THE UNIVERSITY OF MANITOBA

THE HERBICIDAL ACTIVITY OF HALOXYDINE IN SOME MANITOBA SOILS

by

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A dissertation submitted to the Faculty of Graduate Studies of the University of Manitoba in partial fulfillment of the requirements 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 signle 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 $Y = 0.9794 + 0.0311$ C.E.C. $- 0.0101$ sand $+ 0.0119$ 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,

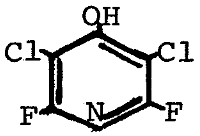
d) soil factors which affect the activity of the herbicide.

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 LD_{50} 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:	$C_5HCl_2F_2NO$
Structural formula:	
Molecular weight:	200.0 (Potassium salt - 238)
Melting point:	102°C
Vapor pressure:	16.1×10^{-3} mm Hg at 30°C 9.2×10^{-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 of 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 wtih 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 degress 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 lenghts 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 ED_{50} values with organic matter content. Cation exchange capacity and clay content were not significantly correlated with ED_{50} 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 $>$ trichlorobenzyl 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 attemtpting 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^{o} rise in temperature from $16^{o}C$ to $30^{o}C$. This response parallels the decomposition of soil organic matter.

Wildung et al. (1963) studied the degradation of $^{14}COOH$-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 $^{14}CO_2$ evolved accounted for 30% of the disappearance of the labelled material and the authors concluded the balance of the $^{14}CO_2$ 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 from the 15" depth compared to the 3" depth. A positive correlation between organic matter content and fenac retention was observed.

Adsorption

Bailey and White (1964) in their review of adsorption describe the adsorption phenomena as due to attraction between a solid surface and a vapor or solution. This attraction results from an interaction of the field of force emanating from the surface of the adsorbent and the molecules or ions of the adsorbate.

Physical adsorption is due to van der Waals forces resulting from: dipole-dipole interactions, induced dipole interactions, ion-dipole interactions, dispersion interactions and Born repulsion interactions. Chemical adsorption is due to coulombic forces and results from bond formation between adsorbent and adsorbate.

Physical adsorption has low binding strength between adsorbent and the herbicide while chemical adsorption has a high binding strength. A soil colloid acts as the adsorbent and since the herbicide is in solution most frequently, there is adsorption of both water and molecules of herbicide on the colloid.

Soil colloids are composed of clay minerals and organic matter. Clays vary in their surface area and cation exchange capacity. Organic matter also varied in its nature depending on parent material and degree of degradation. Thus the nature of the colloid affects herbicide behavior in the soil.

The nature of the herbicide affects the adsorption and within a family of herbicides with a common base there is an inverse relationship between solubility and adsorption. Increased chloro substitution on the benzene ring of 2,4-D decreased solubility and increased adsorption. The nature of the functional group affects adsorption as H-bonding appears to take place between COOH, NH_2 and CH_3 groups and the colloid. Soil solution pH affects adsorption by affecting the degree of dissociation of ionization of the herbicide. The pH also affects the charge on inorganic soil colloids and the formation of stable complexes between herbicides and metallic ions.

Soil moisture affects adsorption since water and herbicide molecules may be competing for adsorption sites on the colloid. High soil moisture frequently has the effect of making the herbicide more available to plant uptake from the soil solution by desorbing the herbicide from the colloid.

Temperature affects adsorption since the process is exothermic; however, increases in temperature have not always shown less adsorption and temperature may be of more importance by its effect on herbicide solubility and vapor pressure.

Doherty and Warren (1969) compared the adsorption of simazine, linuron, pyrazon and prometryne on organic matter and bentonite clay. The three sources of organic matter in order of increasing decomposition were: sphagnum moss, fibrous peat and muck soil. Peat and muck soil were considerably more adsorptive than bentonite and moss. The peat was more adorptive than muck soil for all herbicides except linuron. The variation in adsorption by the three sources of organic matter was thought to be a result of the differences in chemical properties of reactive sites for H-bonding rather than differences in the physical properties of cation exchange capacity and hygroscopic surface area.

Harris and Warren (1964) studied herbicide adsorption on four different mediums: organic muck soil, bentonite clay, anion exchanger and cation exchanger. The chemical properties of the herbicide, temperature and pH affected the adsorption on the various media. Lowering the pH increased adsorption of monuron, atrazine, CIPC and DNBP on bentonite. Diquat adsorption on bentonite did not vary with pH. Adsorption by the bentonite was also greater at 0°C than at 50°C but muck soils did not vary with temperature differences. DNBP was adsorbed by an anion exchanger but not by a cation exchanger; the reverse was true for diquat. Monuron, atrazine and CIPC were adsorbed by both anion and cation exchangers. The nature of the adsorbent affected the herbicide adsorption as atrazine, simazine, amiben and 2,4-D could be desorbed more easily from bentonite than from a muck soil.

Hance (1965) concluded that under aqueous slurry conditions, there is competition between water and diuron for adsorption sites. Organic matter was of greater importance than soil mineral matter surfaces for adsorption of diuron in the presence of water.

Hurle and Freed (1972) studied the effects of chloride salts of NH_4^+, K^+ and Ca^{++} on herbicide solubilities and adsorption. Herbicides studied were atrazine, ametryne, atratone, monuron, diuron and fenuron. Herbicide solubility decreased in the presence of increasing ionic strength solutions. Adsorption increased in the presence of salts, with the monovalent K^+ and NH_4^+ having increased adsorption compared to the Ca^{++} ion. Adsorption also varied with ionic strength. Thus the addition of fertilizers may affect adsorption of herbicides under certain soil moisture conditions.

METHODS AND MATERIALS

Field Experiments

Field experiments were conducted in 1969 with wheat, barley and flax. In 1970 the crops were flax, rape and potatoes. The fields were prepared and planted by the farmer. Two trials were initiated in the fall of 1970 for herbicide movement and residue studies. In 1971 four indicator crops were planted to determine the biological availability of haloxydine on these trials. Trials were conducted on flax and rape in 1971.

A randomized complete block design was used for all experiments. Plot size was 3.6 m x 5.5 m. Four replicates were used in all trials except for rape in 1970. Six replicates were used for rape in 1970 in the interests of additional precision. The herbicide was applied to the central 2.4 m of each plot using a bicycle sprayer equipped with flat fan nozzles delivering 112 liters per hectare (l/ha). All herbicide treatments are given as amounts of active ingredient. Wind velocities are estimated.

Visual ratings were made several times during the growing season to assess weed control and crop phytotoxicity. The ratings were made on a 0 to 9 scale (Table 2).

Table 2. Visual ratings of weed control and crop phytotoxicity.

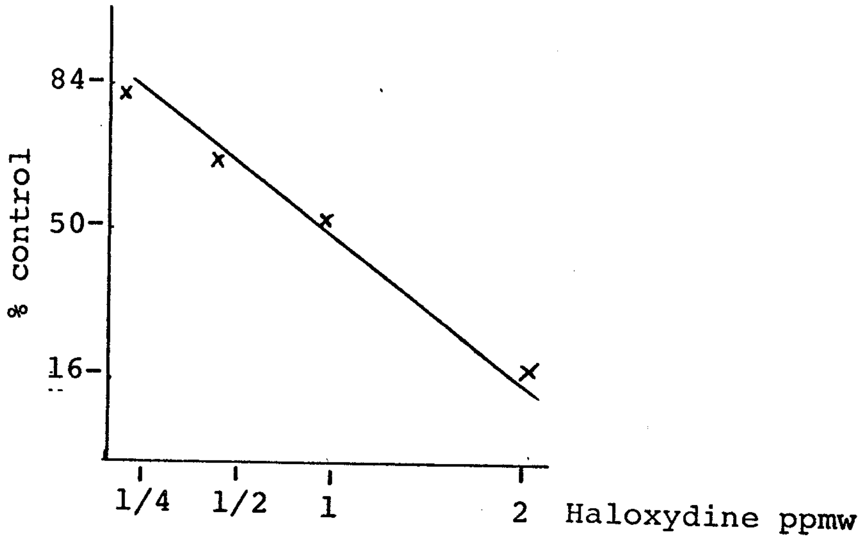
Rating	Degree of weed control	Degree of crop phytotoxicity
0	No control	No tolerance
1	Very poor control	Very severe stand reduction
2	Poor control	Severe stand reduction
3	Slight control	Moderate stand reduction
4	Moderate control	Slight stand reduction
5	Fair control	Plant height reduced
6	Nearly acceptable control	Slight deformity
7	Acceptable control	Acceptable tolerance
8	Very good control	High degree of tolerance
9	Complete kill	No crop injury

Bioassay Experiments

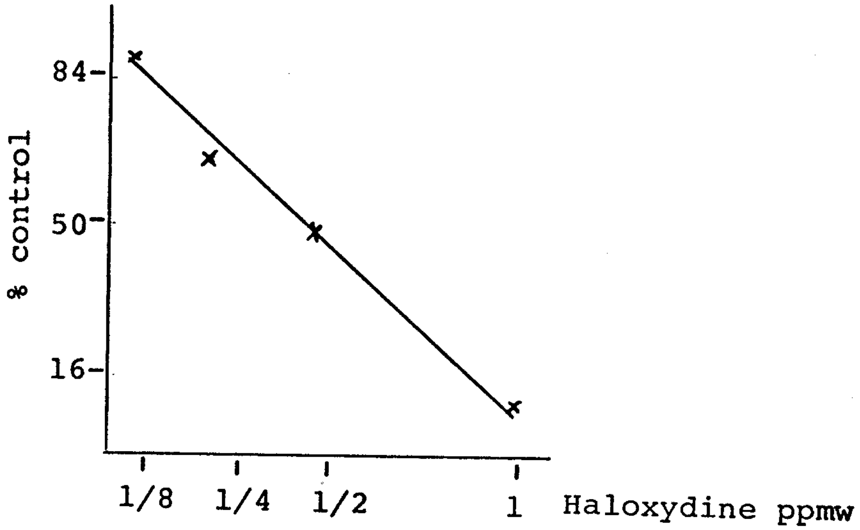
Samples were obtained from the 0-5, 5-10 and 10-15 centimeter (cm) depths from clay soils and from the 0-5, 5-10, 10-15, 15-23, 23-30 and 30-45 cm depths from loam soils. Samples were dug with a garden trowel to minimize mixing. Samples from each replicate were bulked, air dried, ground to pass through a two millimeter (mm) sieve, and thoroughly mixed.

Soil (400 grams) from the field sample was placed in a 7.5 cm diameter glass jar. Soil moisture levels were determined by flooding a sample and allowing the free water to drain. Samples were brought up to 75% of the saturated weight and maintained by daily additions of 10 to 15 grams of water. Wheat (var. Manitou) was used as the bioassay indicator species. Four seeds were planted in each jar and the stand reduced to two uniform seedlings following emergence. Plants were grown in a greenhouse in a completely randomized design. A 16 hour day length of 17,222 lux and $21^{\circ} \pm 3^{\circ}C$ temperature was maintained. The nyctoperiod temperature was $13^{\circ} \pm 3^{\circ}C$. The wheat was harvested two weeks after seeding and dry weights were recorded.

The level of haloxydine residues were calculated from ED_{50} values. Blackman (1962). ED_{50} is the concentration of herbicide required to reduce the dry weight of the plant to 50% of the dry weight of an untreated check. The ED_{50} was calculated by converting the dry weight percentage to a probit value and constructing a standard curve for a range of herbicide concentrations on a logarithmic scale. Blackman (1952). Standard curves were constructed for each soil type. (Figure 1)



Glenlea



Graysville

Figure 1. Haloxydine Standard Curves, Glenlea, Graysville

Bioassay Calculations

Bulk densities and the soil weight of an area of one hectare and 15 cm in depth for the Graysville and Glenlea residue trials are found in Table 3.

An application of 0.56 kg/ha of haloxydine to the 0 to 15 cm depth is equivalent to 0.283 ppmw.

$$\frac{0.56 \text{ kg/ha}}{1.98 \times 10^{6} \text{ kg/ha}} = 0.283 \times 10^{-6} \text{ or } 0.283 \text{ ppmw.}$$

An application of 0.56 kg/ha to a one hectare area 5 cm in depth is equivalent to 0.849 ppmw.

$$\frac{0.56 \text{ kg/ha}}{0.66 \times 10^{6} \text{ kg/ha}} = 0{,}849 \times 10^{-6} = 0.849 \text{ ppmw.}$$

Similarly 1.15 kg/ha - 1.697 ppmw and 2.24 kg/ha - 3.394 ppmw on a one hectare area 5 cm in depth. The appropriate concentration of herbicide (ppmw) in each sample layer of the various soil zones is given in Table 4.

The percent recovery was obtained by dividing the ppmw detected by the bioassay procedure by the ppmw of haloxydine originally applied for the appropriate sample layer. Example: 0.56 kg/ha, 15-23 cm depth

$$\frac{\text{ppmw detected}}{0.473 \text{ ppmw}} \times 100 = \% \text{ recovery}$$

Table 3. Bulk densities and soil weight for the Graysville and Glenlea residue trials.

	Zone	Bulk Density	Soil Weight
Graysville	0-15 cm	1.32 g/cc	1,980,000 kg/ha
	15-30 cm	1.58 g/cc	2,370,000 kg/ha
	30-60 cm	1.69 g/cc	2,535,000 kg/ha
Glenlea	0-15 cm	1.25 g/cc	1,875,000 kg/ha

Table 4. Herbicide concentration (ppmw) in sampled soil layer.

	Depth (cm)	Herbicide application rate		
		0.56 kg/ha	1.12 kg/ha	2.24 kg/ha
Graysville	0-5	0.848	1.697	3.394
	5-10	0.848	1.697	3.394
	10-15	0.848	1.697	3.394
	15-23	0.473	0.945	1.890
	23-30	0.473	0.945	1.890
	30-45	0.221	0.442	0.884
Glenlea	0-5	0.896	1.792	3.584
	5-10	0.896	1.792	3.584
	10-15	0.896	1.792	3.584

Experiment I Wild oat control trials, 1969.

Field trials were conducted in 1969 at Fort Whyte, Portage la Prairie, and Brunkild to investigate control of wild oats. The treatments that were applied at the various crop stages are listed in Table 5. Haloxydine was compared with barban (Carbyne) for wild oat control. Each treatment had four replicates. Crops, soil characteristics and climatic date at application time for each location are listed in Table 6.

Table 5. Wild oat control treatments, 1969.

Treatment	Rate of application (g/ha)	Stage of application
Haloxydine	70	Pre-emerge
Haloxydine	140	Pre-emerge
Haloxydine	280	Pre-emerge
Haloxydine	70	2-3 leaf
Haloxydine	140	2-3 leaf
Haloxydine	280	2-3 leaf
Haloxydine	70	5-6 leaf
Haloxydine	140	5-6 leaf
Haloxydine	280	5-6 leaf
Carbyne	350	2 leaf
Weedy check	-	-

Table 6. Crops, soil characteristics and climatic data for wild oat control trials, 1969.

Location, Crop, Soil	Crop stage and time at application	Climatic conditions			
		°C	RH[1]	Wind[2]	SM[3]
Fort Whyte					
Flax Var. Noralta	Pre-emerge, 16 June, 0930.	12	70%	0-2	18.0%
Seeded 12 June O.M.[5] 7.7%	5-8 cm height, 9 July, 1000.	23	69%	8-16	18.5%
Sand 17.0% Silt 39.0% Clay 44.0%	10-15 cm height 25 July, 1500.	22	56%	12-16	19.5%
Portage la Prairie					
Barley var. Conquest	Pre-emerge 30 May, 2000.	21	60%	10-12	14.0%
Seeded 26 May O.M. 5.8%	2-3 leaf, 15 June, 0700.	11	62%	0-3	14.0%
Sand 20.0% Silt 55.0% Clay 25.0%	5-6 leaf, 11 July, 1100.	27	75%	0-5	14.6%
Brunkild					
Wheat var. Manitou	Pre-emerge, 16 June, 2000.	18	54%	3-8	11.3%
Seeded 13 June O.M. 6.8%	2-3 leaf, 17 July, 1000.	26	68%	0-5	13.2%
Sand 4.0% Silt 41.0% Clay 55.0%	5-6 leaf	Not applied[4]			

1. Relative humidity
2. Kilometers per hour
3. Soil Moisture
4. Site was flooded
5. Organic matter

Experiment II Wild oat and broad-leaved weed control trials, 1969.

Field trials were conducted in 1969 at Portage la Prairie to investigate control of wild oats and broad-leaved weeds. The treatments that were applied are listed in Table 7. Haloxydine was compared with bromoxynil octanate plus MCPA 1:1 (Burctril M) and with 2,4-D plus dicamba plus mecoprop 5:2:1 (Banvel 3) for broad-leaved weed control. Each treatment had four replicates. Crops, soil characteristics and climatic data at application time are listed in Table 8.

Table 7. Wild oat and broad-leaved weed control treatments, 1969.

Treatment	Rate of application (g/ha)
Haloxydine	17.5
Haloxydine	35.0
Haloxydine	70.0
Haloxydine	105.0
Haloxydine	140.0
Haloxydine	280.0
Haloxydine + 2,4-D[1]	17.5 + 420
Haloxydine + 2,4-D	35.0 + 420
Haloxydine + 2,4-D	70.0 + 420
Haloxydine + 2,4-D	105.0 + 420
Haloxydine + 2,4-D	140.0 + 420
Buctril M	560.0
Banvel 3	560.0
Weedy check	-

[1] iso-octyl ester

Table 8. Crops, soil characteristics and climatic data for wild oat and broad-leaved weed control trials, 1969.

Location, Crop, Soil-	Time and crop stage at application	- Climatic conditions
Portage la Prairie		
Barley,var Conquest	6 leaf	21°C, 80% RH[1],
Seeded 26 May	11 July	Wind 6-10[2],
Organic Matter 5.8%	0900	Soil Moisture 14.6%
Sand 20.0%		
Silt 55.0%		
Clay 25.0%		
Portage la Prairie		
Flax, var Noralta	15 cm Height	25°C, 75% RH,
Seeded 24 May	11 July	Wind 8-12,
Organic Matter 5.8%	1030	Soil Moisture 14.6%
Sand 20.0%		
Silt 55.0%		
Clay 25.0%		

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1. Relative Humidity
2. Kilometers per hour

Experiment III Weed control in rape, 1970.

Field trials were conducted in rape *Brassica napus* L. var Target and *Brassica campestria* L. var Echo in 1970. Trials were located at Graysville, Petersfield, and Westbourne. The treatments that were applied are listed in Table 9. Haloxydine treatments were applied pre-emerge to the crop and niclofen (TOK-RM) at the two leaf stage of the crop. Each treatment had six replicates. Varieties, soil characteristics and climatic data at application time are listed in Table 10. Yield samples were harvested from a 0.25 m^2 area and cleaned. A count of the seedling weeds in the weedy check was made at each location. The numbers of weeds present on a 0.25 m^2 area are listed in Table 11.

Table 9. Weed control in rape treatments, 1970.

Treatment	Rate of application (g/ha)
Haloxydine	420
Haloxydine	560
Haloxydine	700
Haloxydine	840
TOK - RM	1350
Weedy check	-

Table 10. Varieties, soil characteristics and climatic data for weed control in rape, 1970.

Location, Variety, Soil	Time and crop stage at application	Climatic conditions
<u>Graysville</u>	Pre-emerge, 4 June, 1630.	32°C, 40%RH[1], Wind 0-3[2].
Target		
Seeded 3 June		
Organic matter 2.3%	2 leaf, 22 June, 0900.	18°C, 70% RH, Wind 6-8.
Sand loam soil		
<u>Petersfield</u>	Pre-emerge, 10 June, 2200.	19°C, 70% RH, Wind 8-11.
Target		
Seeded 10 June		
Organic matter 7.5%	2 leaf, 4 July, 0630.	12°C, 88% RH, Wind 0-3.
Clay soil		
<u>Westbourne</u>	Pre-emerge, 25, June, 1400.	22°C, 64% RH, Wind 8-11.
Echo		
Seeded 24 June		
Organic matter 7.0%	2 leaf, 10 July, 1430.	28°C, 70% RH, Wind 6-8.
Clay loam soil		

1. Relative humidity.
2. Kilometers per hour.

Table 11. Weed count per 0.25 m^2, Weed control in rape, 1970.

Weed Species	Grays-ville	Peters-field	West-bourne
Green foxtail *Setaria viridis* L. Beauv.	42.3	24.0	138.3
Redroot pigweed *Amaranthus retroflexus* L.	1.8	2.5	0.3
Lamb's-quarters *Chenopodium album* L.	0.8	12.8	0.8
Wild buckwheat *Polygonum convolvulus* L.	1.1	1.0	-
Green smartweed *Polygonum scabrum* L. Moench	0.2	1.0	-
Stinkweed *Thlaspi arvense* L.	-	22.8	-
Wild oats	-	5.1	-
Quack grass *Agropyron repens* L. Beauv.	2.8	-	-
Annual sow-thistle *Sonchus oleraceus* L.	1.1	-	-

Experiment IV Weed control in flax, 1970.

Field trials were conducted in flax, var. Noralta at Carberry and MacDonald in 1970. The treatments that were applied at the various crop heights are listed in Table 12. Haloxydine and haloxydine plus the ester formulation of MCPA were compared with Carbyne for wild oat control and with Buctril M for broad-leaved weed control. Each treatment had four replicates. The predominant weeds at the MacDonald trial were wild oats, wild buckwheat and Canada thistle (*Cirsium arvense* L. Scop.). The predominant weeds at Carberry were wild buckwheat, bluebur (*Lappula echimata* Gilib.), lamb's-quarters, green foxtail and wild oats. Soil characteristics and climatic data at the times of herbicide application are listed in Table 13.

Table 12. Weed control in flax treatments, 1970.

Treatment	Rate of application (g/ha)	Crop height at application (cm)
Haloxydine	140	Pre-emerge
Haloxydine	280	Pre-emerge
Haloxydine	560	Pre-emerge
Haloxydine	840	Pre-emerge
Haloxydine	140	5-8
Haloxydine	280	5-8
Haloxydine	560	5-8
Haloxydine	840	5-8
Haloxydine + MCPA	140 + 420	5-8
Haloxydine + MCPA	280 + 420	5-8
Haloxydine + MCPA	560 + 420	5-8
Carbyne[1]	350	3-5
Buctril M	560	7-10
Weedy check	-	-

[1] Carbyne applied in a volume of 56 l/ha using a pressure of 3.2 kg/cm^2.

Table 13. Soil characteristics and climatic data for weed control in flax, 1970.

Location, Soil	Crop stage and time of application	Climatic conditions
<u>MacDonald</u>	Pre-emerge,	19°C, 61% RH[1],
Seeded 1 June	2 June, 0800.	Wind 5.8[2].
Organic matter 8.5%	3-5 cm,	26°C, 64% RH,
Clay soil	15 June, 1900.	Wind 5-8,
	5-8 cm,	19°C, 62% RH,
	24 June, 1800.	Wind 4-6.
	7-10 cm,	24°C, 56% RH,
	26 June, 1500.	Wind 0-5.
<u>Carberry</u>	Pre-emerge,	20°C, 51% RH,
Seeded 1 June	3 June, 2245.	Wind 3-6.
Organic matter 3.0%	3-5 cm,	27°C, 40% RH.
Sandy loam soil	15 June, 1400.	Wind 0-3.
	5-8 cm,	17°C, 78% RH,
	24 June, 0800.	Wind 5-8.
	7-10 cm,	29°C, 78% RH,
	29 June, 1530.	Wind 0-5.

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1. Relative humidity
2. Kilometers per hour

Experiment V Weed control in potatoes, 1970.

Field trials were conducted in potatoes at Carberry and Carman in 1970. Haloxydine and haloxydine plus paraquat (Gramoxone) were compared to Gramoxone and linuron (Lorox) for weed control. Herbicides were applied in a volume of 225 l/ha. Each treatment had four replicates. The treatments that were applied at the various crop stages are listed in Table 14. Post-emerge treatments were applied when the crop was 60% emerged and crop height was 4 to 7 cm. Varieties, soil characteristics and climatic data at application time are listed in Table 15. Hand weeded checks were maintained in a weed free condition using light hoes and the cultivated checks were maintained using the farmers regular tillage program of four to six cultivations until the crop canopy "closed over" the inter-row area. A count of the seedling weeds was made at the time of post-emerge applications. The numbers of weeds present at time of spraying on a 0.25 m^2 area are listed in Table 16. Row spacing was 91 cm and ungraded yields were harvested from the center two rows of each plot.

Table 14. Weed control in potato treatments, 1970.

Treatment	Rate of application (g/ha)	Crop stage at application
Haloxydine	560	Pre-emerge
Haloxydine	1120	Pre-emerge
Haloxydine	560	Post-emerge
Haloxydine	1120	Post-emerge
Haloxydine + Gramoxone	280 + 560	Post-emerge
Haloxydine + Gramoxone	560 + 560	Post-emerge
Gramoxone	840	Post-emerge
Gramoxone	1120	Post-emerge
Lorox	1680	Pre-emerge
Hand weeded check	-	-
Cultivated check	-	-

Table 15. Varieties, soil characteristics and climatic data for weed control in potatoes, 1970.

Location, Variety, Soil	Time and crop stage at application	Climatic conditions
Carberry Netted Gem Planted 1 June	Pre-emerge, 3 June, Time: 0930.	22°C, 48% RH[1], Wind 3-5[2].
Organic matter 8.7% Clay loam soil	Post-emerge, 24 June, Time: 0600.	11°C, 88% RH, Wind 3-5.
Carman Norland Planted 23 May	Pre-emerge, 27 May, Time: 1500.	13°C, 76% RH, Wind 5-8.
Organic matter 4.3% Fine sand loam	Post-emerge, 16 June, Time: 1700.	28°C, 48% RH, Wind 8-10.

[1] Relative humidity

[2] Kilometers per hour

Table 16. Weed count per 0.25m^2, Weed control in potatoes, 1970.

Weed species	Carberry	Carman
Green foxtail	10.0	99.1
Wild buckwheat	1.2	9.5
Redroot pigweed	5.5.	1.1
Lamb's-Quarters	0.1	4.5
Flax	0.5	29.8
Green smartweed	-	8.0

Experiment VI Haloxydine residue trial at Graysville, 1970.

A field trial was initiated on August 28 and haloxydine applied at the following rates (g/ha): 0, 560, 1120, 2240. Each treatment was replicated three times. The soil had 3.5% organic matter, 78% sand, 11% silt, 11% clay and a cation exchange capacity of 18.0 millequivalents per 100 grams of soil.

a) Field Bioassay. On May 14, 1971 wheat (var. Manitou), flax (var. Redwood 65), rape (var. Target), and alfalfa (var. Beaver) were seeded across the plots. The crops were seeded without tilling the soil to prevent soil mixing. Visual ratings of crop phytotoxicity were conducted. Yield samples of wheat and rape were harvested from an area 61 cm x 61 cm.

b) Greenhouse bioassay. Soil samples were taken on November 3, 1970; May 6, 1971; and October 20, 1971. Rainfall records of total amount and amount occuring as rainfall exceeding 0.75 cm on an occasion are listed in Table 17. Levels of haloxydine were determined using a standard curve and a bioassay conducted in the greenhouse. Each sample had four replicates.

Table 17. Total and effective[1] rainfall (cm) at Graysville and Glenlea, 1970, 1971.

	Graysville			Glenlea	
1970	Total rain	Effective rain		Total rain	Effective rain
August	4.44	3.76		2.62	1.73
September	5.61	4.78		8.13	6.82
October	3.33	2.95		4.01	2.54
November	1.78	-		1.57	-
1971					
April	4.37	3.12		5.94	5.32
May	6.35	5.52		2.87	2.38
June	12.29	11.10		5.28	3.43
July	8.52	6.82		8.33	6.38
August	4.15	2.86		3.35	2.72
September	8.33	7.48		6.81	6.03
October	11.73	10.88		6.83	5.45
Rainfall between sample dates					
Aug. 28- Nov.3.	12.70	11.49	Oct.14- Nov.1.	3.70	2.31
Apr. 1 - May 6.	4.37	3.12	Apr.1 - May 13.	6.10	5.32
May 6 - Oct. 20.	48.57	43.90	May 13 - Sept.30.	26.48	20.94

[1] Rainfall exceeding 0.75 cm per day.

Experiment VII Haloxydine residue trial at Glenlea, 1970.

A field trial was initiated on October 14. Haloxydine was applied to fallow ground at the following rates (g/ha): 0,560,1120,2240. Each treatment was replicated four times. The soil had 4.6% organic matter, 6% sand, 35% silt, 59% clay and a cation exchange capacity of 43.1 millequivalents per 100 grams of soil.

a) Field Bioassay. On May 31, 1971 wheat (var. Manitou), flax (var. Redwood 65), rape (var. Target), and alfalfa (var. Beaver) were seeded across the plots. The crops were seeded without tilling the soil to prevent soil mixing. Visual ratings of crop phytotoxocity were conducted. Yield samples of wheat and rape were harvested from an area 61 cm x 61 cm.

b) Greenhouse bioassay. Residue samples were taken on November 1, 1970; May 13, 1971; and September 30, 1971. Rainfall records are listed in Table 17. Levels of haloxydine were determined as in Experiment VI.

Experiment VIII. Weed control trial at Carman, 1971.

Field trials were conducted in rape and flax at Carman in 1971. The potassium salt and the ester formulations of haloxydine were compared to determine whether they had similar selectivities between crops and weed species.

Haloxydine was applied pre-emerge to the rape and post-emerge to the flax (6 to 8 cm). The treatments that were applied are listed in Table 18. Each treatment had four replicates. Varieties, soil characteristics, and climatic data at application time are listed in Table 19. The predominant weed species were green foxtail, wild buckwheat, lamb's-quarters, green smartweed, and redroot pigweed.

Table 18. Carman weed control treatments, 1971.

Treatment	Rate of application (g/ha)	Formulation
Haloxydine	560	Potassium salt
Haloxydine	1120	Potassium salt
Haloxydine	560	Ester
Haloxydine	1120	Ester
Haloxydine	2240	Ester
Weedy check	-	-

Table 19. Varieties, soil characteristics and climatic data for Carman weed control trial, 1970.

Crop, Variety, Soil		Time and crop stage at application	Climatic conditions
Rape (var. Target)		Pre-emerge,	23°C, 90% RH[1],
Seeded 10 June		11 June,	Wind 5-7[2].
Organic matter	2.8%	Time: 1130.	
Sand	86.0%		
Silt	7.0%		
Clay	7.0%		
Flax (var. Redwood 65)		Post-emerge,	16°C, 85% RH,
Seeded 10 June		30 June,	Wind 8-10.
Organic matter	2.8%	Time: 1330.	
Sand	86.0%		
Silt	7.0%		
Clay	7.0%		

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[1] Relative humidity

[2] Kilometers per hour

Experiment IX Haloxydine - soil type interaction trials, 1971

Soil samples were obtained from seven locations and used in a wheat bioassay to determine ED_{50} values of haloxydine. Regression analysis was conducted on the ED_{50} values to determine the soil components affecting herbicide activity.

Samples were collected from several soil horizons to obtain varying levels of organic matter, sand, silt, clay and cation exchange capacities. Each treatment had four replicates. The treatments applied to each soil sample are listed in Table 20.

Techniques used in the bioassay are similar to the greenhouse bioassay; however, experimental units were arranged in a completely randomized design in a growth chamber. A 16 hour day length of 23,680 lux was used with a day temperature of 24°C and a nyctoperiod temperature of 18°C. Soil characteristics of the samples are listed in Table 21.

Table 20. Haloxydine - soil type interaction treatments, 1971.

Treatment	Rate of application (ppmw)		
	Fine textured soil[1]	Coarse textured soil[2]	Silica sand
Check	-	-	-
Haloxydine	0.25	0.125	0.0625
Haloxydine	0.50	0.250	0.0938
Haloxydine	0.75	0.500	0.1250
Haloxydine	1.00	0.750	0.1563

1. Percent sand less than 10%.

2. Percent sand greater than 10%.

Table 21. Soil characteristics of haloxydine - soil type interaction in trials, 1971.

Location,	Depth (cm)	OM%[1]	Sand%	Silt%	Clay%	C.E.C.[2] Meq/100 g
Graysville	0-15	3.5	78	11	11	18.0
	15-30	1.9	75	12	13	14.0
	60-90	0.6	71	14	15	7.6
Carman	0-10	2.8	86	7	7	12.6
Carberry	0-15	4.8	29	46	25	30.9
Sperling	0-15	5.5	38	32	30	29.6
	45-60	1.7	15	50	35	21.2
Glenlea	0-15	4.6	6	35	59	43.1
	30-45	1.3	4	26	70	36.8
Portage la Prairie	0-15	6.6	1	44	55	45.0
	45-60	2.3	6	46	48	35.1
Silica sand	-	0.1	100	-	-	0.3

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1. Organic matter

2. Cation exchange capacity, millequivalents

RESULTS

Experiment I Wild oat control trials, 1969.

The predominant weeds at Fort Whyte were wild buckwheat and green smartweed. Wild oats were absent. There was no control of weeds with pre-emerge applications and very poor control at the higher rates (140 and 280 g/ha) applied at the 5 to 8 cm stage of the flax. Applications at the 10 to 15 cm crop stage showed promising herbicidal activity. Flax was tolerant to all treatments and growing conditions were normal.

The predominant weeds at Portage la Prairie were wild oats, wild buckwheat, green smartweed and green foxtail. There was no weed control from applications at the pre-emerge and 2 to 3 leaf stage of barley. Application of 70 g/ha at the 5 to 6 leaf stage of barley controlled 50% of the wild buckwheat. Application of 140 g/ha controlled 80% of the wild buckwheat, 50% of wild oats in the 3 to 4 leaf stage, and 75% of the green foxtail. Applications of 280 g/ha controlled wild buckwheat and wild oats in the 3 to 4 leaf stage. This rate did not control wild oats in the 5 to 6 leaf stage but did contol 80% of the green foxtail and 80% of the green smartweed. The barley exhibited a slight chlorosis at this rate. Carbyne provided fair control of wild oats.

Growing conditions were normal.

At Brunkild the predominant weeds were green smartweed, wild mustard (Sinapsis arvensis L.) and cocklebur (Xanthium strumarium L.). Wild oats were absent on this site which was late seeded. There was no weed control from pre-emerge applications. Applications at the 2 to 3 leaf stage of the wheat provided slight control at 70 g/ha. Control of 50% of the smartweed and 40% of the wild mustard was obtained at 140 g/ha. The application of 280 g/ha gave 80% control of smartweed and mustard, and a stunting of cocklebur, while the stand of wheat was reduced by 30%.

Barley was more tolerant to haloxydine than was wheat. The margin of selectivity between weeds and cereals was not sufficient to warrant further developmental work.

<u>Experiment II</u> Wild oat and broad leaved weed control trials, 1969.

Visual observations of weed control and crop tolerance are given in Table 22.

The predominant weeds in both the barley and the flax were wild buckwheat, lamb's-quarters, green smartweed, wild oats and green foxtail. Haloxydine at 17.5 and 35 g/ha did not control weeds; however, when 2,4-D was combined with these rates the broad-leaved weed control was better than it was with 2,4-D alone. Broad-leaved weed control using haloxydine at rates of 70 to 280 g/ha was similar to results in Experiment I.

Control of wild oats was similar to Experiment I; however, there was 80% control of wild oats in the 5 to 6 leaf stage with an application of 280 g/ha.

Barley and flax were tolerant to the rates of haloxydine used although the 280 g/ha application rate caused a slight chlorosis on leaf margins. The application of 2,4-D ester to flax caused a delay in maturity.

Table 22. Ratings of weed control and crop tolerance in wild oat and broad-leaved weed control trials, 1969.

Treatment	Rate (g/ha)	Barley W.O.[1]	 B.L.[2]	 Tol.[3]	Flax W.O.	 B.L.	 Tol.
Haloxydine	17.5	0	0	9	0	0	9
Haloxydine	35	0	0	9	0	0	9
Haloxydine	70	0	3	9	0	3	9
Haloxydine	105	0	4	9	0	3	9
Haloxydine	140	3	6	9	2	6	9
Haloxydine	280	6	7	9	6	8	8
Haloxydine + 2,4-D	17.5 + 420	0	3	9	0	3	6
Haloxydine + 2,4-D	35 + 420	0	3	9	0	3	6
Haloxydine + 2,4-D	70 + 420	0	4	9	0	5	6
Haloxydine + 2,4-D	105 + 420	0	5	9	0	5	6
Haloxydine + 2,4-D	140 + 420	3	6	9	3	7	6
Buctril M	560	0	3	9	0	3	9
Banvel 3	560	0	4	9	0	4	5
Weedy check	-	0	0	9	0	0	9

1. Wild oats
2. Broad-leaved weeds
3. Crop tolerance

Experiment III Weed control in rape, 1970.

Visual oberservations of herbicide performance on weed control and crop tolerance at the Graysville, Petersfield and Westbourne locations are given in Tables 23, 24, and 25 respectively. Yield data for the three locations are given in Table 26.

Increasing rates of haloxydine gave better weed control at the Graysville location. The maximum herbicidal effect was not evident at the June 16 rating. Growing condition were good throughout the season and the crop competed vigourously with weed growth. The 420 g/ha application had a yield of 2056 kg/ha and the 840 g/ha application had a yield of 1752 kg/ha. This would suggest a degree of crop phytotoxicity since weed competition to the crop was low (Table 26).

The Petersfield location did not recieve rain for three weeks following haloxydine application. There was no visible effect from the herbicide during this period. Season long weed control was poor since weeds had grown to 8 to 10 cm by the time rainfall occurred causing the herbicide to be moved into the root zone (Table 24). The 420 g/ha application had a yield of 1416 kg/ha and the 840 g/ha application had a yield of 1898 kg/ha. While there was little visible evidence

Table 23. Ratings of weed control and crop tolerance in rape at Graysville, 1970.

Treatment	Rate (g/ha)	16 June				2 July			10 August	
		W.B.[1]	O.W.[2]	G.F.[3]	Tol.[4]	W.B.	O.W.	Tol.	B.L.[5]	Tol.
Haloxydine	420	1	1	0	9	4	4	9	2	9
Haloxydine	560	2	2	2	9	6	6	8	3	8
Haloxydine	700	4	3	2	9	7	7	8	7	8
Haloxydine	840	4	4	3	8	8	8	8	8	8
TOK - RM	1350	-	-	-	-	5	5	9	4	9
Weedy check	-	0	0	0	9	0	0	9	0	9

1. Wild buckwheat
2. Other weeds
3. Green foxtail
4. Crop tolerance
5. Broad-leaved weeds

Table 24. Ratings of weed control and crop tolerance in rape at Petersfield, 1970.

Treatment	Rate (g/ha)	20 July W.B.[1]	20 July O.W.[2]	20 July W.O.[3]	20 July Tol.[4]	12 August W.B.	12 August O.W.	12 August W.O	12 August Tol.
Haloxydine	420	0	0	0	9	0	0	0	9
Haloxydine	560	1	2	0	9	0	1	0	9
Haloxydine	700	1	2	1	9	0	1	1	9
Haloxydine	840	1	2	2	8	0	1	1	9
TOK - RM	1350	0	0	0	9	1	1	0	9
Weedy check	-	0	0	0	9	0	0	0	9

1. Wild buckwheat

2. Other weeds

3. Wild oats and green foxtail

4. Crop tolerance

of weed control with haoxydine this yield data would indicate some suppression of weed growth (Table 26). Weeds were a vigourous competitor with the crop.

The Westbourne location had green foxtail as the predominant weed and very few broad-leaved weeds. There was no rainfall for two weeks following haloxydine application of 700 and 840 g/ha provided some weed control (Table 25). Crop tolerance was good and yield data did not suggest any crop phytotoxicity (Table 26).

Table 25. Ratings of weed control and crop tolerance in rape at Westbourne, 1970.

Treatment	Rate g/ha	23 July B.L.[1]	G.F.[2]	Tol.[3]
Haloxydine	420	2.5	0.0	9.0
Haloxydine	560	3.3	1.0	9.0
Haloxydine	700	4.9	2.4	8.5
Haloxydine	840	6.4	4.4	8.3
TOK - RM	1350	2.1	0.7	9.0
Weedy check	-	0.0	0.0	9.0

1. broad-leaved weeds
2. green foxtail
3. crop tolerance

Table 26. Yield (kg/ha) of weed control in rape, 1970.

Treatment	Rate (g/ha)	Graysville	Petersfield	Westbourne
Haloxydine	420	2056	1416	1835
Haloxydine	560	1928	1658	1780
Haloxydine	700	1864	1718	1989
Haloxydine	840	1752	1898	1809
TOK - RM	1350	1632	1643	1823
Weedy check	-	1612	1386	1665
	L.S.D. 05	N.S.	346	N.S.

Experiment IV Weed control in flax, 1970.

Visual observations of weed control and crop tolerance using haloxydine at Carberry and MacDonald are given in Tables 27 and 28 respectively.

Growing conditions for the summer at Carberry were good. Rainfall occurred six days after pre-emerge applications (0.3 cm) and one day after post-emerge applications (0.6 cm). Post-emerge applications provided better weed control than pre-emerge applications (Table 27.) This may result from the 9.9 cm of rainfall that occurred between pre-emerge and post-emerge applications. On a sandy loam soil this amount of rainfall could move the herbicide out of the zone of germinating weed seeds. There were no differences in crop phytotoxicity in the pre-emerge versus post-emerge haloxydine applications.

Post-emerge haloxydine applications of 140 g/ha and 280 g/ha did not provide adequate weed control. The level of weed control decreased from ratings on 7 July to 7 August. Post-emerge haloxydine applications of 560 g/ha and 840 g/ha provided good control of wild oats and green foxtail as well as broad-leaved weeds throughout the growing season.

The addition of MCPA to haloxydine gave slightly better broad-leaved weed control than the corresponding rates of haloxydine.

The application of the ester formulation of MCPA caused some delay in crop flowering and maturity but did not affect the crop height or stand. The 840 g/ha rate of haloxydine caused a 7 to 10 day delay in crop maturity.

Growing conditions for the summer at MacDonald were normal. Rainfall occurred eight days after pre-emerge applications (1.8 cm) and one day after post-emerge applications (0.8 cm). There were 5.0 cm of rainfall between pre-emerge and post-emerge applications. Post-emerge applications provided slightly better weed control than pre-emerge applications (Table 28). Initial weed control (July 7) was fair but later ratings indicated the herbicidal activity was of a temporary nature and weeds flowered and set seed. Although rainfall was adequate to move the herbicide into the zone of germinating weed seedlings, the herbicide appeared to be adsorbed to the heavy clay soil, rendering the haloxydine non-available for plant uptake. There was crop phytotoxocity at the 840 g/ha rate of application. The weed control of the comparison herbicides (Carbyne and Buctril M) was poor.

Table 27. Ratings of weed control and crop tolerance in flax at Carberry, 1970.

Treatment	Rate (g/ha)	7 July			23 July			7 August			
		B.L.[1]	G.W.[2]	Tol.[3]	B.L.	G.W.	Tol.	B.L.	W.O.[4]	G.F.[5]	Tol.
Haloxydine-pre	140	2	2	9	0	0	9	3	0	1	9
Haloxydine-pre	280	5	3	9	2	1	9	3	2	2	9
Haloxydine-pre	560	6	5	8	4	2	8	3	5	3	8
Haloxydine-pre	840	7	5	6	7	6	6	6	8	6	8
Haloxydine-post	140	5	5	9	3	2	9	2	1	2	9
Haloxydine-post	280	5	7	8	5	6	8	3	3	5	8
Haloxydine-post	560	8	8	8	7	8	8	7	7	8	8
Haloxydine-post	840	8	9	8	8	9	8	8	8	8	8
Haloxydine+MCPA	140+420	6	6	8	4	5	8	3	0	4	8
Haloxydine+MCPA	280+420	7	7	7	6	6	8	4	5	5	8
Haloxydine+MCPA	560+420	8	8	6	8	8	7	8	8	8	8
Carbyne	350	0	3	9	0	4	9	0	6	0	9
Buctril M	560	9	0	9	8	1	9	8	0	0	9
Weedy check	-	0	0	9	0	0	9	0	0	0	9

1. Broad-leaved weeds
2. Grassy weeds
3. Crop tolerance
4. Wild oats.
5. Green foxtail

Table 28. Ratings of weed control and crop tolerance in flax at MacDonald, 1970.

Treatment	Rate (g/ha)	7 July W.B.[1]	7 July W.O.[2]	7 July Tol.[3]	11 August W.B.	11 August W.O.	11 August Tol.
Haloxydine-pre	140	2	1	9	0	0	9
Haloxydine-pre	280	3	1	9	0	0	9
Haloxydine-pre	560	4	3	7	0	0	8
Haloxydine-pre	840	5	4	6	0	0	7
Haloxydine-post	140	3	2	8	0	0	9
Haloxydine-post	280	5	2	8	0	0	8
Haloxydine-post	560	6	3	8	1	0	8
Haloxydine-post	840	6	3	8	2	1	8
Haloxydine+MCPA	140+420	4	3	7	1	0	8
Haloxydine+MCPA	280+420	5	3	6	1	0	8
Haloxydine+MCPA	560+420	7	4	5	1	0	7
Carbyne	350	0	4	9	0	2	9
Buctril M	560	6	0	8	4	0	9
Weedy check	-	0	0	9	0	0	9

1. Wild buckwheat   2. Wild oats   3. Crop tolerance

Experiment V Weed control in potatoes, 1970.

Visual observations of weed control and crop tolerance using Haloxydine at Carberry and Carman are given in Tables 29 and 30 respectively. Yield data for the two locations are given in Table 31.

The Carberry location had good growing conditions throughout the season. The rates of haloxydine used did not cause any crop phytotoxicity (Table 29). Good control of broad-leaved weeds and grassy species was obtained with all haloxydine treatments. Post-emerge applications were slightly superior to pre-emerge applications. The addition of Gramoxone to haloxydine did not give a marked improvement in weed control. Gramoxone and Lorox gave good control initially, however, later germinating seedlings were not controlled. There were no significant yield differences amongst the haloxydine and/or Gramoxone treatments (Table 31). The yield of the Lorox treatments was significantly lower than the yields of the other herbicide treatments. The yield of the hand weeded check was lower than the yield of the cultivated check.

The Carman location had normal growing conditions. Rainfall at Carmn was 7.5 cm less than at Carberry in the June-July period; however, Carman recieved 8.0 cm more rain in August than did Carberry. Haloxydine treatments provided acceptable to excellent weed control

(Table 30). Even though weed populations at Carman were higher than at Carberry (Table 16) the weed control was similar at both locations. Haloxydine treatments were phytotoxic to the potatoes at Carman. Haloxydine at 560 g/ha caused moderate yield reductions and reduction in plant vigour (Table 31). Haloxydine at 1120 g/ha caused a total crop failure as the tuber size obtained was too small to be marketable. The haloxydine at 560 g/ha in combination with Gramoxone did not have as severe yield reduction as did haloxydine used alone. Haloxydine at 280 g/ha in combination with Gramoxone gave good weed control and was not phytotoxic to the potatoes. Gramoxone and Lorox treatments did not provide season-long weed control. At both locations tuber size from the cultivated check treatments was larger than tuber size from the other treatments which did not recieve a "hilling" operation.

Table 29. Ratings of weed control and crop tolerance in potatoes at Carberry,1970.

Treatment	Rate (g/ha)	7 July			23 July			7 August		
		B.L.[1]	G.W.[2]	Tol.[3]	B.L.	G.W.	Tol.	B.L.	G.W.	Tol.
Haloxydine-pre	560	7	7	8	5	6	9	7	7	9
Haloxydine-pre	1120	8	8	8	7	7	8	8	8	9
Haloxydine-post	560	9	9	8	8	8	9	8	8	9
Haloxydine-post	1120	9	9	7	9	9	8	8	8	9
Haloxydine+GM[4]	280+560	9	9	8	8	8	9	8	8	9
Haloxydine+GM	560+560	9	9	8	9	8	9	9	8	9
Gramoxone	840	8	9	8	8	9	9	7	8	9
Gramoxone	1120	8	9	8	9	8	9	6	7	8
Lorox	1680	4	6	8	1	5	9	1	2	9
Handweeded check	-	3	4	9	6	6	9	8	6	9
Cultivated check	-	0	0	9	6	8	9	8	8	9

1. Broad-leaved weeds
2. Grassy weeds
3. Crop tolerance
4. Gramoxone

Table 30. Ratings of weed control and crop tolerance in potatoes at Carman, 1970.

Treatment	Rate (g/ha)	2 July			17 July			10 August		
		B.L.	G.W.	Tol.	B.L	G.W.	Tol.	B.L.	G.W.	Tol.
Haloxydine-pre	560	9	8	7	8	7	7	8	5	7
Haloxydine-pre	1120	9	9	3	9	9	2	9	9	1
Haloxydine-post	560	8	7	8	8	7	8	7	6	7
Haloxydine-post	1120	9	7	7	9	8	6	9	9	2
Haloxydine+GM[4]	280+560	8	8	8	8	7	9	7	5	8
Haloxydine+GM	560+560	9	8	8	8	7	8	8	7	7
Gramoxone	840	7	7	8	2	3	9	3	3	9
Gramoxone	1120	8	8	8	3	4	9	4	3	8
Lorox	1680	6	4	9	2	2	9	1	1	6
Handweeded check	-	7	6	9	7	5	9	6	5	9
Cultivated check	-	7	6	9	8	6	9	7	8	9

1. Broad-leaved weeds
2. Grassy weeds
3. Crop tolerance
4. Gramoxone

Table 31. Yield (Quintals/ha) of weed control in potatoes, 1970.

Treatment	Rate (g/ha)	Carberry	Carman
Haloxydine-pre-emerge	560	220.2	115.5
Haloxydine-pre-emerge	1120	231.2	17.4
Haloxydine-post	560	221.9	107.0
Haloxydine-post	1120	215.6	31.7
Haloxydine+Gramoxone	280+560	225.6	182.9
Haloxydine+Gramoxone	560+560	221.9	115.7
Gramoxone	840	220.6	154.3
Gramoxone	1120	202.9	137.8
Lorox	1680	170.2	168.6
Handweeded check	-	185.7	162.8
Cultivated check	-	215.8	218.1
	L.S.D.05	30.5	36.1

Experiment VI Haloxydine residue trial at Graysville, 1970.

a) Field Bioassay. Visual observations of crop tolerance on the crops grown in 1971 are given in Table 32. Yield data for wheat and rape grown in 1971 is given in Table 33.

Haloxydine caused some phytotoxicity to wheat at 560 and 1120 g/ha (Table 32). There appeared to be a reduction in wheat yield at these rates of application (Table 33). At 2240 g/ha, haloxydine caused very severe stand reduction to wheat as well as a reduction in plant height. The few plants surviving tillered well and produced a higher than expected yield.

Flax plants were slightly reduced in height and delayed in maturity by the 560 g/ha application of haloxydine (Table 32). An application of 1120 g/ha gave a slight reduction in stand while the 2240 g/ha application resulted in a severe stand reduction.

Rape appeared completely tolerant to rates of 560 and 1120 g/ha of haloxydine (Table 32). Yield figures from these two treatments are equivalent to the check (Table 33). The application of 2240 g/ha of haloxydine caused a mild chlorosis early in the growing season and yields were reduced.

Alfalfa was not tolerant to the 1120 and 2230 g/ha of haloxydine (Table 32). The 560 g/ha application of haloxydine caused a very severe stand reduction of the alfalfa.

Table 32. Ratings of crop tolerance, Graysville residue trials, 1970.

Treatment	Rate (g/ha)	17 June				6 July				21 July				3 Sept.			
		W.[1]	F.[2]	R.[3]	A.[4]	W.	F.	R.	A.	W.	F.	R.	A.	W.	F.	R.	A.
Haloxydine	560	7	7	8	1	8	8	9	1	7	8	9	1	8	7	9	1
Haloxydine	1120	6	4	8	0	6	5	9	0	5	4	9	0	5	3	9	0
Haloxydine	2240	1	2	7	0	1	2	8	0	1	2	9	0	1	1	8	0
Check	-	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9

[1] Wheat

[2] Flax

[3] Rape

[4] Alfalfa

Table 33. Yield (Quintals/ha) of Graysville residue trial, 1970.

Treatment	Rate (g/ha)	Wheat	%[1]	Rape	%
Haloxydine	560	29.4	80.3	23.6	97.8
Haloxydine	1120	29.0	79.2	22.1	91.4
Haloxydine	2240	9.6	26.2	20.9	86.4
Check	-	36.6	100.0	24.2	100.0
	L.S.D$_{05}$	13.4		3.3	

[1] Percentage of control

b) Greenhouse bioassay. The amounts of herbicide detected by the greenhouse bioassay are given in Table 34.

Haloxydine residues were detected in the 15 to 23 cm zone in the fall sampling in 1970 (Table 34). The herbicide had moved to this depth at all rates of application. The total amounts of herbicide recovered indicated that it had not been degraded. Rainfall occuring between application and sampling was 11.5 cm.

Spring sampling in 1971 detected haloxydine at the same soil depths as the previous fall sampling (Table 34). Rainfall between April 1 and spring sampling was 3.1 cm. Total amounts recovered were less than the 1970 results indicating that degredation had occurred.

Fall sampling in 1971 detected haloxydine to the 30 cm depth with the 560 g/ha application and to the 45 cm depth for higher rates of application (Table 34). The total amount recovered indicated an increased rate of degredation for the 2240 g/ha application compared to the 560 and 1120 g/ha rates of application. There were similar amounts of haloxydine in the 0-15 cm zone for all rates of application.

Table 34. Ppmw and % recovery of haloxydine on three occasions, Graysville, 1970.

a) Fall sample 3/11/70 — Rate of application (g/ha)

Depth (cm)	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-5	.205	24.2	.233	13.7	.705	20.8
5-10	.250	29.5	.925	54.5	1.068	31.5
10-15	.214	25.2	.387	22.8	.847	25.0
15-23	.102	21.6	.163	17.3	.230	12.2
23-30	-	-	-	-	-	-
		100.5		108.3		89.5

b) Spring sample 6/5/71 — Rate of application (g/ha)

Depth (cm)	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-5	.195	23.0	.203	12.0	.656	19.3
5-10	.250	29.5	.487	28.7	.742	21.9
10-15	.200	23.6	.256	15.1	.638	18.8
15-23	.042	8.9	.238	25.2	.288	15.2
23-30	-	-	-	-	-	-
		85.0		81.0		75.2

c) Fall sample 20/10/71 — Rate of application (g/ha)

Depth (cm)	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-15	.106	37.5	.122	21.6	.129	11.4
15-23	.098	20.7	.092	9.7	.108	5.7
23-30	.090	19.0	.182	19.3	.267	14.1
30-45	-	-	.082	18.6	.094	10.6
		77.2		69.2		41.8

Experiment VII Haloxydine residue trial at Glenlea, 1970.

a) Field bioassay. Visual observations of crop tolerance on the crops grown in 1971 are given in Table 35. Yield data for wheat and rape grown in 1971 are given in Table 36.

Crop phytotoxicities were similar to results obtained in Experiment VI on a sandy loam soil. Wheat had a high degree of tolerance to the 560 g/ha application of haloxydine, while the 1120 g/ha application was slightly phytotoxic (Table 35). The 2240 g/ha application caused a very severe reduction of the stand of wheat. There was a significant yield reduction of the wheat at the 2240 g/ha application (Table 36).

Flax was slightly deformed by the 560 g/ha application of haloxydine (Table 35). The 1120 g/ha application caused a slight stand reduction was well as a reduction in crop height. Flax was not tolerant to the 2240 g/ha application after the initial germination and the flax plants died in the cotyledon stage.

Rape was tolerant to applications of 560 and 1120 g/ha of haloxydine (Table 35); however, the 2240 g/ha applications caused a significant yield reduction and some phytotoxicity to the crop (Table 36).

Alfalfa was not tolerant to the 1120 and 2240 g/ha rates of haloxydine (Table 34). Plants germinated on

Table 35. Ratings of crop tolerance, Glenlea residue trial, 1970.

Treatment	Rate (g/ha)	22 June				20 July				8 Sept.			
		W.[1]	F.[2]	R.[3]	A.[4]	W.	F.	R.	A.	W.	F.	R.	A.
Haloxydine	560	8	6	9	1	8	7	9	1	8	6	9	1
Haloxydine	1120	6	4	8	0	6	3	9	0	8	3	9	0
Haloxydine	2240	1	0	6	0	1	0	8	0	1	0	8	0
Check	-	9	9	9	9	9	9	9	9	9	9	9	9

1. Wheat
2. Flax
3. Rape
4. Alfalfa

Table 36. Yield (Qintals/ha) of Glenlea residue trial, 1970.

Treatment	Rate (g/ha)	Wheat	%[1]	Rape	%
Haloxydine	560	24.0	82.8	18.7	94.6
Haloxydine	1120	21.5	74.1	17.8	90.3
Haloxydine	2240	15.7	54.2	14.2	72.0
Check	-	29.0	100.0	19.8	100.0
	L.S.D.$_{05}$	10.0		2.5	

[1] Percentage of control

all treatments but became severely chlorotic in the cotyledon stage. The 560 g/ha application had a very severe stand reduction.

b) <u>Greenhouse bioassay</u>. The amounts of herbicide detected by the greenhouse bioassay are given in Table 37.

The fall 1970 sampling indicated increasing depth of herbicide penetration with an increasing rate of herbicide application (Table 37). There was a relatively short period of time between application and sampling (two weeks) during which 2.3 cm of rain were recorded. Soil moisture levels were high at the time of initial applications. There did not appear to be any herbicide degradation during this interval and the low recovery of the 560 g/ha application may be attributed to the nature of the bioassay (biological variation of the material used in the bioassay).

The spring 1971 sampling did not indicate any further movement of the herbicide. The total amounts recovered would indicate that degradation was starting to take place and at a higher rate on the higher application (2240 g/ha).

The fall 1971 sampling indicated further movement of the 560 g/ha application into the 5 to 10 cm level while residues were not detected in the 10 to 15 cm level of the 2240 g/ha application (Table 37). There were similar levels of haloxydine in the 0-5 cm level at all three rates of application. The highest rate of application had the greatest degradation.

Table 37. Ppmw and % recovery of haloxydine on three occasions, Glenlea, 1970.

a) Fall Sample 1/11/70

Depth (cm)	Rate of application (g/ha)					
	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-5	.740	82.6	1.312	73.2	1.750	48.8
5-10	-	-	.480	26.8	1.362	38.0
10-15	-	-	-	-	.542	15.1
		82.6		100.0		101.9

b) Spring sample 13/5/71

Depth (cm)	Rate of application (g/ha)					
	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-5	.717	80.0	1.037	57.9	1.415	39.5
5-10	-	-	.577	32.2	.929	25.9
10-15	-	-	-	-	.497	13.9
		80.0		90.1		79.3

c) Fall sample 29/9/71

Depth (cm)	Rate of application (g/ha)					
	560		1120		2240	
	ppmw	%	ppmw	%	ppmw	%
0-5	.300	33.5	.300	16.7	.425	11.9
5-10	.313	34.9	.744	41.5	.313	8.7
10-15	-	-	-	-	-	-
		68.4		57.2		20.6

Experiment VIII Weed control trial at Carman, 1971.

Visual observation of haloxydine weed control and crop tolerance in rape are given in Table 38. The soil at this location contained 86% sand and 14% silt and clay. Haloxydine was more phytotoxic to rape at Carman than had previously been observed in Experiment III. This increased phytotoxicity may be attributed to the low silt and clay content of the soil. The ester formulation of haloxydine was less phytotoxic to rape than was the potassium salt formulation (Table 38). The ester formulation was less effective than the potassium salt formulation in controlling the seeds present, particularly the grassy weeds. The 2240 g/ha application of potassium haloxydine very severely reduced the stand of rape.

Visual observations of weed control and crop tolerance of flax are given in Table 39. The results of applications of ester and potassium formulations of haloxydine to flax were similar to the results with rape. Both crops were grown in the same field and the potassium formulation was more phytotoxic to flax than was the ester formulation. The weed control was slightly less than with rape and this may be a result of the more vigorous crop competition occurring with rape.

Table 38. Ratings of weed control and crop tolerance of rape, Carman, 1971.

Treatment	Rate (g/ha)	17 June		6 July			22 July			3 Sept.
		B.L[1]+G.W.[2]	Tol.[3]	B.L.	G.W.	Tol.	B.L.	G.W.	Tol.	Tol.
Haloxydine - K[4]	560	9	7	9	9	3	8	8	3	5
Haloxydine - K	1120	9	7	9	9	1	9	9	1	2
Haloxydine-ester	560	8	9	8	7	8	7	5	8	6
Haloxydine-ester	1120	9	8	8	8	5	7	7	5	5
Haloxydine-ester	2240	9	8	9	9	3	9	8	3	4
Weedy check	-	0	9	0	0	9	0	0	9	9

1. Broad-leaved weeds

2. Grassy weeds

3. Crop tolerance

4. Potassium

Table 39. Ratings of weed control and crop tolerance of flax at Carman, 1971.

Treatment	Rate (g/ha)	6 July			21 July			3 September		
		B.L.[1]	G.W.[2]	Tol.[3]	B.L.	G.W.	Tol.	B.L.	G.W.	Tol.
Haloxydine-potassium	560	8	8	7	8	8	6	8	8	5
Haloxydine-potassium	1120	8	8	5	9	9	2	9	9	1
Haloxydine-ester	560	5	4	7	6	3	7	5	3	8
Haloxydine-ester	1120	8	7	5	8	6	5	7	6	5
Haloxydine-ester	2240	8	8	2	8	8	3	8	6	3
Weedy check	-	0	0	9	0	0	9	0	0	9

1. Broad-leaved weeds

2. Grassy weeds

3. Crop tolerance

Experiment IX Haloxydine - soil type interaction trial, 1971.

The ED_{50}values (ppmw) of the soil samples as determined by a bioassay are listed in Table 40. A multiple regression analysis of the independent variables; organic matter, cation exchange capacity, sand, silt and clay upon the dependent variable ED_{50} was conducted to determine the soil compenents affecting herbicide activity. The multiple correlation coefficients "R^2" and "t" values of the independent variables are listed in Table 41. R^2 is the proportion of the total sums of squares attributable to regression and the "t" value is a test of significance of the coefficients of the independent variables.

A multiple regression analysis was also conducted on variables affecting cation exchange capacity. Cation exchange capacity is determined by the reaction sites present in organic matter and clay, therefore there is a correlation between cation exchange capacity and the other variables used in the analysis of the ED_{50} values. Table 42 lists "R^2" and "t" values of the independent variables organic matter, sand, silt and clay upon the dependent variable cation exchange capacity.

In the analysis of ED_{50} the R^2 value of the five independent variables is .9322 (Table 41). Cation exchange capacity is the single variable which has the highest R^2 value and is responsible fo 86.6% of the total regression. The regression equation of ED_{50} (Y) on cation exchange capacity is

$$Y = 0.0790 + 0.0184 \text{ C.E.C.}$$

When two independent variables are used to explain the regression, the highest R^2 value is obtained with the cation exchange capacity and clay variables (Table 41). The regression equation of ED_{50} (Y) on cation exchange capacity and clay is

$$Y = 0.0360 + 0.0329 \text{ C.E.C.} - 0.0102 \text{ clay.}$$

The regression coefficient of clay has a negative value as a result of the strong correlation of clay with the cation exchange capacity. The R^2 value of clay on cation exchange capacity is 0.8063 (Table 42).

The use of organic matter and sand to explain the ED_{50} values results in a R^2 value of 0.8746 which explains 93.8% of the total regression (Table 41). The regression equation of ED_{50} (Y) on organic matter (O.M.) and sand is

$$Y = 0.4216 + 0.0898 \text{ O.M.} - 0.0037 \text{ sand.}$$

Organic matter and sand are preferable to cation exchange capacity and organic matter as the former has a higher level of significance in testing the "t" values.

The use of three variables, cation exchange capacity, sand and silt in explaining the ED_{50} results in a R^2 value of 0.9319 which explaisn 99.96% of the total regression (Table 41). The regression equation of ED_{50} (Y) on cation exchange capacity, sand and silt is

$$Y = 0.9794 + 0.0311 \text{ C.E.C.} - 0.0101 \text{ sand} + 0.0119 \text{ silt.}$$

These three variables are used in preference to others on the basis of the highest level of significance of the "t" values.

The use of four variables did not result in any additional explanation of the total regression.

Table 40. ED_{50} values (ppmw) in haloxydine - soil type interaction trials, 1971.

Location	Depth (cm)	ED_{50} (ppmw)
Graysville	0-15	0.403
	15-30	0.376
	60-90	0.125
Carman	0-10	0.380
Carberry	0-15	0.925
Sperling	0-15	0.675
	45-60	0.438
Glenlea	0-15	0.993
	30-45	0.510
Portage la Prairie	0-15	0.855
	45-60	0.623
Silica sand	-	0.066

Table 41. R^2 and "t" values of the independent variables on ED_{50}.

a) One variable

X_1	R^2	"t" value X_1	X_2	X_3
C.E.C.[1]	.8072	6.470^{xx}		
O.M.[2]	.6983	4.811^{xx}		
Sand	.5681	3.627^{xx}		
Silt	.5425	3.444^{xx}		
Clay	.4283	2.737^{x}		

b) Two Variables

X_1	X_2	R^2	"t" value X_1	X_2
C.E.C.	+ clay	.9270	7.841^{xx}	3.843^{xx}
C.E.C.	+ O.M.	.8917	4.011^{xx}	2.652^{x}
O.M.	+ sand	.8746	4.689^{xx}	3.556^{xx}
O.M.	+ clay	.8481	4.987^{xx}	2.978^{x}
C.E.C.	+ sand	.8355	3.824^{xx}	1.244
O.M.	+ silt	.8324	3.946^{xx}	2.685^{x}
C.E.C.	+ silt	.8139	3.623^{xx}	0.572
Silt	+ clay	.5887	1.873	1.005
Sand	+ clay	.5887	1.873	0.670
Sand	+ silt	.5887	1.005	0.670

c) Three Variables

X_1	X_2	X_3	R^2	"t" value X_1	X_2	X_3
C.E.C.	+ sand	+ silt	.9319	6.350^{xx}	3.723^{xx}	3.366^{xx}
C.E.C.	+ sand	+ clay	.9319	6.350^{xx}	0.760	3.366^{xx}
C.E.C.	+ silt	+ clay	.9319	6.350^{xx}	0.760	3.723^{xx}
C.E.C.	+ O.M.	+ clay	.9280	2.979^{x}	0.331	2.006^{x}
C.E.C.	+ O.M.	+ silt	.9018	2.377^{x}	2.676^{x}	0.906
C.E.C.	+ O.M.	+ sand	.8922	1.146	2.053	0.192
O.M.	+ silt	+ clay	.8747	4.272^{xx}	1.303	1.641
O.M.	+ sand	+ silt	.8747	4.272^{xx}	1.641	0.080
O.M.	+ sand	+ clay	.8747	4.272^{xx}	1.303	0.080
Sand	+ silt	+ clay	.5887	0.004	0.010	0.006

d) Five variables

C.E.C. + O.M. _ sand + silt + clay $R^2 = .9322$

[1] C.E.C. cation exchange capacity

[2] O.M. organic matter

xx Significant at .01 level of probability

x Significant at .05 level of probability

Table 42. R^2 and "t" values of the independent variables on cation exchange capacity.

		"t" values			
	R^2	X_1	X_2	X_3	X_4
a) One variable					
X_1					
Sand	.8366	7.155^{xx}			
Clay	.8063	6.453^{xx}			
Silt	.5781	3.702^{xx}			
Organic matter	.4878	3.086^{x}			
b) Two variables					
X_1 X_2					
Organic matter+clay	.9751	7.805^{xx}	13.264^{xx}		
Organic matter+sand	.9374	3.806^{xx}	8.038^{xx}		
Clay +silt	.8510	4.059^{xx}	1.641		
Sand +silt	.8510	4.059^{xx}	0.931		
Clay +sand	.8510	0.931	1.641		
Organic matter+silt	.7121	2.046	2.648^{x}		
c) Three variables					
X_1 X_2 X_3					
Organic matter+sand+silt	.9783	6.859^{xx}	9.916^{xx}	3.890^{xx}	
Organic matter+clay+silt	.9783	6.859^{xx}	9.916^{xx}	1.098	
Organic matter+clay+sand	.9783	6.859^{xx}	3.890^{xx}	1.098	
Sand +silt+clay	.8510	0.001	0.006	0.011	
d) Four variables					
X_1 X_2 X_3 X_4					
Organic+sand+silt+clay	.9783	7.584	0.002	0.003	0.028

DISCUSSION

Haloxydine is primarily a soil acting herbicide, thus the availability of haloxydine to plant species is dependent in a large part on the soil characteristics. The results of Experiment IX indicate a ninefold difference in amounts of haloxydine required to reach the ED_{50} value on a variety of Manitoba soils. This great range of haloxydine required to obtain a uniform biological response was obtained on naturally occurring soils rather than on artificial mediums.

Elliott (1970) found that three different triazine herbicides had approximately 10% to 50% differences in amounts required to reach an ED_{50} value. These herbicides were tested on four different soils with sand contents ranging from 3% to 76% and cation exchange capacities ranging from 14.5 to 31.6 millequivalents per 100 grams of soil.

Thus under Manitoba conditions haloxydine tends to have a more variable response than some other soil acting herbicides.

The results of Experiment IV confirm this variable response where a given rate of haloxydine provided good weed control at Carberry and very poor weed control was obtained at MacDonald. An estimate of the ED_{50} values would be 0.9 ppmw of haloxydine at MacDonald and 0.4 ppmw at Carberry. This large amount of haloxydine required to reach an ED_{50} at MacDonald explains the poor weed control obtained.

Soil moisture is important in making haloxydine available to the plant. Herbicides are frequently moved into the soil profile via a downward mass movement with water, Harris (1967). The Petersfield location in Experiment III did not receive rainfall for several weeks following haloxydine applications, resulting in poor weed control of established plants. When rainfall occurred, haloxydine moved into the soil profile and became effective. Weed control was good following rainfall and there appeared to be little breakdown of haloxydine on the soil surface.

Soil moisture affects the adsorption of herbicides on a colloidal surface. When soil moisture is low, a herbicide is strongly adsorbed and less available for plant uptake, Bailey and White (1964). The results with haloxydine in 1969 appeared to be affected by soil moisture levels. Poor weed control was obtained with applications in May and June when soil moisture was low. Applications in July when soil moisture had improved gave good weed control.

Rainfall affected the movement of haloxydine through the soil profile. The results of Experiment VI indicate that haloxydine moved to the 23 cm depth of a sandy soil during a two month period receiving 11.5 cm of rainfall. In contrast, Elliott (1970) found a maximum penetration to 15 cm in one case and more frequently, the 10 cm level was maximum depth of penetration. This rapid movement of haloxydine is a probable cause of the poorer weed control obtained with pre-emerge applications at Carberry in Experiment IV. It appeared that haloxydine was leached out of the zone of germinating weeds and late germinating weeds were not controlled.

The overall performance of haloxydine was dependent on adequate moisture and on soil characteristics. If the soil contained large amounts of clay and organic matter or rainfall was inadequate, then there was little biological activity as haloxydine was not available to the plants (MacDonald - Experiment IV and Petersfield - Experiment III).

In cases of excess rainfall and very sandy soils, the weed control obtained with haloxydine was of a temporary nature due to the rapid leaching of haloxydine through the soil profile (Carberry - Experiment IV and Experiment VIII).

However, in cases where haloxydine was used on loam soils under adequate rainfall, haloxydine proved to be an effective herbicide (Carberry - Experiment V).

The results of Experiment IX indicate that various soil parameters can be used in explaining the amounts of haloxydine required to obtain a given biological response. Cation exchange capacity is a valuable predictor of haloxydine activity. Other soil characteristics have been documented for large areas of Manitoba and in this regard, organic matter and sand contents can be of more practical benefit than cation exchange capacities in drawing up a recommendation for a given area.

Haloxydine movement through the soil profile is primarily affected by sand content of a soil. The results of Experiment VI and VII indicate haloxydine movement to the 45 cm and 15 cm depths on soils containing 78% and 6% sand, respectively. Haloxydine leached very quickly and was detected at the 23 cm depth on a sandy soil after 67 days and the 15 cm depth on a clay soil after 17 days. These rapid rates of haloxydine movement could offer an explanation for the poorer weed control obtained with pre-emerge treatments at Carberry in Experiment IV.

Haloxydine persists in the soil to the extent that susceptible crops can be severely damaged when grown one year after treatment (Experiment VI and Experiment VII). There was very little degradation between application and sampling dates in Experiment VI. Sampling a year after application detected approximately 70% of the original amounts of haloxydine applied at the 560 g/ha and 1120 g/ha rates of haloxydine (Experiment VI and Experiment VII). The highest rate of application (2240 g/ha) also had sufficient haloxydine present to damage succeeding crops. After one year the 0 cm to 15 cm soil zone had similar levels of haloxydine for all rates of application.

The results of Experiment VIII would suggest that the ester formulation of haloxydine did not appear to have a wider range of selectivity than did the potassium formulation. At equivalent rates the ester formulation was less phytotoxic to crops; however, it was also less effective in controlling weeds compared to the potassium formulation. Compared to the potassium formulation, the ester formulation had a higher threshold level to obtain biological activity but did not have increased selectivity.

The relative crop tolerance to haloxydine was rape wheat = flax alfalfa in Experiments VI and VII. In comparing the crops grown where selective weed control with haloxydine was attempted a relative placing of crop tolerance is rape > potatoes = flax > wheat and barley. Slater (1968) and the Oregon State University, Spring, (1969) also found that rape had good tolerance to haloxydine. Even though rape was the most tolerant crop to haloxydine the results of Experiment III indicated a slight phytotoxicity in a sandy loam soil (Graysville) but not in a loam (Westbourne) or clay (Petersfield) soil. The results of Experiment V with potatoes and Experiment VIII with rape indicate that the likelihood of crop damage is much higher on sandy soils.

Crop tolerances as well as weed control were highly dependent on soil moisture and soil characteristics.

SUMMARY AND CONCLUSIONS

Haloxydine activity on crops and weed species is highly dependent on soil type and climatic factors. Wide variations in haloxydine performance were experienced from one soil type to another and between various crops. This variation with soil type presents difficulties in making a recommendation of rates of application to be used for weed control in a given crop.

Soils requiring large amounts of haloxydine to obtain and ED_{50} response appear to have less capacity to "buffer" the amount of haloxydine available to a plant. The margin of selectivity of haloxydine, between weed and crop species, was less in soils with high ED_{50} values (clay soils) than in soils with low ED_{50} values (sandy soils).

Soil moisture is required in amounts necessary to enable haloxydine to be taken up by the plants; however, if heavy rainfalls occur, poor results are often obtained in sandy soils.

A determination of soil parameters can be a valuable aid in predicting the performance of haloxydine in Manitoba soils. It is not known if these results can be extrapolated to other areas where soils have been formed under different conditions. However, such a technique would appear feasible for recommending amounts of haloxydine once a series of standards has been determined. Since haloxydine appears to be largely soil acting, a useful study would be the determination of the basis for selectivity between crops and weed species. If this knowledge were available, the development of a technique for using haloxydine as a selective herbicide would be facilitated.

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