha) to highest (9.0 kg/ha) rate of application. The sediment concentrations ranged from 0.33 to 6.23 ppm for the respective rates of application. Ritter et al. (26) applied atrazine, propachlor, and diazinon to watersheds ranging in size from 1.9 to 3.8 acres. The average slope was 10-15%. Half of the watersheds were surface contoured and planted to corn and half were bedded and planted to corn. Pesticide losses were much greater from the surface-contoured watersheds than the ridged watersheds. Generally, the pesticide concentrations were higher on the sediment than in the water; however, greater total losses were associated with the greater volume of water. Atrazine losses of 15 to 16% were reported. These losses are quite high and would be doubtful under conditions of lesser slope. No runoff was obtained in the propachlor studies before it degraded. Losses of diazinon were insignificant.

Several factors influencing the magnitude of herbicide losses from soil have already been discussed. An extensive literature review on pesticide-water-sediment interactions is presented by Pionke and Chesters (24). Because runoff is generated primarily at the soil surface, the pesticides that persist at the surface are most likely to be transported in this manner. Rainfall immediately following application usually produced larger losses than did delayed rainfalls (36). Epstein and Grant (14) found that concentrations and amounts of DDT, endrin and endosulfan were lower in runoff from a rotation of potato (Solanum tuberosum L) and oats (Avena sativa L.) and sod than under continuous potatoes. Trichell et al. (31) found losses of dicamba and picloram were greater from sod plots than fallow plots, whereas 2,4,5-T losses were about equal. Barnett et al. (3) studied 2,4-D washoff using simulated rainfall. Antecedent soil moisture did not influence the amount of butyl ester 2,4-D recovered, but did influence the amount of an amine formulation recovered. When the amine (a water soluble formulation) was used the 2,4-D concentration was less than 1 ppm whereas concentrations of isooctyl ester as high as 4.2 ppm were measured. It was suggested that initial soil penetration, accomplished by use of a more soluble form, greatly reduced runoff losses. Munn et al. (23) used small plots and simulated rainfall to determine the effect of soil, cover, slope, and rainfall factors on phosphorus and soil movement. The quantity of runoff water, eroded solids and phosphorus in the runoff increased with the degree of slope and rainfall intensity. A high correlation was found between total phosphorus in the runoff and the quantity of soil eroded. Plant cover was effective in reducing runoff volume, soil erosion and phosphorus

losses. Weber and Best (35) studied the movement of 13 herbicides across the soil surface. Ratings were made depending on the lateral movement of the herbicide as reflected by the inhibition of indicator crops and weeds in adjacent plots. Prometryn was classified as low movement and fluometuron was classified as intermediate on their scale.

Pesticide Leaching

Vertical herbicide movement through the profile is important from the standpoint of runoff and erosion of the pesticide as well as from the standpoint of the pesticide availability to plants. In addition to studying herbicide runoff as influenced by several environmental factors, the movement of the herbicide into the profile is also of interest.

An extensive review on the movement of s-triazine herbicides and the substituted urea herbicides is presented by Bailey and White (2) and Helling (20). This review will be restricted to only the herbicides or the environmental factors pertinent to this study. Bailey and White (2) state that the total amount of water received, and its intensity and frequency all appear to effect movement of a pesticide in the soil.

Upchurch et al. (33) used soil columns to study the effect of the quantity, intensity and frequency of simulated rainfall on the movement of monuron applied at 40 lb/A. Increased amounts of rainfall increased leaching, but intensity had little effect. Increased frequency increased the movement. One explanation offered is that leaching involves two steps: entrance of the herbicide into solution and adsorption onto the soil. Rainfall intensity apparently effects

adsorption but not entry into solution. Frequency apparently effects both steps. Harris (17) found a positive effect of intensity. Movement of diphenamid or dicamba was greater with either 5 or 10 surface inches of water when the water was applied in 0.25 inch increments applied 30 minutes apart versus 1.0 inch increments applied 30 minutes apart. Increased volume also increased movement. Davidson and Santelmann (9) found the rate at which fluometuron moved through a water saturated glass bead or uniformly packed soil column was a function of the water flux or average pore velocity. Rogers (25) used leaching columns to study the influence of various frequencies and rates of simulated rainfall on the leaching of four triazines. He found a given quantity of simulated rainfall applied in 1 week was more effective in leaching a given herbicide than the same amount applied in 2 to 4 weeks. Upchurch et al. (34) found that the movement of monuron in soil columns was little effected by antecedent soil moisture.

The movement of a herbicide through the soil profile may be influenced by the compound's adsorption, desorption and solubility characteristics. Talbert and Fletchall (29) and Harris (18) have shown prometryn to be strongly adsorbed to the soil particles. Harris suggests that the SCH_3 radical in prometryn influences the electron density of the molecule to cause strong binding forces between herbicide and soil particles. In an upward movement leaching column study, prometryn remained predominantly in the lower 5 cm with all soil types used. In another study Harris (19) found prometryn to have low mobility, compared to the substituted ureas such as fluometuron. Talbert et al. (30) reported that prometryn was leached to a greater depth in a coarse textured soil than in a fine-textured clay soil when a leaching column

study was conducted. Different amounts of applied water had little effect on depth of leaching.

The movement of a herbicide within a soil profile owing to its adsorption-desorption characteristics is reported by Davidson et al. (10), and Davidson and McDougal (11), and Hornsby and Davidson (21). A nonsingularity between adsorption and desorption of several herbicides is reported. This nonsingularity may cause a tailing effect or the absorbed concentration of herbicides behind the invading herbicide front to remain quite high. Davidson and McDougal (11) also reported prometryn to be more strongly adsorbed than fluometuron.

Past research on herbicide runoff, generally has varied widely in both methods and results. Very little information is available on fluometuron or prometryn, two widely used cotton herbicides, under realistic field conditions. Very little research has been conducted to determine some environmental factors that effect herbicide runoff in the field. Most of the research of this type has been greenhouse work. In addition, little research is available on the effect of environmental conditions on the movement of fluometuron and prometryn under field conditions. This field research was conducted to determine the effect of rainfall frequency, antecedent soil moisture conditions, and rainfall subsequent to treatment on fluometuron and prometryn runoff and leaching.

TABLE I
COMMON AND CHEMICAL NAMES OF PESTICIDES

Common Names	Chemical Names
aldrin	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4-endo, exo-5,8-dimethanonaphthalene
atrazine	2-chloro-4-ethylamino-6-isopropylamino-s-triazine
DDT	dichlorodiphenyltrichloroethane
diazinon	0,0-diethyl 0-2(2-isopropyl-4-methyl-6-pyrimidinyl) phosphorothioate
dicamba	2-methoxy-3,6-dichlorobenzoic acid
dieldrin	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo, exo-5,8-dimethanonaphthalene
diphenamid	N,N-dimethyl-2,2-diphenylacetamide
diuron	3,(3,4 dichlorophenyl)-1,1-dimethylurea
endosulfan	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a,-hexahydro-6,9-methano-2,4,3-benzodioxathiepien-3-oxide
endrin	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo-endo-5,8-dimethanonaphthalene
fluometuron	1,1-dimethyl-3-($\alpha\alpha\alpha$, -trifluoro-m-tolyl) urea
methoxychlor	2,2-bis (P-methoxyphenyl)-1,1, 1-trichloroethane
monuron	3-(P-chlorophenyl)-1-,1-dimethylurea
picloram	4-amino-3,5,6-trichloropicolinic acid
prometryn	2-4-bis(isopropylamino)-6-methylmercaptos-triazine
propachlor	2-chloro-N-isopropylacetanilide
toxaphene	a mixture of octachloro camphene isomers
trifluralin	$\alpha\alpha\alpha$ -trifluoro-2,6,dinitro-N,N-dipropyl-P-toluidine
2,4-D	(2,4-dichlorophenoxy) acetic acid
2,4,5-T	(2,4,5-trichlorophenoxy) acetic acid

CHAPTER III

MATERIALS AND METHODS

Field Experiments

Field experiments were conducted on the Oklahoma State University Agronomy farm near Perkins, in 1973. Specially constructed field plots were used to study the influence of rainfall frequency, antecedent soil moisture and subsequent rainfall on the movement of fluometuron and prometryn thru and across the soil. The dissipation of these herbicides during the growing season was also studied.

The experiments were conducted on Tellar sandy laom soil having the following characteristics: C.E.C. - 7.3 meq/100 gm, clay - 20%, silt - 20%, sand - 60%, organic matter - 1.2% and pH - 6.6. The average surface slope of the experimental area was 1%. Prior to the start of each runoff experiment, the desired area was plowed 15 cm deep and worked to good seedbed condition with a dish and spring-toothed harrow. Cotton (Gossypium hirsutum L. 'Westburn 70') was seeded in rows 1.02 m apart the day the plots were constructed.

All experimental plots were 1.52 X 4.57 m in size with 1.52 m between plots. Each plot was arranged so that a row of cotton and its corresponding wheel track occupied the center of the plot and ran the entire length of the plot. Each plot was bordered with stainless steel lawn edging that extended approximately 12 cm below the soil surface. A sheet metal catchment device 15 cm wide, 20 cm deep and 1.5 m long was

installed at the lower end of the plot such that the opening extended the full width of the plot. A catchment of this type was chosen to minimize lateral flow and possible accelerated flow near the point of catchment. A sheet of plastic was attached to the full width of the catchment opening, extended out 10 cm in front of the catchment, and was buried into the soil. The soil was carefully sealed around the plastic. Thus, the last 10 cm of flow to the catchment was on plastic. This technique was found to be most effective in insuring that the runoff flowed into the catchment as opposed to washing under it. The 10 cm of plastic was not considered in the 4.57 m length of the plot. The catchment device was shielded to prevent direct entry of rainfall. After the installation of the border and catchment was complete, the plot was hand raked and smoothed around the inside border. The planter furrow and wheel track down the center of the plots were left intact. The catchment device was vacuummed prior to runoff to remove soils that could have entered during installation.

Herbicides were applied to the plots with a calibrated tractor-mounted plot sprayer. The boom extended across the plot while the tractor was driven outside the plot. The herbicides were applied in a spray volume of 374 l/ha. The 80% wettable powder commercial formulation of both herbicides was used throughout the studies.

Immediately after herbicide application, soil samples were taken for soil moisture and initial herbicide concentration determinations. Soil moisture samples were taken from the 0-8, 8-15, and 15-30 cm depths. Five soil samples for initial herbicide concentrations were taken from the 0-5 cm depth.

Simulated rainfall was applied with Sears-Roebuck and Company

oscillating lawn sprinklers. The output of these sprinklers was approximately 1.2 cm/hr if continuously operated. This particular sprinkler was chosen for its uniformity of coverage and output. One sprinkler was used to cover three plots in each experiment unless otherwise indicated.

The different variations in the rainfall application will be presented with the individual experiments. However, in all experiments where runoff was studied, water was applied to each plot until a total of 45.4 liters of runoff were obtained. This represents approximately 1 cm or $\frac{1}{4}$ inch of runoff. The total amount of simulated rainfall applied to each plot will be presented in the results. The first 3.8 liters of runoff was collected from each plot and set aside. The next 41.6 liters of runoff were composited in a stainless steel barrel. A one liter sample was taken from the first 3.8 liter of runoff. The 41.6 liter contents in each barrel was thoroughly stirred, and a one liter sample taken. This sample was to serve a two-fold purpose. First, the herbicide content of the water was determined. Second, by weighing the amount of sediment in the 1 liter sample the total amount of sediment removed from the plot was calculated. After the water sample was taken from each barrel, the suspended clays were flocculated by adding approximately 200 gm CaCl_2 . Upon flocculating sediment samples were collected from each barrel for freezing and analysis. Prior to analysis, water samples were filtered through one sheet of Whatman 42 filter paper. The sediment obtained was oven dried and weighed. The amount of sediment lost per plot was calculated from these weights. Sediment samples collected for analysis were air dried and ground prior to analysis.

The previous procedures apply to all runoff studies. Individual studies were conducted to determine the effect of rainfall frequency antecedent soil moisture and subsequent rainfall on runoff and leaching of prometryn and fluometuron. The methods unique to each experiment are presented below.

The Effect of Rainfall Frequency

The plots were constructed and three relative frequencies of rainfall were simulated - designated fast, intermediate and slow. Each frequency was duplicated.

Fast: Water was applied at a rate of 1.25 cm/hr until all runoff samples were obtained.

Intermediate: Water was applied in 0.6 cm aliquots until 2.5 cm had been applied. After a 3 hr. period of infiltration, water was applied at 1.25 cm/hr until all runoff was obtained.

Slow: Water was applied in 0.3 cm aliquots, over a 4 day period, until 6.3 cm total water had been applied. No lateral runoff was allowed to occur.

Two days after water application, 0-5, 5-10 and 10-20 cm deep, soil samples were taken from each plot to determine the vertical movement of each herbicide. The initial herbicide rates applied in this experiment was 2.8 kg/ha for both fluometuron and prometryn.

The Effect of Antecedent Soil Moisture

Two antecedent soil moisture conditions wet and dry were studied on duplicated plots. The plot area was dried by tillage prior to

constructing the plots. Plots to be designated dry were covered, and the remaining plots received 4.5 cm of rainfall over a two day period. The plots were then allowed to dry for 24 hours and the herbicides were applied at a rate of 2.2 kg/ha. Simulated rainfall was then applied to all plots at a rate of 1.25 cm/hr until all runoff was obtained. Two days subsequent to runoff, soil samples were taken from 0-5, 5-10 and 10-20 cm depths to determine the movement through the profile.

The Effect of Subsequent Rainfall

To determine the effect of rainfall subsequent to treatment on herbicide runoff and leaching, one subsequent simulated rainfall and one subsequent natural rainfall was studied in duplicated plots. Forty days subsequent to treatment of the dry antecedent soil moisture plots, they received a second simulated rainfall. Soil samples were taken immediately prior to this rainfall application to determine the herbicide concentration in the profile at this time. Another set of samples were taken subsequent to the water application to determine any further vertical movement due to the second simulated rainfall. Water and sediment samples were collected from the second runoff as previously described. Runoff water was obtained from a natural rainfall 10 days subsequent to the second simulated rainfall. No sediment was obtained from this sampling.

Dissipation & Movement During the Growing Season

The dissipation and movement through the profile of prometryn and fluometuron during the cotton growing season was studied by sampling

the soil at various times during an eighty-four day period subsequent to the herbicide application. The plots studied were those from the rainfall frequency experiment. However, some additional information was obtained from the samples taken during the subsequent rainfall experiment.

The first sampling date was immediately subsequent to the runoff study. As stated previously, the sampling depths were 0-5, 5-10 and 10-20 cm. In addition, soil samples 24 and 84 days subsequent to the first date were also taken. The sampling depths for these dates were 0-5, 5-10, 10-20 and 20-30 cm.

Analytical Methods

Chemical analytical methods and bioassays were used to determine the concentration of herbicide in the water and soil samples. The chemical methods were used as the primary method with the bioassays used as a secondary or check method.

Fluometuron Chemical Analysis

The basic principle for the analysis of free and bound fluometuron and its metabolites is hydrolysis to 3-trifluoromethylaniline by strong alkali. The hydrolysis product is extracted by steam distillation into isooctane. The extracted aniline is diazotized and coupled with N-ethyl-1-naphthyl-amine to produce a colored compound. The concentration of the colored compound may be determined spectrophotometrically. A Bliedner distillation extraction head (5) modified by Heizler (8) is required for this procedure. The basic procedure is taken from Ciba-Geigy Res. Bulletin No. CF-R5 (8).

For water analysis, a 100 ml sample of filtered water is placed in a 1000 ml heavy wall boiling flask, and 300 ml of 5N sodium hydroxide were added. Several boiling chips (Hengar granules) and a small amount of Antifoam-A spray were added to the flask to inhibit foam formation during hydrolysis. The strong alkali will etch the walls of several brands of boiling flasks. Labglass and Kimax brand flasks were found to be suitable for prolonged use. The flask were placed in a 1000 ml heating mantle and are ready for attachment to the hydrolysis side of the distillation extraction head. The distillation extracts head was prepared by filling with water and isooctane (8). The 1000 ml flask was connected to the hydrolysis side and a 250 or 500 ml flask containing 100 ml of isooctane (A.C.S. reagent grade) was connected to the extraction side of the distillation-extraction head. The isooctane flask was also fitted with a heating mantle of the appropriate size. The distillation-extraction head must be fitted with a condenser. The coiled type condenser such as the Graham were found to be unsuitable for this. A Liebig or Ahllin condenser worked quite well. The flasks were heated to boiling and maintained at the proper temperature with a variable voltage transformer (Variac). A separate Variac must be used for the hydrolysis flask and the isooctane flask. The proper mantle temperature was determined by observing the "solvent sausages" which are continuously formed in the feeding capillary of the head. The "solvent sausages" were to be maintained at a nearly uniform size by adjusting the heat of one or both of the flasks by altering the Variac setting. A voltage of 100 volts on the hydrolysis flask and 70 volts on the isooctane flask was a good starting point. Once the flasks reached the proper temperature, continuous distillation-extraction was

continued for 4 hours.

After completing the distillation-extraction, the isooctane was cooled and transferred to a 250 ml separatory funnel. The isooctane was extracted three times with 10 ml quantities of 1.0 N HCl and adjusted to a final volume of 50 ml with 1.0 N HCl. The HCl extract was transferred to a 125 ml Erlenmeyer flask. A 2 ml aliquot of 2.0% sodium nitrite, aqueous solution, was added and allowed to stand ten minutes. To decompose the excess sodium nitrite, 2.0 ml of 10% sulfamic acid, aqueous solution, was added to the flask and vigorously shaken. After ten minutes, 2.0 ml of 1% N-ethyl-1-naphthylamine absolute ethanolic solution was added to the flask and allowed to stand 30 minutes. It was essential that the sodium nitrite, sulfuric acid and N-ethyl-1-naphthylamine solutions be prepared fresh daily.

After the flasks had stood 30 minutes a pink color developed if any fluometuron was present. The solution was transferred to a 250 ml separatory funnel and extracted with 20 ml of N-butanol. The colored butanol extract was transferred to a test tube and 10 g anhydrous sodium sulfate granules were added to remove the water. The sample was transferred to a 1 or 4 cm cuvette, depending on the color intensity, and quantified on a Beckman spectrophotometer at 525 nm. The sample was read against a N-butanol reference. Since no clean-up procedure was used in this procedure, several samples containing untreated water were analyzed to obtain a correction for any color not due to fluometuron or its metabolites. Each sample was determined in duplicate.

Standards were prepared by spiking samples of water obtained from check plots with analytical fluometuron. A standard curve ranging from 0.01 to 0.6 ppm was used. All standard determinations were made in a

4 cm cuvette.

Soil samples were air dried and ground to pass a 1.0 mm screen. A 50 g or 25 g sample was taken for analysis depending on the herbicide concentration present. The soil sample is added to a 1000 ml boiling flask and the same procedure outlined for water was followed. Soil standards were prepared by spiking soil samples with analytical fluometuron. The herbicide spike was added to enough water to bring the soil to 10% soil moisture by weight. The sample was mixed thoroughly, allowed to dry, and screened through a 1 mm screen. The standard curve was from 0.1 to 6.0 ppm.

The data for fluometuron water and soil standards are presented as corrected absorbance, from the spectrophotometer, versus the concentration of fluometuron present (Table II). These standard readings were utilized to determine the fluometuron concentrations in all field samples. In order to determine the dependability of the assay method, three water and three soil standards containing different concentrations of fluometuron were analyzed to determine the standard deviation at each concentration. Six replicate samples were analyzed at each concentration. For the water analysis method, the standard deviations were determined to be ± 0.002 at $.05 \mu\text{g/ml}$, ± 0.005 at $0.1 \mu\text{g/ml}$ and ± 0.03 at $0.4 \mu\text{g/ml}$. For the soil analysis, the standard deviations were less than ± 0.05 at $0.4 \mu\text{g/g}$, ± 0.07 at $2 \mu\text{g/ml}$ and ± 0.45 at $6 \mu\text{g/g}$. These standard deviations apply to all chemical analysis data presented.

Prometryn Chemical Analysis

The principle of the method involved extraction of the prometryn

TABLE II

ABSORBANCE OF LIGHT AS COMPARED WITH THE CONCENTRATIONS
OF FLUOMETURON EXTRACTED FROM
WATER AND SOIL

Water			Soil		
Fluometuron Conc. ($\mu\text{g/ml}$).	μg Fluometuron	Absorbance ^{1/} @ 525 nm.	Fluometuron Conc. ($\mu\text{g/g}$)	μg Fluometuron	Absorbance ^{2/} @ 525 nm.
.01	1	0.032	0.2	5	0.03
.02	2	0.060	0.4	10	0.06
.05	5	0.165	1.0	25	0.17
.10	10	0.320	2.0	50	0.35
.20	20	0.620	4.0	100	0.68
.40	40	1.240	8.0	200	1.37

^{1/}Water read in a 4 cm cuvette, soil in a 1 cm cuvette.

^{2/}Means are an average of 2 replications.

from the soil or water, the extract evaporated to dryness, redissolving in carbon tetrachloride, cleaning, and conversion of the prometryn to hydroxytriazine by acid treatment. Quantitative measurements are made spectrophotometrically in the ultraviolet region. The basic procedure is from Ciba-Geigy analytical Bulletin No. 10 (1).

Prometryn was extracted from water with methylene chloride. A 100 ml sample of filtered water was placed in a 250 ml separatory funnel equipped with a teflon stopcock. The water was extracted with 25 ml methylene chloride and the methylene chloride was filtered through a 25 g pad of anhydrous sodium sulfate into a 250 ml Erlenmeyer flask equipped with a 24/40 joint. The extraction was repeated with 25 ml of methylene chloride and passed through the same sodium sulfate pad. The sodium sulfate pad was then washed with 25 ml methylene chloride into the same Erlenmeyer flask. This combined extract was then evaporated on a rotary evaporation with the water bath at 45°C.

The columns used for clean-up were 18 mm I.D. X 400 mm with a fritted disk in the bottom. The aluminum oxide used in the columns was Woeln basic alumina (ICN Pharmaceuticals, Cleveland, Ohio). The activity grade 4.4 aluminum oxide was prepared by mixing 88 g of the activity grade I aluminum oxide with 12 ml of distilled water. The water and alumina were mixed thoroughly and allowed to stand overnight in a tightly closed bottle. The dry packed column was prepared by placing a small glass wool plug in the bottom of the column. Twenty five (25) grams of alumina (activity 4.4) were added to the column and lightly tapped to eliminate channeling. A small plug of glass wool was carefully placed on top of the alumina.

The sample residue from above was dissolved in 10 ml of carbon

tetrachloride, transferred to the column and allowed to penetrate the alumina. The flask was washed with 10 ml carbon tetrachloride, transferred to the column and allowed to penetrate as before. This was repeated with 5 ml and then 80 ml of carbon tetrachloride. When the last of the 80 ml of solvent had penetrated the column, a clean 250 ml Erlenmeyer (with 24/40 joint) was placed as a receiver and 100 ml of 5% diethyl ether in carbon tetrachloride was added. The total 100 ml of eluant solution was collected for analysis. The ether-carbon tetrachloride eluate was evaporated to approximately 15 ml on a rotary evaporator with the bath at 45°C. The vacuum in this laboratory was found to be inadequate to evaporate carbon tetrachloride and other solvents at low water bath temperatures. Vacuum produced by a water aspirator corrected this problem. The 15 ml evaporated eluate was placed in a special test tube made from a 24/40 outer joint. This tube was placed in a hot water bath (50°C), and the solution was evaporated to dryness using a gentle stream of clean, dry air. Upon reaching dryness, the residue was redissolved by adding 10 ml of 1.0 N H₂SO₄ to the tube. An air condenser, made by joining 7 mm glass tubing to a 24/40 inner joint (30 cm total length) was fitted on the tube, and the water bath heated to boiling. The tube remained in the boiling water bath for 3 hr to insure complete conversion to hydroxy triazine. The tube was then cooled to room temperature, and the solution was transferred to a 125 ml separatory funnel equipped with a teflon stopcock. The solution was washed with 25 ml of 20% diethyl ether in chloroform. The aqueous layer was transferred to a second separatory funnel and was washed by shaking with 25 ml of diethyl ether. The aqueous layer was transferred to a clean test tube. It has been

reported (1) that some kinds of diethyl ether give high reagent blanks and must be washed with H_2SO_4 . In this research, Mallinkrodt (No. 3434) Nanograde diethyl ether gave excellent results with no washing necessary.

A reagent blank was prepared by washing 10 ml of 1.0 N H_2SO_4 with the 25 ml of 20% diethyl ether in chloroform followed with 25 ml of diethyl ether as above. The aqueous solution from above was transferred to a 1 or 4 cm silica cell. The sample was read against a reagent blank in a Beckman spectrophotometer. The absorbance was determined at 225, 240 and 255 nm. The net absorbance (E) was determined at 240 nm using a baseline technique according to the equation:
$$E = A^{240} \left(\frac{A^{225} + A^{255}}{2} \right)$$
. This E value was then corrected for check samples. This prometryn procedure is very accurate, but it is extremely sensitive to impurities due to its determination in the uv. spectrum. All glassware must be scrupulously clean, and high purity chemicals must be used throughout the determination. Teflon stopcocks were used in all separatory funnels to avoid contamination by stopcock grease. Clean silica cells were an absolute necessity for reproducible results. The cells were cleaned, filled with distilled water, and placed in the spectrophotometer. The instrument was then properly zeroed at 240 nm. By scanning the wavelength from 220 to 240 to 260 nm, the cells were determined to be dirty if a needle deflection of ± 0.005 was noted. If the deflection exceeded 0.005 absorbance the cells were recleaned and the procedure repeated. When the cells were determined clean with distilled water, the cells were filled with reagent blank and the scanning repeated.

Prometryn standards were prepared by dissolving prometryn in

methylene chloride. The methylene chloride was evaporated to dryness and the residue carried through the complete procedure. A plot of corrected E against μg of prometryn was prepared. By running spiked water samples in an identical manner, the percentage of recovery of prometryn from water was obtained.

Prometryn extraction from soil was done at reflux temperature using 10% water-acetonitrile as the solvent (22). A 100 g soil sample (air dried and ground to pass a 1 mm mesh screen) was placed in a 500 ml boiling flask fitted with a 24/40 joint. Three hundred (300) ml of 10% water-acetonitrile (v/v) was added to the flask, and the flask was fitted with a condenser. The mixture was heated to reflux with a heating mantle and maintained at reflux temperature for 1 hr. Upon cooling the supernatant was filtered through two kinds of filter paper. A sheet of Reeves-Angel Grade 802 (32 cm) was placed in a long stem funnel followed by a sheet of Whatman 2V (32 cm) paper. The supernatant was poured into the funnel, collected, and mixed. A 75 ml aliquot (equivalent to 25 gm of soil) was transferred to a 1000 ml separatory funnel equipped with a teflon stopcock. The aliquot was diluted with 700 ml distilled water, and 20 ml of saturated sodium sulfate solution was added. This solution was extracted with 50 ml of methylene chloride. The phases were allowed to separate, and the methylene chloride was filtered through a 30 g pad of anhydrous sodium sulfate into a 250 ml Erlenmeyer flask equipped with a 24/40 joint. The extraction was repeated with 50 ml methylene chloride and filtered through the same sodium sulfate pad. The sodium sulfate pad was washed with 25 ml methylene chloride into the flask. The solvent was evaporated to dryness on a rotary evaporation with the bath temperature

of 45°C. The sample was then carried through the previously outlined procedure.

Soil standards were prepared by spiking soil samples with analytical prometryn in a 10% ethanol-water solution. The percentage of recovery was determined by comparing with the prometryn analytical standard dissolved in methylene chloride.

Prepared analytical standards in methylene chloride, water and soil were analyzed for prometryn over a range of concentrations. By comparing the corrected readings obtained from the methylene chloride standards with the readings obtained from the extracted water standards (Table III), the percent recovery of prometryn from water was calculated to be 90 %. By comparing the corrected readings from the extracted soil standards (Table IV) with the readings from the methylene chloride standards, the recovery of prometryn from soil was calculated to be 88 %. All unknown determinations from soil and water were made from the methylene chloride standard curve and corrected for their respective recovery percentage. Standard deviations were determined for six replicate samples at two concentrations. The standard deviation was ± 0.006 at 0.1 $\mu\text{g/g}$ and ± 0.2 at 2.0 $\mu\text{g/g}$. These standard deviations apply to all chemical analysis data presented.

Bioassay Analysis

As a back up for the chemical analysis for both water and soil, bioassays were conducted. Soil samples were bioassayed by placing 225 g soil in styrofoam cups and planting to oats. The oats were grown under continuous light for a period of 14 days in most cases. In soils known to have a high herbicide concentration, the studies were

TABLE III
 ABSORBANCE VERSUS CONCENTRATION FOR
 TECHNICAL PROMETRYN STANDARDS

$\mu\text{g Prometryn}^{1/}$	$E^{2/}$	
	CH_2Cl_2	Water ^{3/}
5	$0.047^{4/}$	0.042
10	0.087	0.078
20	0.165	0.160
50	0.430	0.380
100	0.880	0.790
200	1.75	1.50

^{1/} $\mu\text{g prometryn per 100 ml methylene chloride solution as 100 ml water solution.}$

^{2/} Background corrected $E = A_{240} - \frac{A_{225} + A_{225}}{2} - E_{\text{check}}$ where A = absorbance.

^{3/} Water samples were extracted with CH_2Cl_2 and analyzed concurrent with the CH_2Cl_2 standards.

^{4/} All means are an average of 2 replications.

TABLE IV
 ABSORBANCE VERSUS THE CONCENTRATION OF PROMETRYN
 EXTRACTED FROM SOIL STANDARDS

μg Prometryn ^{1/}	Sample Size	Conc. $\mu\text{g/g}$	E ^{2/}
13.3	33.3g	0.4	0.104 ^{3/}
33.3	33.3g	1.0	0.243
66.6	33.3g	2.0	0.50
133.2	33.3g	4.0	1.02

^{1/} μg of prometryn in the soil sample extract taken for analysis.

^{2/} Background corrected $E = A_{240} - \frac{A_{225} + A_{225}}{2} - E \text{ Check}$, where $A =$ absorbance.

^{3/} All means are an average of 2 replications.

terminated at 12 days. Soil standards were prepared from soil taken from the plot area. The soil standards were spiked with a known amount of herbicide in a carrier volume of water sufficient to bring the soil up to 10% moisture by weight. The soil and herbicide solution was thoroughly mixed, allowed to dry, and screened to pass a 1 mm screen. A standard curve was conducted with each bioassay. Upon termination of the studies, the plants were harvested and fresh weights were recorded. The data are expressed as μg prometryn per gram of soil.

Water samples were analyzed using a floating disc technique described by Truelove et al. (32). Pumpkin (Cucurbita pepo L 'Big Tom') seeds were planted in vermiculite and grown under continuous light for 10 days. The cotyledons were excised and cut into discs with a No. 2 cork cutter. Each disc was then halved and transferred to a beaker of distilled water. A 100 ml sample of water to be tested was placed in a 250 ml beaker and 1 ml each of a 1 M potassium phosphate buffer pH 6.5 and a 1% solution of Triton X-207 surfactant was added. Fifty of the half discs were then transferred to each beaker of water herbicide solution. All treatments and standards were replicated three times. The beakers were covered with petri dish lids, and transferred to a lighted, oscillating platform at 25°C. At regular time intervals, shaking was halted and the number of half-discs which had sunk were recorded.

CHAPTER IV

RESULTS AND DISCUSSION

Fluometuron

The Effect of Rainfall Frequency on Fluometuron Runoff and Leaching

Studies were conducted to determine the effect of rainfall frequency on fluometuron loss in surface runoff. The data for this study and all the field studies to follow are presented as individual plots. Plots for each treatment were duplicated but the data were not averaged. Due to differences in water amounts required to obtain runoff, slight differences in border and catchment installation and a one day interval in water applications to the two duplicate plots, they were not considered replications. As a result, it was felt the data from each individual plot should be presented alone.

The runoff losses are expressed as concentration of fluometuron (Table V). To calculate the amount of herbicide actually lost from the plot, these data were converted to a percent of the actual fluometuron applied. Rainfall frequency had little effect on fluometuron runoff. In general, the first 3.8ℓ of runoff water contained a higher concentration of fluometuron than did the composite of the next 41.6ℓ; indicating the fluometuron concentration decreased with the increased volume of runoff water. The sediment concentrations of

TABLE V
 THE EFFECT OF RAINFALL FREQUENCY ON THE CONCENTRATION
 AND THE PERCENT OF THE APPLIED FLUOMETURON
 IN RUNOFF WATER AND SEDIMENT

Rainfall Frequency	Initial ^{1/} Concentration	Water				Sediment		Total % Loss
		3.8ℓ ^{2/}		3.8-45.4ℓ		μg/ml	%	
		μg/ml	%	μg/ml	%	μg/ml	%	
Fast	5.7	0.12	.02	0.12	.16	4.1	.02	.20
Fast	4.1	0.10	.02	0.06	.12	2.8	.01	.15
Intermediate	5.7	0.09	.01	0.03	.04	2.9	.01	.06
Intermediate	4.1	0.09	.01	0.05	.09	2.3	.01	.11

^{1/}The concentration of fluometuron (μg/g) chemically determined to be in the top 5 cm of soil sampled immediately after herbicide application.

^{2/}The concentration and percent loss of fluometuron in the first 3.8ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of fluometuron in the next 41.6ℓ composite of runoff water.

fluometuron were much higher than the water concentrations. The sediment fraction consisted primarily of suspended clays. The fluometuron concentration ratio between the water and sediment was higher than would be obtained by adsorption isotherm data (21). However, it has been shown by Hornsby and Davidson (21) that adsorption and desorption of fluometuron are not single valued. That is, once adsorbed the herbicide does not desorb as easily. Most likely, the fluometuron was adsorbed to the soil particles while they were still on the plot. Once the soil particles were suspended in runoff, the herbicide evidently did not desorb back into the water solution readily. The relationship between the herbicide and sediment during runoff appears to be more closely related to desorption than adsorption. The greatest amount of herbicide was lost in the 41.6% composite fraction of water due to the larger amount of total water lost. Although the sediment concentrations were quite high, very little herbicide was lost because very little sediment was removed. In this experiment, all individual fluometuron losses were less than one-fourth of one percent of that applied.

Data for the chemical and bioassay analyses of vertical fluometuron movement thru the soil are presented in Tables VI and VII respectively. In both tables, the data are presented as $\mu\text{g/g}$ of fluometuron present in samples taken from three soil depths. The coefficient of variation in the bioassay analysis was 21%. In general, the bioassay analysis indicated a lesser concentration of fluometuron present than did the chemical analysis method. A partial explanation will be presented later in the discussion. The general trends shown by the two assay methods were the same. A trend toward increased movement with increased

TABLE VI
 CHEMICAL ANALYSIS OF FLUOMETURON LEACHING TO DIFFERENT
 SOIL DEPTHS AS INFLUENCED BY RAINFALL FREQUENCY

Rainfall Frequency	Rainfall Amount (cm)	Initial Concentration($\mu\text{g/g}$)	Soil Depth (cm)		
			0-5	5-10	10-20
Fast	7.5	5.7 ^{1/}	3.0 ^{2/}	1.5	0.8
Fast	6.3	4.1	1.7	1.2	0
Intermediate	6.3	5.7	2.8	0.6	0
Intermediate	5.6	4.1	2.1	0.9	0
Slow	6.3	3.5	2.3	0.4	0
Slow	6.3	4.5	2.8	1.1	0

^{1/} Fluometuron concentration ($\mu\text{g/g}$) immediately after application, in the top 5 cm of the profile.

^{2/} Fluometuron concentration expressed as $\mu\text{g/g}$ of soil.

TABLE VII
 BIOASSAY ANALYSIS OF FLUOMETURON LEACHING TO DIFFERENT
 SOIL DEPTHS AS INFLUENCED BY RAINFALL FREQUENCY

Rainfall Frequency	Rainfall Amount (cm)	Initial Concentration($\mu\text{g/g}$)	Soil Depth (cm)		
			0-5	5-10	10-20
Fast	7.5	4.0 ^{1/}	2.2 ^{2/}	1.9	0.5
Fast	6.3	2.5	1.5	1.4	0
Intermediate	6.3	4.0	2.3	1.0	0
Intermediate	5.6	2.9	1.0	1.1	0
Slow	6.3	3.2	1.3	<.5	0
Slow	6.3	4.1	2.4	1.4	0
C.V. = 21%					
Std. Dev. = 0.3					

^{1/} Fluometuron concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/} Fluometuron concentration expressed as $\mu\text{g/g}$ of soil.

rainfall frequency was present. However, this trend was not well defined. The deeper movement in one replication at the fast intensity could be due to more water being applied to the plot. Water was applied until 1 cm ($\frac{1}{4}$ inch) of runoff was obtained, and this amount varied among plots. At the fast frequency it may also have been possible to exceed equilibrium adsorption; causing the deeper movement. Deeper fluometuron movement into a soil column at high water flow rates has been reported by Hornsby and Davidson (21). At the slow rainfall frequency the rainfall was applied over a three day period, and some water was lost due to evaporation. As a result, less water was available for movement through the profile. With all treatments, fluometuron moved into the 5-10 cm soil depth.

The Effect of Antecedent Soil Moisture on Fluometuron Runoff and Leaching

Antecedent soil moisture influenced both the concentration of fluometuron in the runoff and the percent of the applied fluometuron lost in the runoff (Table VIII). A lower initial concentration of fluometuron was applied to the plots in this experiment compared to the rainfall frequency experiment. As in the rainfall frequency study, the runoff from the dry plots contained very low herbicide concentrations in the water, but a higher concentration was found on the sediment. The runoff losses from the dry plots were only 0.10 percent. However, when fluometuron was applied to a wet plot and irrigated, the runoff losses were higher. The concentrations of fluometuron in the first 3.8 ℓ of water removed were slightly above 0.6 $\mu\text{g}/\text{ml}$, but the concentration decreased substantially in the next 41.6 ℓ of water. The herbicide

TABLE VIII

THE EFFECT OF ANTECEDENT SOIL MOISTURE ON THE CONCENTRATION
AND THE PERCENT OF THE APPLIED FLUOMETURON IN
RUNOFF WATER AND SEDIMENT

Antecedent Soil Moisture	Concentration ^{1/}	3.8 ℓ ^{2/}		3.8-45.4 ℓ ^{3/}		Sediment		Total % Loss
		$\mu\text{g/ml}$	%	$\mu\text{g/ml}$	%	$\mu\text{g/g}$	%	
Dry	2.2	.03	.01	.02	.07	1.9	0.2	.10
Dry	2.8	.09	.02	.04	.07	1.6	.01	.10
Wet	2.0	.61	.22	.15	.59	2.9	.02	.83
Wet	2.4	.65	.19	.27	.88	3.3	.02	1.09

^{1/}The concentration of fluometuron ($\mu\text{g/g}$) chemically determined to be in the top 5 cm of soil sampled immediately after herbicide application.

^{2/}The concentration and percent loss of fluometuron in the first 3.8 ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of fluometuron in the next 41.6 ℓ composite of runoff water.

concentration in the sediment was approximately twice those from the dry plots. The percent of the applied fluometuron lost was 0.83 and 1.09 % from the two wet plots. The wet plots required only one half the volume of simulated rainfall to obtain runoff compared to the dry plots. As a result, less herbicide moved into the soil profile. Because less herbicide moved into the profile, more was available at the surface to be removed by runoff. Although sediment concentrations were high, most of the herbicide was removed in the water portion of the runoff.

The chemical and bioassay analysis for fluometuron leaching are presented in Table IX. Rainfall was only applied to the plots until $\frac{1}{4}$ acre-inch of runoff was obtained, and less water was required on the wet plots. Both the chemical and bioassay analysis indicates that no herbicide moved into the 5-10 cm depth on the wet plots. The lesser amount of water applied would explain this. The coefficient of variation was 19% in the bioassay experiment. The limit of detectability for the bioassay was 0.5 $\mu\text{g/g}$. In general the chemical and bioassay analysis were in close agreement.

The Effect of Subsequent Rainfall on

Fluometuron Runoff and Leaching

The highest concentrations of fluometuron in the runoff and the highest percent losses resulted from the first rainfall that produced runoff (Table X). The initial simulated rainfall was applied July 13 and the second was applied August 21. In the interim period, 11.3 cm of natural rainfall occurred, but runoff samples were not collected. In general, the rainfall occurred in small amounts. Natural rainfall

TABLE IX
 CHEMICAL AND BIOASSAY ANALYSIS OF FLUOMETURON LEACHING
 TO DIFFERENT SOIL DEPTHS AS AFFECTED BY
 ANTECEDENT SOIL MOISTURE

Antecedent Soil Moisture	Rainfall Amount (cm)	Initial Concentration ^{1/}		Soil Depth (cm)			
		Chem	Bio	0-5		5-10	
				Chem	Bio	Chem	Bio
Dry	5.6	2.2	1.9	1.8	2.1 ^{2/}	0.2	<0.5
Dry	5.0	2.8	2.7	2.2	2.1	0.4	0.5
Wet	3.1	2.0	1.9	2.0	1.3	0.0	0.0
Wet	2.5	2.4	2.7	2.4	2.3	0.0	0.0

Bioassay C.V. = 19%

Bioassay Std. Dev. = 0.3

^{1/} Fluometuron concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm. of the profile.

^{2/} Fluometuron concentrations expressed as $\mu\text{g/g}$ of soil.

TABLE X

THE EFFECT OF SUBSEQUENT RAINFALL ON THE CONCENTRATION AND THE PERCENT
OF THE APPLIED FLUOMETURON IN RUNOFF WATER AND SEDIMENTS

Treatment	7/13 ^{1/}						8/21						9/4	Total % Loss
	Water			Sediment			Water			Sediment			3.8ℓ	
	3.8ℓ ^{2/} μg/ml	%	3.8-45.4ℓ ^{3/} μg/ml	%	μg/g	%	3.8ℓ μg/ml	%	3.8-45.4ℓ μg/ml	%	μg/g	%	3.8ℓ μg/ml	
Fluometuron	.03	.01	.02	.07	1.9	.02	.01	<.01	<.01	-	3.3	.01	<.01	0.11
Fluometuron	.09	.02	.04	.07	1.6	.01	<.01	-	<.01	-	2.5	.01	<.01	0.11

^{1/}The initial runoff producing simulated rainfall was applied 7/13, followed by another simulated rainfall 8/21, and a natural rainfall 9/4.

^{2/}The concentration and percent loss of fluometuron in the first 3.8ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of fluometuron in the next 41.6ℓ composite of runoff water.

for the summer is shown as Appendix Table XXIV. Fluometuron could not be detected in the water from runoff produced by the second simulated rainfall, but a quite high concentration was found in the sediment. The runoff producing rainfall on September 4 was natural, and it resulted in an undetectable concentration in the water. No sediment was collected. The two subsequent rainfalls resulted in an additional .01% of the applied fluometuron lost in the runoff. A total of 0.11% was accounted for from each plot due to runoff.

Chemical and bioassay analysis of fluometuron movement is presented in Table XI. The herbicide movement due to the first simulated rainfall is represented by the data for July 15 and shows that it moved into the 5-10 cm soil layer. The natural rainfall of 11.3 cm occurring between 7/15 and 8/21 resulted in no additional movement. However, the simulated rainfall applied on 8/21 resulted in a higher concentration of fluometuron moving into the 5-10 cm soil depth. Apparently the frequency and volume of the natural rainfall were not sufficient to cause movement into the deeper soil depths.

Bioassay Analysis of Water Samples

A set of analytical standards of fluometuron in water and two selected runoff water samples were bioassayed using a floating disc technique (32). The purpose of the bioassay was to evaluate the herbicide concentration in the runoff water by an alternate method. The data (Table XII) for the two runoff water samples indicated the concentration to be in close agreement with that determined chemically. As a result of this agreement, no further bioassays were conducted for water.

TABLE XI

CHEMICAL AND BIOASSAY ANALYSIS OF FLUOMETURON LEACHING TO DIFFERENT
SOIL DEPTHS AS INFLUENCED BY SUBSEQUENT RAINFALL

Initial Concentration ^{1/}		7/15 ^{2/}				8/21 ^{3/}				8/24 ^{4/}			
		0-5 cm		5-10 cm		0-5 cm		5-10 cm		0-5 cm		5-10 cm	
Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio
2.2	1.9	1.8	2.1	0.2	<.5	1.8	1.2	0.2	0.5	1.1	1.0	0.6	0.7
2.8	2.7	2.2	2.1	0.4	0.5	2.1	2.1	0.4	0.7	1.6	1.3	0.7	0.8

Bioassay C.V. = 19%

Bioassay Std. Dev. = 0.2

^{1/}Fluometuron concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/}Sampled after the initiated simulated rainfall of 5 cm.

^{3/}Natural rainfall totaling 11.3 cm occurred between 7/5 and 8/21.

^{4/}Simulated rainfall of 3.75 cm. applied on 8/21 and sampled 8/24.

TABLE XII
 BIOASSAY OF ANALYTICAL STANDARDS AND
 TWO SELECTED RUNOFF WATER SAMPLES
 CONTAINING FLUOMETURON

Treatment Standards	Conc. $\mu\text{g/ml}$	Percent sinking of half discs after 45 hours
1	0	4
2	.05	10
3	.10	34
4	.50	54
5	1.0	100
Water Samples ^{1/}		
1		20
2		82
C.V. = 21%		

^{1/}Water samples 1 and 2 were chemically determined to contain 0.09 and 0.65 $\mu\text{g/ml}$ respectively.

Fluometuron Dissipation and Movement over
an 84 Day Period

The plots for the rainfall frequency experiment were sampled on three dates, to determine the movement and dissipation of fluometuron. Data for the chemical and bioassay analysis of the samples are shown in Tables XIII and XIV. However, bioassay analysis was not conducted for the soil samples taken from the 20-30 cm depth. In general, the bioassay showed a lower concentration of fluometuron present than did the chemical analysis. Two possible explanations are offered: First, the chemical analysis will detect any breakdown product between trifluoromethyl aniline and the intact fluometuron molecule. Thus at the later sampling dates a less toxic breakdown product could be detected chemically but not biologically. Second, it is possible that the fluometuron was more available in the freshly prepared herbicide standards compared to field soils that had undergone several wetting and drying cycles. The standard soils were wetted and dried once after the herbicide was added to simulate field conditions. If the herbicide in the standards was more available, it would reflect as lower concentrations present in the field samples of unknown concentrations. Both analytical methods indicated the same trends. By the final sampling date, the herbicide was chemically shown to be in the 10-20 cm deep soil layer on all plots and the 20-30 cm zone on one plot. This was not indicated, in all cases, by the bioassay. However, most of the concentrations in the 10-20 cm depth were below the limit of detectibility of the bioassay. Although dissipation was slowly taking place, the fluometuron was still present in phytotoxic levels to oats

TABLE XIII

CHEMICAL DETERMINATION OF FLUOMETURON MOVEMENT AND DISSIPATION OVER AN
EIGHTY-FOUR DAY PERIOD

Initial Concentration ^{1/}	Initial Rainfall Frequency and amount (cm)	7/2			7/26				9/24			
		0-5	5-10	10-20	0-5	5-10	10-20	20-30	0-5	5-10	10-20	20-30
5.7	Fast-7.5	3.0 ^{3/}	1.5	0.8	3.0	1.5	0.8	0.3	2.6	1.9	1.3	0.3
4.1	Fast-6.3	1.7	1.2	0	1.5	0.9	0	0	1.6	1.1	0.2	0
5.7	Int-6.3	2.8	0.6	0	2.8	1.0	0	0	2.3	0.8	0.1	0
4.1	Int-5.6	2.1	0.9	0	2.3	0.6	0	0	1.4	0.8	0.1	0
3.5	Slow-6.3	2.3	0.4	0	2.3	0.8	0	0	1.7	0.9	0.2	0
4.5	Slow-6.3	2.8	1.1	0	2.1	0.6	0	0	1.4	1.1	0.2	0

^{1/} Fluometuron concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/} Applied immediately after herbicide application by simulated rainfall. For natural rainfall, see Appendix Table.

^{3/} Fluometuron concentrations in $\mu\text{g/g}$ of soil.

TABLE XIV

BIOASSAY DETERMINATION OF FLUOMETURON MOVEMENT AND DISSIPATION
OVER AN EIGHTY-FOUR DAY PERIOD

Initial Concentration ^{1/}	Initial Rainfall Frequency and amount (cm) ^{2/}	7/2			7/26			9/24		
		0-5	5-10	10-20	0-5	5-10	10-20	0-5	5-10	10-20
4.0	Fast-7.5	2.2 ^{3/}	1.9	0.5	1.1	0.7	0.5	0.9	0.7	0.6
2.5	Fast-6.3	1.5	1.4	0	0.8	0.5	0	0.8	0.5	0
4.0	Int-6.3	2.3	1.0	0	2.8	1.0	0	1.6	1.1	0
2.9	Int-5.6	1.0	1.1	0	1.6	0.9	0	1.0	1.0	0.5
3.2	Slow-6.3	1.3	<.5	0	1.3	0.4	0	0.6	0.7	0
4.1	Slow-6.3	2.4	1.4	0	2.6	1.6	0	1.6	0.9	0.4
C.V. = 19%										
Std. Dev. = 0.3										

^{1/} Fluometuron Concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/} Applied immediately after herbicide application by simulated rainfall. For natural rainfall, see Appendix Table XXIV.

^{3/} Fluometuron concentrations in $\mu\text{g/g}$ of soil.

eighty-four days after application

Prometryn

The Effect of Rainfall Frequency on

Prometryn Runoff and Leaching

Rainfall frequency had little effect on the concentration of prometryn in the runoff water or sediment (Table XV). However, a twofold difference between replications occurred. The fast and intermediate plots having the higher concentrations received the simulated rainfall one day subsequent to the other plots. During the one day delay, 0.6 cm of natural rainfall occurred. This delay period may have caused the difference between replicated plots. The first 3.8 ℓ contained a higher concentration of prometryn than did the next 41.6 ℓ . The sediment contained a much higher concentration of prometryn than did the water. The relative concentrations of prometryn on the sediment and in the water indicated the herbicide was adsorbed and little desorption back into the solution occurred. The concentrations of prometryn are comparable to the concentrations of fluometuron obtained in this study. However, a lower initial concentration of prometryn was indicated compared to fluometruon. When the data was converted to percent of the applied prometryn recovered from the runoff, the difference between replications was again evident. Although the prometryn concentration was lowest in the 41.6 ℓ composite, more herbicide was lost in this fraction of the runoff. The sediment only accounted for an .01% loss. The percent losses were from 0.25 to 0.51%. Of the total herbicide lost in runoff, over 90% was lost in the water portion. The percent losses for prometryn was somewhat higher

TABLE XV
 THE EFFECT OF RAINFALL FREQUENCY ON THE CONCENTRATION
 AND THE PERCENT OF THE APPLIED PROMETRYN IN
 RUNOFF WATER AND SEDIMENT

Rainfall Frequency	Initial Concentration ^{1/}	3.8 ℓ ^{2/}		3.8-45.4 ℓ ^{3/}		Sediment		Total % Loss
		$\mu\text{g/ml}$	%	$\mu\text{g/ml}$	%	$\mu\text{g/g}$	%	
Fast	3.5	0.23	.05	0.17	.38	3.0	.01	.44
Fast	3.5	0.11	.02	0.10	.22	1.7	.01	.25
Intermediate	3.3	0.24	.05	0.19	.45	3.5	.01	.51
Intermediate	3.3	0.10	.02	0.10	.24	1.8	.01	.27

^{1/}The concentration of prometryn ($\mu\text{g/g}$) chemically determined to be in the top 5 cm of soil sampled immediately after herbicide application.

^{2/}The concentration and percent loss of prometryn in the first 3.8 ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of prometryn in the next 41.6 ℓ composite of runoff water.

than the fluometuron losses in the same experiment. Apparently, a higher concentration of the less-mobile prometryn remained near the soil surface during infiltration. This could leave a higher concentration available for runoff losses.

Prometryn leaching, as affected by rainfall frequency, showed that no prometryn leached to the 5-10 cm soil depth in any plot (Table XVI). The bioassay analysis showed lesser concentrations of prometryn than did the chemical analysis. In the bioassay experiment, differences between higher rates of prometryn were difficult to distinguish because the higher concentrations (2, 3, and 4 $\mu\text{g/g}$) killed the plants.

The Effect of Antecedent Soil Moisture on Prometryn Runoff and Leaching

Antecedent soil moisture influenced the concentrations of prometryn in the runoff water and sediment (Table XVII). Higher concentrations of prometryn were present in the water and sediment analyzed from plots wetted before treatment. The wet plots also had a much higher percent of loss of prometryn. The percent losses ranged from 0.36 to 3.67%. As in the previous study, most of the total loss was in the water portion of the runoff. The concentration of prometryn on sediment from the dry plot was much lower than that of the wet plot. However, the percent losses were approximately equal because less sediment was removed from the antecedent wet plot. The highest prometryn concentrations in the runoff water occurred in the first 3.8 ℓ removed.

Antecedent soil moisture had no effect on prometryn leaching into the 5-10 cm soil layer (Table XVIII). Prometryn was not detected at this depth, either chemically or biologically. The prometryn

TABLE XVI
 CHEMICAL AND BIOASSAY ANALYSIS OF PROMETRYN LEACHING
 TO DIFFERENT SOIL DEPTHS AS INFLUENCED
 BY RAINFALL FREQUENCY

Rainfall Frequency	Rainfall Amount (cm)	Initial Concentration ^{1/}		Soil Depth (cm)			
		Chem	Bio	0-5		5-10	
				Chem	Bio	Chem	Bio
Fast	6.3	3.5	1.8	3.2	1.9 ^{2/}	0	0
Fast	5.6	3.5	2.4	3.5	2.4	0	0
Intermediate	6.3	3.3	1.8	3.0	1.9	0	0
Intermediate	5.6	3.3	1.9	3.0	1.9	0	0
Slow	6.3	3.8	2.0	3.2	1.8	0	0
Slow	6.3	3.8	1.9	3.2	1.9	0	0

Bioassay C.V. = 17%

Bioassay Std. Dev. = 0.2

^{1/} Prometryn concentration ($\mu\text{g/g}$), immediately after application,
 in the top 5 cm of the profile.

^{2/} Prometryn concentrations expressed as $\mu\text{g/g}$ of soil.

TABLE XVII

THE EFFECT OF ANTECEDENT SOIL MOISTURE ON THE
CONCENTRATION AND THE PERCENT OF THE APPLIED
PROMETRYN IN THE RUNOFF WATER AND SEDIMENT

Antecedent Soil Moisture	Initial Concentration ^{1/}	3.8ℓ ^{2/}		3.8-45.4ℓ ^{3/}		Sediment		Total % Loss
		μg/ml	%	μg/ml	%	μg/g	%	
Dry	2.0	.12	.04	.08	.31	2.4	.01	0.36
Dry	2.4	.16	.05	.10	.33	1.5	.02	0.39
Wet	2.0	1.32	.47	0.81	3.18	5.7	.02	3.67
Wet	2.9	1.35	.33	0.78	2.11	7.2	.01	2.75

^{1/}The concentration of prometryn (μg/g) chemically determined to be in the top 5 cm of soil sampled immediately after herbicide application.

^{2/}The concentration and percent loss of prometryn in the first 3.8ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of prometryn in the next 41.6ℓ composite of runoff water.

TABLE XVIII

CHEMICAL AND BIOASSAY ANALYSIS OF PROMETRYN LEACHING
TO DIFFERENT SOIL DEPTHS AS INFLUENCED
BY ANTECEDENT SOIL MOISTURE

Antecedent Soil Moisture	Rainfall Amount (cm)	Initial Concentration ^{1/}		Soil Depth (cm)			
		Chem	Bio	0-5		5-10	
		Chem	Bio	Chem	Bio	Chem	Bio
Dry	5.6	2.0	1.5	1.6	1.4 ^{2/}	0	0
Dry	5.0	2.4	1.6	1.7	1.5	0	0
Wet	3.3	2.0	1.5	1.4	1.4	0	0
Wet	2.5	2.9	1.7	1.9	1.6	0	0

Bioassay C.V. = 17%

Bioassay Std. Dev. = 0.2

^{1/} Prometryn concentration ($\mu\text{g/g}$), immediately after application,
in the top 5 cm of the profile.

^{2/} Prometryn concentrations expressed as $\mu\text{g/g}$ in soil.

concentrations indicated by bioassay were somewhat lower, but the indicated trends were the same.

The Effect of Subsequent Rainfall on
Prometryn Runoff and Leaching

The first runoff producing rainfall after herbicide application produced the highest concentrations and percent losses of prometryn in the runoff (Table XIX). No prometryn could be detected in the water from runoff sampled from a simulated rainfall on 8/21 and a natural rainfall on 9/4. The sediment from the 8/21 runoff had a moderate concentration of prometryn adsorbed to it. However, this only accounted for an additional 0.01% loss from the plot, and only 0.37 and 0.40% of the applied prometryn was accounted for in the runoff from all sampling dates. A prometryn concentration of 1.4 $\mu\text{g/g}$ remained in the top 5 cm of soil at the second simulated rainfall application date. Prometryn adsorbed to surface soil particles probably accounted for the high sediment concentration. However, essentially none of this herbicide was apparently available to go into the water solution.

The initial runoff producing rainfall and all subsequent rainfall occurring before 8/24 failed to leach prometryn into the 5-10 cm soil depth (Table XX). Both analytical methods indicate some dissipation during this period, but the herbicide remained at concentrations phototoxic to oats. The natural rainfall occurring between the simulated rainfalls on 7/13 and 8/21 totaled 11.3 cm.

Selected standards and runoff water samples were bioassayed using the technique described for fluometuron bioassay from water. The concentrations of prometryn determined by the bioassay in two selected

TABLE XIX

THE EFFECT OF SUBSEQUENT RAINFALL ON THE CONCENTRATION AND THE PERCENT
OF THE APPLIED PROMETRYN IN RUNOFF WATER AND SEDIMENT

Treatment	7/13 ^{1/}						8/21						Total % Loss	
	Water				Sediment		Water				Sediment			
	3.8ℓ ^{2/}		3.8-45.4ℓ ^{3/}		μg/g	%	3.8ℓ		3.8-45.4ℓ		9/4 3.8ℓ			
	μg/ml	%	μg/ml	%			μg/ml	%	μg/ml	%	μg/g	%		μg/ml
Prometryn	.12	.04	.08	.31	2.4	.01	<.01	-	<.01	-	2.1	.01	<.01	0.37
Prometryn	.16	.05	.10	.33	1.5	.01	<.01	-	<.01	-	1.4	.01	<.01	0.40

^{1/}The initial runoff producing simulated rainfall was applied 7/13, followed by another simulated rainfall 8/21, and a natural rainfall 9/4.

^{2/}The concentration and percent loss of Prometryn in the first 3.8ℓ of runoff removed from the plot.

^{3/}The concentration and percent loss of Prometryn in the next 41.6ℓ composite of runoff water.

TABLE XX

CHEMICAL AND BIOASSAY ANALYSIS OF PROMETRYN LEACHING TO DIFFERENT
SOIL DEPTHS AS INFLUENCED BY SUBSEQUENT RAINFALL

Initial Concentration ^{1/}		7/15 ^{2/}				8/21 ^{3/}				8/24 ^{4/}			
Chem	Bio	0-5 cm		5-10 cm		0-5 cm		5-10 cm		0-5 cm		5-10 cm	
Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio	Chem	Bio
2.0	1.5	1.6	1.4	0	0	1.4	0.9	0	0	1.4	1.0	0	0
2.4	1.6	1.7	1.5	0	0	1.4	1.0	0	0	1.4	0.9	0	0

Bioassay C.V. = 17%

Bioassay Std. Dev. = 0.2

^{1/}Prometryn concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/}Sampled after the initial simulated rainfall of 5 cm.

^{3/}Natural rainfall totaling 11.3 cm occurred between 7/15 and 8/21.

^{4/}Simulated rainfall of 3.75 cm applied on 8/21 and sampled 8/24.

water samples (Table XXI) were found to agree with the concentrations determined chemically. In this research, the pumpkin cotyledon discs were much slower to react than those described by Truelove (32). However, the method was found to be accurate for quantitating prometryn concentrations. The method was not as sensitive to small concentration differences as was the chemical assay technique.

Prometryn Dissipation and Movement over
an 84 Day Period

Soil samples were collected on three different dates during an 84 day period. Consistent with previous results, the bioassay indicated a lesser concentration present than did the chemical analysis (Tables XXII and XXIII). The herbicide never moved into the 5-10 cm soil depth in detectable concentrations during the period studied. Both analytical methods indicate some dissipation of prometryn during this period, but the chemical remained in phytotoxic concentrations after 84 days. The data indicates that more breakdown occurred between 7/2 and 7/26 than occurred between 7/26 and 8/24. This would be expected if first order reaction degradation is occurring. However, the lack of dissipation in August may partially be explained by the low rainfall for August (Appendix Table XXIV). Only 6.4 cm of rainfall occurred in August, and 5.7 cm of this rainfall occurred on the same day. As a result, August was very dry, and microbial degradation may have been retarded. The fact that the bioassay showed the herbicide concentrations after the first rainfall to be higher than the initial concentration on some plots was attributed to biological variation in the bioassay.

TABLE XXI
 BIOASSAY ANALYSIS OF ANALYTICAL STANDARDS
 AND TWO SELECTED RUNOFF WATER SAMPLES
 CONTAINING PROMETRYN

Treatment Standards	Conc. $\mu\text{g/ml}$	Percent sinking of half discs	
		32 hours	75 hours
1	0	0	0
2	0.1	0	40
3	0.5	18	86
4	1.0	68	100
5	2.0	86	100
Water Samples ^{1/}			
1	-	26	94
2	-	0	44
C.V. = 17%			

^{1/}Water samples 1 and 2 were chemically determined to contain 0.81 and 0.16 $\mu\text{g/ml}$ prometryn respectively.

TABLE XXII
 CHEMICAL DETERMINATION OF PROMETRYN MOVEMENT
 AND DISSIPATION OVER AN EIGHTY-
 FOUR DAY PERIOD

Initial Concentration ^{1/}	Initial Rainfall frequency and amount (cm)	7/2		7/26		9/24	
		0-5	5-10	0-5	5-10	0-5	5-10
3.5	Fast-6.3	3.2 ^{2/}	0	1.7	0	1.6	0
3.5	Fast-5.6	3.5	0	2.2	0	1.7	0
3.3	Int-6.3	3.0	0	1.7	0	1.4	0
3.3	Int-5.6	3.0	0	1.8	0	1.7	0
3.8	Slow-6.3	3.2	0	2.3	0	2.0	0
3.8	Slow-6.3	3.2	0	2.0	0	1.7	0

^{1/}Prometryn concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/}Prometryn concentration in $\mu\text{g/g}$ of soil.

TABLE XXIII
 BIOASSAY DETERMINATIONS OF PROMETRYN MOVEMENT AND
 DISSIPATION OVER AN EIGHTY-FOUR DAY PERIOD

Initial Concentration ^{1/}	Initial Rainfall Frequency and Amount (cm)	7/2		7/26		9/24	
		0-5	5-10	0-5	5-10	0-5	5-10
1.8	Fast-6.3	1.9 ^{2/}	0	1.0	0	1.1	0
2.4	Fast-5.6	3.0	0	1.4	0	1.2	0
1.8	Int.-6.3	1.9	0	1.0	0	1.1	0
1.9	Int.-5.6	1.9	0	1.0	0	1.3	0
2.0	Slow-6.3	1.8	0	1.1	0	1.4	0
1.9	Slow-6.3	1.8	0	1.3	0	1.2	0

C.V. = 17%

Std. Dev. = 0.2

^{1/} Prometryn concentration ($\mu\text{g/g}$), immediately after application, in the top 5 cm of the profile.

^{2/} Prometryn concentrations in $\mu\text{g/g}$ of soil.

CHAPTER V

SUMMARY

Field studies were conducted to determine the effect of specific environmental factors on fluometuron and prometryn runoff and leaching. Dissipation of herbicides was also studied.

The first 3.78% of water that ran off the treated plots contained a higher concentration of herbicide than did a composite of the next 41.6%. The sediment carried a much higher concentration of herbicide than did the runoff water. In terms of the total herbicide lost, however, most of the herbicide was lost in the liquid runoff portion because sediment removal from the plot area was slight. In general, prometryn losses were greater than fluometuron losses. However, in all experiments that were applied to a dry soil, fluometuron or prometryn losses were only 0.5% or less of the amount of herbicide originally applied. Fluometuron was easily leached into the 5-10 cm soil depth, and low concentrations were found at the 10-20 cm depth. Prometryn was never detected below 5 cm in the soil.

Rainfall frequency had little effect on herbicide runoff losses. Fluometuron leaching appeared to be influenced by rainfall frequency. Runoff losses were highest when the herbicides were applied to a wet rather than a dry soil. The highest runoff losses also occurred at the first runoff producing rainfall after herbicide application. One month subsequent to application, neither prometryn nor fluometuron

could be detected in the runoff water.

After 84 days, both herbicides remained in the soil at levels phytotoxic to oats used as a bioassay. However dissipation of the herbicides was occurring during this period. In general, bioassay analysis of the soils indicated lower concentrations of herbicide present than did the chemical analysis methods.

In this research, the runoff losses from the dry soil were considered to be very small. Further research is necessary to determine the extent of the losses under other conditions.

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APPENDIXES

TABLE XXIV
NATURAL RAINFALL DATA FOR JUNE THROUGH
SEPTEMBER, 1973

Date	Amount (cm)
6/5	6.45
6/14	0.48
6/18	2.26
6/30	0.56
7/10	1.78
7/11	1.10
7/25	1.65
7/26	0.51
7/28	2.97
8/9	5.74
8/30	0.48
9/2	1.09
9/4	3.23
9/5	1.14
9/6	0.30
9/8	1.83
9/13	1.37
9/17	0.46
9/22	1.02

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