

THE UNIVERSITY OF MANITOBA

THE HERBICIDAL ACTIVITY OF HALOXYDINE IN SOME MANITOBA SOILS

by

ALAN DUNCAN MCMILLAN

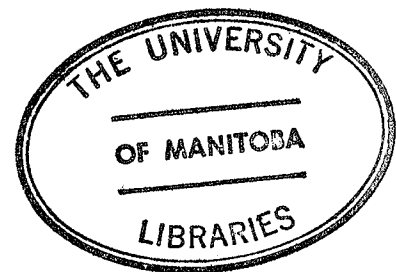
A THESIS

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A dissertation submitted to the Faculty of Graduate Studies of
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of the degree of

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ABSTRACT

McMillan, Alan Duncan, M.Sc. The University of Manitoba, May 1975. The herbicidal activity of haloxydine on some Manitoba soils. Advisor Dr. E. H. Stobbe, Department of Plant Science.

Haloxydine, an experimental herbicide was tested in the field for selective control of broad-leaved and grassy weeds in wheat Triticum aestivum L., barley Hordeum vulgare L., flax Linum usitatissimum L., rape Brassica napus L. and potatoes Solanum tuberosum L. Applications of 280 g/ha of haloxydine controlled weeds at some locations with barley, flax and rape not showing any phytotoxicity, however wheat stands were reduced by 30%. Rape and flax were tolerant to applications of haloxydine of 840 g/ha. Potatoes were severely damaged by applications of 560 g/ha and 1120 g/ha at Carman but not at Carberry. Post-emergent treatments were more effective than pre-emergent treatments in controlling weeds.

Weed control ranged from nil to excellent from one site to another. This was attributed to variations in soil properties. A comparison of formulations of haloxydine at Carman indicated the ester form had less biological activity than the potassium salt.

Haloxydine residue trials were established in the fall of 1969. A field bioassay was conducted in the spring of 1970 using wheat, flax, rape and alfalfa Medicago sativa L. Rape was the only crop that could be safely grown following fall application of haloxydine.

Persistence and movement of haloxydine applied in the fall was studied. Samples collected 18 and 68 days after application indicated a 100% recovery of haloxydine and maximum movement to a depth of 15 and 23 cm in a clay soil and a sandy loam soil, respectively. Samples collected the following spring indicated an 80% recovery and no downward movement during the intervening winter. One year after application, haloxydine had penetrated to a depth of 45 cm in the sandy loam soil but no further movement had occurred in the clay soil. After one year, 42% to 77% of the haloxydine persisted in the sandy loam soil and 21% to 68% in the clay soil, with high rates having a greater percentage degraded.

A bioassay of haloxydine-soil type interaction indicated ED_{50} values of 0.125 ppm to 0.993 ppm. Multiple regression analysis using the variables cation exchange capacity, organic matter, % sand, % silt, % clay on observed ED_{50} values yielded an $R^2 = .9322$. The best single variable was cation exchange capacity with $R^2 = .8072$. Cation exchange capacity plus clay had an $R^2 = .9270$. However, organic matter plus sand with an $R^2 = 0.8745$ is preferred since clay is highly correlated with cation exchange capacity. The use of three variables, cation exchange capacity (C.E.C.), sand and silt yields an equation predicting ED_{50} values of $Y = 0.9794 + 0.0311 \text{ C.E.C.} - 0.0101 \text{ sand} + 0.0119 \text{ silt}$. These three variables are responsible for 99.96% of the total regression.

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INTRODUCTION

The development of a herbicide suitable for commercial use involves many procedures to test the efficacy of the product.

A compound is synthesized in a laboratory and is initially tested for biological activity. If the compound has botanical activity it is screened on a wide variety of plant species and parameters are established to define its range of suitability.

Field testing of a compound is conducted at various levels depending on the knowledge of the compound's activity and anticipated usage. Crop species, weed spectrum, rates and methods of application are examined since herbicide performance can vary greatly from one region to another under varying environmental conditions.

This study was conducted with an experimental herbicide, haloxydine. The objectives of the study were to obtain information on haloxydine in the following areas:

- a) crops which were tolerant to the herbicide,
- b) weed species which were controlled by the herbicide and rates of application necessary to obtain this control,
- c) the persistence and movement of the herbicide under field conditions,
- d) soil factors which affect the activity of the herbicide.

LITERATURE REVIEW

Review of Haloxydine

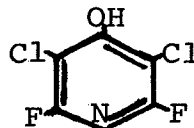
Plant Protection Ltd. (1969) reported that haloxydine (PP 493) acted mainly through the soil and had a residual effect of two to three months. They suggested that the chlorosis that was produced in plants indicated that haloxydine interfered with chlorophyll formation. The toxicological data indicated an oral LD₅₀ of 800 mg/kg for rats. Haloxydine mode of action is an uncoupling of oxidative phosphorylation. Weed Abstracts (1969).

Table 1. The chemical and physical properties of haloxydine

Chemical Name: 3,5-dichloro-2,6-difluoro-4- hydroxypridine

Molecular formula: C₅HCl₂F₂NO

Structural formula:



Molecular weight: 200.0 (Potassium salt - 238)

Melting point: 102°C

Vapor pressure: 16.1 x 10⁻³ mm Hg at 30°C
9.2 x 10⁻³ mm Hg at 25°C

Appearance: White crystalline solid

Stability: Completely stable. Forms a stable salt with alkalis.

Solubility: Slightly soluble in water. Soluble in aqueous alkalis alcohols (10%), acetone (10%), chloroform.

Slater (1968) reported kale Brassica oleracea L. was tolerant to rates of haloxydine from 35 g/ha to 280 g/ha applied pre-emerge to the kale. Herbicidal activity was noted on both broad-leaved and grassy weeds. There was no injury to red clover Trifolium pratense L. on plots treated with haloxydine the previous year.

The Oregon State University, New Herbicide evaluation test, Summer (1968) reported that haloxydine used at rates of 560 g/ha to 2240 g/ha did not appear to possess any true selectivity to any of the twenty-eight species included in the screening trial. Pre-emerge applications appeared to have more herbicidal activity than post-emerge applications.

The Oregon State University, New Herbicide evaluation test, Spring (1969) reported testing haloxydine at rates of 140 g/ha to 560 g/ha. The pre-emerge applications at 140 g/ha had good broad-leaved weed control combined with crop tolerance to twelve crop species. At the 560 g/ha application only two crops had partial tolerance to haloxydine. Tests conducted in the summer of 1969 at Oregon State University indicated appreciable broad-leaved weed control with a 70 g/ha pre-emerge application.

The Oregon State University, Field Bioassay, (1969-1970) reported the degree of phytotoxicity to indicator crops planted six months after application of haloxydine ranging from 70 g/ha to 560 g/ha. There was no injury at rates of 70 g/ha and slight injury at rates of 140 g/ha and 280 g/ha.

An application of 560 g/ha caused moderate injury to the indicator crop even though in excess of 75 cm of rain were recorded between haloxydine application and planting of the crop.

The Oregon State University, Screening Trial, Spring, (1970) examined wild oat Avena fatua L. control in wheat and barley. Applications of 140 g/ha and 70 g/ha were not phytotoxic to wheat or barley and offered appreciable stand reduction and growth reduction of the wild oats.

Bioassay

Crafts (1935) used oats Avena sativa L. in a bioassay to determine the phytotoxicity of arsenical compounds in the soil. Since that time a variety of plants have been used as indicator crops to determine the presence and amounts of herbicide in a soil.

Plant responses used in herbicide bioassays include the following: fresh shoot weights, dry shoot weights, plant heights, elongation of seedling shoots, visual injury ratings, root weights, root lengths and seedling germination counts. Santelmann et al. (1971).

Scifres et al. (1972) compared the detection of picloram by bioassay technique using field beans, Phaseolus vulgaris L., soybeans Glycine max L., sunflowers Helianthus annus L., cucumbers Cucumis sativus L. to detection using gas liquid chromatograph techniques. Results from both detection methods were closely correlated with certain indicator

plants being less suitable than others. Symptom ratings were more effective than fresh weights or oven dry weights in estimating concentrations of picloram that varied from 4 ppb to 250 ppb. Response versus log of concentration was the most efficient method of comparing bioassay estimates of picloram with gas liquid chromatograph values.

Parker (1965) developed a bioassay technique to detect herbicides inhibiting photosynthesis. Lemna minor L. was the test species used and the reaction with the photosynthetic inhibitor, paraquat, was used to determine unknown amounts of triazine, uracil and substituted urea herbicides. These classes of herbicides normally exhibit their effect on a test species after the seed reserves of the plant are depleted in 3 to 4 weeks. However using a 24 hour pre-treatment for the effects of paraquat to develop considerably shortened the time required to conduct the bioassays.

Dowler (1969) used cucumber as an indicator to determine the herbicide residue levels of bromacil, dicamba, diuron, fenac, picloram and prometryne. The range of detectability varied from 0.002 ppmw to 2.0 ppmw depending on the particular herbicide being tested. The response curve varied with different herbicides and not all herbicides had the same range of detectability. Visual injury ratings could detect lower levels of herbicide concentrations than could depressions in fresh weight.

A rapid bioassay to detect pebulate was used by Horowitz

(1966) that entailed measuring the shoot elongation of oat seedlings after 48 hours. Pre-germinated oats were placed in a petri dish, sealed and held 15 degrees from the vertical position for a 24 hour incubation period. Shoot elongation was measured after 24 and 48 hours following the incubation period. Bioassays frequently require growing the indicator species from 14 to 28 days, thus this technique offers considerable saving in time and equipment.

Hardcastle and Wilkinson (1970) used a 50% reduction in root lengths of rice Oryza sativa L. germinating in a herbicide test solution as a bioassay. They measured antagonistic, synergistic and additive responses of combinations of nine different herbicides.

Hartley and Park (1954) used a bioassay technique to determine the leaching of a herbicide through a soil column. The soil column was laid in a horizontal position, the case opened and rows of indicator species seeded the length of the column. The extent of leaching was indicated by the distance through which the indicator species showed a growth depression.

Bioassays are limited in their ability to detect a wide range of herbicide concentrations. To overcome this, Talbert and Fletchall (1964) used various plants as indicator species in a bioassay to obtain a sensitive response.

Holly and Roberts (1963) used a dilution factor with untreated soil to obtain a soil composite where the herbicide

exceeded the upper range of detectability. In this manner the original amounts of herbicide present could be calculated.

Santelmann et al. (1971) evaluated the accuracy and precision of bioassay techniques since there is a wide variation in responses measured using different bioassays. An "unknown" amount of prometryne was applied to a soil and distributed among various researchers. The results from the different bioassay techniques varied from 147% below to 234% above the actual concentration of prometryne in the sample. By standardizing conditions of temperature, light, moisture and handling procedures the bioassay determinations varied from 32% below to 0% above the actual concentration. Best results were obtained when more than one type of response was used to measure herbicide activity.

Dawson et al. (1968) determined the persistence of monuron, diuron and simazine in a silt loam using oats as an indicator species. Samples were obtained to a depth of 12" by excavating a large hole at each collection site. Soil samples were removed at appropriate depths by making horizontal cuts into the smooth side of the large hole. Soil from samples in each plot were combined before testing.

Biological Activity

Grover (1966) modified the soil characteristic of a heavy clay soil by additions of peat moss and silica sand. Oats were used to detect the biological availability of simazine. Soil moisture conditions were also varied. The effect of

added organic matter (peat moss) was to reduce the availability of simazine. At high moisture levels, the effect of added clay did not reduce herbicidal availability. Thus the adsorptive forces in clay were not as strong as in organic matter. As moisture conditions were reduced, the simazine toxicity was reduced as a result of more adsorption on hydrophilic surfaces.

Upchurch et al. (1966) studied five herbicides, simazine, diuron, CIPC, CDEC and CDAA at seventeen field locations with organic matter contents varying from 0.7% to 48.8%. Phytotoxic responses were determined on soybean, cotton Gossypium spp. and annual grassy weeds. Fourteen factors were measured representing soil, climatic and biotic characteristics. The highest simple correlation was obtained with organic matter; for each 1% increment in organic matter between 1% to 40%, the additional pounds per acre required to maintain a 50% grass control level were 0.46, 0.16 and 0.99 for simazine, diuron and CIPC, respectively. The response of the five herbicides varied widely and there were different factors involved in the reaction with the three test species. Each herbicide had to be treated on an individual basis to fully explain the herbicide behavior.

Grover (1968) determined the ED_{50} values of picloram on seven Saskatchewan soils using sunflowers as an indicator species. He found a four fold difference in ED_{50} values of 0.068 ppm to 0.265 ppm and a significant simple correlation

coefficient of ED₅₀ values with organic matter content. Cation exchange capacity and clay content were not significantly correlated with ED₅₀ values.

Parker (1966) developed a technique to measure the relative importance of root and shoot uptake of soil acting herbicides using sorghum Sorghum spp. seedlings. EPTC, diallate and possibly CDAA require entry via the shoot to exhibit their toxic effect. Trifluralin, CIPC and dichlobenil can act directly on the shoot but appeared more effective with root uptake.

O'Brien and Prendeville (1972) studied the uptake of four herbicides, linuron, diuron, simazine and atrazine applied at 4.5 kg/ha to peas Pisum sativum L. Herbicide uptake in the region of the first internode did not cause any significant reduction in the dry weight of the plant. Herbicide uptake in the region of the second and third internode areas caused a 50% reduction in dry weight. Radioactive tracer studies with atrazine indicated that the first internode area did absorb herbicide but not in sufficient quantities to reduce growth. The second and third internode areas had a two to three fold increase in uptake and movement of the herbicide. The differential selectivity was attributed to greater maturity of the first internode area with fuller epidermal and cuticle development.

Parker (1963) studied the site of diallate uptake in oats, wheat and barley. The most sensitive area was the region 10 to 15 mm above the coleoptile node at a time just

prior to the emergence of the first leaf from the coleoptile. The basis of selectivity between oats and the cereals was based on shoot morphology wherein oats has an elongation of the mesocotyl which placed the sensitive stem apex in a zone of herbicide treated soil. Wheat and barley do not possess a mesocotyl and elongation of the sub-crown internode does not occur until a later date at which time the plant is more resistant to diallate. There was little root uptake of diallate by any of the test species.

Kuratle et al. (1969) determined that the basis of linuron selectivity between carrot Daucus carota L. and common ragweed Ambrosia artemisiifolia L. was the phytotoxicity of the metabolites within the plant. In carrot, 87% of the linuron was metabolized to non-phytotoxic derivatives compared to 13% in the ragweed. None of the four breakdown products were toxic to ragweed.

Stickler et al. (1969) found the effectiveness of some herbicides increased, others decreased or remained constant as soil moisture increased from 25% to 31% to 37% using a silty clay loam. Atrazine and EPTC phytotoxicity increased as the soil moisture increased from 25% to 31% but was not affected by higher levels. Amiben response increased linearly and trifluralin decreased linearly with increasing moisture levels. Propachlor and CP50144 activities were not affected by moisture levels varying from 25% to 37%.

Movement and Leaching

Green et al. (1969) studied the effects of varying soil water amounts on the adsorption of atrazine on three horizons of a latosolic soil. They concluded that only on low adsorption soils will water content variations alter significantly the herbicide concentration in the soil solution. The principal effect of soil water content on herbicide phytotoxicity probably is associated with herbicide transport which is more sensitive to changes in water content than is the concentration of herbicide in the soil solution.

Herr et al. (1966) applied picloram to three soil types. When residual amounts were determined after 9 and 15 months in heavy and medium textured soil, the greatest amounts of herbicide were recovered in the top six inches of soil. In a light textured soil after 15 months the greatest amounts of picloram were detected at the 18" to 24" level, the deepest level sampled. Picloram was dissipated faster at low rates in all three soil types studied. The greatest amounts of picloram detected at the end of the study were in the heavy textured soil with the largest amount of organic matter. Soil organic matter was most influential in retaining picloram from leaching by percolating water.

Burnside et al. (1969) studied the persistence of five herbicides under semi-arid conditions (Nebraska 14" to 34" average annual precipitation). Persistence varied with the herbicide applied: isocil) propazine) atrazine) trichloro-benzyl chloride (TCBC)) linuron. Soil texture differences

had a greater influence on herbicide residue carry-over than did climatic differences, with coarse textured soils and dry conditions exhibiting the maximum residues. Leaching of herbicides into the soil profile was a mode of dissipation.

Phillips (1968) determined that 2,3,6-TBA penetrated up to 8' in depth with the greatest concentration in the 3' to 6' depth. The herbicide was detectable in a silty clay loam soil 11 years after application. Persistence and movement did not appear to be related to temperature or moisture but herbicide residues of fall applications were slightly greater than spring applications.

Wiese and Davis (1964) studied herbicide movement in a silty clay loam under laboratory conditions. Herbicides moved the following distances: amine salt of 2,3,6-TBA and PBA, 20"; amine salt of 2,4-D and sodium salt of fenac, 15"; ester of fenac, fenuron, monuron, amine silvex and amine 2,4,5-T, 9"; esters of silvex, 2,4,5-T and 2,4-D remained in the top 3" of soil. Relatively soluble herbicides were not affected by method of water application whereas relatively insoluble herbicides were moved deeper when subjected to a "flushing" technique simulating flood irrigation.

Degradation

Comes and Timmons (1965) used oats to determine the toxicity of atrazine, simazine, diuron, fenuron and monuron exposed to sunlight on a soil surface. Experiments were conducted in March with soil temperatures of 90°F to 120°F and in August with temperatures of 140°F to 180°F. Elevation

was 7200'. In the spring losses varied from 13% for monuron to 49% for fenuron after 25 days. After 60 days losses varied from 33% for monuron to 73% for atrazine. In the summer there were no significant losses between treatments of 25 and 60 days for any of the herbicides. The overall levels of losses, 65% to 82%, were higher than those encountered in the spring. Appreciable losses of herbicides also took place from soil samples kept in the dark. In attempting to determine losses due to photodecomposition, the soil temperature and air movements must be carefully controlled.

Jordan et al. (1965) examined the effects of far, middle and near ultraviolet light on monuron, diuron, neburon, fenuron, simazine, atrazine, bromacil and isocil. The far ultraviolet light caused more breakdown of all herbicides than did the near ultraviolet light which is more representative of sunlight. The most decomposition occurred with fenuron where 30% of the initial amount remained after 500 hours exposure and the least with simazine where 75% remained after 500 hours.

McCormick (1966) studied the microbial decomposition of atrazine and diuron in a loamy sand and clay loam. Herbicide inactivation was directly related to metabolism of soil organic carbon. More herbicide was inactivated per unit of soil organic carbon in the sandy soil compared to the loamy soil. This was attributed to less specific surface area with the sand and, consequently, a greater

concentration of herbicide on the soil particles. When additional energy sources were added, the breakdown of both herbicides increased, indicating a non-preferential involvement in microbial metabolism. Decomposition of atrazine and diuron doubled and tripled, respectively, for each 10° rise in temperature from 16°C to 30°C . This response parallels the decomposition of soil organic matter.

Wildung et al. (1963) studied the degradation of $^{14}\text{COOH}$ -labelled chloramben on three soil types. Two rates of herbicide were perfused through soil columns for 160 days. The rate and extent of degradation was similar for all three soil types; however, the amount of herbicide degraded was larger, 40% to 56%, for the low herbicide rate compared to the high herbicide rate with 23% to 39% degradation. A lag phase indicated soil microflora were responsible for degradation and the mechanism was inferred to be decarboxylation on the basis of the loss of ^{14}C activity of the labelled COOH group. A measure of $^{14}\text{CO}_2$ evolved accounted for 30% of the disappearance of the labelled material and the authors concluded the balance of the $^{14}\text{CO}_2$ was retained by the soil or incorporated into microbial tissue.

Harris et al. (1969) determined the dissipation of atrazine and fenac at 3", 9" and 15" soil depths at various locations in the United States after 3 months. Increasing soil organic matter, depth of placement and decreasing temperatures caused the herbicides to be more persistent. There was 61% more atrazine and 41% more fenac recovered