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PERSISTENCE AND PHYTOTOXICITY OF DINITROANILINE HERBICIDES IN MANITOBA SOILS

by

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TITLE: Persistence and Phytotoxicity of Dinitroaniline Herbicides in Manitoba Soils

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The persistence and phytotoxicities of trifluralin (α, α, α-trifluro-2, 6-dinitro-N, N-dipropyl-p-toluidine), profluralin (N-(cyclopropylmethyl) -α, α, α-trifluro-2, 6-dinitro-N-propyl-p-toluidine), fluchloralin (N-(2-chloroethyl) -2, 6-dintro-N-propyl-4-(trifluromethyl) aniline), and dinitramine (N^4, N^4-diethyl -α, α, α-trifluro-3, 5-dinitrotoluene-2, 4-diamine) were compared in Manitoba soils. Analytical determinations and field bioassay results, one year after application of the chemicals on light soil, indicated the order of persistence as: profluralin > trifluralin ≥ fluchloralin > dinitramine. On heavy soils, the order of dinitroaniline persistence was: profluralin > dinitramine > trifluralin > fluchloralin, one year after application of the chemicals. Organic matter increased persistence of trifluralin and fluchloralin while clay content increased persistence of profluralin and dinitramine. The phytotoxicities of dinitroanilines were: dinitramine > trifluralin > profluralin =

fluchloralin. The phytotoxicities of all chemicals decreased with increased organic matter.

Under low soil temperatures (4°C), trifluralin caused a greater inhibition of wild oat (*Avena fatua* L.) roots than the other chemicals while dinitramine and fluchloralin were the most phytotoxic herbicides at the higher temperatures (8°C). Root length was the best criterion for detection of dinitroaniline herbicide activity. Shoot growth was inhibited most by trifluralin at all temperatures tested but shoot dry weight was a poor indicator of herbicide injury.

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INTRODUCTION

Substituted dinitroaniline chemicals have been introduced as preplant, soil-incorporated herbicides for the control of annual grasses and certain broadleaf weeds in many agronomic and horticultural crops.

Trifluralin, a dinitroaniline herbicide, has been used commercially since 1963 and extensive research has been conducted concerning its soil persistence. However, relatively little is known about the persistence and phytotoxicity of three recently introduced dinitroaniline herbicides, profluralin, fluchloralin, and dinitramine. Knowledge of the persistence characteristics expressed by each of these herbicides under Manitoba conditions of climate and edaphic factors is essential when utilizing them in a normal crop rotation program. Information on persistence is required when selecting a herbicide which will not persist in the soil in large enough quantities to injure susceptible crops grown the year following application.

An indication of the phytotoxicity of these chemicals at low soil temperatures is useful when considering their herbicidal activity during periods of low soil temperatures early in the growing season. The performance of the herbicide under these conditions may determine the level of weed control obtained in the current crop year.

Experiments were conducted to determine differential persistence and phytotoxicity between trifluralin, profluralin, fluchloralin, and dinitramine* as affected by soil type and application rate under Manitoba conditions. The phytotoxicities of the herbicides at low soil temperatures was also studied.

*Chemical structures of the dinitroaniline herbicides used in this study are given in the appendix.

LITERATURE REVIEW

INTRODUCTION

Dinitroaniline herbicides are most effective on annual grassy weeds but also control some broadleaf weeds (Harvey, 1973a). Although dinitroanilines are similar chemically, they differ in effectiveness in controlling weeds (Murray *et al.*, 1973; Harvey, 1973a; Harvey, 1973b).

Dinitroaniline herbicides are applied and incorporated into the soil and their interactions with soil components greatly affect their behaviour under field conditions (Harvey, 1973b). Persistence of these herbicides depends on specific properties of the chemical and on climatic and edaphic factors which affect the rate of disappearance (Horowitz, 1969).

Murray *et al.* (1973) noted that there appears to be a definable variation in phytotoxicity as the chemical structure of dinitroaniline compounds is changed and that there appears to be an inherent phytotoxicity in each of these herbicides with the differential selectivity being controlled primarily by herbicide rate selection.

Work done by Rahman (1973) showed that the phytotoxicity of trifluralin can vary with temperature and soil type.

ANALYTICAL TECHNIQUES

According to Messersmith *et al.* (1971), interpretation of small changes in phytotoxicity of a herbicide from a biological assay is difficult because a change of some factors such as soil moisture and soil pH can affect availability of a compound for plant absorption. They used corn* dry weight as an indicator for trifluralin presence in sandy loam and silty clay loam soils, and noted that corn was sensitive to 8 ppmw of trifluralin in the silty clay loam soil and to only 4 ppmw in sandy loam soil.

Burnside (1974) also found that corn dry weight was not a definitive indicator for trifluralin in silty clay loam soil in the greenhouse as ED_{50} was 4 ppmw. For field studies, he used millet top growth and oat seed production as indicators of trifluralin persistence, again in silty clay loam soil.

Horowitz (1969), working with several herbicides including trifluralin, found that oats, growing in very low concentrations of herbicides in the greenhouse, were frequently taller and had a greater fresh weight than the control plants.

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*Generic names for all plants cited in literature review are presented in the Appendix.

Oat fresh weights were used by Savage and Barrentine (1969) in a greenhouse bioassay of trifluralin on field samples of a silt loam soil. The bioassay failed to show differences in phytotoxic residues from various incorporation depths, but it did show the reduced residual phytotoxicity of a 4.48 kg/ha surface application of trifluralin. Gas chromatographic (GC) analysis of these same soils showed a highly significant trend to increasing persistence of trifluralin with increasing incorporation depth.

Weber *et al.* (1974a), using both a bioassay, measuring fresh weight of foxtail millet shoots, and GC analysis for detection of trifluralin in a loamy sand, achieved a good correlation between the two methods in the soil with no additive and in the soil with a clay additive, but found that only 15% of the trifluralin present was biologically detectable in soil fortified with high levels of organic matter.

When studying pH effects on trifluralin persistence in a very fine sandy loam, Savage (1973) noted a good correlation between GC analysis and biological assay detection when employing shoot heights of sorghum as the bioassay criteria. However, he pointed out the fact that bioassays measure only the biologically available herbicide in the soil.

Menges and Tamez (1974) conducted both laboratory

bioassays using shoot length of pre-germinated lettuce, redroot pigweed, and Italian millet as indicators, and field bioassays with a visual rating of the growth reduction of sorghum and Italian millet as criteria for trifluralin persistence in a sandy loam soil. Upon comparison of the bioassays to GC analysis, the concentration of trifluralin indicated by the bioassays was greater than that by GC analysis and they assumed biologically active decomposition products of trifluralin were detected in the bioassay.

Visual ratings of sorghum and Japanese millet were used by Miller *et al.* (1975) in greenhouse bioassays for trifluralin in fine sandy loam soil. Horowitz *et al.* (1974), also using sorghum found 1, 2, and 4 ppmw trifluralin reduced shoot length and shoot fresh weight of plants grown in treated clay soil by 62, 90, and 97% respectively.

Visual ratings of oat injury, soybean plant counts and foliage and seed yield, as well as visual ratings and dry weights of velvetleaf and giant foxtail shoots were bioassay indicators used by Harvey (1973a) for the persistence of several dinitroaniline herbicides in a field study on silt loam soil.

Inhibition of crabgrass has been used by Probst *et al.* (1967) as an index for the trifluralin content in a silty clay loam soil under laboratory conditions.

Phytotoxicity studies by Murray *et al.* (1973) revealed that oats were more resistant to dinitroaniline injury than was sorghum. This was concluded from ED_{50} values (herbicide concentration reducing plant growth by 50%) obtained by evaluations of the fresh weight reduction and root injury caused by these herbicides.

SOIL PERSISTENCE AND PHYTOTOXICITY

Savage and Barrentine (1969) determined from GC analysis, that residue levels of trifluralin increase with deeper incorporation, probably due to less volatilization and less rapid gas exchange at these greater depths. By using bioassays (sorghum and Italian millet height and vigor) and analytical methods on soil samples from the field, Menges and Tamez (1974) also found longer persistence as depth of incorporation was increased.

Vapor loss of trifluralin, according to Bardsley *et al.* (1968) who used a charcoal vapor trap, was much greater from a loamy sand surface than when the chemical was applied subsurface, allowing the soil to act as an adsorptive barrier to volatilization.

Upon exposure of surface applied trifluralin to sunlight under greenhouse conditions, Wright and Warren (1965) noticed a decrease in the biological activity of the herbi-

cide on fresh weight of Italian millet shoots, due to photodecomposition and volatilization of the chemical. Nilles and Zabik (1974) determined by use of thin-layer soil plates and subsequent GC analysis, that photodestruction of fluchloralin at potential environmental concentrations is rapid.

Grover (1974) stated that the efficacy of the soil-applied herbicides is dependent on their relative availability in the soil, the latter being regulated by the extent of adsorption on soil colloids, especially the soil organic matter.

Weber *et al.* (1974a), after fortifying loamy sand soil with clay or organic matter, concluded, by means of a bioassay using foxtail millet fresh weights and GC analysis, and after consideration that only a portion of trifluralin present was biologically detectable in high organic matter soils, that the clay content of soil does not alter residual trifluralin but organic content does increase persistence. They found that of a total of 3.2 kg/ha applied over 2 years, approximately 2% remained in unmodified soil and in soil modified with clay only, while 11% remained in soil modified with organic matter. This increased persistence due to organic matter additions is likely due to the increased adsorptive capacity of the organic materials and their ability to retain vapors, according to Bardsley *et al.* (1967).

Soil pH, according to Savage (1973) who worked with a very fine sandy loam soil, seems to have an influence on soil persistence of trifluralin since he found greater persistence under acid soil conditions as indicated by reduced shoot heights of sorghum and GC analysis. But he suggests the response to pH may be reflecting a physico-chemical change in the adsorptive-desorptive complex of the herbicide in the soil rather than actual herbicide degradation.

Leaching studies by Anderson *et al.* (1968) on a clay loam soil showed that large water volumes failed to move trifluralin any appreciable distance into the soil. These results were achieved by use of soil columns with injury to junglerice and shoot height and root development of sorghum as biological indicators.

Menges and Tamez (1974) also found by bioassays using sorghum and millet height and vigor as indicators, and by analytical methods, that trifluralin remained within the original zone of incorporation in a sandy loam soil, regardless of rainfall. Miller *et al.* (1975) also confirmed the limited movement of this herbicide in a fine sandy loam by both GC and a bioassay using visual ratings of sorghum and millet as indicators.

Trifluralin degradation has been found, by Messersmith *et al.* (1971), to increase with higher soil moistures

on silty clay loam and sandy loam soils, as determined from the effect on corn dry weight. Harvey (1973a), by using several measurements on oats, soybeans, velvetleaf, and giant foxtail, reached a similar conclusion after finding that the length of persistence of trifluralin, profluralin, fluchloralin, and dinitramine appeared to be reduced by increased rainfall.

Horowitz *et al.* (1974), using sorghum leaf length as an indicator, observed that trifluralin degraded more rapidly in clay soil when left undisturbed than in soil which had been dried and mixed periodically. In applying this to cultural practices, they assumed that the soil drying and aeration effect of cultivation could extend the persistence and hence effectiveness of a herbicide application.

Burnside (1974) noticed that plowing appeared to reduce long term carry-over of trifluralin in silty clay loam soil and suggested it may result from detoxification of the herbicide when incorporated into more moist soil. In these studies, topgrowth of millet and oats were used as field bioassay.

Increased vaporization occurring in moister silt loam soils enhances movement of trifluralin vapors and a subsequent loss of the herbicide from the soil, according to Hollist and Foy (1971). Bardsley *et al.* (1968) determined that great-

er volatilization of trifluralin took place from a loamy sand soil at the maximum retentive capacity than from the same soil at field capacity due to greater herbicide-crystal dissolution, more vaporization of a free liquid than a liquid under tension, and the competition of water with trifluralin for soil adsorption sites.

Hamilton and Arle (1972) noticed damage to sugar beets and sorghum, 1, 2, or more years following application of trifluralin to desert soils because of low soil moisture and/or low temperatures.

High temperatures result in increased volatilization of trifluralin from a sandy loam soil causing increased phytotoxicity to wheat seedlings (Rahman, 1973). Savage and Barrentine (1969), using a charcoal vapor trap over treated silt loam soil, also observed increased volatilization of trifluralin with increasing soil temperatures.

Horowitz *et al.* (1974) determined that the rate of degradation of trifluralin in clay soil approximately doubled as incubation temperatures of the soil, at 50% field capacity, rose by 10°C increments between 10 and 40°C. His bioassay criteria were leaf length and fresh shoot weight of sorghum.

By analytical methods, Probst *et al.* (1967) also found trifluralin breakdown in silty clay loam and fine sand soils to be temperature dependent under laboratory conditions

and noted the rate of disappearance under controlled conditions to reduce considerably compared to studies conducted in the field.

It has been noted by Probst *et al.* (1967) that microorganisms may contribute to the eventual destruction of trifluralin, in a silt clay loam soil, to simpler compounds but this cannot be considered the major pathway of trifluralin degradation. They also observed that trifluralin-treated soils in a controlled environment showed no build-up of specific microorganisms which could be credited with the degradation of trifluralin. Savage (1973) reached the same conclusion, that microbial degradation is not a major mode of trifluralin loss from soil as autoclaved and non-autoclaved soils were similar in herbicide persistence.

By use of radioactive ^{14}C-trifluralin, Messersmith *et al.* (1971) also found no build-up of specific microorganisms capable of degrading trifluralin in silty clay loam and sandy loam soils and noted that high concentrations of the chemical inhibited the activity of specific organisms which could metabolize the herbicide.

Dinitramine, however, is degraded primarily by the microbial populations within soil as seen from the significantly reduced degradation rate in steam sterilized soils as compared to non-sterilized soils (Smith *et al.*, 1973).

Degradation pathways under aerobic and anaerobic soil conditions in a controlled environment have been postulated for trifluralin and dinitramine by Probst *et al.* (1967) and Smith *et al.* (1973), respectively.

Residue analysis by Probst *et al.* (1967), using ^{14}C-labelled trifluralin, indicated neither trifluralin nor its degradation products are incorporated into the leaves, seeds, or fruit of soybeans and cotton. However, they found roots of these crops contained some residues of trifluralin in the region of soil incorporation, while onions and garlic contained residues only in their outer shell. Carrot roots, according to these researchers, do incorporate trifluralin to some extent.

Studies by Burnside (1974) and Parka and Tepe (1969) of repeated annual applications of trifluralin to various soil types at several locations in the United States have shown no build-up of the herbicide in the soil. Even with applications at twice the recommended rate, Miller *et al.* (1975) observed no accumulation of trifluralin in fine sandy loam by either GC analysis or field and greenhouse bioassay studies which employed visual ratings of sorghum and millet.

Horowitz *et al.* (1974) determined that after 1 month, growth reduction of sorghum shoot length due to 1 ppm trifluralin fell to less than 20% in undisturbed clay soil in

the greenhouse and that complete detoxification was achieved at 4 months.

The concentration of radioactive trifluralin, in field studies on silt loam soil, fell to 10-15% within ½ - 1 year, in experiments by Probst *et al.* (1967). Menges and Tamez (1974) found no appreciable amount of trifluralin persisting after 1 year in a Texas sandy loam soil using sorghum and millet height and vigor bioassays.

A field comparison of dinitroaniline herbicides by Harvey (1973a) in a Wisconsin silt loam soil with 3.5 - 4% organic matter, showed trifluralin to be the most persistent of the four herbicides under consideration in this project. It was followed by fluchloralin and profluralin with dinitramine being the least persistent. The results were obtained over a 2 year period employing a visual estimate of injury to oats planted over the plot area.

Murray *et al.* (1973) demonstrated, by measurement of fresh shoot weights and root evaluations of sorghum and oats, that dinitramine showed the highest degree of phytotoxicity followed by trifluralin, profluralin, and fluchloralin, in decreasing order.

The phytotoxicity of trifluralin to both wheat and green foxtail appeared to decrease with increasing organic matter content of the soil in studies by Rahman (1973). He

used shoot dry weights of wheat and foxtail as indicators. Weber *et al.* (1974b) also noted the reduced phytotoxicity of trifluralin to grassy and broadleaf weeds caused by organic matter.

LOW TEMPERATURE EFFECTS ON PHYTOTOXICITY

Negi and Funderburk (1968) suggested trifluralin vapors play a role in herbicide activity when the herbicide is incorporated into the soil. Using proso millet shoot length as an indicator, Swann and Behrens (1972) concluded that shoot entry of trifluralin in the gaseous phase may be a major, if not the major, means of trifluralin uptake by germinating seedlings in the soil under field conditions.

Decreasing soil temperatures, as reported by Rahman (1973), decreased volatilization of trifluralin and hence reduced the toxicity of the chemical to wheat in silt loam soil. The lowest temperature reported in this work was 16^{o}C.

Harvey (1974) concluded that differences in volatilization of the dinitroaniline herbicides from silt loam soil probably have the greatest influence of the effectiveness of soil applications. Based on in vitro studies measuring foxtail millet root and shoot length on treated or untreated agar, he found trifluralin, profluralin, fluchloralin, and dinitramine to be relatively volatile.

LOW TEMPERATURE GERMINATION OF WILD OATS

Work conducted by Friesen and Shebeski (1961) demonstrated that under controlled conditions at 10°C, wild oats did not emerge until 14 days after placement and after 28 days, emergence was only 55%. They found no emergence at 4.4°C and indications of induced seed dormancy.

In contrast, Leggett and Banting (1959) found the largest percentage of wild oat seeds germinate between 1.1°C and 10°C.

SECTION 1

PERSISTENCE AND PHYTOTOXICITY OF DINITROANILINE HERBICIDES IN MANITOBA SOILS

Abstract. The persistence and phytotoxicity of trifluralin (α, α, α-trifluro-2, 6-dinitro-N, N-dipropyl-p-toluidine), profluralin (N-(cyclopropylmethyl) -α, α, α- trifluro-2, 6-dinitro-N-propyl-p-toluidine), fluchloralin (N-(2-chloroethyl) -2, 6-dinitro-N-propyl-4-(trifluromethyl) aniline), and dinitramine (N^4, N^4-diethyl-α, α, α-trifluro -3, 5-dinitrotoluene -2, 4-diamine) were compared in 3 Manitoba soils: sandy loam, clay loam, and clay. Analytical determinations and field study results, one year after application of the chemicals on light soils, indicated the order of persistence as: profluralin > trifluralin ≥ fluchloralin > dinitramine. On heavy soils, the order of dinitroaniline persistence was: profluralin > dinitramine > trifluralin > fluchloralin one year after application of the chemicals. Increased organic matter increased persistence of trifluralin and fluchloralin while profluralin and dinitramine persistence increased with increased clay content. The phytotoxicities of all chemicals decreased with increased organic matter. The phytotoxicities of the dinitroanilines were: dinitramine > trifluralin > profluralin = fluchloralin.

INTRODUCTION

The dinitroaniline herbicides, trifluralin, profluralin, fluchloralin, and dinitramine are used primarily for control of annual weeds in several agronomic and horticultural crops when applied as a preplant, soil-incorporated treatment. The persistence of these chemicals in soil is influenced by soil type, application rate, chemical and microbial degradation, photodecomposition, volatility and climatic factors of temperature and rainfall (5, 6, 11, 12, 13, 15, 17, 18, 19).

The clay content of soils does not alter residue levels of trifluralin whereas organic matter increases persistence (18). Field applications of trifluralin at twice the recommended rate have shown no build-up of the herbicide in the soil (7). Time-rate degradation studies with trifluralin have indicated an initial rapid decrease in concentration to 10-15% within ½ to 1 year with small quantities persisting as long as 2 years (12). Greater persistence of dinitroanilines under low soil moisture has been reported by several researchers (1, 3, 6).

Field studies under Wisconsin conditions indicated trifluralin to be the most persistent of these herbicides followed by fluchloralin and profluralin with dinitramine exhibiting the most rapid disappearance (3).

Dinitramine has been found the most phytotoxic herbicide followed by trifluralin, profluralin, and fluchloralin in decreasing order (8).

Dry weight of oats (15) and sorghum fresh shoot weights (5, 7) have been used for the bioassay detection of trifluralin in soil. Analytical determinations have been used by several researchers (2, 9, 10, 11, 16). A good correlation was observed between fresh weight of foxtail millet bioassays and analytical determinations but high organic matter reduced detectability in soil bioassays (18). A good correlation between the two methods was also noted when using sorghum shoot height as the bioassay indicator (14).

The purpose of this study was to determine phytotoxicities of trifluralin, profluralin, fluchloralin, and dinitramine and to compare the persistence of these herbicides as affected by soil type and application rate under Manitoba conditions.

MATERIALS AND METHODS

Field Experiment. Experimental sites were selected at 3 locations; Carman (Almasippi very fine sandy loam), Roland (Myrtle heavy fine clay loam), and Glenlea (Red River clay). The physical characteristics of these soils are given in

Table 1.

Prior to herbicide application, the sites were cultivated or rotovated to a depth of 15 cm. A randomized block experimental design was used. There were four replicates per treatment with a plot size of 2 x 6 M on the sandy loam soil and 4 x 12 M on the clay loam and clay soils. Trifluralin, profluralin, fluchloralin, and dinitramine were applied to the treatment areas at the rates and dates shown in Table 2. The rates applied were at the recommended rate, and twice and four times the recommended rate, with rate adjustments made depending on soil type (exception on clay loam soil, 2, 4, and 8 times recommended rate). The rates selected represent the minimum and maximum amount of chemical that would be applied to a given soil. Recommended rates vary according to soil type. Plots were sprayed twice at half strength to ensure even chemical distribution. The herbicides were applied in 220 ℓ/ha water using a bicycle sprayer operated at 4.8 kph and 240 kPa. Immediately following application, the chemicals were incorporated to a 10 cm depth with a rotovator.

Soil samples were collected from the upper 10 cm of each treatment at each site 3, 8, 16, and 50 weeks following application and were placed in plastic bags and frozen at -20^{o}C until analysis. All sites were treated with para-

Table 1. Location and physical characteristics of soils[a].

Location	Texture	% Organic matter	Inorganic separates (%)			CEC[b]
			Sand	Silt	Clay	
Carman	Sandy loam	4.3	75.2	3.9	20.9	18.4
Roland	Clay loam	8.3	30.8	22.0	47.2	39.6
Glenlea	Clay	6.8	4.3	20.3	75.4	47.9

[a]Analysis by the Canada, Manitoba Soil Survey Laboratory, University of Manitoba.

[b]Cation exchange capacity in meq/100g.

Table 2. Rates and dates of herbicide application at each location.

Location	Date of Application	Application Rate*	Treatment Rate (kg/ha)			
			Trifluralin	Profluralin	Fluchloralin	Dinitramine
Carman (Sandy loam)	June 21, 1974	1 x	0.84	1.12	1.12	0.75
		2 x	1.68	2.24	2.24	1.50
		4 x	3.36	4.48	4.48	3.00
Roland (Clay loam)	June 17, 1974	2 x	2.24	2.80	2.80	1.68
		4 x	4.48	5.60	5.60	3.36
		8 x	8.96	11.20	11.20	6.72
Glenlea (Clay)	June 20, 1974	1 x	1.12	1.40	1.40	0.84
		2 x	2.24	2.80	2.80	1.68
		4 x	4.48	5.60	5.60	3.36

*1 x represents recommended rate; 2 x, twice recommended rate; 4 x, four times recommended rate; and 8 x, eight times recommended rate.

quat or mowed in early August, 1974 to kill any weed growth.

Relative phytotoxicities. To determine the relative phytotoxicities of dinitroaniline herbicides, several rates of each herbicide were applied to each soil type and ED_{50} values in sorghum (*Sorghum bicolor* (L.) Moench x *Sorghum sudanense* (Piper) Stapf.) and oats (*Avena sativa* (L.) var. Harmon) were determined under growth chamber conditions. ED_{50} is the herbicide concentration required to inhibit plant growth by 50% as compared to untreated plants.

The respective herbicides were applied to 200 g untreated, air-dry soil in 10 ml water with an atomizer to give concentrations ranging from 0 to 3 ppmw. The soil was thoroughly mixed in a plastic bag to ensure adequate distribution of the herbicide. The soil was placed into 8 cm, 200 ml plastic cups and after a 20 hr equilibration period, sorghum or oats were planted. Cups were placed in the growth chamber at a constant temperature of 22°C and a 24 hr photoperiod at 1060 lux. There were 6 replications per treatment. Every 2-3 days, each cup was weighed and brought up to field capacity. Seedlings were thinned to 5 plants per cup after 5 days, and one week after planting, each cup was fertilized with 150 ppm of nitrogen, phosphorus, and potassium. At the end of 3 weeks, the aboveground plant parts were harvested, oven dried, and weighed. A probit transformation

of percent growth reduction due to herbicide injury was plotted against herbicide concentration. The ED_{50} values obtained were expressed in ppmw. The results could not be analyzed statistically as the values were obtained from a series of independent experiments.

Field Study. Approximately one year following herbicide application, four crops, wheat (*Triticum aestivum* (L.) var. Selkirk), oats, sorghum, and corn (*Zea mays* (L.) 'Weather Master CDO') were sown across each replicate at each plot site with a 3-row planter. Planting dates were June 24, June 25, and July 4, 1975 on the sandy loam, clay loam, and clay soil plots, respectively. After 5 weeks, a one meter row from each crop in each treatment was harvested and dry weights were obtained and compared to the check. Tukey's honestly significant difference test was used to compare all means in each table at the 5% level of significance. Values followed by the same letter are not significantly different at the 5% level of significance.

Analytical Determination of Residues. Standard curves for each chemical were obtained by preparing standard solutions in n-hexane with concentrations ranging from 0.005 ng/µl to 0.3 ng/µl. Standard curves of peak height (in linear response region) versus herbicide concentration were determined for

each chemical by analyzing 1.5 µl aliquots of each standard on a gas chromatograph. A Varian Aerograph Series 1800 gas chromatograph was used with a tritium foil, electron capture detector. The 1.2 M glass column had an inside diameter of 4 mm (on column injection) and was packed with 5% SE-30 on 80-100 mesh chromosorb W. Prepurified nitrogen was used as the carrier gas at 40 ml/min. Injection port, column oven, and detector temperatures were 230, 170, and 220°C, respectively. Retention times for trifluralin, profluralin, fluchloralin, and dinitramine were 1.57, 2.55, 2.35, and 2.45 minutes, respectively.

Extraction efficiency for each herbicide in each soil type was determined as follows. To 50 g samples of untreated, sieved, air-dry soil in 400 ml screw-capped, glass bottles were added 5 ml of the respective herbicide stock solution in hexane to give concentrations of 0.5 or 1.0 ppmw. After mixing to ensure distribution of the chemical, the soil was placed into plastic bags and left exposed to the air for 6 hrs to allow the hexane to evaporate from the sample. The bags were then sealed and frozen at -20°C. After 7 days, the herbicide was extracted from 2 replicates of each herbicide concentration in each soil type.

The extraction procedure is according to Smith (16) with modifications. Ten grams of the air-dry soil sample

was placed into a 100 mℓ capacity beaker to which was added 30 mℓ of 10% aqueous acetonitrile and extracted for 2.5 min using an Artek Sonic Dismembrator at maximum power. The sample was transferred to a centrifuge tube and centrifuged for 10 min. Ten millilitres of the acetonitrile solution was added to 50 mℓ of an aqueous sodium sulfate solution (2:15 v/v saturated aqueous sodium sulfate solution and distilled water) in a separatory funnel and extracted with 25 mℓ n-hexane. For high concentrations of herbicide (1 to 5 ppmw), 50-75 mℓ hexane were used. The aqueous layer was discarded and the hexane phase dried over anhydrous sodium sulfate in a stoppered, round-bottom flask. Two 1.5 µℓ aliquots from each sample were analyzed using the gas chromatograph. An external standard was injected regularly and the concentration of the herbicide present in the sample was determined by comparing the sample peak height with that of the standard. Recovery data are given in Table 3.

Soil samples from the field plots were analyzed for herbicide residue as follows. Treatments were bulked and paired representative samples were analyzed on the gas chromatograph. The results were corrected for extraction efficiency according to the recovery percentages and expressed as a percentage of the applied herbicide. The results were also expressed in ppmw. As replicates were bulked before analysis,

Table 3. Recovery of dinitroanilines from fortified soils using ultrasonic extraction, 7 days following fortification.

Soil type	Mean % recovery ± standard deviation			
	Trifluralin	Profluralin	Fluchloralin	Dinitramine
Sandy loam	87.1 ± 4.2	95.8 ± 4.1	107 ± 6.2	104.9 ± 4.4
Clay Loam	94.6 ± 4.6	98.9 ± 7.4	103 ± 4.4	106.5 ± 5.1
Clay	100.8 ± 2.8	96.2 ± 3.6	100.9 ± 3.1	107.2 ± 7.6

the results could not be analyzed statistically.

RESULTS AND DISCUSSION

Relative Phytotoxicities. The ED_{50} values in sorghum and oats determined for each herbicide in each soil type are presented in Tables 4 and 5. Oats were found to be more resistant than sorghum to dinitroanilines as seen from the larger ED_{50} values for oats. Other researchers have found sorghum to be more susceptible than oats to dinitroaniline injury (8).

Of the dinitroanilines studies, dinitramine showed the greatest degree of phytotoxicity in sorghum and oats on all soil types with the exception of oats in the sandy loam soil where fluchloralin appeared the most phytotoxic. Trifluralin followed dinitramine in phytotoxicity, and profluralin and fluchloralin were the least phytotoxic. The dinitroanilines were the least effective on the clay loam soil. Previous studies indicated dinitramine as being the most phytotoxic of the four herbicides to sorghum and oats followed by trifluralin, profluralin, and fluchloralin in order of decreasing phytotoxicity (8).

Tables 4 and 5 indicate that the phytotoxicities of the dinitroaniline herbicides were lower in high organic matter soils than in low organic matter soils. Clay does not

Table 4. Comparative ED_{50} values of dinitroaniline herbicides to sorghum in various soil types.

Herbicide	ED_{50} (ppmw)		
	Sandy loam	Clay loam	Clay
Trifluralin	0.49	0.96	0.98
Profluralin	0.55	----*	0.80
Fluchloralin	0.56	1.45	0.71
Dinitramine	0.31	0.71	0.46

*Value unobtainable from results.

Table 5. Comparative ED_{50} values of dinitroaniline herbicides to oats in various soil types.

Herbicide	ED_{50} (ppmw)		
	Sandy loam	Clay loam	Clay
Trifluralin	1.20	3.38	1.50
Profluralin	1.51	5.60	2.10
Fluchloralin	1.00	3.71	3.18
Dinitramine	1.31	1.74	1.42

appear to be an important factor in decreasing the phytotoxicities of the dinitroanilines. It has been suggested that organic matter and clay may be synergistic in decreasing the phytotoxicities of these chemicals (4). These differential phytotoxicities were considered at time of field application and the chemicals were applied at rates based on equivalent phytotoxicity with adjustments made for soil type.

Field Bioassays. Tables 6-17 present the results for the shoot dry weights of wheat, oats, sorghum, and corn grown in the 3 soil types, one year after herbicide application.

After one year, residue from all rates of all herbicides in all soils injured wheat, except dinitramine at the 1 x rate on the clay soil and at the 2 x and 4 x rates on the clay loam soil, and trifluralin at the 2 x rate on the clay loam soil. Plots treated with the 1 x rate of chemical showed oat injury from dinitramine and fluchloralin on the clay soil and from all chemicals at the 2 x rate, except dinitramine, on the clay loam soil. However, on the sandy loam soil, only profluralin and fluchloralin injured oats even at the 4 x rate. Fluchloralin was the only chemical to injure sorghum on the sandy loam soil at the 1 x rate. The 1 x rate of all chemicals on the clay soil did not damage sorghum while injury to sorghum resulted from the 2 x rate of all treatments on the clay loam soil. Corn was injured at the

Table 6. Dry weight of wheat grown in herbicide treated sandy loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	13.08a				
1 x		7.79 bcd	8.74 b	7.05 cd	8.54 bc
2 x		7.54 bcd	6.79 de	6.25 def	7.64 bcd
4 x		7.85 bcd	4.69 f	5.23 ef	8.96 b

Table 7. Dry weight of oats grown in herbicide treated sandy loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	8.43ab				
1 x		8.09 ab	6.29 abc	5.58 abc	6.49 abc
2 x		6.51 abc	6.58 abc	5.51 abc	9.10 a
4 x		4.96 bcd	1.29 d	3.49 cd	5.63 abc

Table 8. Dry weight of sorghum grown in herbicide treated sandy loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	44.76 bc				
1 x		61.49 a	65.06 a	32.93 d	49.39 b
2 x		17.89 e	29.66 d	47.48 bc	37.31 cd
4 x		16.81 e	3.81 f	12.84 ef	70.74 a

Table 9. Dry weight of corn grown in herbicide treated sandy loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	63.93 bcd				
1 x		65.94 bc	69.15 b	53.28 def	31.64 g
2 x		61.21 bcd	57.41 cde	65.85 bc	43.04 f
4 x		49.24 ef	21.54 g	42.85 f	82.85 a

Table 10. Dry weight of wheat grown in herbicide treated clay loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	7.80 b				
2 x		9.36 a	5.84 cd	5.23 de	7.04 bc
4 x		4.96 de	4.09 ef	3.29 f	6.80 bc
8 x		0.53 h	1.69 gh	2.58 fg	3.10 fg

Table 11. Dry weight of oats grown in herbicide treated clay loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	5.85 a				
2 x		2.34 bcd	3.00 b	2.13 cd	5.09 a
4 x		1.80 d	2.24 bcd	1.64 d	2.85 bc
8 x		0.04 e	0.10 e	0.60 e	1.69 d

Table 12. Dry weight of sorghum grown in herbicide treated clay loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	32.06 a				
2 x		26.32 b	10.31 d	17.63 c	26.68 b
4 x		8.80 be	12.94 d	10.78 d	31.19 a
8 x		1.51 f	0.48 f	5.78 e	17.93 c

Table 13. Dry weight of corn grown in herbicide treated clay loam soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	51.69 a				
2 x		49.95 a	51.62 a	19.74 cde	32.53 b
4 x		28.99 bc	27.90 bcd	11.43 e	32.31 b
8 x		18.76 de	18.43 de	33.08 b	28.25 bcd

Table 14. Dry weight of wheat grown in herbicide treated clay soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	8.19 a				
1 x		5.58 bc	4.70 cd	5.44 bc	8.11 a
2 x		6.43 b	3.88 d	6.95 ab	4.41 cd
4 x		3.68 d	1.48 e	1.90 e	1.31 e

Table 15. Dry weight of oats grown in herbicide treated clay soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifloralin	Profluralin	Fluchloralin	Dinitramine
0	5.90 a				
1 x		5.08 ab	5.05 ab	4.39 bc	3.78 c
2 x		2.56 d	2.33 de	4.21 bc	2.13 de
4 x		1.26 ef	0.85 f	1.29 ef	0.94 f

Table 16. Dry weight of sorghum grown in herbicide treated clay soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	6.96 b				
1 x		5.24 bc	5.65 bc	9.71 a	5.51 bc
2 x		5.49 bc	1.59 ef	3.15 de	4.04 cd
4 x		0.48 f	0.70 f	0.40 f	0.84 f

Table 17. Dry weight of corn grown in herbicide treated clay soil one year following application.

Rate	Dry weight per meter row (g)				
	Check	Trifluralin	Profluralin	Fluchloralin	Dinitramine
0	22.00 a				
1 x		8.84 def	12.38 cd	17.05 abc	13.50 bcd
2 x		13.61 bcd	6.49 efg	18.60 ab	10.48 de
4 x		5.10 fg	3.29 g	2.68 g	3.29 g

2 x rate of dinitramine on the sandy loam soil and by fluchloralin and dinitramine on the clay loam soil at the 2 x rate. All treatments, except fluchloralin at the 1 x and 2 x rates, injured corn on the clay soil.

The field studies indicated that dinitramine exhibited the least persistence of the four herbicides. However, clay soil tended to increase dinitramine persistence relative to the rest of the chemicals. Profluralin and fluchloralin expressed similar persistence in all soils, however, profluralin was more persistent than fluchloralin in heavier soils. Fluchloralin appeared to have lower persistence than did the rest of the chemicals in the clay soil after one year. Trifluralin showed less persistence than profluralin and fluchloralin on all soils. The persistence of trifluralin was greater than dinitramine in the clay loam and sandy loam soils but less than dinitramine in the clay soil.

The field studies showed that dinitroaniline herbicides applied at the recommended rates can injure susceptible crops planted one year following application of these chemicals. Wheat may be injured if planted on sandy loam soil one year following application of any of these chemicals at the 1 x rate. Wheat planted on clay soil one year after application of 1 x rates of trifluralin and proflur-

alin may be injured. Injury to oats by fluchloralin and dinitramine, and to corn by trifluralin, profluralin, and dinitramine may occur if these crops are planted on a clay soil one year following application of these chemicals at the recommended rate.

Analytical Determinations. The analytical determinations of residue levels of the herbicides, expressed as a percent of applied herbicide remaining in the sandy loam, clay loam, and clay soils, are presented in Tables 18, 19 and 20, respectively. The same results expressed in ppmw remaining are given in the Appendix.

The percent of applied herbicide remaining in the three soils, three weeks after application indicate that initial degradation of these chemicals is rapid and is influenced by soil type. The residues of all chemicals showed an average of 37% of the total applied chemical remained in the sandy loam soil three weeks after application and approximately 50% of the total applied chemical remained in the clay loam and clay soils three weeks after application.

Results indicate dinitramine as the least persistent of the four chemicals except in the clay soil where residue levels of dinitramine were second only to profluralin in percentage of applied chemical remaining after 50 weeks.

Table 18. Percent herbicide remaining in sandy loam soil sampled periodically following application.

Herbicide application rate (kg/ha)	Herbicide remaining (%)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
0.84	41	24	19	17
1.68	57	34	27	18
3.36	40	23	15	17
Average	46	27	20	17
Profluralin				
1.12	28	32	33	22
2.24	41	31	26	19
4.48	39	43	23	27
Average	36	35	27	23
Fluchloralin				
1.12	35	26	14	72*
2.24	36	26	18	13
4.48	41	34	16	20
Average	37	29	16	17
Dinitramine				
0.75	33	19	18	6
1.50	26	17	13	8
3.00	24	20	11	10
Average	28	19	14	8

*Not included in average.

Table 19. Percent herbicide remaining in clay loam soil sampled periodically following application.

Herbicide application rate (kg/ha)	Herbicide remaining (%)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
2.24	52	34	35	27
4.48	49	51	27	25
8.96	51	47	34	27
Average	51	44	32	26
Profluralin				
2.80	53	50	48	32
5.60	48	45	40	20
11.20	42	39	33	23
Average	48	45	40	25
Fluchloralin				
2.80	43	40	30	19
5.60	49	34	31	24
11.20	47	22	40	16
Average	46	32	34	20
Dinitramine				
1.68	51	39	22	17
3.36	46	29	21	16
6.72	69	41	40	17
Average	55	36	28	17

Table 20. Percent herbicide remaining in clay soil sampled periodically following application.

Herbicide application rate (kg/ha)	Herbicide remaining (%)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
1.12	27	22	19	17
2.24	25	20	16	16
4.48	40	29	22	27
Average	31	24	19	20
Profluralin				
1.40	47	48	40	37
2.80	78	48	40	28
5.60	56	44	43	26
Average	60	47	41	30
Fluchloralin				
1.40	36	41	31	18
2.80	48	41	20	13
5.60	51	47	28	11
Average	45	43	26	14
Dinitramine				
0.84	51	49	24	25
1.68	67	43	26	24
3.36	73	43	37	29
Average	64	45	29	26

Profluralin was the most persistent of these chemicals when all soil types were considered. Trifluralin persisted longer in the clay loam soil than in the other soil types and was second only to profluralin in amount of residue after 50 weeks when all soil types were considered. Fluchloralin was the least persistent chemical on the clay soil and less persistent than trifluralin and profluralin on the sandy loam and clay loam soils. Profluralin and dinitramine persistence appeared to increase with increasing clay content and trifluralin and fluchloralin persistence appeared to increase with increasing organic matter.

In general, the rate of application had no affect on the rate of disappearance of the chemicals but dinitramine tended to show a reduced rate of degradation at the highest rate applied. Degradation over the winter period (October to June) for all chemicals in all soil types accounted for only 7% average loss in herbicide activity.

A comparison of the field results and the analytical determinations suggests differences in persistence of dinitroaniline herbicides and an influence of soil type on persistence. Trifluralin and profluralin appeared to be the most persistent chemicals on the sandy loam and clay loam soils. Profluralin showed greater persistence on the sandy loam and clay soils than trifluralin while trifluralin

appeared more persistent than profluralin on the high organic, clay loam soil. It has been found that increasing organic matter increases trifluralin persistence while the clay content of soil has no affect on persistence of this chemical (18). Fluchloralin was less persistent than trifluralin and profluralin on the sandy loam and clay loam soils and the least persistent of all chemicals on the clay soil. Dinitramine was the least persistent of the chemicals on the sandy loam and clay loam soils but was second only to profluralin in persistence on the clay soil.

The reduced percentage degradation of dinitramine, when applied at high application rates may be attributed to a limited number of microorganisms which can metabolize dinitramine. Dinitramine is degraded primarily by the microbial population within the soil (17). Therefore, microorganisms which can use dinitramine as an energy source may not be present in soil in large enough quantities to degrade large amounts of herbicide.

It should be noted that the residue levels obtained from these experiments should represent the maximum amount of herbicide persistence that would occur under normal cropping practices. Rainfall during the summer following application of the chemicals was below normal and persistence of these herbicides has been found to be increased by reduced

amounts of moisture (3). The length of the winter period during which the soil is frozen is important when evaluating persistence of soil-applied herbicides under Manitoba conditions. Several researchers (1, 5, 12) have found trifluralin breakdown to decrease with decreasing temperatures.

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SECTION 2

LOW TEMPERATURE EFFECTS ON PHYTOTOXICITY OF DINITROANILINES

Abstract. The phytotoxicities of trifluralin (α, α, α-trifluro-2, 6-dinitro-N, N-dipropyl-p-toluidine), profluralin (N-(cyclopropylmethyl)-α, α, α- trifluro-2, 6-dinitro-N-propyl-p-toluidine), fluchloralin (N-(2-chloroethyl)-2, 6-dinitro-N-propyl-4-(trifluromethyl) aniline), and dinitramine (N^4, N^4-diethyl -α, α, α-trifluro-3, 5-dinitrotoluene -2, 4-diamine) to wild oats (*Avena fatua* L.) were compared at soil temperatures of 4-8°C. Trifluralin showed the greatest inhibition to wild oat root growth at the lower temperatures while dinitramine and fluchloralin exhibited the greatest toxicities to wild oat roots at the higher temperatures. Trifluralin was the most phytotoxic chemical to shoot growth. Root length was the best criterion for detection of dinitroaniline herbicide activity while shoot dry weights were a poor indication of injury.

INTRODUCTION

The activity of soil-applied herbicides is influenced by the edaphic factors of soil type, organic matter, pH and soil moisture and by soil temperature.

Decreasing temperature has been shown to decrease the volatility of trifluralin (5, 6). The phytotoxicity

of trifluralin can be attributed primarily to the vapor phase (4, 7). Harvey (2) found these herbicides to be relatively volatile.

In Western Canada, wild oats have escaped the phytotoxic effect of trifluralin when this chemical has been applied in the fall or early spring. The purpose of this study was to determine if the phytotoxicity of trifluralin, profluralin, fluchloralin, and dinitramine was adequate to inhibit growth of wild oats at the lowest temperature at which wild oat growth can occur.

MATERIALS AND METHODS

The soil used in these experiments was an Almasippi very fine sandy loam containing 79% sand, 7% silt, 14% clay, and 3.6% organic matter.

Using an atomizer, 10 mℓ of an herbicide solution, prepared in distilled water, of commercial grade trifluralin, profluralin, fluchloralin, or dinitramine, was applied to 225 g air-dry, sieved soil. Final concentrations for all chemicals was 1 ppmw. Water was added to bring the soil moisture up to 68% field capacity. The soil was mixed in a plastic bag to ensure adequate distribution of the chemical and water, then placed in 16 cm, 450 mℓ glass jars.

Wild oat seeds were imbibed by placing seeds on

filter paper in petri dishes containing 3 mℓ water. Dishes were covered, wrapped to exclude light, and placed in a growth cabinet at 22°C for 48 hours. Germination of most seeds had begun at this time. Ten seeds were placed into each jar at a depth of 2 cm and the soil was covered with 1.5 cm of vermiculite to act as a thermal insulator. The jars were placed into a water-glycol bath at 4, 5, 6, 7, or 8°C in a completely randomized design with 5 replications per treatment. The bath temperature was maintained by a cooling coil and a thermostatically controlled circulating pump. The experiments were conducted under laboratory conditions of air temperature and light.

After 21 days, 5 plants from each jar were harvested and root lengths, root dry weights, and shoot dry weights determined. The results were graphed and treatments compared to the control plants. Tukey's honestly significant difference test was used to compare treatments at each temperature at the 5% level of significance. Curvilinear regression analysis was performed on each treatment response curve at the 5% level of significance.

RESULTS AND DISCUSSION

The effect of dinitroaniline phytotoxicity on root length, root dry weight, and shoot dry weight of wild oats

at several temperatures is presented in Figures 1, 2, and 3 respectively.

Root lengths showed a significant difference between the check and all treatments over the temperature range tested with no significant differences between treatments (Figure 1). Of the four herbicides, trifluralin appeared to show the greatest root length inhibition at the lower temperatures while profluralin seemed the least phytotoxic to wild oat roots at all temperatures. At the highest temperature, 8°C, dinitramine exhibited more phytotoxicity than the other chemicals. In all cases, except for trifluralin, root lengths of wild oats were greater at 4°C than at 5°C, suggesting an interaction between temperature and herbicidal activity. Above 5°C, root length increased, in all treatments, with increasing temperature.

Measurements of root dry weight (Figure 2) yielded trends similar to those of root length. Dry weights were significantly lower than the control for all treatments at all temperatures, except at 4°C where only trifluralin produced a marked reduction from the control. As with root lengths, there appeared to be a temperature-herbicide interaction at 4°C as determined from the slope at the lower end of the treatment response curves. Root dry weight increased with temperature in all treatments except dinitramine where

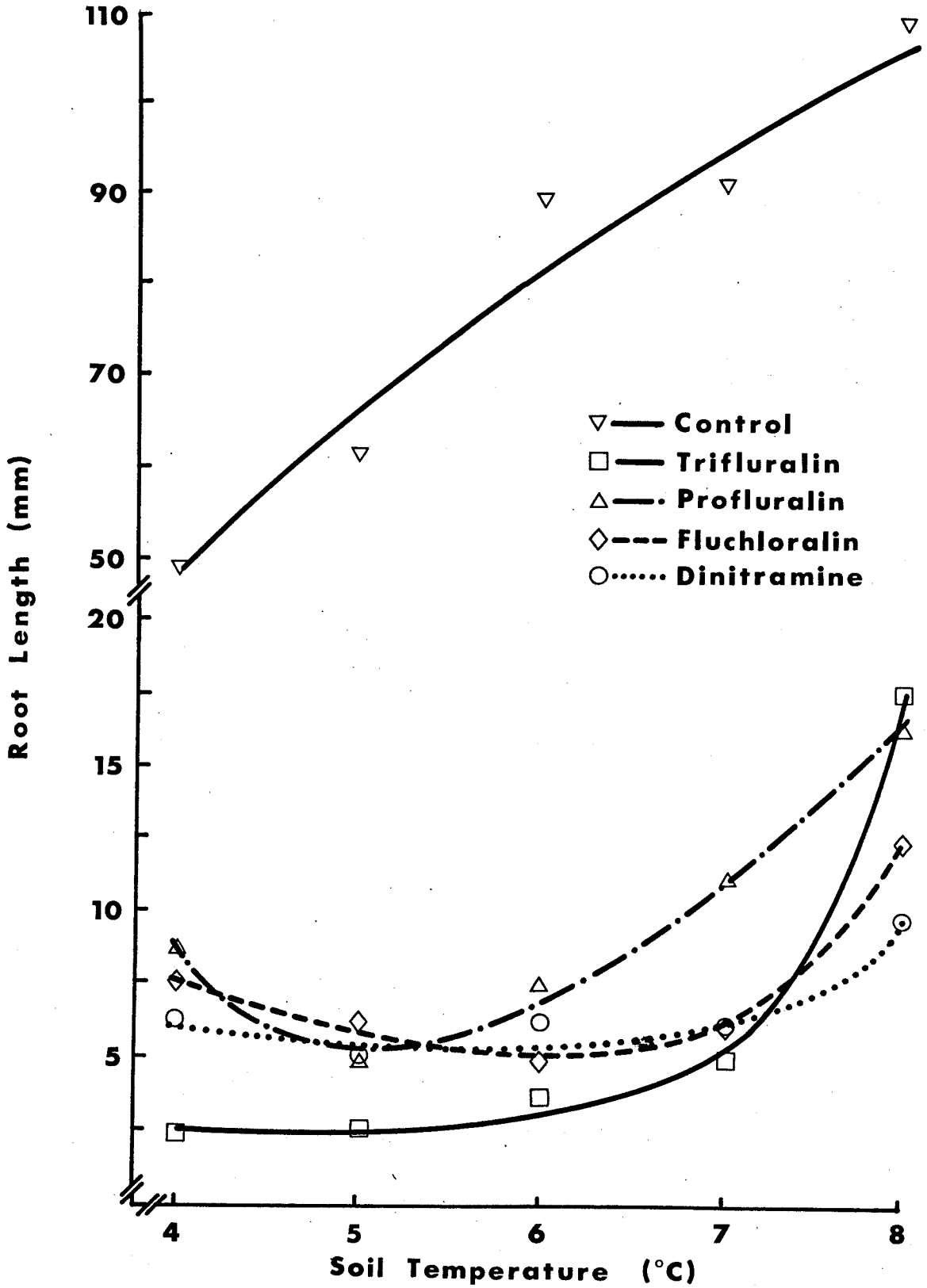

Figure 1. Effect of dinitroanilines on root length of wild oats grown 3 weeks in 1 ppmw treated soil (Note: Two scales for root length).

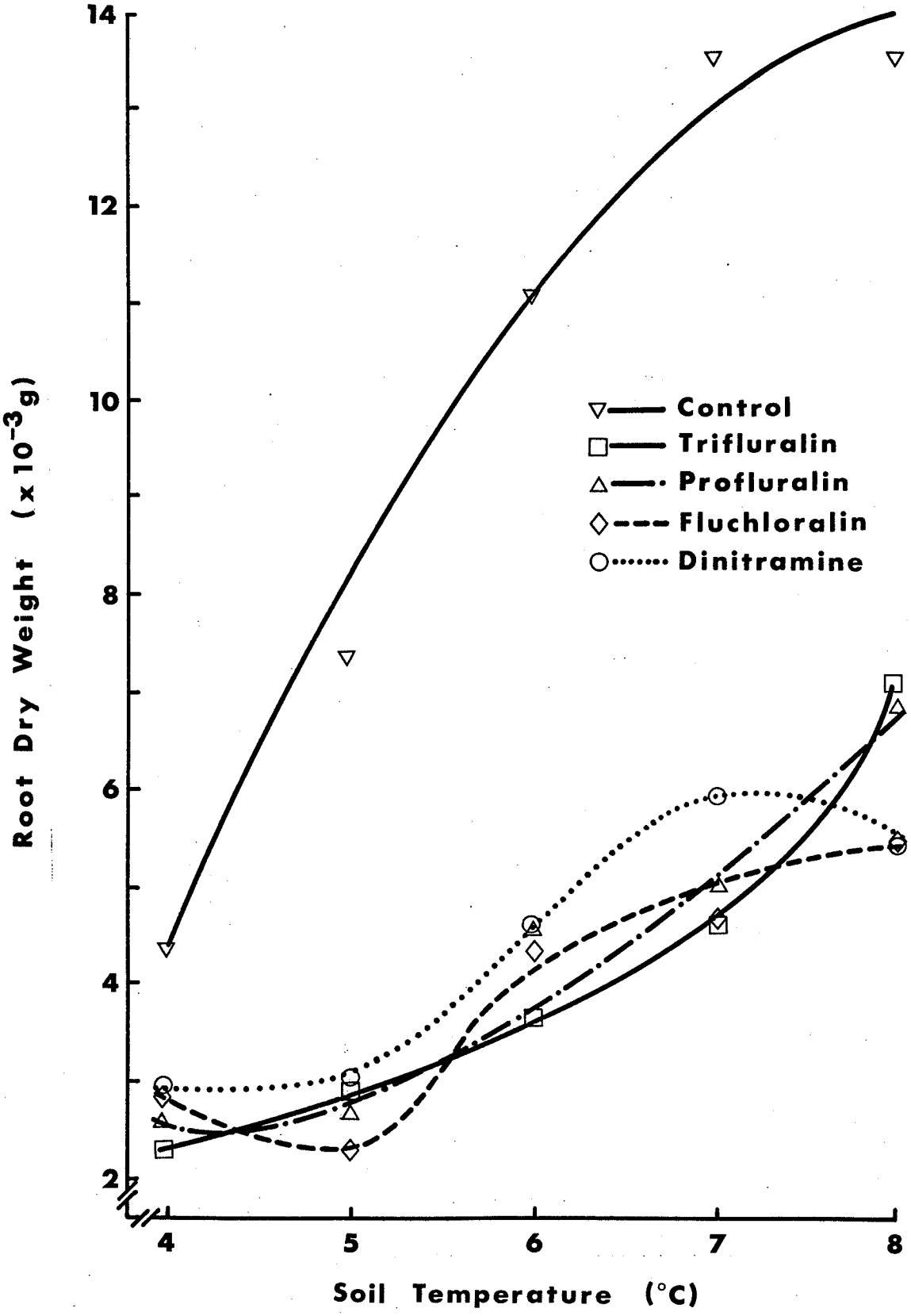

Figure 2. Effect of dinitroanilines on root dry weight of wild oats grown 3 weeks in 1 ppmw treated soil.

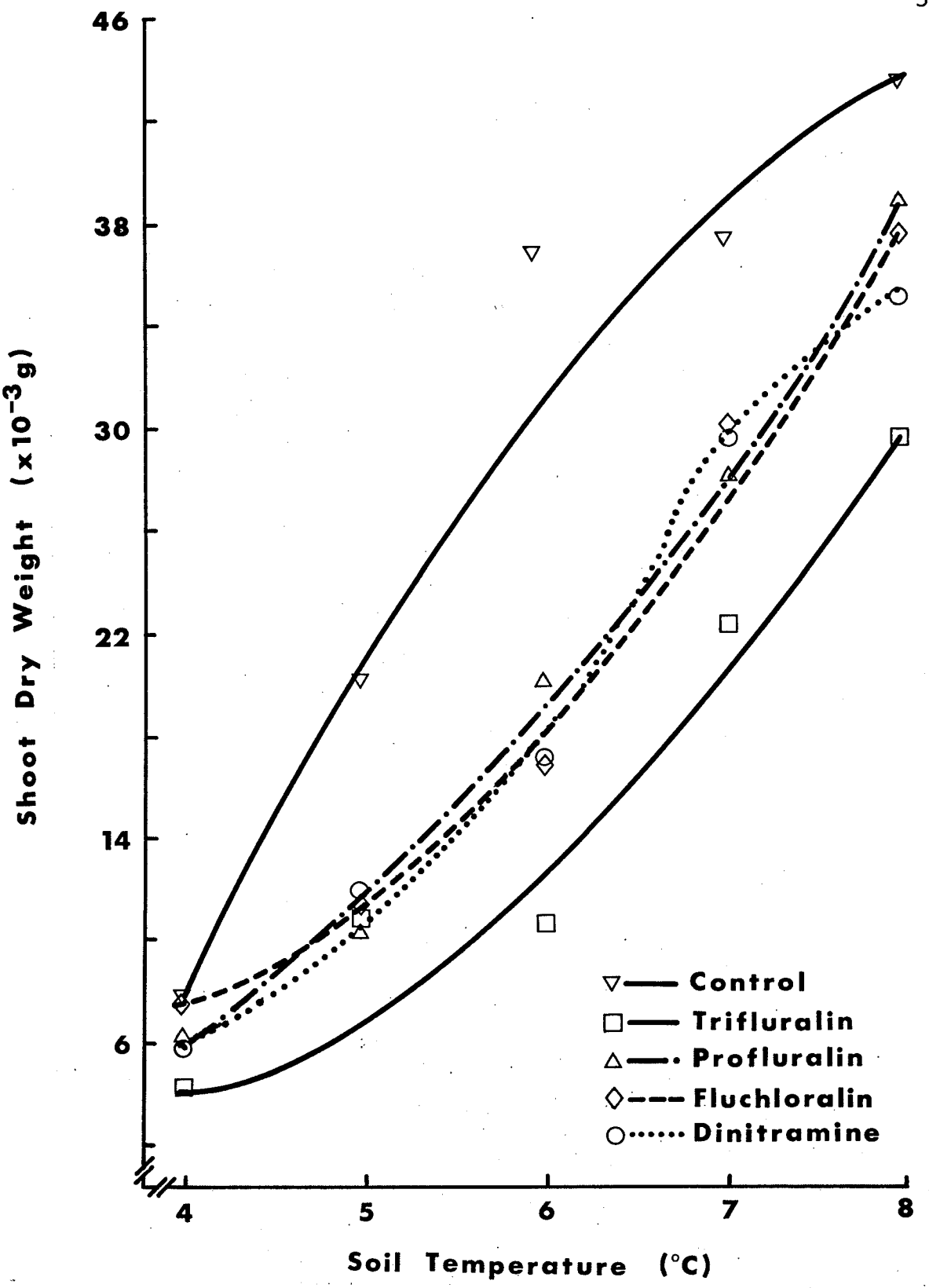

Figure 3. Effect of dinitroanilines on shoot dry weight of wild oats grown 3 weeks in 1 ppmw treated soil.

a decrease in dry weight was noted at 8°C. At the highest temperature, 8°C, dinitramine and fluchloralin caused the greatest dry weight reductions.

Shoot dry weight for all treatments increased with increasing temperature (Figure 3) and the treatment curves paralleled the control curve more closely than did the root length and root dry weight measurements. At all temperatures, except 4°C, there were significant differences between the control and treatments. Trifluralin showed the greatest shoot weight reduction of the four herbicides with the other three chemicals very similar to each other in response. However, at 8°C, shoot dry weight for dinitramine was not significantly different from that of trifluralin.

Comparison of the data from the three measurements of wild oat growth indicates differential phytotoxicity between chemicals which is influenced by soil temperature. At the highest temperature tested, dinitramine and fluchloralin exhibited the greatest phytotoxicity to wild oat roots when compared to the other chemicals. In vitro studies on treated agar at 30°C, have shown that dinitramine and fluchloralin reduce root length of soybeans (*Glycine max* (L.) Merr. var Corsoy) and velvetleaf (*Abutilon theophrasti* Medic) to a greater extent than trifluralin and profluralin (1). At the lower temperatures, root length of wild oats was in-

hibited more by trifluralin than by the other herbicides. Of the chemicals tested, trifluralin was the most phytotoxic to shoot growth of wild oats. At 25°C, dinitramine has been found to be the most phytotoxic chemical to the fresh shoot weight of sorghum (*Sorghum bicolor* (L.) Moench var. OK612) and oats (*Avena sativa* (L.) var. Cimarron) on a sandy loam soil, followed by trifluralin, profluralin, and fluchloralin in order of decreasing phytotoxicity (3).

The present observations of the effects of dinitroanilines on wild oat roots tend to indicate reduced herbicidal activity of all chemicals, except trifluralin, at 4°C. The slopes of the curves of profluralin, fluchloralin, and dinitramine between 4 and 5°C suggest increasing phytotoxicity with increased temperature. Prolonged periods of cool temperatures following early spring applications of these herbicides could result in reduced weed control if soil temperatures are low enough to reduce the herbicidal activity of these chemicals. Trifluralin appeared to express activity at lower soil temperatures than the other compounds.

The quadratic nature of the shoot dry weight, root dry weight, and root length curves for trifluralin indicate that trifluralin may have a two part effect on development of wild oats at low soil temperatures. In all three measure-

ments taken of wild oat growth at low soil temperatures, the response curves for trifluralin are distinctly different from the other three herbicides in both shape and position.

The large differences in root length between the treated and untreated plants indicate root length as being a better biological indicator of the concentration of dinitroanilines on wild oats at reduced soil temperatures than root or shoot dry weight. Thorough washing of soil from roots, necessary when measuring root dry weights, can cause error in root dry weight measurements due to breakage and loss of roots. Adherence of clay particles to roots of plants grown in soil having a high clay content makes thorough washing difficult. Shoot dry weight, though the easiest to use, is the least useful as an indicator of dinitroaniline activity at low soil temperatures because of the proximity of the curves of the control and treated plants to each other. After the 3 week bioassay period, topgrowth of the wild oats was not extensive, even in the check plants, suggesting that food reserves in the seed could be accounting for most of the shoot growth in both treated and untreated plants. It appears that trifluralin may have an effect on the mobilization of seed reserves as indicated by the quadratic nature of the shoot dry weight curve.

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SECTION 3

BIOASSAY DETERMINATION OF DINITROANILINE PERSISTENCE IN MANITOBA SOILS

INTRODUCTION

As an alternate method to analytical determinations, bioassays have been used for the quantitation of dinitroaniline herbicides in soil. Comparison of the bioassay and analytical determinations has indicated both good and poor correlations between the two methods depending on soil type and the indicator plant used (1, 2, 3, 4, 5, 7).

Bioassays for trifluralin soil residues, using foxtail millet (*Setaria italica* (L.) Beauv.), showed a good correlation with analytical determinations for soil low in organic matter but the biological detectability of trifluralin was significantly reduced in soil high in organic matter (7). The sensitivity of corn (*Zea mays* L., var. H49T ms x N6) to trifluralin has been found to vary depending on soil type (1, 3). Under greenhouse conditions, a good correlation was achieved between bioassays, using sorghum (*Sorghum bicolor* L., var. Dekalb 57E) shoot length, and analytical determinations (4). Oats (*Avena sativa* L., var. Mulga) grown in very low concentrations of several herbicides, including trifluralin (α, α, α-trifluro-2, 6-dinitro-N, N-dipropyl-p-toluidine), in the greenhouse, were frequent-

ly taller and had a greater fresh weight than the control plants (2). Bioassays of stored (frozen) and of freshly prepared standard series of some chemicals have been found to differ significantly in topgrowth in the indicator plant (6).

The purpose of these experiments was to determine trifluralin, profluralin (N-(cyclopropylmethyl)-α, α, α-trifluro-2, 6-dinitro-N-propyl-p-toluidine), fluchloralin (N-(2-chloroethyl)-2, 6-dinitro-N-propyl-4-(trifluromethyl) aniline), and dinitramine (N^4, N^4-diethyl-α, α, α-trifluro -3, 5-dinitrotoluene-2, 4-diamine) residues in field soil by growth cabinet bioassays.

MATERIALS AND METHODS

Experimental sites were selected at 3 locations; Carman (Almasippi very fine sandy loam), Roland (Myrtle heavy fine clay loam), and Glenlea (Red River clay). The physical characteristics of these soils are given in Table 1.

Prior to herbicide application, the sites were cultivated or rotovated to a depth of 15 cm. A randomized block experimental design was used. There were four replicates per treatment with a plot size of 2 x 6 M on the sandy loam soil and 4 x 12 M on the clay loam and clay soils. Triflur-

alin, profluralin, fluchloralin, and dinitramine were applied to the treatment areas at the rates and dates shown in Table 2. The rates applied were at the recommended rate, and twice and four times the recommended rate, with rate adjustments made depending on soil type (exception on clay loam soil, 2, 4, and 8 times recommended rate). The rates selected represent the minimum and maximum amount of chemical that would be applied to a given soil. Recommended rates vary according to soil type. Plots were sprayed twice at half strength to ensure even chemical distribution. The herbicides were applied in 220 ℓ/ha water using a bicycle sprayer operated at 4.8 kph and 240 kPa. Immediately following application, the chemicals were incorporated to a 10 cm depth with a rotovator.

Soil samples were collected from the upper 10 cm of each treatment at each site 3, 8, 16, and 50 weeks following application and were placed in plastic bags and frozen at -20°C until analysis. All sites were treated with paraquat or mowed in early August, 1974 to kill any weed growth.

Standard curves for sorghum (*Sorghum bicolor* (L.) Moench x *Sorghum sudanense* (Piper) *Stapf.*) and oats (*Avena sativa* L., var. Harmon) were prepared for each herbicide in each soil type to be used for the determination of dinitroaniline residues in the field samples. Several rates of the

respective herbicides were applied to 200 g untreated sieved, air-dry soil in 10 mℓ water with a Windex sprayer to give concentrations ranging from 0 to 3.0 ppmw. The soil was thoroughly mixed in a plastic bag to ensure adequate distribution of the herbicide. The soil was placed into plastic cups and after a 20 hr equilibration period, sorghum or oats were planted. Cups were watered to field capacity and placed in a growth chamber, in a completely randomized design, at a constant temperature of 22°C and a 24 hr photoperiod at 1060 lux. There were 6 replications per treatment. Every 2-3 days, each cup was weighed and brought up to field capacity. Seedlings were thinned to 5 plants per cup after 5 days, and one week after planting, each cup was fertilized with 150 ppm of nitrogen, phosphorus, and potassium. At the end of 3 weeks, the aboveground plant parts were harvested, oven-dried, and weighed. A probit transformation of percent growth reduction due to herbicide injury was plotted against herbicide concentration to give standards curves for sorghum and oats for each herbicide in each soil type.

Before analysis, field soil samples were air-dried and passed through a 2 mm sieve. From each sample, four subsamples of 200 g each were placed into 8 cm, 200 mℓ plastic cups. Seeds of sorghum or oats were planted into each

cup. Sorghum was used as an indicator plant for herbicide concentrations up to approximately 1 ppmw, while oats were used as an indicator for the higher rates. Cups were placed in the growth chamber under the conditions described for the standard curves. After 3 weeks, plants were harvested, dried, and weighed. A probit transformation of percent growth reduction due to herbicide injury was compared to the appropriate standard curve and the herbicide concentration in ppmw was obtained. Statistics were not used to compare values.

RESULTS AND DISCUSSION

The bioassay determinations of herbicide residues are presented in the Appendix. Although certain trends were noted in the results, the bioassays lacked consistency. Only at the high chemical concentrations did either sorghum or oats exhibit significant growth reductions when compared to untreated plants. This is indicated by the response of oats 3 weeks after herbicide application. The concentrations of all chemicals even at the lowest rates should have been large enough to prevent oat growth but the results indicate several of the treatments having no herbicide present. Previous research (2) has indicated that when oats (*Avena sativa* L., var. Mulga) were grown in soil containing

low concentrations of a herbicide, the plants often had a greater fresh weight than the control plants. Others (6) have found that bioassay results from stored (frozen) and from freshly prepared standard series of linuron and nitralin differed significantly in topgrowth of the indicator plant indicating that freezing of the samples has some effect on availability of the herbicide or on the general characteristics of the soil. The differences between frozen and freshly prepared samples were most pronounced in the controls and in samples treated with low rates of the chemicals.

The limited sensitivity of the shoot dry weights of the indicator plants to the herbicide in the soil may be attributed to the optimum growing conditions of light, humidity, and soil moisture in the growth chamber rather than to a lack of herbicidal activity. Root observations indicated significant growth reductions in most treatments but comparative reductions in shoot growth were not evident. As determined from the study on the effect of low soil temperatures on phytotoxicities of these chemicals reported previously in this work, root length is a more definitive indicator of the herbicidal activity of dinitroanilines than is shoot dry weight, under laboratory conditions. Light, moisture, or nutrient stress may be necessary if shoot growth is to be used as a bioassay indicator for these herbi-

cides.

The data, however, did indicate certain differences in phytotoxicities and persistence of the dinitroanilines and also the role soil type has in expression of these differences. Dinitramine was the most phytotoxic chemical to oat growth 3 weeks after application, on all soil types. Profluralin was the least phytotoxic of all chemicals on the sandy loam and clay loam soils 3 weeks following herbicide application.

Fifty weeks after application of the chemicals, dinitramine was the least persistent chemical on sandy loam soil and appeared to be the most persistent chemical in the clay soil. Profluralin and fluchloralin were the least persistent chemicals in the clay loam soil. Trifluralin, profluralin, and fluchloralin appeared equally persistent in the sandy loam and clay soils. Trifluralin and dinitramine were the most persistent chemicals on the clay loam soil.

Shoot dry weights of sorghum on the sandy loam soil were not reduced by the 2 x rates of any of the chemicals, 50 weeks after application. The 1 x rate of all chemicals on the clay soil and the 2 x rate of all chemicals on the clay loam soil injured sorghum, fifty weeks after application.

Therefore, dinitramine appeared to be the most phy-

totoxic chemical to oats 3 weeks following application but was the least persistent after 50 weeks except on clay soil where persistence was greater than for the other chemicals.

The results obtained from these experiments that are in agreement with results from the field bioassays and analytical determinations presented previously in this report are:

1. dinitramine was the least phytotoxic chemical on the sandy loam soil 50 weeks after application of the herbicides.

2. dinitramine persistence increased with increased clay content.

3. trifluralin and fluchloralin were similar in persistence on the sandy loam soil 50 weeks after herbicide application.

4. the 2 x rate of all chemicals on the clay loam soil injured sorghum 50 weeks after application.

Growth chamber bioassays as described for these experiments do not appear to be a good indicator of dinitroaniline residues in soil. Measurements of roots, or the introduction of a stress, such as light, moisture, or nutrients, to plants grown in the growth chamber, may provide a better determination of dinitroaniline activity.

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GENERAL DISCUSSION

Results from these experiments indicate that the efficacy of dinitroaniline herbicides in weed control under normal cropping practices depends on soil type, chemical nature of the herbicide, susceptibility of the target plants, and climate. Increasing organic matter was found to decrease the phytotoxicity of trifluralin, profluralin, fluchloralin, and dinitramine to sorghum and oats. Rahman (1973) and Weber *et al.* (1974b) observed that organic matter reduced the phytotoxicity of trifluralin. Adsorption of the herbicides to organic matter in the soil is instrumental in reducing the amount of chemical that is biologically available, thereby decreasing the herbicides effectiveness.

Sorghum and oats were found to differ in susceptibility to the dinitroaniline herbicides, oats being more resistant than sorghum to injury by these chemicals. These results agreed with Murray *et al.* (1973). The phytotoxicities of these herbicides to susceptible plants is dependent on the physiological and morphological characteristics of the plants, which, together with soil type, determine the amount of chemical required for adequate weed control.

Low soil temperature (4^{o}C) decreased the herbicidal activity of profluralin, fluchloralin, and dinitramine. Re-

duced phytotoxicities of dinitroaniline herbicides resulting from low soil temperatures early in the growing season following application of the herbicides may allow weeds to "escape" the herbicides' effects and result in poor weed control in the current cropping year. Results from the growth chamber bioassays indicated that an environmental stress, such as light, moisture, or nutrients, may be necessary before dinitroaniline phytotoxicity is expressed in the form of reduced shoot growth.

The field bioassays, using wheat, oats, sorghum, and corn as indicator plants, showed that residues of dinitroaniline herbicides may persist in soil in large enough quantities to injure susceptible crops grown the year following a normal application of these herbicides.

Persistence of dinitroanilines was shown to be influenced by soil type, soil temperature, and possibly soil moisture. Profluralin and dinitramine persistence increased with increasing clay content while organic matter was responsible for increasing the persistence of trifluralin and fluchloralin. Adsorption of the chemicals by the organic and/or inorganic constituents of soil decreases the rate of disappearance of the chemicals by making them less available for biological and non-biological degradation and by reducing losses due to volatilization. Several researchers have shown

that trifluralin degradation is reduced by low soil temperatures (Horowitz *et al.*, 1974; Hamilton and Arle, 1972; Probst *et al.*, 1967). Analytical determinations of dinitroaniline residues in all soils studied indicated an average of only 7% loss of chemical over a seven month period from October to June. This limited amount of degradation was attributed not only to the reduced rate of degradation generally observed when approximately 20% of the applied herbicide remains, but also to the low soil temperatures occuring throughout this period. Reduced soil temperatures retard the chemical and microbial degradation of these chemicals which becomes a veryimportant consideration when evaluating dinitroaniline persistence in regions experiencing long, cool winter periods. Low amounts of rainfall following application of the chemicals may account for some of the injury observed in the indicator crops used for the detection of dinitroaniline residues in the field bioassays. Harvey (1973a) indicated that reduced soil moisture reduced the rate of disappearance of dinitroaniline herbicides. Low soil moisture induces an aerobic soil environment which impedes the degradation of dinitroaniline herbicides. Probst *et al.* (1967) noted less trifluralin degradation under aerobic soil conditions than under anaerobic soil conditions. Smith *et al.* (1973) observed that degradation of dinitramine

is also less under aerobic soil conditions than under anaerobic soil conditions.

Injury to crops planted the year following application of dinitroaniline herbicides depends on the amount of chemical persisting in the soil, the sensitivity of the crop to the chemical, and the growing conditions encountered. The field bioassay results showed that, although dinitroaniline residues were present in soil (according to the analytical determinations), detectable injury to susceptible crops occurred only if the residues were present in amounts greater than a certain critical concentration. As noted from the growth chamber bioassays, stress conditions of light, moisture, or nutrients may influence the degree to which the herbicide injury is expressed.

SUGGESTIONS FOR FURTHER WORK

The role of organic matter and soil type in dinitroaniline persistence should be further considered as indications from this study suggest profluralin and dinitramine persistence is affected by clay content while trifluralin and fluchloralin persistence is affected by organic matter. Previous research (Weber *et al.*, 1974a; Savage, 1973) has discussed the role of soil type in trifluralin persistence but information is lacking on the role of soil type in the persistence of profluralin, fluchloralin, and dinitramine.

Further investigation into the effects of low soil temperature on the phytotoxicity of dinitroaniline herbicides is necessary in order to explain the apparent reduced herbicidal activities of profluralin, fluchloralin, and dinitramine at low temperatures (4°C), as found in these experiments. The growth response curves of shoot dry weight and root length for trifluralin at low soil temperatures suggest that trifluralin may have a two-part effect on wild oat development. Perhaps trifluralin is affecting two or more physiological processes in the young seedlings rather than just inhibition of root development.

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APPENDIX Table 1. Common and generic names of bioassay plants cited in literature review

Common name	Generic name	Reference
Carrot	*Daucus carota* L., var. *sativa*	Probst *et al.* (1967)
Corn	*Zea mays* L., var. H49T x N_6	Burnside (1974), Messersmith *et al.* (1971)
Cotton	*Gossypium hirsutum* L.	Probst *et al.* (1967)
Crabgrass	*Digitaria* sp. L.	Probst *et al.* (1967)
Foxtail, giant	*Setaria faberi* Herrm.	Harvey (1973a)
Foxtail, Green	*Setaria viridis* (L.) Beauv.	Rahman (1973)
Garlic	*Allium sativum* L.	Probst *et al.* (1967)
Junglerice	*Echinochloa colonum* (L.) Link	Anderson *et al.* (1968)
Lettuce	*Lactuca sativa* L., var. *capitata* cv. Valverde	Menges and Tamez (1974)
Millet	*Panicum miliaceum* L., var. Panhandle	Burnside (1974)
	Panicum miliaceum L., var. White	Swann and Behrens (1974)
Millet, Italian (foxtail)	*Setaria italica* (L.) Beauv.	Harvey (1974), Menges and Tamez (1974), Weber *et al.* (1974a), Wright and Warren (1965)

APPENDIX Table 1. Continued

Common name	Generic name	Reference
Millet, Japanese	*Echinochloa crusgalli* (L.) Beauv. var. *frumentarea* (Roxb.) W.F. Wight	Miller *et al.* (1975)
Oats	*Avena sativa* L.	Savage and Barrentine (1969)
	Avena sativa L., var. Cimarron	Murray *et al.* (1973)
	Avena sativa L., var. Lodi	Harvey (1973a)
	Avena sativa L., var. Mulga	Horowitz *et al.* (1969)
	Avena sativa L., var. Neal	Burnside (1974)
Onion	*Allium cepa* L.	Probst *et al.* (1967)
Pigweed, redroot	*Amaranthus retroflexus* L.	Menges and Tamez (1974)
Sorghum	*Sorghum bicolor* (L.)	Menges and Tamez (1974), Miller *et al.* (1975)
	Sorghum bicolor (L.) Moench var. Dekalb 57E	Savage (1973)
	Sorghum bicolor (L.) Moench var. OK 612	Murray *et al.* (1975)
	Sorghum vulgare L., var. Amak R-12	Anderson *et al.* (1968)

APPENDIX Table 1. Continued

Common name	Generic name	Reference
	Sorghum vulgare L., var. Hazera 6078	Horowitz *et al.* (1974)
Soybean	*Glycine max* L.	Probst *et al.* (1967)
	Glycine max (L.) Merr. var. Corsoy	Harvey (1973a)
Sugar beet	*Beta vulgaris* L.	Hamilton and Arle (1972)
Velvetleaf	*Abutilon theophrasti* Medic.	Harvey (1973a)
Wheat	*Triticum aestivum* L., var. Manitou	Rahman (1973)
Wild oats	*Avena fatua* L.	Friesen and Shebeski (1961), Leggett and Banting (1959)

Appendix Table 2. Analytical determination of herbicide remaining in sandy loam soil sampled periodically following application.

Herbicide application rate (ppmw)	Herbicide concentration (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
0.75	0.31	0.18	0.14	0.13
1.5	0.86	0.51	0.41	0.27
3.0	1.21	0.69	0.46	0.52
Profluralin				
1.0	0.28	0.32	0.33	0.22
2.0	0.82	0.61	0.51	0.38
4.0	1.57	1.72	0.90	1.06
Fluchloralin				
1.0	0.35	0.26	0.14	0.72
2.0	0.76	0.51	0.35	0.26
4.0	1.62	1.35	0.63	0.81
Dinitramine				
0.67	0.22	0.13	0.12	0.04
1.33	0.34	0.22	0.17	0.11
2.67	0.65	0.53	0.30	0.26

Appendix Table 3. Analytical determination of herbicide remaining in clay loam soil sampled periodically following application.

Herbicide application rate (ppmw)	Herbicide concentration (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
2.0	1.03	0.68	0.70	0.54
4.0	1.97	2.03	1.07	0.98
8.0	4.08	3.78	2.69	2.13
Profluralin				
2.5	1.33	1.26	1.20	0.79
5.0	2.40	2.24	1.98	1.02
10.0	4.18	3.94	3.26	2.32
Fluchloralin				
2.5	1.07	1.00	0.74	0.47
5.0	2.44	1.69	1.55	1.20
10.0	4.71	2.16	3.97	1.61
Dinitramine				
1.5	0.76	0.58	0.33	0.25
3.0	1.37	0.87	0.64	0.47
6.0	4.15	2.46	2.38	1.01

Appendix Table 4. Analytical determination of herbicide remaining in clay soil sampled periodically following application.

Herbicide application rate (ppmw)	Herbicide concentration (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
1.0	0.27	0.22	0.19	0.17
2.0	0.49	0.39	0.32	0.32
4.0	1.58	1.15	0.89	1.09
Profluralin				
1.25	0.59	0.60	0.50	0.46
2.5	1.94	1.20	1.01	0.70
5.0	2.81	2.21	2.16	1.31
Fluchloralin				
1.25	0.45	0.51	0.39	0.22
2.5	1.20	1.02	0.50	0.33
5.0	2.57	2.34	1.40	0.57
Dinitramine				
0.75	0.38	0.37	0.18	0.19
1.5	1.00	0.65	0.39	0.36
3.0	2.18	1.28	1.12	0.88

Appendix Table 5. Bioassay determination of herbicide remaining in sandy loam soil sampled periodically following application.

Herbicide application rate (ppmw)	Herbicide concentration (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
0.75	0.34	0.06	0.21	0.0
1.5	0.87	0.09	0.49	0.06
3.0	1.64	0.51	0.72	0.58
Profluralin				
1.0	0.40	0.24	0.28	0.0
2.0	0.51	0.0	0.19	0.0
4.0	1.93	0.79	0.97	0.85
Fluchloralin				
1.0	0.28	0.18	0.0	0.15
2.0	0.43	0.0	0.32	0.15
4.0	2.63	1.02	1.06	0.55
Dinitramine				
0.67	0.60	0.08	0.16	0.0
1.33	0.95	0.13	0.28	0.0
2.67	2.15	1.01	0.56	0.11

Appendix Table 6. Bioassay determination of herbicide remaining in clay loam soil sampled periodically following application.

Herbicide application rate (ppmw)	Herbicide remaining (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
2.0	1.48	0.81	1.15	0.24
4.0	6.70	4.31	2.81	1.77
8.0	15.07	10.50	6.58	49.06
Profluralin				
2.5	1.65	----*	----	----
5.0	4.06	5.06	----	----
10.0	17.66	14.4	4.96	----
Fluchloralin				
2.5	2.15	4.66	1.14	0.08
5.0	9.96	3.55	5.45	3.28
10.0	20.32	11.86	6.72	12.17
Dinitramine				
1.5	1.28	11.08	0.60	0.09
3.0	2.78	3.01	2.16	1.49
6.0	7.58	8.10	2.89	6.55

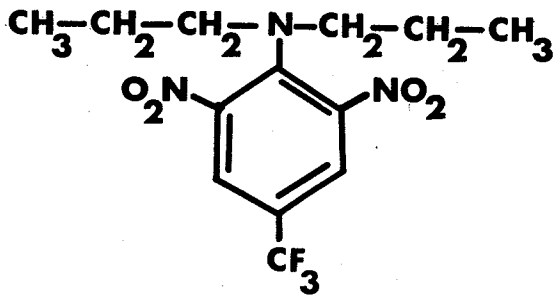
*Values missing because of inadequate standard curve.

Appendix Table 7. Bioassay determination of herbicide remaining in clay soil sampled periodically following application.

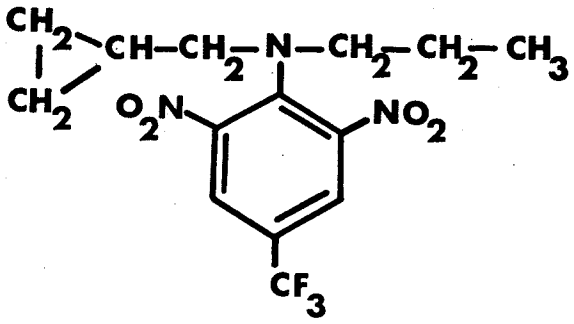
Herbicide application rate (ppmw)	Herbicide remaining (ppmw)			
	3 weeks	8 weeks	16 weeks	50 weeks
Trifluralin				
1.0	0.0	0.0	0.99	0.71
2.0	0.54	0.45	1.60	1.09
4.0	2.03	1.39	1.51	3.35
Profluralin				
1.25	0.0	0.40	0.63	0.42
2.5	1.25	0.23	1.23	0.71
5.0	2.85	2.48	4.10	1.10
Fluchloralin				
1.25	0.0	0.31	0.58	0.58
2.5	1.01	0.32	0.68	0.51
5.0	8.93	3.93	4.92	1.41
Dinitramine				
0.75	0.66	0.28	0.51	0.31
1.5	1.47	1.66	1.28	0.76
3.0	2.52	4.04	12.31	4.28

APPENDIX TABLE 8. Chemical structures of dinitroanilines.

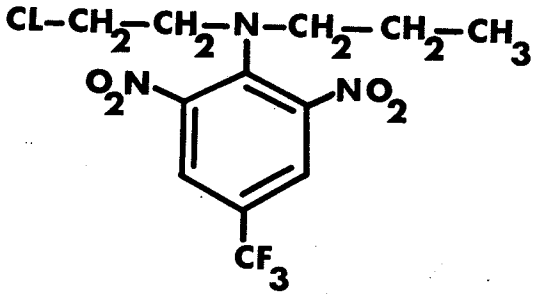
TRIFLURALIN



PROFLURALIN



FLUCHLORALIN



DINITRAMINE