Soil movement and residual aspects of chlorsulfuron and its control of Canada thistle (Cirsium arvense (L.) Scop.)
by William Edward Dyer

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Agronomy
Montana State University

Abstract:
Chlorsulfuron (2-chloro-N-[(4-methoxy-6-methyl-l,3,5-triazin-2-yl)aminocarbonyl] benzenesulforamide) is a new, residual herbicide used for the control of broadleaf weeds in small grains. Chlorsulfuron controls several 2,4-D [(2,4-dichlorophenoxy)acetic acid] resistant weeds and Canada thistle [Cirsium arvense (L.) Scop.]. Suggested use rates, which depend upon soil pH, range from 9 to 26 g a.i./ha. Chlorsulfuron is mobile in the soil, and has a high potential for leaching. Degradation of chlorsulfuron, which is accomplished through chemical hydrolysis, is slower in alkaline soils.

The movement of chlorsulfuron was measured in soil columns. Bioassays using foxtail millet [Setaria italica (L.) Beauv. 'Golden German'] indicated that chlorsulfuron was present to the bottom of 22.5 cm soil columns, after leaching the soil to field capacity. Under similar leaching conditions, 14C-labeled chlorsulfuron was found in decreasing amounts with increasing depth in soil columns. Delays after herbicide application and before leaching with water had little effect on the degree of chlorsulfuron movement. 14C-labeled chlorsulfuron moved as a concentrated front on soil thin-layer chromatography (TLC) plates. Increased movement on soil TLC plates was attributed to the high herbicide:soil ratio used.

The effect of soil residues of chlorsulfuron on 11 rotational crops grown in Montana was measured. Weed control and crop injury ratings, and crop dry weights were taken for 3 successive years after applications of 35, 70, and 140 g a.i./ha. Dry weight was reduced for all crops tested by all rates of application except wheat (Triticum aestivum), barley (Hordeum vulgare), and oats (Avena sativa) 36 months after herbicide application. The rate of degradation of chlorsulfuron in two Montana soils appeared to be consistent with a reported half-life of 60 days.

The effect of chlorsulfuron on Canada thistle regrowth was measured. Chlorsulfuron, applied at 18, 35, and 70 g a.i./ha, reduced Canada thistle stems/m2 1 year after application. Applications at the 5 leaf stage of Canada thistle appeared to be slightly more effective than treatments applied at the bud stage. There was no reduction of Canada thistle stems/m2 by any rate 2 years after application. Treatments applied in 2 successive years at lower rates provided more control of Canada thistle regrowth after 2 years than single applications at higher rates.

SOIL MOVEMENT AND RESIDUAL ASPECTS OF CHLORSULFURON AND ITS CONTROL OF CANADA THISTLE [*Cirsium arvense* (L.) Scop.]

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ABSTRACT

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CHAPTER 1

LITERATURE REVIEW

Movement of Chlorsulfuron in Soil

Movement of herbicides in soil is affected by (1) adsorption, (2) physical properties of the soil, and (3) climatic conditions (Bailey and White, 1970). Each of these factors must be considered before a discussion of chlorsulfuron (2-chloro-N-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl] benzenesulfonamide) and its behavior in the soil can progress.

Adsorption

The relationship between adsorption and herbicide movement is not fully understood. Upchurch and Pierce (1957, 1958) proposed that two steps are involved in the leachability of a herbicide: (1) entrance of the molecule into solution and (2) adsorption of the molecule to soil colloids. Entrance of a herbicide into the soil solution can take place either from the dissolution of the herbicide particle or from desorption from a colloidal surface. Once the herbicide molecule is present in the soil solution, factors including the soil or colloid type, the chemical nature of the herbicide molecule, soil reaction (pH), temperature, and the nature of the herbicide formulation will influence its adsorption onto soil colloids. Each of these factors will be discussed as they apply to the adsorption and leaching potential of chlorsulfuron.

Soil colloids are generally divided into the mineral and organic fractions. The expanding 2:1 clay minerals, such as montmorillonite (which is the predominant clay in Montana) (Anon., 1975), have a high cation exchange capacity and large surface area. These properties impart a high adsorption capacity. However, because chlorsulfuron is weakly acidic (Shea, 1982), it would tend to be negatively adsorbed to clay colloid surfaces (Carringer et al., 1975). Further, clay surfaces in natural soils are usually coated with organic matter, which reduces the number of clay sites available for adsorption (Hance, 1969). If this is the case, chlorsulfuron is not adsorbed to a significant degree by clay colloids.

Adsorption of herbicides to organic matter is strongly correlated to retention against leaching (Helling et al., 1971). Goring (1967) cited 41 references attesting to the importance of organic matter in soil adsorption of pesticides. Chlorsulfuron, which is classified as a sulfonylurea herbicide, is related to the triazine and urea families of herbicides (Figure 1). There are several reports (Williams, 1968; Walker and Crawford, 1970; Wu and Santelmann, 1975; Lambert et al., 1965) which showed that triazine herbicides were adsorbed more and leached less in heavy, high organic matter soils than in light, sandy soils. The substituted urea herbicides have been shown to behave in a similar manner (Harris and Warren, 1964; Hilton and Yuen, 1963; Upchurch and Pierce, 1958; Sherburne et al., 1956; Grover, 1975; Yuen and Williams, 1962; Rhodes et al., 1970). There should therefore be more adsorption and less leaching potential for chlorsulfuron on high organic matter soils, as has been reported by Palm et al. (1980).

The chemical nature of a herbicide molecule has a direct influence on its capacity to be adsorbed. Adsorption is increased by the presence of such functional groups as R_3N+, $-CONH_2$, $-OH$, $-NHCOR$, $-NH_2$, $-OCOR$, and $-NHR$ on the adsorbate molecule (Helling et al., 1971). Amino groups may become protonated and be adsorbed as cations. Carbonyl and amino groups may participate in hydrogen bonding. Chlorsulfuron, which contains two amino groups, should be adsorbed. However, polarity and polarizability (a measure of the ease by which the positive and negative charges in a molecule can be displaced with respect to each other in the presence of an electric field) are also important forces in

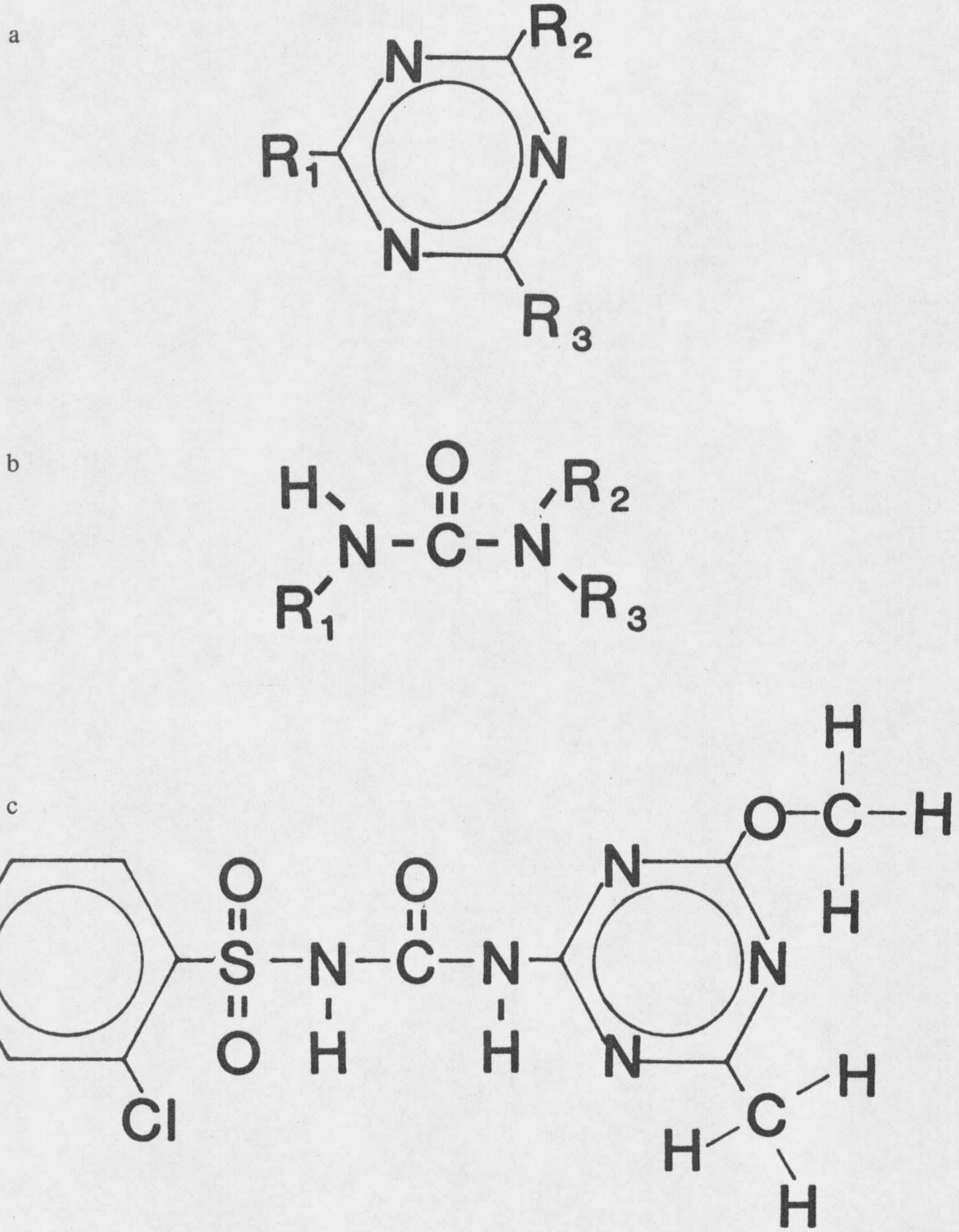

Figure 1. Chemical structures of the *s*-triazine (a) and urea (b) herbicide families, and the structure of chlorsulfuron (c).

adsorption (Bailey and White, 1970). The chlorsulfuron molecule appears to be highly polar, and at the same time, somewhat polarizable (Shea, 1982) (Figure 2).

The polar nature of the molecule would increase the degree to which it is solvated in the soil solution, which, in turn would increase the overall energy required for adsorption to occur. Water molecules can effectively compete with polar herbicide molecules for adsorption sites on clay, which would reduce adsorption.

The polarizability, or delocalization of charges, of the chlorsulfuron molecule suggests that it can participate in hydrophobic or charge transfer bonds (Shea, 1982), which are significant in acidic or neutral environments (Bailey and White, 1970). These types of bonding reactions would be less significant in alkaline soils which predominate in Montana.

Molecular size or parachor has been used as a measure for adsorption of herbicides on soil (Lambert, 1967). This relationship, however, assumes that organic matter is the primary adsorbing medium in the soil and that the adsorbate or solute is an uncharged molecule. The attempt to relate the molecular weight of the polar chlorsulfuron molecule (GMW. 358) to adsorption potential would thus be questionable, using Lambert's method.

The dissociation constant (pka or pkb) of a herbicide molecule is an important factor in determining the extent of adsorption and the ease of desorption by colloidal systems. The dissociation constant is reported to be the major factor controlling the magnitude of adsorption for the s-triazine and substituted urea herbicides (Bailey et al., 1968). The pka of chlorsulfuron is 3.8 (Shea, 1982). Frissel (1961) noted that negative adsorption for such herbicide acids as 2,4-D [(2,4-dichlorophenoxy)acetic acid] and 2,4,5-T [2-(2,4,5-trichlorophenoxy)propanoic acid] occurred as the pH of a clay-water system approached the pka of the particular compound (starting from neutral). As the pH continued to decrease, positive adsorption commenced and increased gradually. Bailey et al. (1968) reported that positive adsorption of acidic compounds did not begin until the pH of the bulk solution was 1 to 1.5 pH units above the pka of the acid. Thus chlorsulfuron, which is

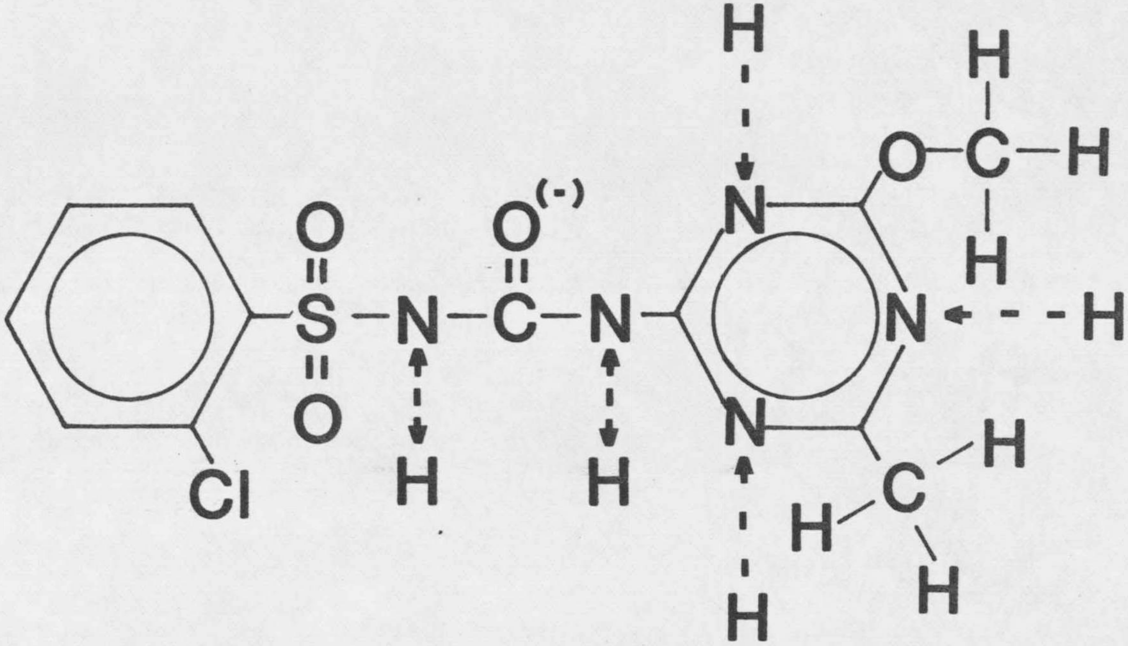

Figure 2. Polar and polarizable characteristics of the chlorsulfuron molecule.

a weak to moderate acid, would not be strongly adsorbed under the alkaline soil conditions in Montana. This trend has been noted by Palm et al. (1980) and Shea (1982).

The effect of the pka on herbicide adsorption appears to be closely related to the soil reaction (pH of the soil solution). In a study of 22 structurally diverse herbicides, Bailey et al. (1968) found that, regardless of the chemical character of the herbicide molecule, adsorption was greater on a highly acidic hydrogen-montmorillonite adsorbent (pH 3.35) than on nearly neutral sodium-montmorillonite (pH 6.8). Adsorption of acidic herbicides is generally more dependent on the pH of the soil solution than on the pka of the molecule itself. Shea (1982) reported that chlorsulfuron is adsorbed less in alkaline solutions.

The relationship between the water solubility of a herbicide molecule and adsorption is unclear. Several authors (Briggs, 1969; Hance, 1965; Harris and Warren, 1964) found no relationship between the degree of water solubility and the extent of adsorption for members of the substituted urea family. Others (Frissel, 1961; Yuen and Hilton, 1962; Hilton and Yuen, 1963; Wolf et al., 1958) found that the order of adsorption of the substituted ureas was inversely related to their water solubilities. These results are in direct contrast to the results of Bailey et al. (1968), who reported a direct relationship between water solubility and adsorption of the substituted ureas on H-montmorillonite.

In a review of the relationship between water solubility and adsorption, Bailey and White (1970) concluded that within a chemical family or analogue series basic in chemical character, the magnitude of adsorption was governed by the degree of water solubility. Comparisons which are based on water solubility alone made among structurally diverse herbicides are generally not valid (Bailey et al., 1968). In addition, several of the above studies were carried out on natural soils, while other adsorption measurements were made on purified clay or synthetic adsorbents. Caution must be exercised when comparing data obtained from 'pure' clay or soil constituents to results obtained with natural soils (Grover, 1975).

The water solubility of chlorsulfuron (125 ppm) is an average value for soil-active herbicides (Bailey et al., 1968). Helling et al. (1971) stated that the water solubility of a herbicide molecule, when taken alone, does not appear to be a valid indicator of leachability. Hance (1967) and Briggs (1969) reported that π (a measure of the hydrophobic/hydrophilic balance of a molecule) was a good predictor of adsorptive behavior of herbicides in soil systems. A π value for chlorsulfuron is not available at this time.

Adsorption is an exothermic process and desorption is endothermic, so an increase in temperature would be expected to reduce adsorption and favor desorption. However, Harris and Warren (1964) reported greater adsorption of simazine [2-chloro-4,6-bis(ethylamino)-s-triazine], atrazine [2-chloro-4-(ethylamino)-6-(isopropylamino)-s-triazine, and monuron [3-(p-chlorophenyl)-1,1-dimethylurea mono(trichloroacetate)] at 0° than at 50°C. Valid generalizations about the effect of temperature on herbicide adsorption are difficult to develop, due to interactions with other factors.

Herbicide formulation and the presence of surfactants can influence adsorption. Gaynor and Volk (1976) found that addition of 1.0% v/v of a cationic surfactant increased adsorption of picloram (4-amino-3,5,6-trichloropicolinic acid) at pH 5.0. Others (Bayer, 1967; Smith and Bayer, 1967) reported similar results for the substituted urea herbicides. All workers reported no effect or slightly decreased adsorption with the addition of a nonionic or anionic surfactant. The explanation for increased adsorption with the addition of a cationic surfactant was that the positively charged surfactant molecules were being readily adsorbed, and creating a lipophilic layer on the colloid surfaces. Herbicide molecules were then easily adsorbed on the lipophilic surfaces. Chlorsulfuron is applied with the addition of 0.25% v/v surfactant. Since most surfactants presently in use are nonionic or anionic, little effect on adsorption would be expected to occur.

Adsorption in soils is usually measured empirically by the Freundlich equation,

$$x/m = KC^{1/n}$$

where x/m is the amount of herbicide (x) adsorbed per unit amount of adsorbent (m); K and n are constants describing adsorption, and C is the equilibrium solution concentration. The Freundlich isotherm is utilized graphically by plotting log x/m vs. log C. If the equation is applicable to the data, a straight line is obtained with a slope of 1/n. K values are directly correlated to adsorption.

Measured Freundlich K values for chlorsulfuron are .68 on a Flanagan silt loam (aquic argiudoll, 5% OM, pH 5.0) and .4 on a Keyport silt loam (aquic hapludult, 2.1% OM, pH 5.8) (DuPont, 1980). The variation of these values reflects the dependence of the Freundlich isotherm on soil type. Although substituted urea herbicides generally conform to the Freundlich equation (Kozak and Weber, 1983), chlorsulfuron K values determined on more alkaline soils would probably be considerably lower, due to decreased adsorption. Also, K values are highly dependent on the ratio of soil:water used in the determination (Grover and Hance, 1970). They found a 6-fold increase in the K value for linuron [3-(3,4-dichlorophenyl)-1-methoxy-1-methylurea] when the soil:water ratio was increased from 4:1 to 1:10. Likewise, K values for atrazine increased by a factor of 3 when determinations were made on the same range of soil:water ratios. K values, then, are useful when comparing herbicide leaching potentials if the measurements are all made under the same experimental conditions. K values alone are of limited use in describing the behavior of a particular herbicide under various soil conditions.

The adsorption of chlorsulfuron on soil colloids, then, appears to be primarily correlated to the amount of organic matter and the soil pH (Palm et al., 1980). Clay content does not appear to be highly related to adsorption.

Soil and Climatic Factors

While herbicide adsorption is the primary factor concerning the potential for herbicide movement in the soil, soil characteristics and climatic conditions also affect movement.

It is generally assumed that herbicides move in soil via mass flow and diffusion (Helling et al., 1971). Movement by diffusion is insignificant since it accounts for only a few centimeters of displacement in soil (Hartley, 1960). Since chlorsulfuron is essentially nonvolatile (vapor pressure 4.6×10^{-6} mm Hg at 25°C), little movement via diffusion would be expected.

Mass flow of herbicides in the soil solution is affected by any soil factor which affects water movement. Soil pore size and pore-size distribution appear to be the most significant factors, since they affect the rate of water movement in soil (Bailey and White, 1970). Sherburne et al. (1956) reported that the degree of leaching of chloroxuron [3(p-chlorophenyl)-1,1-dimethyl urea] was directly related to the rate of water flux in soil columns. Upchurch and Pierce (1957, 1958) reported increased leaching of monuron in a Lakeland sand soil as the amount and rate of applied water increased. Davidson et al. (1968) found that two substituted urea herbicides behaved similarly in both water-saturated glass bead columns and packed soil columns.

The potential exists, then, for significant leaching of chlorsulfuron under high water flow conditions. Hageman and Behrens (1980) and Nilsson (1982) conducted field experiments which support this conclusion. Hageman and Behrens reported that chlorsulfuron leached readily in a silty clay soil with 2.8% OM. Nilsson found chlorsulfuron at depths of 15 cm on all soil types tested and at 25 cm on a clay soil 10 months after applications of 10 g/ha. He also reported indications of upward movement of chlorsulfuron by capillary action, and lateral movement into untreated plots. Unfortunately, precipitation levels were not reported in either case.

O'Sullivan (1982) reported that chlorsulfuron was readily leached in a silt loam soil (13.7% OM, pH 6.8) in soil columns made of poly-vinyl chloride (PVC) pipe. Using Tartary buckwheat [*Fagopyrum tataricum* (L.) Gaertn.] as a bioassay plant, he found chlorsulfuron at depths of 11 and 17 cm following applications of water which were equivalent to

38 and 75 mm of rainfall. He suggested that leaching of chlorsulfuron, coupled with persistence, may be responsible for observations of increased injury to sensitive crops from older chlorsulfuron soil residues.

The relationship between initial soil moisture condition and herbicide movement is unclear. Several substituted urea and acid herbicides were leached more deeply in moist rather than dry soils (Wiese and Davis, 1962). Another study, however, showed that the mobility of monuron was not affected by soil water content (Upchurch and Pierce, 1958). Chlorsulfuron would probably be leached more deeply in high moisture content soils, due to a low affinity for soil adsorptive sites.

Several methods have been used to study the movement of herbicides in soil. Soil columns have been used extensively since their use was first reported by Ogle and Warren (1954). Intact soil cores have been used (Poelstra and Frissel, 1967), and probably most closely reflect actual field conditions. Lambert et al. (1965) developed a variation of the soil column which he termed the "slotted tube technique" to test the applicability of chromatographic theory to the movement of herbicides in soil. He concluded that leaching depended on soil type, amount of organic matter, and soil water content. Harris (1967) used subirrigated soil columns designed to reduce the rate of water flow which occurs with gravity flow columns.

Soil thick-layer and thin-layer chromatography are more recent techniques. The thick-layer chromatography technique utilizes a shallow tray filled with soil in which herbicides are leached with water (Gerber et al., 1970). A plant bioassay is carried out on the soil after leaching is complete.

Soil thin-layer chromatography (TLC) was first described by Helling and Turner (1968). The technique uses a soil matrix applied to a glass plate, and is analogous to conventional TLC. Radioactive labeled herbicides are spotted onto the plate and are leached by the capillary action of ascending water. Results are visualized by autoradiography, and can be quantified by oxidation and liquid scintillation counting. The soil TLC technique has been used by Helling (1971a, 1971b, 1971c) and others (Rhodes et al., 1970) to compare the mobility of several herbicides when exposed to different leaching conditions. Rhodes et al. (1970) reported that the soil TLC method provided information about herbicide leaching that was not obtained from Freundlich K values. Autoradiographs of soil TLC plates showed whether herbicides moved as a compact band or exhibited "tailing." Also, soil TLC provided an estimate of the lateral diffusion of herbicides in soil.

Wu and Santlemann (1975) compared repeatability, ease of preparation, and time required for the three leaching techniques. They concluded that Rf values from soil thick-layer and thin-layer chromatography were almost identical, while herbicides moved somewhat farther in slotted columns. The soil TLC technique required the shortest preparation time, but required the longest drying and development time (21 days). Recent advances in X-ray film have reduced the development time to 3 to 4 days, which would be substantially less than the time required for the other two methods, which depend on the growth of bioassay plants.

Chlorsulfuron Persistence in the Soil

Chlorsulfuron is the first member of the sulfonylurea family of herbicides to be marketed. This family combines characteristics from two related herbicide families, the substituted ureas and the triazines.

A substantial amount of research has been carried out on the persistence of the substituted ureas and their breakdown in the soil. Geissbuhler et al. (1975) listed 51 references concerning field observations of persistence of the substituted ureas and biological pathways for their decomposition by soil microbes. Likewise, Esser et al. (1975) noted that several reviews (Harris et al., 1968; Jordan et al., 1970; Kaiser et al., 1970; Kaufman and

Kearney, 1970; Sheets, 1970) have been compiled concerning the persistence and degradation of the triazine herbicides in soil. Both the substituted ureas (Geissbuhler et al., 1975) and the triazines (Esser et al., 1975) appear to be degraded primarily by soil microorganisms. Jordan et al. (1970), however, noted that nonbiological pathways may be more important in the degradation of triazines than previously thought.

Chlorsulfuron is broken down almost exclusively by chemical hydrolysis, with little effect of soil microbes on the parent molecule (Palm et al., 1980). Because of this method of degradation, comparisons between the hydrolytic breakdown of chlorsulfuron and the microbially-degraded ureas or triazines are generally not valid.

Any soil factor which influences chemical reactions will influence the rate of degradation of chlorsulfuron. The three most significant soil factors regulating the rate of breakdown of chlorsulfuron are temperature, soil moisture, and soil pH (Palm et al., 1980). Generally, the rate of degradation of any soil active herbicide increases with an increase in temperature if adequate soil moisture is available (Hurle and Walker, 1980). These generalizations appear to be valid for chlorsulfuron (Walker and Brown, 1983).

The relationship between soil pH and chlorsulfuron degradation is not well documented, but there appears to be less hydrolysis of chlorsulfuron in alkaline soils (Palm et al., 1980; Walker and Brown, 1983). At alkaline pH, the relative absence of H^+ ions would prevent significant nucleophilic attack at the carbon atom and subsequent hydrolysis of the chlorsulfuron molecule.

Several instances of chlorsulfuron persistence in field situations have been reported. Hageman and Behrens (1980) reported that applications of 125 and 250 g/ha[1] persisted for more than one year in Minnesota and severely injured sugar beet (*Beta vulgaris*) and sunflower (*Helianthus annuus*). Residue levels were higher in a clay loam soil (pH 8.3) than in a silt loam soil (pH 6.1). They suggested that low levels of precipitation may have further

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[1] All herbicide rates are expressed in g/ha of active ingredient (a.i.).

inhibited chlorsulfuron degradation in the high pH soil. Salembier and Gomand (1981) reported slight injury to winter barley (*Hordeum vulgare*) planted six months after applications of 48 to 480 g/ha chlorsulfuron, on silty and sandy soils. In another trial, chlorsulfuron rates of 48 to 280 g/ha severely injured winter barley and vetch crops, planted six months after herbicide application. Sugar beets planted in the fall, 1980 showed toxicity symptoms from chlorsulfuron at 240 and 320 g/ha applied in the spring, 1978. Van Himme et al. (1981) reported 100% injury to sugar beet, spinach (*Spinacia oleracia*), onion (*Alium cepa*), and red clover (*Trifolium incarnatum*) planted in April, 1981 from 25 g/ha chlorsulfuron, applied the previous October. Several other broadleaf crops were severely injured. In related studies, they observed more injury from 10-month old residues than from treatments applied six months previously, possibly due to herbicide leaching, as was suggested by O'Sullivan (1982). Brenchley (1981) reported that 10 g/ha chlorsulfuron, applied to fallow ground on May 20, 1980 severely injured potato (*Solanum tuberosum*), sugar beet, lettuce (*Lactuca sativa*), dry bean (*Phaseolus vulgaris*), alfalfa (*Medicago sativa*), and corn (*Zea mays*) planted in May, 1981. Norris et al. (1981) observed severe injury to sugar beet, onion, alfalfa, and carrot (*Daucus carota*) 10 months after applications of 17, 35, and 70 g/ha of chlorsulfuron.

Nilsson (1982) carried out bioassays on chlorsulfuron-treated field soil samples from various parts of Sweden and detected residues from 10 g/ha chlorsulfuron 11 months after application. Palm et al. (1980) reported that spring applications of 30 g/ha in England, France, and West Germany injured sugar beet planted 12 months later. Fall applications, however, of 80 g/ha in these same three countries did not injure sugar beet planted 18 months later. In contrast, Brewster et al. (1981) reported marked injury to sugar beet in Oregon from 35 g/ha applied 18 months previously.

The conflicting reports of chlorsulfuron's residual period are undoubtedly due to climatic and soil type differences at the various locations. The colder soil temperatures in northern Minnesota, for example, would contribute to increased persistence of chlorsulfuron. Walker and Brown (1983) have developed a mathematical model to predict levels of chlorsulfuron residues following applications at different times or locations. Bioassays (Bond and Roberts, 1976) were carried out every two weeks for 98 days on spiked soil samples (sandy loam, 1.2% OM, pH 7.1) that were held at 2 moisture contents and 3 temperature regimes. Soil samples were also taken at the same intervals from treated field plots, and subjected to the bioassay. Degradation curves were constructed from these data which showed that the rate of chlorsulfuron loss was affected by both temperature and soil moisture content. The data were then used in a previously reported computer program (Walker and Barnes, 1981) to predict possible chlorsulfuron residues after various time intervals. The predicted levels of residue were in general agreement with field results reported by Palm et al. (1980). The model, however, failed to account for the effects of soil type and soil pH on the rate of degradation of chlorsulfuron. This omission would limit any predicted residual levels of chlorsulfuron to soils that were characteristically similar to the experimental soil used for this work. The model does, however, illustrate the potential usefulness of the technique to evaluate herbicide persistence in the soil.

Control of Canada Thistle with Herbicides

Canada thistle [*Cirsium arvense* (L.) Scop.] is an aggressive perennial weed which reproduces from seed and by an extensive root system. Seed production may vary from 3 to 103 seeds/head (Amor and Harris, 1974). The plant is dioecious, and insects are the primary means of pollination (Dershied and Schultz, 1960). Germination of freshly harvested seed can range from 53 to 96%, and some authors (Bakker, 1960; Kolk, 1947) have described a dormancy period for Canada thistle seed. Once in the soil, seed can remain viable for as long as 21 years (Hodgson, 1968).

The root system of Canada thistle allows the plant to spread rapidly once it has become established in an area. Segments from any part of the root system as small as 5 mm thick and 8 mm in length can produce new shoots from adventitious buds (Prentiss, 1889). Canada thistle roots have been shown to penetrate the soil to a depth of 2.4 m (Paczoskij and Berg, 1917), although the majority of roots are concentrated in the upper 1.3 m of the soil profile (McAllister and Haderlie, 1981). Roots at these depths are well below the reach of tillage implements. Carbohydrate reserves in an established Canada thistle root system enable the plant to initiate new shoots for about 1.5 growing seasons despite repeated cultivations that prevent production and transfer of new food to the roots (Seely, 1952).

Canada thistle is widely distributed in the northern half of the United States (Anon., 1970) and southern Canada (Moore, 1975). Blankenship (1901) recorded infestations of Canada thistle in only five locations in Montana in 1901. By 1956, Heikes (1956) reported more than 253,000 ha of Canada thistle in the state. Currently, it is estimated that Canada thistle infests over 450,000 ha of cropland in Montana (Jackson, personal communication). Canada thistle is spreading and infests additional land each year (Alley, 1981).

Canada thistle is extremely competitive with crops for water and nutrients. Yield losses from Canada thistle are difficult to determine due to the uneven, patchy infestations of the weed. In Canada thistle infestations of 50 shoots/m^2, crop yield losses often exceed 70% (Behrens and Elakkad, 1981). Brown (1959) reported a wheat (*Triticum aestivum*) yield loss of 34% from a Canada thistle stand density of 14 shoots/m^2. Similarly, Hodgson (1971) reported that a Canada thistle density of 25 shoots/m^2 reduced wheat yields by 59%.

The cost of these yield losses is significant. Messersmith (1981) estimated annual losses in wheat of $18.3 million in 1978 and $18.8 million in 1979 in North Dakota. Several authors (Amor and Harris, 1975; Bendall, 1975; Stachon and Zimdahl, 1980;

Wilson, 1981) have suggested that suppression of plants growing in Canada thistle infestations might be due to allelopathy.

Common herbicides used for controlling or suppressing Canada thistle include 2,4-D, dicamba (3,6-dichloro-o-anisic acid), picloram and glyphosate [N-(phosphonomethyl) glycine].

Results from spraying Canada thistle with 2,4-D amine have varied from a 100% reduction in density after two years (Alley et al., 1981), to a range of 16 to 80% reduction after four years of spraying (Hodgson, 1958; McKay et al., 1959). The combination of herbicides with tillage and cropping has been shown to be more effective in reducing Canada thistle regrowth than herbicide treatments alone (Hodgson, 1958; McKay et al., 1959; Dershied et al., 1961). Hodgson (1958) reported that Canada thistle density was reduced to 14, 3, and 1% of the original stand after 1, 2, and 3 years of cropping with spring wheat and yearly applications of .84 Kg/ha of 2,4-D amine. When the field was mechanically fallowed during the years before and after the spring wheat plus 2,4-D treatment, the Canada thistle density was reduced to 0.3, 0.7, and 0.0% of the original stand after 1, 2, and 3 years. Rasmussen (1956) noted no significant difference in efficacy from applications of 2,4-D amine at the bud, bloom, and mature growth stages of Canada thistle. Applications at the 5 leaf stage were less effective. He concluded that applications made at the bud stage would be preferable because they would prevent seed formation during the year of treatment. Continuous, yearly treatments of 2,4-D amine alone have been shown to select for tolerant ecotypes of Canada thistle (Hodgson, 1970; O'Rear and Whitworth, 1969; Saidak, 1966).

Response of Canada thistle to dicamba has generally been similar to the pattern of the phenoxy herbicides (Marriage, 1981). The period of maximum sensitivity appears to be between the vegetative shoot and bud stages (Carson and Bandeen, 1975; Hodgson, 1977;

Saidak, 1970). Rates of 2.2 to 3.3 Kg/ha of dicamba applied in the fall have shown to effectively control regrowth the year after treatment (Gale, 1972; Hodgson, 1971).

Picloram and especially 3,6-dichloropicolinic acid, which is an analogue of picloram, have been shown to be very effective in controlling Canada thistle regrowth (Whitesides and Appleby, 1978). Neither stage of growth of the Canada thistle nor time of application appeared to significantly affect the degree of control (VandenBorn and Schraa, 1974; Warden, 1965). However, the residual nature and subsequent crop injury potential of dicamba and picloram, especially at the higher rates needed for regrowth control, limit their use as Canada thistle herbicides.

Glyphosate, a non-residual herbicide, has given effective control of Canada thistle, especially when applied at the bud-to-bloom growth stage. The non-selective nature of glyphosate precludes its use in a crop situation, although fall treatments of Canada thistle in cereal stubble have given from 88 to 100% control (Hamill, 1975; Marriage, 1978). Significantly less control has occasionally been observed (Keys, 1977), due to insufficient regrowth (and reduced spray interception) after harvest and before treatment.

Chlorsulfuron has shown the ability to control Canada thistle (Palm et al., 1980). Messersmith and Lym (1981) and Harrison (1981) have reported that chlorsulfuron provided 90% or more control of Canada thistle regrowth with no damage to wheat planted one year after application. Zimdahl (1981) reported that 70 g/ha of chlorsulfuron controlled 95% of Canada thistle regrowth in several experiments. O'Sullivan (1982) reported excellent topgrowth control during the year of treatment and excellent regrowth control one year after application of 50 g/ha chlorsulfuron. Canada thistle stand densities were reduced from 71 to 17 shoots/m^2 and shoot fresh weights were reduced from 402 to 32 g/m^2, one year after application. He suggested that Canada thistle regrowth was controlled by soil residues of chlorsulfuron rather than by downward phloem transport and accumulation of the herbicide in the roots. Alley (1981) concluded that rates of 200 g/ha or higher were required for 90% control of Canada thistle regrowth in Wyoming.

CHAPTER 2

CHLORSULFURON MOVEMENT IN SOIL

Abstract

The leaching characteristics of chlorsulfuron were studied in soil columns and on soil thin-layer chromatography (TLC) plates. Non-labeled chlorsulfuron was detected to the bottom of 22.5 cm soil columns, after leaching the soil to field capacity. ^{14}C-labeled chlorsulfuron was found in decreasing amounts with increasing depth in soil columns. Delays after herbicide application and before leaching with water had little effect on the degree of herbicide movement. ^{14}C-labeled chlorsulfuron moved as a concentrated front on TLC plates. The lack of adsorption and increased movement on soil TLC plates was attributed to the high herbicide:soil ratio used.

Introduction

Chlorsulfuron has a high potential for leaching in the soil (Palm et al., 1980; O'Sullivan, 1982; Nilsson, 1982; Shea, 1982). The potential for mobility appears to decrease as the herbicide ages in the soil (DuPont, 1980).

The objectives of these studies were to determine if a delay before leaching, or initial soil moisture content would influence the leaching and eventual immobilization of chlorsulfuron in the soil.

Materials and Methods

Leaching columns were constructed from 10 cm diameter poly-vinyl chloride (PVC) pipe. The pipe was cut into 2.5 cm rings, which were taped together to create 15 cm tall columns. Nylon screen (16 mesh) was glued on the bottom, and the columns were filled with Bozeman silt loam soil (pachic cryoboroll, fine, mixed, 1.3% OM, pH 7.6). Formulated chlorsulfuron (75% a.i.) was applied to the column surface using a moving belt sprayer at a rate equivalent to 18 g/ha. The soil contained 3.5% moisture at the time of treatment. The herbicide was applied in 117 l/ha of water at 2.4 Kg/cm^2 pressure. There were 2 replications of treated and control columns.

The columns were leached after 1 hour with enough water to bring the soil to field capacity (400 ml). Water was applied to the soil columns using a flat fan nozzle (8001E, Spray Systems Co., Wheaton, IL), at a rate of 100 ml/hour. After equilibrating overnight, the columns were separated and each 2.5 cm section of soil was transferred to 10 cm tall paper cups. Ten seeds of green foxtail [*Setaria viridis* (L.)] were planted per cup, and after 7 days the seedlings were thinned to 5 plants per cup. The plants were grown in the greenhouse at approximately 25°C day and 20°C night with 16 hour daylength. Natural light was supplemented with incandescent and fluorescent lighting. The pots were fertilized weekly with 20 ml of Peters 20-20-20 soluble fertilizer (W. R. Grace and Co., Allentown, PA). Sixteen days later, the plants were harvested, dried at 50°C for 3 days, and weighed.

In a second study, 22.5 cm columns were constructed as described above and treated with 9 g/ha chlorsulfuron. The columns were leached 1, 24, 96, and 168 hours after herbicide application, in order to determine if chlorsulfuron would become immobilized with increasing time. The soil contained 3.5% moisture when the herbicide was applied. There were 3 replications. Foxtail millet [*Setaria italica* (L.) Beauv. 'Golden German'] was used as the bioassay indicator species. The plants were grown as described above. Plant height measurements were taken after 21 days.

A third leaching study was carried out to determine if chlorsulfuron immobilization was affected by the soil moisture content during the period between herbicide application and leaching. Columns (22.5 cm) were constructed and treated with chlorsulfuron as described above. One-half of the columns was leached with water 4, 7, and 12 days after herbicide application. Water was applied to other columns to wet the soil to a depth of 1 cm, immediately after herbicide application. The columns were then leached after 4, 7, and 12 days. There were 3 replications. Foxtail millet was used as the bioassay plant, and plant heights were measured after 30 days.

There was extreme variability in the growth of bioassay plants in response to chlorsulfuron, so column leaching experiments using ^{14}C-phenyl labeled chlorsulfuron (4.01 μCi/mg specific activity) were established. The ^{14}C-labeled herbicide was dissolved in acetone and 0.1 μCi aliquots were withdrawn. Each aliquot was mixed thoroughly with 1 g of soil. The soil was spread uniformly on the surface of 5 cm diameter by 25 cm tall columns. The equivalent herbicide concentration was 16.6 μg per column.

Some of the columns were leached after 1, 7, and 12 days. Water was applied to other columns to wet the soil to a depth of 1 cm. These columns were leached after 7 and 12 days. After equilibrating overnight, the columns were separated and each 2.5 cm section of soil was dried at 30°C for 3 days and weighed. One g subsamples from each soil section were combusted in a biological material oxidizer (R. J. Harvey Instruments Co., Hillsdale, NJ) and the evolved $^{14}CO_2$ was trapped in 15.0 ml of scintillation fluid containing toluene: 2-methoxyethanol:ethanolamine (10:7:3 v/v/v) plus 5 g PPO and 0.5 g dimethyl-POPOP/L. A combustion efficiency curve was constructed (Appendix A). Radioactivity per sample was quantified by counting in a liquid scintillation counter (Packard Instruments Company, Inc., Downers Grove, IL). Background radiation levels were subtracted.

Soil thin-layer chromatography (TLC) was used for further studies in order to reduce the volume of soil contaminated with radioactivity. TLC plates were constructed using the soil described above, which was sieved to < 420 μ using a 35 mesh soil screen. The soil was mixed with distilled water (1:2 soil:water v/v) and the slurry was spread onto 20 cm by 20 cm glass plates by rolling a glass rod over masking tape along the plate edges. The resulting air-dried soil layer was 250 μ thick. Radio-labeled chlorsulfuron was dissolved in acetone and 0.02 μCi (3.4 μg chlorsulfuron) was spotted 2 cm from the bottom edge of the plate. Oxy-acetic ^{14}C-labeled 2,4-D amine (56.8 μCi/mg specific activity) (ICN Chemicals and Radioisotopes Div., Irvine, CA) was spotted on the plates as a reference compound. The plates were leached with ascending distilled water in closed chromatography tanks for 4 hours. After the plates had dried, X-ray film (XAR-OMAT, Eastman Kodak Co., Rochester, NY) was placed on the plate and both were wrapped in a closed folder. The film was exposed for 3 days, and was developed using Kodak GBX developer (Yamaguchi and Crafts, 1958). To quantify the movement of ^{14}C-labeled chlorsulfuron, soil from the leached plates was scraped in 2 cm increments. Each soil sample was oxidized and counted as described above.

TLC plates were spotted with herbicides and leached with water after 7, 14, and 21 days. Some of the plates were maintained at ambient relative humidity (28 ± 5%) and the others were held at 100% relative humidity before leaching. There were 2 replications.

Results and Discussion

Dry weight of plants grown in soil from all depths of the treated columns was reduced, when water was applied 1 hour after herbicide application (Figure 3).

In the control columns, plants grown in soil from the lower segments were slightly larger than other control plants, indicating leaching of nutrients from the upper levels. The plants grown in treated soil did not exhibit this effect. The dry weight of plants grown

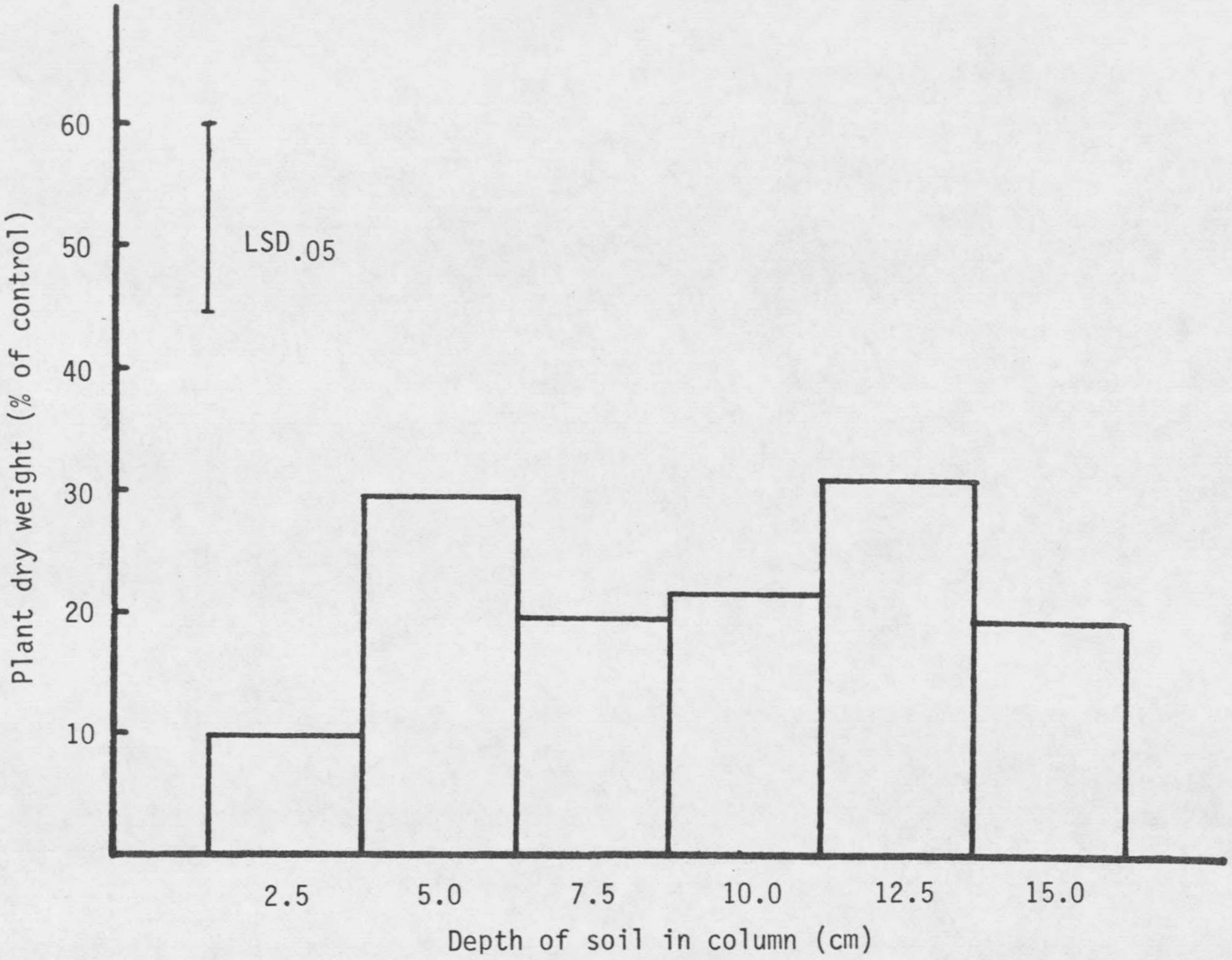

Figure 3. Dry weight (% of control) of green foxtail (*Setaria viridis*) plants grown in soil from 6 depths of chlorsulfuron-treated soil columns. The columns were leached to field capacity immediately after herbicide application.

in the upper 2.5 cm of soil from the treated columns was reduced more than from the other column depths. Therefore, there was more adsorption of chlorsulfuron in the upper 2.5 cm of the soil columns. However, sufficient amounts of chlorsulfuron were leached throughout the column to reduce plant growth at all soil depths. Chlorsulfuron did not appear to move as a concentrated front during leaching, since growth of the indicator plants was reduced similarly from all depths of treated soil.

In the second study, leaching was delayed for 1, 24, 96, or 168 hours after chlorsulfuron application (Figure 4). Plant heights from all treatments were reduced 80 to 85%, when grown in soil from the 2.5 cm column depth. When leaching was delayed for 1 hour, lower amounts of chlorsulfuron were detected at the 5.0, 7.5, and 20.0 cm soil depths. When leaching was delayed for 24 and 96 hours, a similar reduction in plant height was observed from the 2.5 to 12.5 cm depths. At depths of 20.0 and 22.5 cm, there was less reduction from the 96 hour than from the 24 hour delay. The 168 hour delay in leaching appeared to cause less leaching of chlorsulfuron into the 5.0 to 12.5 cm zones than any of the other treatments. At levels from 12.5 to 20.0 cm, plant height reductions were similar to the 24 and 96 hour delay treatments. There was less chlorsulfuron in the 22.5 cm zone from the 168 hour delay than from any other treatment.

In a similar study, leaching was delayed for 4, 7, and 12 days after chlorsulfuron application. The columns were either left at the initial moisture condition (Figure 5), or water was added to a depth of 1 cm immediately after chlorsulfuron application (Figure 6). When the columns were left dry after chlorsulfuron application, all treatments reduced plant heights 88 to 94% at the 2.5 cm depth. Reductions in plant height generally decreased in progressively deeper soil samples, indicating reduced amounts of chlorsulfuron. When leaching was delayed for 12 days, plant heights were reduced less at the 20.0 and 22.5 cm zones than from any other treatment.

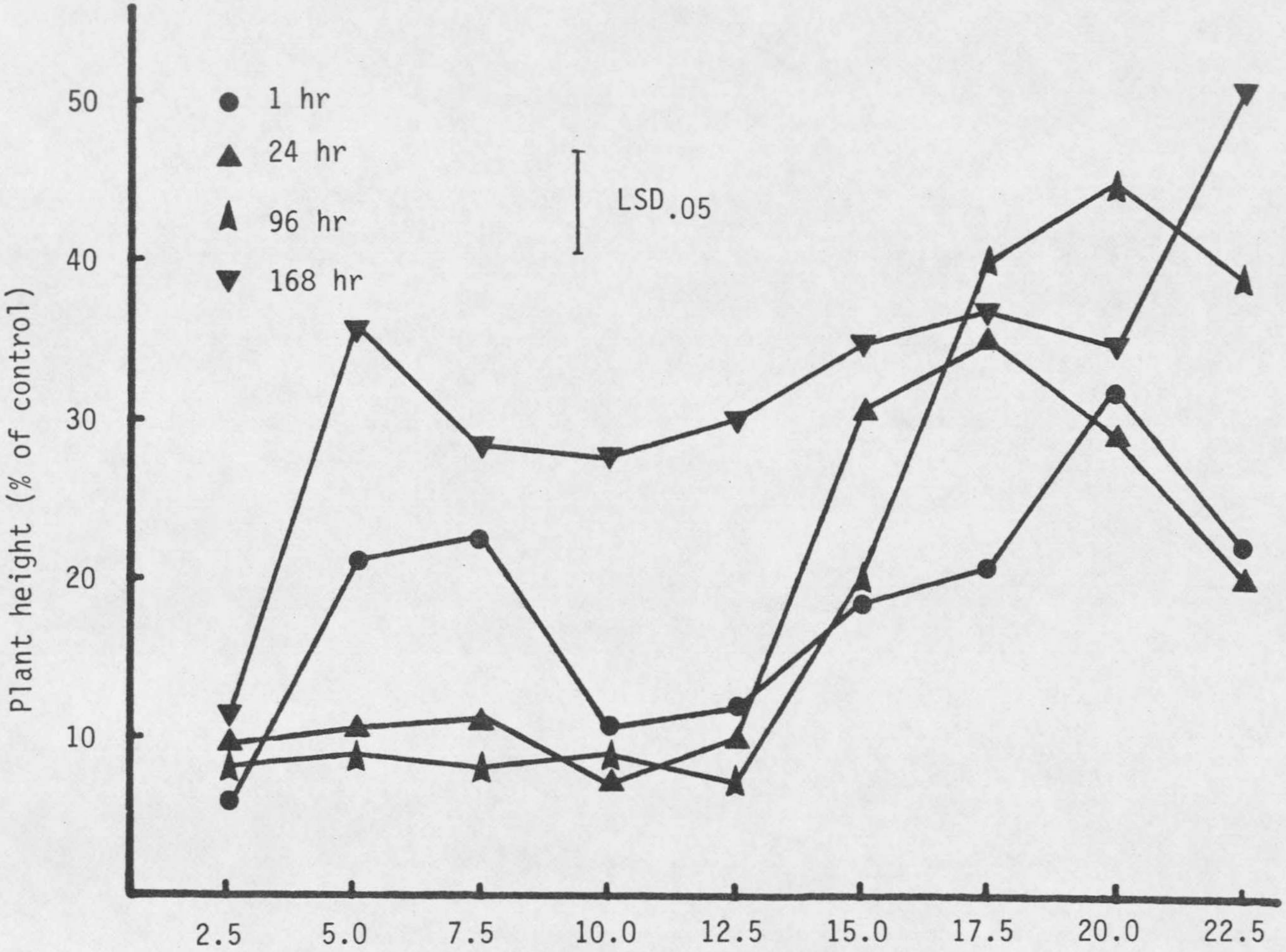

Figure 4. Plant height (% of control) of foxtail millet (*Setaria italica*) plants grown in soil from 9 depths of chlorsulfuron-treated soil columns. The columns were leached 1, 24, 96, or 168 hours after herbicide application.

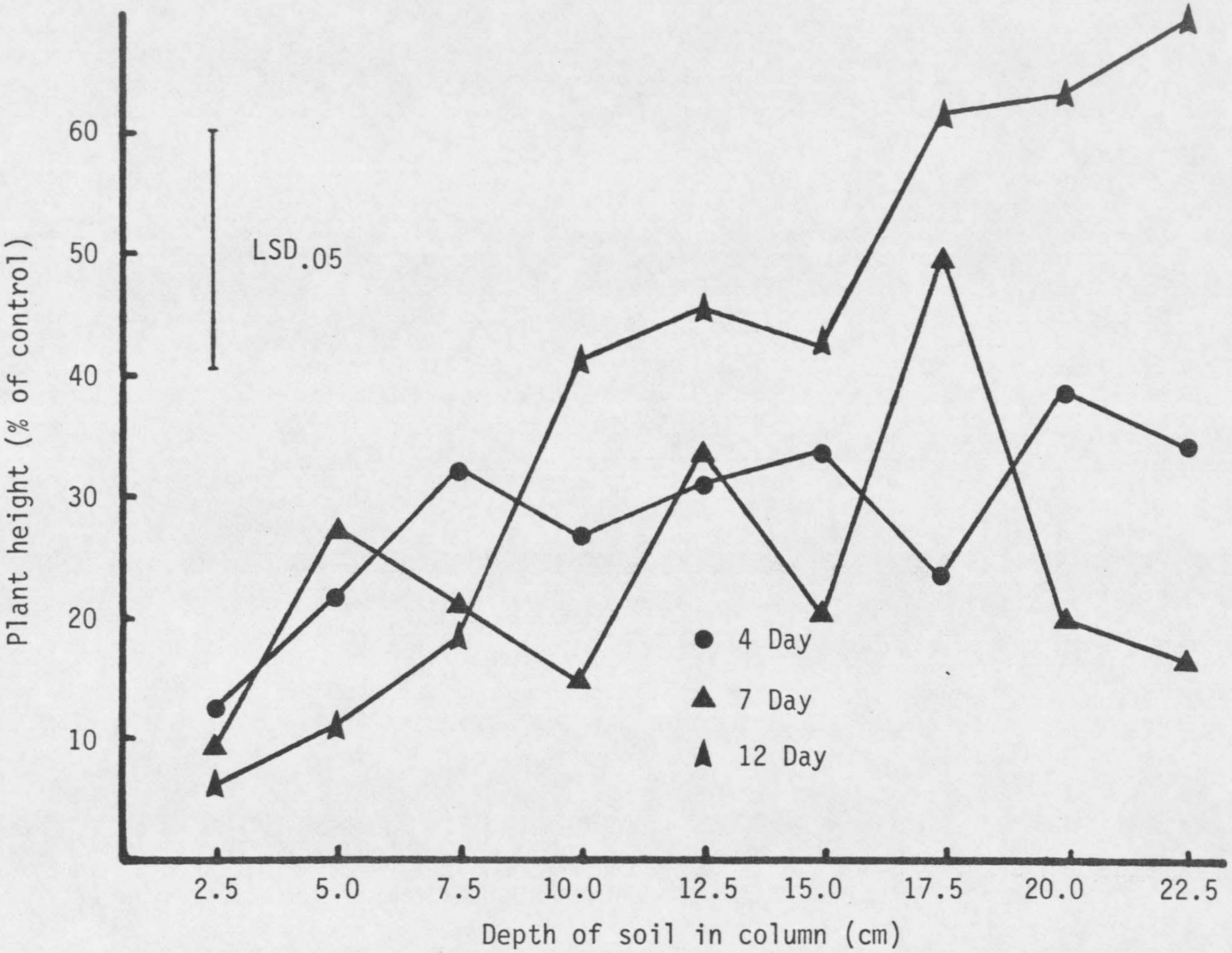

Figure 5. Plant height (% of control) of foxtail millet (*Setaria italica*) plants grown in soil from 9 depths of chlorsulfuron-treated soil columns. The columns were leached 4, 7, or 12 days after herbicide application.

The addition of a small volume of water prior to the total column leaching did not appear to alter the pattern of chlorsulfuron movement (Figure 6). All treatments resulted in a 93 to 95% reduction of plant height at the 2.5 cm soil depth. Different delays before leaching did not appreciably alter the pattern of movement.

The choice of foxtail millet as a bioassay indicator plant came as the result of many attempts to develop a consistent bioassay for chlorsulfuron soil residues (Dyer et al., 1982). Bioassays using 3 vascular plants (corn, barley, sunflower), algae (*Chlorella sorokiniana*), mammalian cells (BHK 21), virus (Mengovirus), and soil bacteria and fungi were not effective. Results from the corn root bioassay developed by the E. I. DuPont de Nemours and Co., Inc. (Hutchinson, 1980) were excessively variable when the Bozeman silt loam soil was used. The use of the corn root bioassay for different soils has since been discontinued by the DuPont Co. (Groves, personal communication). The foxtail millet bioassay gave a reasonable, linear response in plant height to chlorsulfuron concentrations from 0 to 4 ppb in the Bozeman silt loam soil (Appendix B). However, the same bioassay was not successful when used in a clay loam soil (4.9% OM, pH 7.5). The difficulties encountered in establishing a bioassay system and the high degree of variability of data in the 4 leaching studies suggest that the chlorsulfuron-soil-water equilibrium reaction is complex.

There were several general conclusions drawn from leaching studies conducted with non-labeled chlorsulfuron. There were relatively higher levels of chlorsulfuron at the surface of soil columns than at lower depths, indicating increased adsorption. Secondly, there appeared to be decreasing amounts of chlorsulfuron present in lower levels of the columns. Chlorsulfuron was mobile in the column system, since sufficient quantities were detected at all soil depths to significantly reduce the growth of bioassay plants. Delays before leaching did not appear to alter the pattern of movement of chlorsulfuron.

The leaching of ^{14}C-labeled chlorsulfuron in soil columns followed the same pattern as in the experiments described above. When leaching was carried out on the same day as

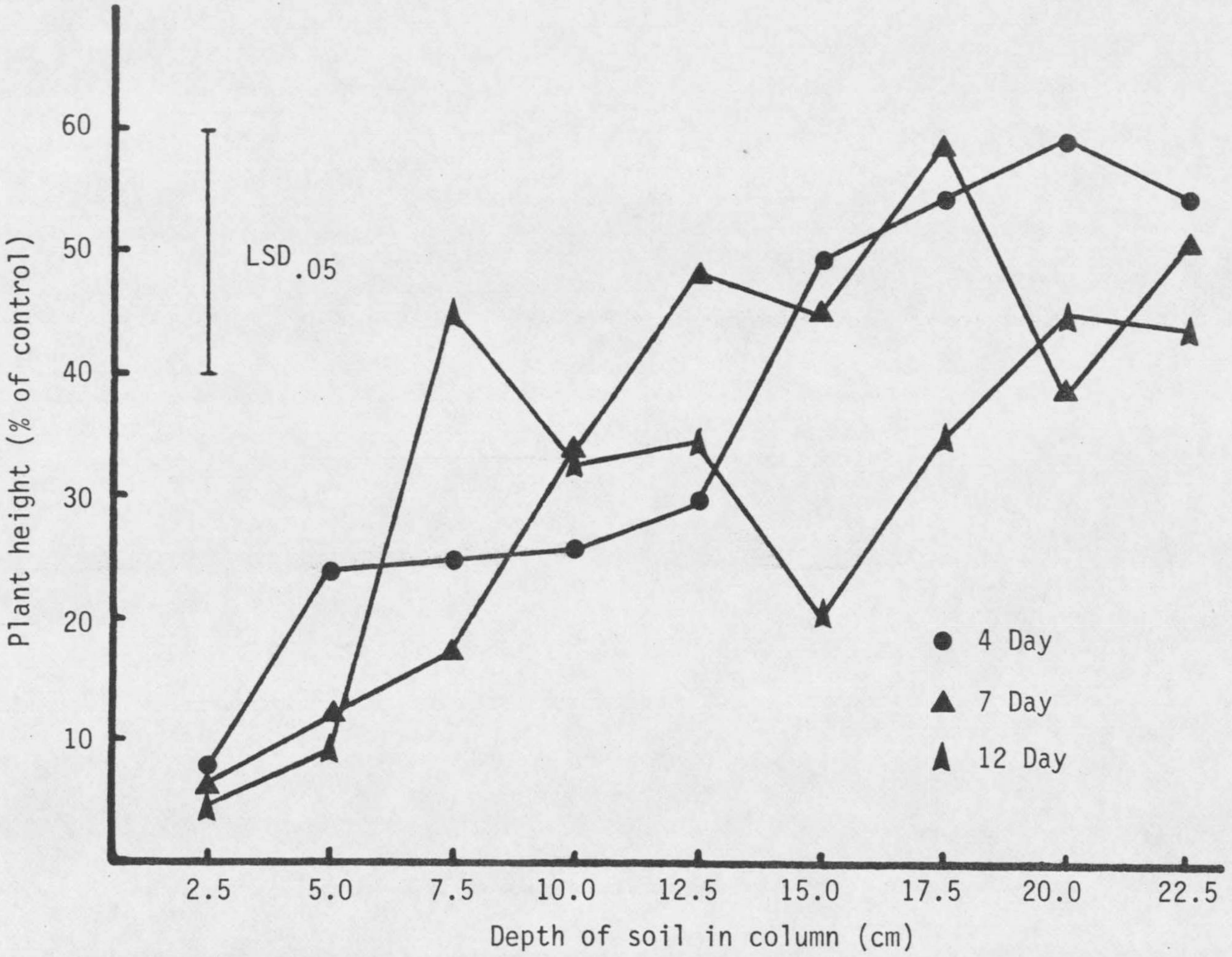

Figure 6. Plant height (% of control) of foxtail millet (*Setaria italica*) plants grown in soil from 9 depths of chlorsulfuron-treated soil columns. The columns were leached to a depth of 1.0 cm immediately after herbicide application, and were totally leached after 4, 7, or 12 days.

chlorsulfuron application, there was more radioactivity recovered in the 5.0 cm zone than when leaching was delayed for 7 or 12 days (Figure 7). Amounts of radioactivity in all other zones from all treatments were similar. The delay of 7 or 12 days before leaching did not alter the movement of labeled chlorsulfuron, below 2.5 cm. From 30 to 37% of the recovered radioactivity was found in the top 2.5 cm of the soil columns. Radioactivity levels decreased with increasing depth in the column. Very little of the recovered radioactivity was found below 15.0 cm.

When a small amount of water was added prior to total column leaching, 40 to 52% of the total applied radioactivity was recovered in the top 2.5 cm of the columns (Figure 8). More radioactivity was recovered in the 2.5 cm zone after a 7 day delay than a 12 day delay before leaching. As in the dry columns, radioactivity levels decreased with increasing depth in the column.

Direct quantification of ^{14}C-labeled chlorsulfuron movement was possible in these studies, due to oxidation and liquid scintillation counting of the samples. The resulting data provided confirmation of the trends observed in the bioassay trials. The same pattern of movement was found in the labeled as in the non-labeled chlorsulfuron studies. Thirty to 50% of the recovered radioactivity was found at the soil surface, and decreasing amounts were found with increasing column depth. There was no apparent increase in adsorption of chlorsulfuron with increasing delays before leaching. The expected decrease in mobility from aged herbicide residues did not occur probably due to restricted adsorption on the dry soil surface of the columns.

Labeled chlorsulfuron moved as a concentrated front on soil TLC plates. Autoradiographs of leached plates showed that almost all of the herbicide was present at the water front, with a slight degree of tailing. More than 80% of the recovered radioactivity was found in the zone 2 to 4 cm behind the water front, when plates were kept dry for 7 days before leaching (Figure 9). Movement on plates kept at 100% relative humidity for 7 days

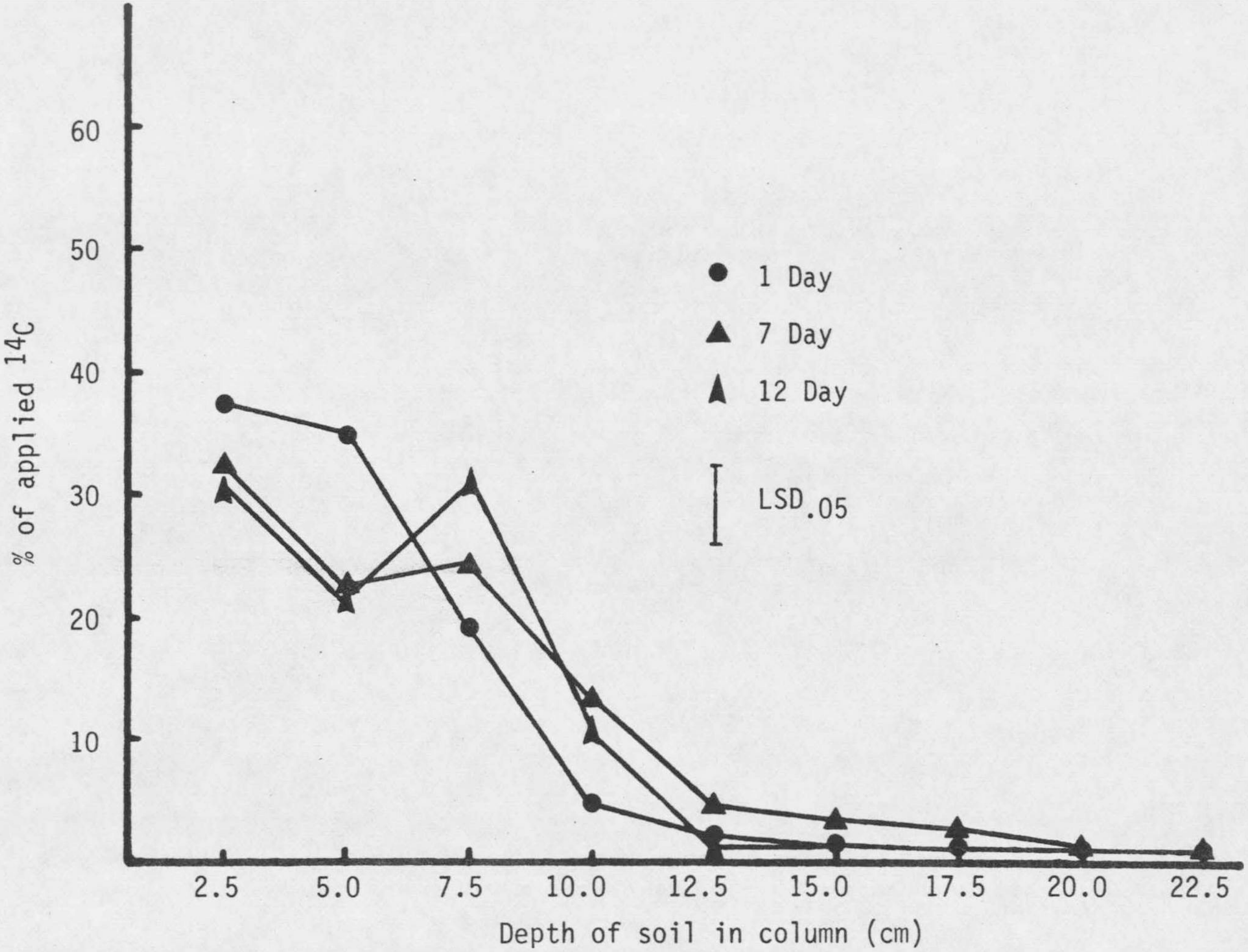

Figure 7. Percent of applied ^{14}C-labeled chlorsulfuron in 9 depths of soil columns that were leached 1, 7, or 12 days after herbicide application.

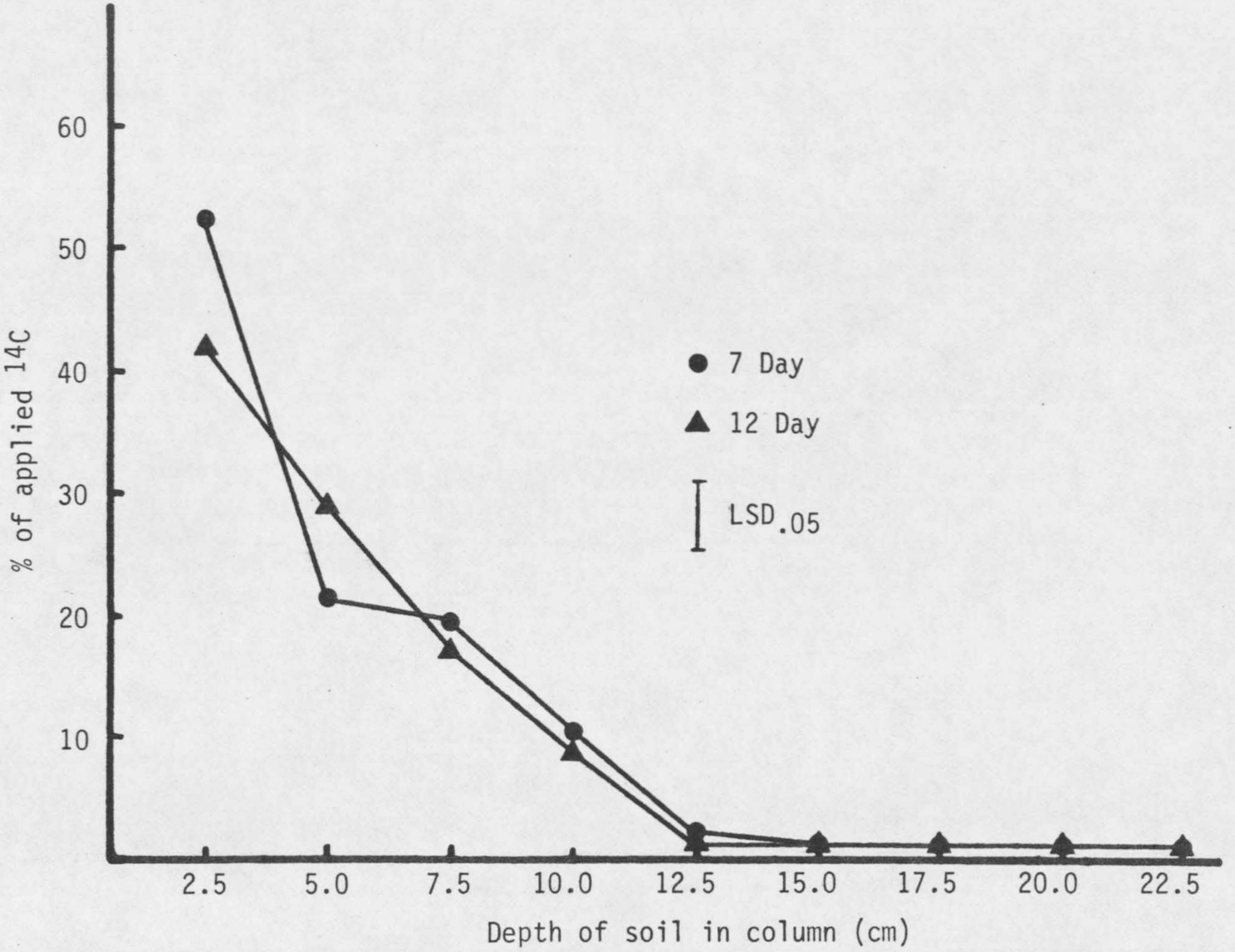

Figure 8. Percent of applied ^{14}C-labeled chlorsulfuron in 9 depths of soil columns that were leached to a depth of 1.0 cm immediately after herbicide application, and were totally leached after 7 or 12 days.

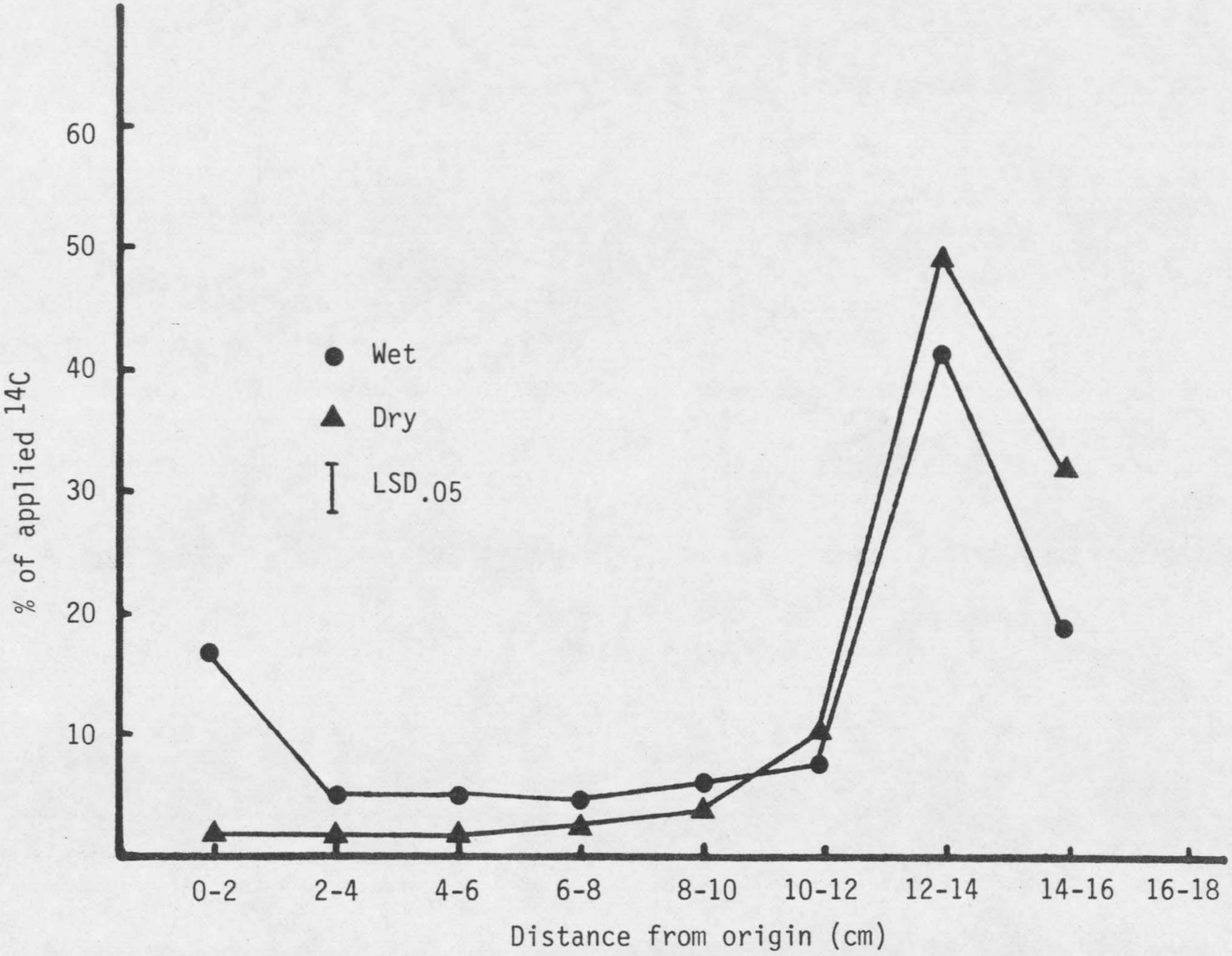

Figure 9. Percent of applied ^{14}C-labeled chlorsulfuron in 2.0 cm segments of soil from soil TLC plates. The plates were maintained at 100% (wet) or 28 ± 5% (dry) relative humidity for 7 days prior to leaching.

before leaching was similar. However, 17 times more radioactivity remained at the origin on moist plates than on dry plates. This increased amount of radioactivity near the origin indicates that chlorsulfuron diffused from the origin and was immobilized during the moist period. Autoradiographs supported this conclusion.

When leaching was delayed for 14 days, the pattern of movement was similar (Figure 10). The majority of radioactivity was found in the 12 to 16 cm zone on both moist and dry plates. Immobilization again occurred near the origin on plates which were kept at 100% relative humidity prior to leaching.

When there was a 21-day delay between chlorsulfuron application and leaching, the pattern of movement remained the same (Figure 11). Immobilization occurred at the origin on plates stored under moist conditions, and there also appeared to be more radioactivity present at all depths from 2 to 14 cm on the moist plates, although differences were not significant. The amount of radioactivity in the 14 to 16 cm zone was much less on the moist plates, due to the increased adsorption of chlorsulfuron at levels closer to the origin.

The disparity between results from the soil column and soil TLC systems is of interest since the soil type, temperature, and water flow rate were the same in both systems. However, a major difference existed in that the herbicide:soil ratios were not comparable. In the soil column system, 0.1 μCi of radioactive chlorsulfuron was applied to each column. This amount was chosen to provide a sufficient level of radioactivity per column section for counting purposes. This amount of radioactivity is equivalent to 16.6 μg chlorsulfuron per column, which was initially mixed with 1 g of soil, when it was applied to the column surface. In a field application of 18 g/ha of chlorsulfuron, the herbicide:soil concentration on the soil surface would be approximately 20 μg chlorsulfuron per g of soil (.86 Kg of soil = 1 m^2 surface area). Therefore, the ratio used in the column studies closely approximated an actual field situation.

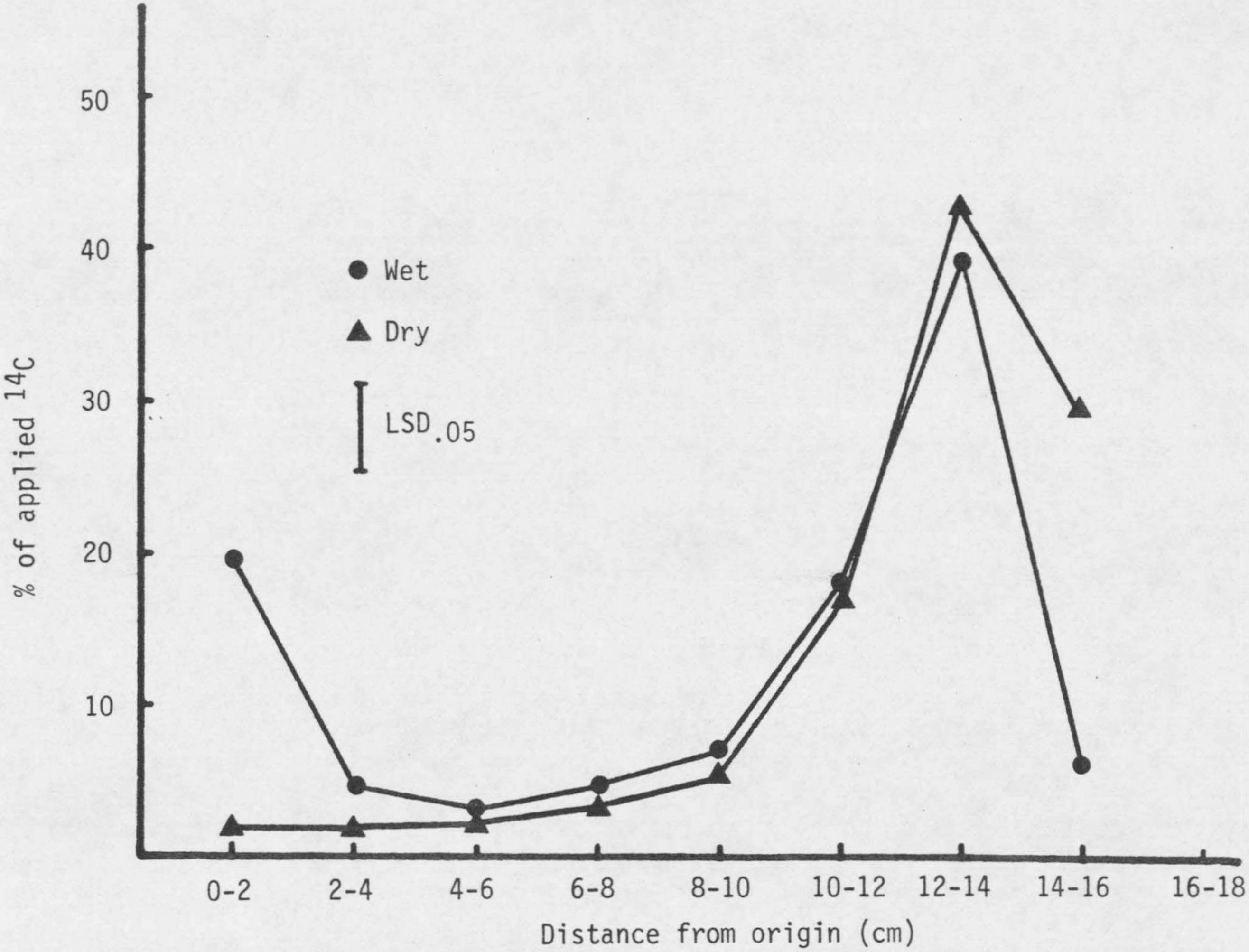

Figure 10. Percent of applied ^{14}C-labeled chlorsulfuron in 2.0 cm segments of soil from soil TLC plates. The plates were maintained at 100% (wet) or 25 ± 5% (dry) relative humidity for 14 days prior to leaching.

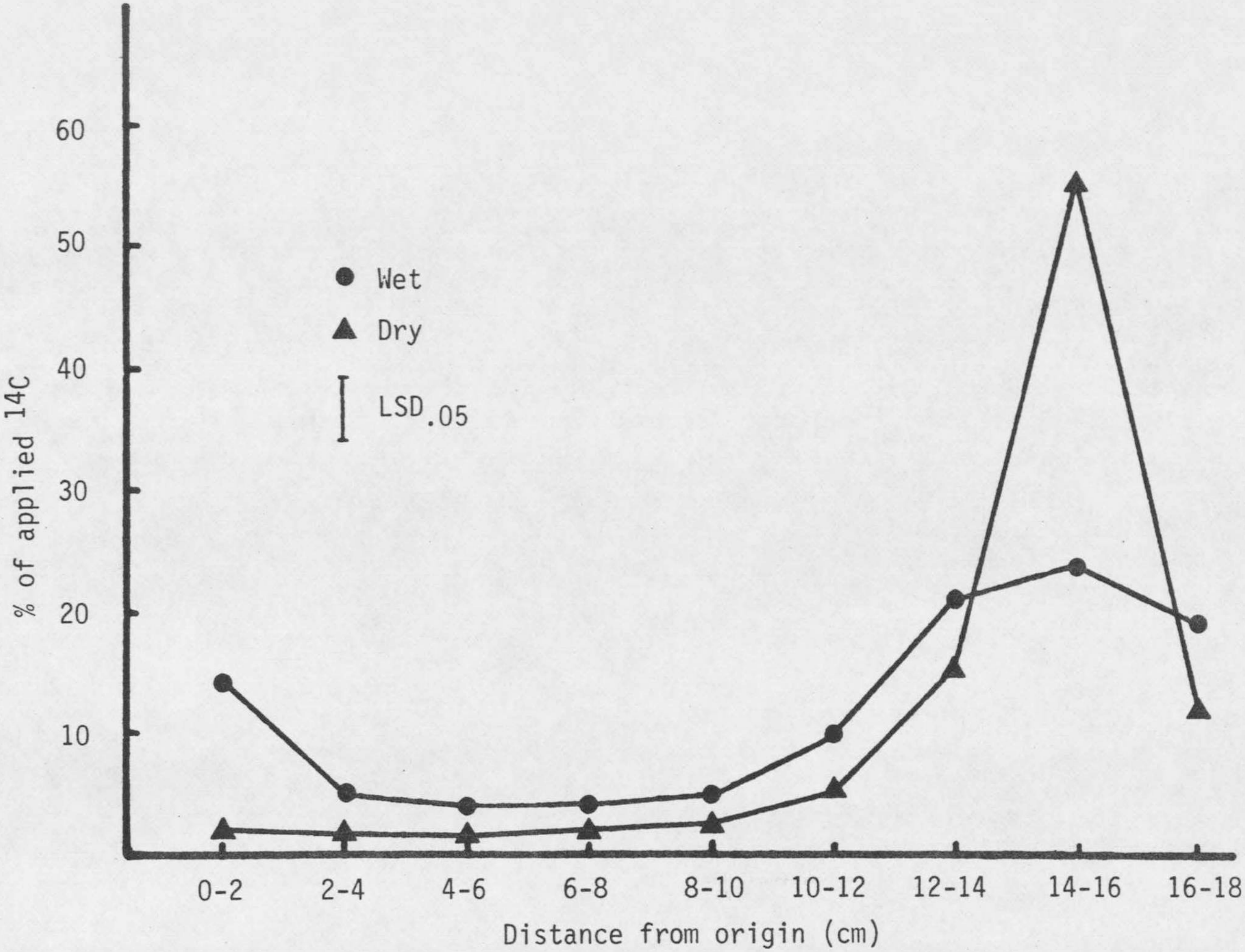

Figure 11. Percent of applied ^{14}C-labeled chlorsulfuron in 2.0 cm segments of soil from soil TLC plates. The plates were maintained at 100% (wet) or 28 ± 5% (dry) relative humidity for 21 days prior to leaching.

For the soil TLC system, 0.02 μCi of radioactive chlorsulfuron was applied to each plate, according to the procedure described by Helling (1971). This amount was determined to be sufficient to expose X-ray film after a reasonable time period (unpublished results). The initial ratio of chlorsulfuron to soil at the origin, based on this amount of radioactivity, was 330 μg chlorsulfuron per g of soil. This ratio of herbicide:soil is approximately 20 times the ratio used in the soil column studies.

It is possible that the amount of labeled chlorsulfuron applied in the soil TLC studies dominated the soil adsorptive sites at the point of application. While some adsorption was observed when the chlorsulfuron diffused away from the origin under moist conditions, most of the chlorsulfuron could not be adsorbed because of adsorption site saturation. When leaching began, the majority of chlorsulfuron was freely mobile and moved as a front with the water flow.

Movement on soil TLC plates cannot be directly compared to field or soil column experiments (Helling, 1970). Sheets (1964) reported that the speed of leaching of monuron was positively correlated to herbicide concentration. The soil TLC technique used did not reflect the behavior of chlorsulfuron in an actual field situation.

The degree of leaching of chlorsulfuron in field soils would be positively correlated with the soils' water holding capacity. Percolating water from rainfall or irrigation would carry chlorsulfuron into the soil, probably as far as the water itself moved. As the chlorsulfuron was leached, it would become more diluted and would have greater opportunity to become adsorbed, once water movement stopped. If excessively large amounts of water were applied, chlorsulfuron would probably continue to move through the soil profile. Since chlorsulfuron is applied at rates of 9 to 18 g/ha, deeper leaching in the soil would soon reduce chlorsulfuron residues to non-detectable levels. The potential for detectable levels of chlorsulfuron to be leached into the water table would be severely restricted.

CHAPTER 3

THE EFFECT OF CHLORSULFURON ON ROTATIONAL CROPS GROWN IN MONTANA

Abstract

Chlorsulfuron was applied at 35, 70, and 140 g/ha in September, 1980 to plots at Bozeman and Great Falls, Montana. Eleven crops were planted into the subsequent soil residues of chlorsulfuron. Weed control and crop injury ratings were taken for 3 successive years. Dry weight was reduced for all crops tested except wheat, barley, and oats (*Avena sativa*) by all application rates of chlorsulfuron. The rate of degradation of chlorsulfuron in Montana soils was consistent with the reported half-life of 60 days.

Introduction

Chlorsulfuron is labeled for use in Montana on wheat (including durum) and barley. It may be applied pre emergence or post emergence to the crop, however, the recommended use rates depend upon soil pH. Chlorsulfuron may be applied at rates up to 26 g/ha on soils with a pH below 6.5. On soils with a pH between 6.6 and 7.5, chlorsulfuron may be applied at rates up to 18 g/ha. Applications are not permitted on soils above pH 7.5.

The breakdown of chlorsulfuron in soil is accomplished primarily through chemical hydrolysis (Palm et al., 1980). The rate of hydrolysis is increased by higher soil moisture, temperature, and organic matter, and by decreased soil pH. Hydrolysis is slower in more alkaline soils. The half-life of chlorsulfuron is reported to be 4 to 6 weeks under normal summer field conditions. Chlorsulfuron residues might be expected to be more persistent in Montana since many of the agricultural soils in the state are alkaline (above pH 7.3) and annual precipitation levels are low (under 40 cm).

While wheat and barley are the predominant crops in Montana, other rotational crops such as oats, alfalfa, safflower (*Carthamus tinctorius* L.), sunflower, sugar beet, lentil, potato, and dry bean are also grown. All of these rotational crops except oats appear to be sensitive to residues of chlorsulfuron (Walker and Brown, 1982). Because of this potential for injury to rotational crops, field experiments were carried out to determine the tolerance of each crop to chlorsulfuron residues in the soil.

Materials and Methods

A field trial was initiated in May, 1980 to determine the effect of rate and time of application of chlorsulfuron on the yield of weed-free spring wheat. 'Pondera' spring wheat was seeded on May 23, 1980 and chlorsulfuron was applied at 0, 70, and 140 g/ha using a CO_2-pressurized backpack sprayer in 98 l/ha of water. The 2.1 by 7.6 m plots were arranged in a randomized complete block design with 3 replications. The treatments were applied approximately once a week, corresponding to the 4 leaf stage through the soft dough stage of the wheat. Crop yields were measured on September 1, 1980 using a small plot combine.

A similar trial was carried out on winter wheat to determine the effect of rate and time of application of chlorsulfuron on crop yield. Chlorsulfuron was applied at 9, 28, and 53 g/ha approximately once a week to weed-free 'Redwin' winter wheat. The treatments were applied using a CO_2-pressurized backpack sprayer in 140 l/ha of water to 2.1 by 7.6 m plots, in a randomized complete block design. There were 3 replications. Applications were made from pre emergence through the 6 leaf stage of the crop. Crop yields were harvested using a small plot combine. The experiment was repeated in 1982.

Two plot experiments were initiated in the fall of 1980 to determine the effects of chlorsulfuron soil residues on subsequent crops. Chlorsulfuron was applied at 35, 70, and 140 g/ha to 3.3 by 18.3 m plots in a randomized complete block design. The treatments were applied using a CO_2-pressurized backpack sprayer in 102 l/ha of water on September 10 and 20, 1980, to wheat stubble at Great Falls and Bozeman, respectively. The soil type at the Bozeman location was a silt loam (argic pachic cryoboroll, fine, mixed, mesic). At Great Falls, the soil was a Lawther silty clay (vertic haploboroll, fine, montmorillonitic). There were 4 replications at Bozeman and 3 replications at Great Falls. Soil characteristics for both locations are given in Table 1.

Table 1. Soil Characteristics from Plot Locations at Bozeman and Great Falls, Montana.

Location	Organic Matter (%)	pH	CEC meq/100 g
Bozeman	1.3	8.1	31.1
Great Falls	2.0	7.9	43.9

The plots were disked in the fall and spring at both locations. The plot area at Bozeman was cultivated using a 'Triple-K' cultivator (Kongskilde Co., Exeter, Ontario, Canada) on April 28, 1981. 'Pondera' spring wheat was seeded on May 15, 1981 with a double disk drill. The number of wheat plants/m of row and plant height measurements were taken on July 2, 1981. The number of tillers/3 m of row and seeds/head counts were taken on August 10, 1981. Crop yields were harvested on September 22, 1981 using a small plot combine. The plots at Great Falls were left fallow in 1981 and weed control ratings were taken on May 14 and July 30, 1981.

The experimental areas at both locations were disked in the fall of 1981 and in May of 1982. The plot area was cultivated with a 'Triple-K' cultivator on May 1, 1982 at the Bozeman location and the following crops were seeded on May 7, 1982: 'Piroline' barley, 'Pondera' spring wheat, 'UI 111' pinto beans, and 'Ladak' alfalfa. Spring wheat and barley were seeded with and without a seed treatment of 0.5% w/w 1,8 napthalic anhydride (NA). Visual crop injury ratings (0 = no injury; 100 = complete kill) were taken on June 28 and July 30, 1982. Ten plants/species/plot of all crops except wheat and barley were harvested by hand on August 20, 1982. The plant material was dried at 30°C for 6 days and weighed. The wheat and barley were harvested on September 7, 1982 using a small plot combine. At the Great Falls location, the plots were disked twice on May 21, 1982 and the crops listed above and 'Betaseed 1443' sugar beets were seeded the same day. Alfalfa was not planted at Great Falls. Ten plants/species/plot were harvested by hand on August 16, 1982. The plant material was dried at 30°C for 5 days and weighed. The small grain crops were harvested with a small plot combine.

The plots at both locations were disked in the fall, 1982 and again in May, 1983. The following crops were planted on May 23, 1983 at Bozeman and on May 29, 1983 at Great Falls: 'Marberg' spring wheat, 'Piroline' barley, 'Chilian' lentils, 'Betaseed 1443' sugar beets, 'Hartman' safflower, 'Culbert' flax (*Linum usitatissum*), 'Viva' pink beans, 'Ackerperle' faba beans (*Phaseolus vulgaris*), 'UC-5' garbanzo beans (*Cicer arietinum*), 'IS 984' sunflower, 'Ladak' alfalfa, 'NK 199' corn, and 'Otana' oats. Potatoes were planted at the Bozeman location. Crop injury ratings were taken on July 12 and August 18, 1983 at Bozeman and on August 31, 1983 at Great Falls. All crops were harvested at Bozeman on August 25, 1983 and on August 31, 1983 at Great Falls. Plant material from all crops except wheat, barley, and oats was dried at 30°C for 7 days and weighed. The small grain crops at both locations were not harvested in 1983 due to bird damage at Great Falls and hail damage at Bozeman.

Results and Discussion

Spring wheat yields were not affected by post emergence treatments of chlorsulfuron at any rate or time of application (Table 2). The lack of reduction in yield indicates that chlorsulfuron can be safely applied to spring wheat at rates up to 140 g/ha during a relatively long period in the spring. The herbicides presently in common use in small grains have narrow application timings. The wider latitude of application timing with the use of chlorsulfuron will be a valuable advantage to producers, who are rarely able to apply herbicides at an exact time, due to weather or schedule conflicts.

Table 2. Spring Wheat Yields After Application of Chlorsulfuron at 7 Wheat Growth Stages in the Spring of 1981.*

Wheat Growth Stage at Application		Yield Kg/ha
4 leaf		4109
2-3 tillers		4136
3-4 tillers		4140
jointing		4149
boot		4146
headed		4157
soft dough		4149
control		4136
	CV	.57
	LSD .05	54.7

*Yields are means of plots treated with 2 rates (70 and 140 g/ha).

Plant heights were visually observed on June 9, 1981. The height of plants treated on the second date of application was reduced by 7 cm. Rain (3.2 cm) occurred within 3 days after the second treatments were applied. The stunting of the wheat plants may have been the result of leaching of chlorsulfuron into the crop root zone. Wheat plants may not be able to metabolize chlorsulfuron effectively when a large amount is absorbed through the roots. The stunting injury was not reflected in reduced crop yield.

Similarly, winter wheat yields were not affected by any rate or time of application of chlorsulfuron tested (Table 3). The first 3 treatments were applied to the crop in the fall, prior to the onset of winter dormancy. The ability to apply chlorsulfuron in the fall to winter wheat would be particularly advantageous to farmers in Montana. Chlorsulfuron's residual nature would permit extended control of winter annual and spring weeds. Present weed control practices of applying herbicides to winter wheat in the spring often interfere with other cultural practices, with the result that herbicide applications are often delayed. Under these conditions, winter annual weeds are often too large in the spring to be successfully controlled. Fall applications of chlorsulfuron to winter wheat would circumvent these problems.

Table 3. Winter Wheat Yields After Application of Chlorsulfuron at 6 Growth Stages.*

Crop Stage at Time of Application		Yield Kg/ha
pre emergence		2878
1-1.5 leaf		2704
1.5-2 leaf		2926
2-3 leaf		2811
4 leaf		3120
5-6 leaf		3060
control		2812
	CV	12.4
	LSD .05	513.9

*Crop yields are means of plots treated with 3 rates of application (35, 70, and 140 g/ha).

Spring wheat was injured by soil residues from 70 and 140 g/ha chlorsulfuron, applied 10 months previously (Table 4). Tillering, plant height, and number of seeds/head were reduced in comparison to control plants. Plant counts/m of row were not affected by any rate of chlorsulfuron tested, but all other measurements reflected crop injury from chlorsulfuron applications of 70 and 140 g/ha. These rates are 4 and 8 times greater than the highest recommended use rate in Montana. Spring wheat injury was not observed from soil

Table 4. Spring Wheat Plants/m of Row, Plant Height, Tillers/3 m of Row, Seeds/Head, and Yields 10 Months After Application of 3 Rates of Chlorsulfuron.

Rate g/ha	Plants/m of Row	Plant Height (cm)	Tillers/3 m of Row	Seeds/ Head	Yield g/m of Row
35	29.8	18.0	36.3	34.3	309.2
70	31.3	16.5	31.3	32.5	265.3
140	32.3	15.8	27.3	25.0	183.7
control	32.3	18.8	40.5	37.3	406.6
CV	6.4	4.7	13.8	5.9	18.3
LSD .05	3.2	1.3	7.5	3.1	98.6

residues of 35 g/ha of chlorsulfuron, therefore spring wheat can safely be seeded the year after application of recommended use rates of chlorsulfuron.

Weed control ratings taken at the Great Falls location reflected the presence of chlorsulfuron residues 7 and 9 months after application (Table 5). Jointed goatgrass (*Triticum cylindrica*), which is genetically similar to wheat, was not greatly affected by chlorsulfuron soil residues. Control of downy bromegrass (*Bromus tectorum*) was noted on May 14 but control was not observed on the second date of rating. Annual sowthistle (*Sonchus oleraceus*) and Canada thistle were effectively controlled by the two highest rates of application.

On June 28, 1982, crop injury was evident on all crops planted at Bozeman, especially sugar beets and corn (Table 6). Canada thistle was also controlled at rates of 70 and 140 g/ha.

Crop injury was more pronounced by July 30, 1982 (Table 7). All crops except wheat and barley were showing severe stunting and chlorosis, especially in the plots treated with 70 and 140 g/ha of chlorsulfuron. Nearly complete weed control was observed at the two highest rates of application, however, Canada thistle plants were beginning to reinfest the plots treated with 35 g/ha of chlorsulfuron.

Table 5. Percent Control of 4 Weed Species 8 and 10 Months After Application of 3 Rates of Chlorsulfuron at Great Falls.*

	May 14, 1981		July 30, 1981		
Rate g/ha	Downy Bromegrass	Jointed Goatgrass	Annual Sowthistle	Canada Thistle	Jointed Goatgrass
	Weed control (%)				
35	45	5	72	62	8
70	62	13	96	77	15
140	72	20	99	98	32
CV	6.7	45.2	9.2	4.3	41.5
LSD .05	6.0	8.6	12.3	5.0	11.4

*0 = no control; 100 = complete control.

Table 6. Percent Crop Injury of 7 Crops and Percent Control of Canada Thistle on June 28, 1982 from Residues of Chlorsulfuron Applied on September 20, 1980 at Bozeman.

Rate g/ha	Spring Wheat	Barley	Sunflower	Safflower	Sugar Beet	Pinto Bean	Corn	Canada Thistle
	Crop injury (%)							
35	0	0	7	14	24	5	19	20
70	8	14	19	19	72	16	47	73
140	8	15	29	26	88	21	70	99
CV	41.1	31.0	18.6	19.9	9.5	18.2	13.3	9.1
LSD .05	2.5	3.6	4.1	4.0	7.0	6.8	7.2	6.8

Table 7. Percent Crop Injury of 7 Crops on July 30, 1982 from Residues of Chlorsulfuron Applied on September 20, 1980 at Bozeman.

Rate g/ha	Spring Wheat	Barley	Sunflower	Safflower	Sugar Beet	Pinto Bean	Corn
	Crop injury (%)						
35	0	0	10	2	40	7	17
70	5	10	85	36	98	30	46
140	10	26	97	59	100	73	63
CV	56.7	35.8	11.8	13.2	6.1	16.4	11.3
LSD .05	3.5	5.3	9.1	5.2	5.8	7.2	5.7

Twenty-one months after herbicide application, dry weights of sunflower, safflower, bean, beet, and corn were reduced by all rates of chlorsulfuron (Table 8). All crops except wheat and barley were sufficiently injured 2 years after application of 35 g/ha of chlorsulfuron to suggest that chlorsulfuron should not be used in rotation systems involving these crops.

Table 8. Dry Weight (g/10 plants/plot) of 5 Crops on August 20, 1982 Planted in Soil Residues of Chlorsulfuron Applied on September 20, 1980 at Bozeman.

Rate g/ha	Sunflower	Safflower	Sugar Beet	Pinto Bean	Corn
	------------------ Plant dry weight (g/10 plants) ----------------				
35	59.4	124.7	6.6	34.5	47.4
70	42.1	106.6	.4	24.0	38.0
140	9.1	45.0	.4	15.1	31.0
control	791.7	414.1	36.5	71.8	255.1
CV	54.0	23.0	55.0	29.7	30.3
LSD .05	194.7	63.6	9.6	17.2	45.0

One, 8 napthalic anhydride (NA) has been used to protect crops from herbicide residues in soil (Stephenson and Chang, 1978). The compound is effective when applied as a seed treatment to crops which are marginally tolerant to a herbicide present in the soil at the time of planting. NA has been very effective in reducing injury to corn from soil residues of EPTC (S-ethyl dipropylthiocarbamate) (Burnside et al., 1971). Parker et al. (1980) reported that corn was protected from soil residues of chlorsulfuron by seed treatment with NA. NA was tested as a seed treatment for barley to determine if it would ameliorate crop injury from chlorsulfuron soil residues. Barley yields from untreated seed were reduced by the highest rate of chlorsulfuron tested, while yields from treated seed were not reduced (Table 9).

Spring wheat yields were not affected by the NA seed treatment, and were not reduced by any rate of chlorsulfuron tested. Both small grain crops appeared to possess

Table 9. Wheat and Barley Yields (Kg/ha) With and Without Seed Treatment of 0.5% w/w 1,8 Napthalic Anhydride (NA) on August 7, 1982.*

Rate g/ha	Wheat	Wheat +NA	Barley	Barley +NA
	Crop yields (Kg/ha)			
35	1949	1700	1548	1505
70	1881	1714	1156	1602
140	1713	1546	957	1209
control	1667	1868	1468	1317
CV	19.8	14.4	21.2	20.2
LSD .05	571.2	389.8	435.6	451.6

*The crops were planted into residues from chlorsulfuron applications made on September 20, 1980 at Bozeman.

sufficient tolerance to applications of 35 g/ha of chlorsulfuron under conditions at Bozeman to permit seeding 12 months after application of chlorsulfuron. Barley was less tolerant to soil residues of chlorsulfuron than wheat. NA showed some potential in protecting barley against injury from soil residues of chlorsulfuron.

Crop injury ratings were taken at Great Falls on August 2, 1982. Severe injury was observed on all crops except wheat (Table 10). Barley was injured by the 140 g/ha rate of chlorsulfuron. Ratings of field pennycress (*Thlaspi arvense*) and annual sowthistle control were also taken on the same date (Appendix C). Weed control was continuing in all plots treated with 70 or 140 g/ha of chlorsulfuron. At the rate of 35 g/ha, weed control was erratic, but sufficient chlorsulfuron residue was present to injure all crops except wheat and barley.

Spring wheat and barley yields in 1982 were not reduced by any rate of chlorsulfuron tested (Table 11). Dry weights of the other crops tested were reduced by all rates of chlorsulfuron.

Crop injury ratings were taken at the Bozeman location on July 12, 1983 (Table 12). Lentils, sugar beets, alfalfa, and corn were injured by all levels of chlorsulfuron soil residues. Potatoes, garbanzo beans, and faba beans were injured by soil residues from the 70

Table 10. Percent Injury of 7 Crops on August 2, 1982 from Soil Residues of Chlorsulfuron Applied on September 10, 1980 at Great Falls.

Rate g/ha	Wheat	Barley	Sunflower	Safflower	Sugar Beet	Pinto Bean	Corn
	-----------------------			Crop injury (%)			-----------------------
35	3	0	57	10	53	25	53
70	10	3	98	47	100	27	80
140	13	40	100	85	100	80	99
CV	69.6	53.3	5.4	14.9	4.6	15.8	9.6
LSD .05	9.3	11.5	6.9	10.6	5.8	10.4	11.2

Table 11. Dry Weight (g/10 plants/plot) of 5 Crops Harvested on August 30, 1982 and Yield (Kg/ha) of Wheat and Barley.*

	(Kg/ha)		(g/10 plants/plot)				
Rate g/ha	Wheat	Barley	Sunflower	Safflower	Sugar Beet	Pinto Bean	Corn
35	1727	1236	209.3	54.8	7.7	21.0	20.4
70	1882	1290	20.4	24.9	.0	9.9	6.1
140	1929	1236	8.7	11.9	.0	5.5	3.3
control	1613	1360	852.7	67.0	52.2	27.4	52.0
CV	17.9	20.8	13.3	11.2	48.0	33.8	45.4
LSD .05	641.1	532.8	72.3	8.9	14.4	10.8	18.6

*The crops were planted in soil residues of chlorsulfuron applied on September 10, 1980 at Great Falls.

Table 12. Percent Crop Injury of 11 Crops on July 12, 1983 from Soil Residues of Chlorsulfuron Applied September 20, 1980 at Bozeman.

Rate g/ha	Potato	Lentil	Sugar Beet	Safflower	Flax	Pinto Bean	Faba Bean	Garbanzo Bean	Sunflower	Alfalfa	Corn
	-----------------------					Crop injury (%)					-----------------------
35	10	79	59	4	2	3	6	11	5	16	31
70	31	93	55	8	3	6	36	67	7	40	47
140	67	94	74	45	44	7	61	76	20	92	60
CV	27.1	9.2	29.2	24.1	82.8	81.9	43.6	27.4	78.8	25.9	23.6
LSD .05	11.7	9.8	21.9	17.1	16.3	5.1	18.1	17.0	10.2	15.4	13.1

and 140 g/ha chlorsulfuron treatments. Safflower, flax and sunflower were injured by 140 g/ha chlorsulfuron. Spring wheat and barley were not injured by any rate of chlorsulfuron (Appendix D).

By August 18, 1983, injury had become more apparent on all crops except the small grains (Table 13). Lentils, alfalfa and corn were severely injured by all levels of chlorsulfuron soil residues. The soil residue of chlorsulfuron applied at 35 g/ha 36 months prior to harvest was present at sufficient levels to injure 11 of the 14 crops. The residue of the herbicide persisted longer than had been previously anticipated. Crop injury became more apparent later in the summer, indicating increased root contact with the herbicide in the soil. Leaching of chlorsulfuron may have increased its distribution in the soil profile (O'Sullivan, 1982).

Table 13. Percent Crop Injury of 11 Crops on August 18, 1983 from Soil Residues of Chlorsulfuron Applied September 20, 1980 at Bozeman.

Rate g/ha	Potato	Lentil	Sugar Beet	Safflower	Flax	Pinto Bean	Faba Bean	Garbanzo Bean	Sunflower	Alfalfa	Corn
	Crop injury (%)										
35	35	87	45	14	14	20	21	31	35	49	41
70	55	100	60	20	24	26	31	55	51	83	66
140	70	100	84	35	46	44	47	61	80	97	86
CV	18.6	10.8	20.8	44.0	34.7	39.2	47.6	24.4	17.9	14.0	11.3
LSD .05	11.7	12.4	15.7	12.1	11.6	14.1	19.0	14.4	11.9	12.8	8.8

Dry weight of all crops except spring wheat and barley was determined after harvesting 5 plants/species/plot on August 25, 1983 (Table 14). Dry weight of all crops except potato tubers was reduced by soil residues of all rates of chlorsulfuron tested, 36 months after application. No visible crop injury was noted to wheat, oats, or barley, which were not harvested due to hail damage.

At the Great Falls location, plots were harvested on August 31, 1983, and dry weights were determined (Table 15). Dry weight of all harvested crops was reduced by soil residues

Table 14. Dry Weight (g/5 plants/plot) of 11 Crops Harvested on August 25, 1983.*

Rate g/ha	Potato	Sugar Beet	Saf-flower	Sun-flower	Corn	Pinto Bean	Gar-banzo Bean	Alfalfa	Faba Bean	Lentil	Flax
	---------- Plant dry weight (g/5 plants) ----------										
35	572	116	248	1340	648	79	61	3	116	2	51
70	335	46	209	1262	367	69	28	2	71	2	26
140	257	16	167	367	235	59	23	1	40	1	11
control	748	538	465	3378	1896	159	252	18	307	34	104
CV	35.8	41.3	33.6	36.9	23.3	28.3	42.4	56.2	84.5	70.3	29.9
LSD .05	273.9	118.4	146.2	945.7	293.5	41.5	61.9	5.5	180.0	11.1	22.8

*The crops were planted into soil residues of chlorsulfuron, applied on September 20, 1980 at Bozeman.

Table 15. Dry Weight (g/5 plants/plot) of 9 Crops Harvested on August 31, 1983.*

Rate g/ha	Sugar Beet	Sun-flower	Saf-flower	Garbanzo Bean	Alfalfa	Faba Bean	Lentil	Flax	Pinto Bean
35	90	246	121	53	5	43	3	11	87
70	20	117	169	21	0	29	1	10	54
140	6	90	74	9	0	17	1	3	22
control	319	981	219	374	15	103	25	24	183
CV	29.3	11.7	21.7	42.2	43.0	19.9	44.8	41.4	27.0
LSD .05	63.7	83.6	63.2	96.2	4.5	18.9	6.9	9.9	46.9

*The crops were planted into soil residues of chlorsulfuron, applied on September 10, 1980 at Great Falls.

of all rates of chlorsulfuron tested. Corn was not harvested at Great Falls due to poor establishment. No visible injury was noted to wheat, oats or barley, which were not harvested due to bird damage.

The highest labeled use rate for chlorsulfuron in Montana is 18 g/ha. Soil residues from application of 35 g/ha persisted at levels that were injurious to all crops other than small grains for at least 36 months at 2 locations in Montana.

The rate of degradation of chlorsulfuron has been shown to follow first-order kinetics in a sandy loam soil (Walker and Brown, 1983). A half-life of about 60 days was calculated in this soil at 10°C. Palm et al. (1980) reported an average half-life of 1 to 2 months during the summer from several soil types and locations. If the value of 60 days is applied to the soils used in the present studies, then a no-effect level of chlorsulfuron can be determined.

Crop dry weights from 1982 and 1983 at Bozeman were compared as percent of control values, for the 35 g/ha rate of chlorsulfuron application in 1980 (Figure 12). Sunflower and safflower dry weight was significantly increased in 1983 over 1982, and the other crops showed a similar trend. There is a 120-day period per year in which soil temperatures are above 10°C in Bozeman (Anon., 1981). Using this value and the 60 day half-life, the calculated level of chlorsulfuron soil residue was 0.51 g/ha at the end of 1983.

Walker and Brown (1983) reported a 70% reduction in red beet (*Beta vulgaris*) fresh weight from soil levels in 0.5 g/ha of chlorsulfuron. At 0.35 g/ha, red beet fresh weight was reduced by 20%, and there was no effect from 0.20 g/ha. At Bozeman, sugar beet dry weight was reduced by 74.5% in 1983. If the sensitivity of sugar beet and red beet are equal, then there should be no injury to sugar beet by July, 1984.

A similar comparison was made for crops grown at Great Falls (Figure 13). No significant differences were obtained between years for any of the crops. The plot area at Great Falls was not maintained in a weed-free condition in 1983, which resulted in substantial weed competition to several of the crops. Dry weight reductions from soil residues of chlorsulfuron were thus confounded.

Chlorsulfuron's extended residue must be weighed carefully when considering the use of this herbicide. The lack of injury to spring or winter wheat from pre emergence or post emergence applications of chlorsulfuron indicates that chlorsulfuron has excellent potential for use in cropping systems using small grains only. The lessened tolerance of barley to chlorsulfuron soil residues needs to be further explored, particularly at low application rates. NA may have potential for protecting barley from soil residues of chlorsulfuron.

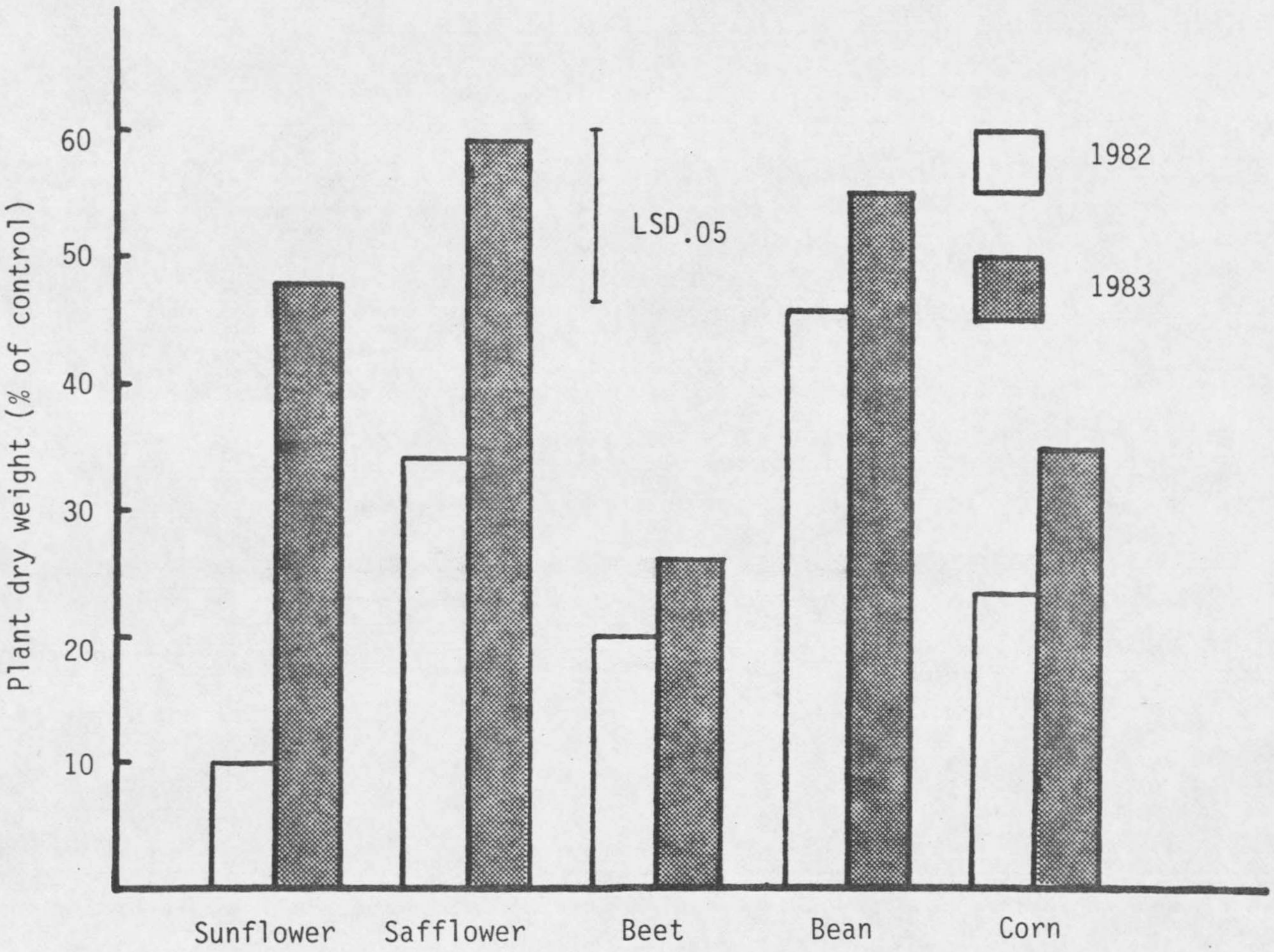

Figure 12. Plant dry weight (% of control) of 5 crops in 1982 and 1983 that were grown in soil treated with 35 g/ha chlorsulfuron on September 20, 1980 at Bozeman, Montana.

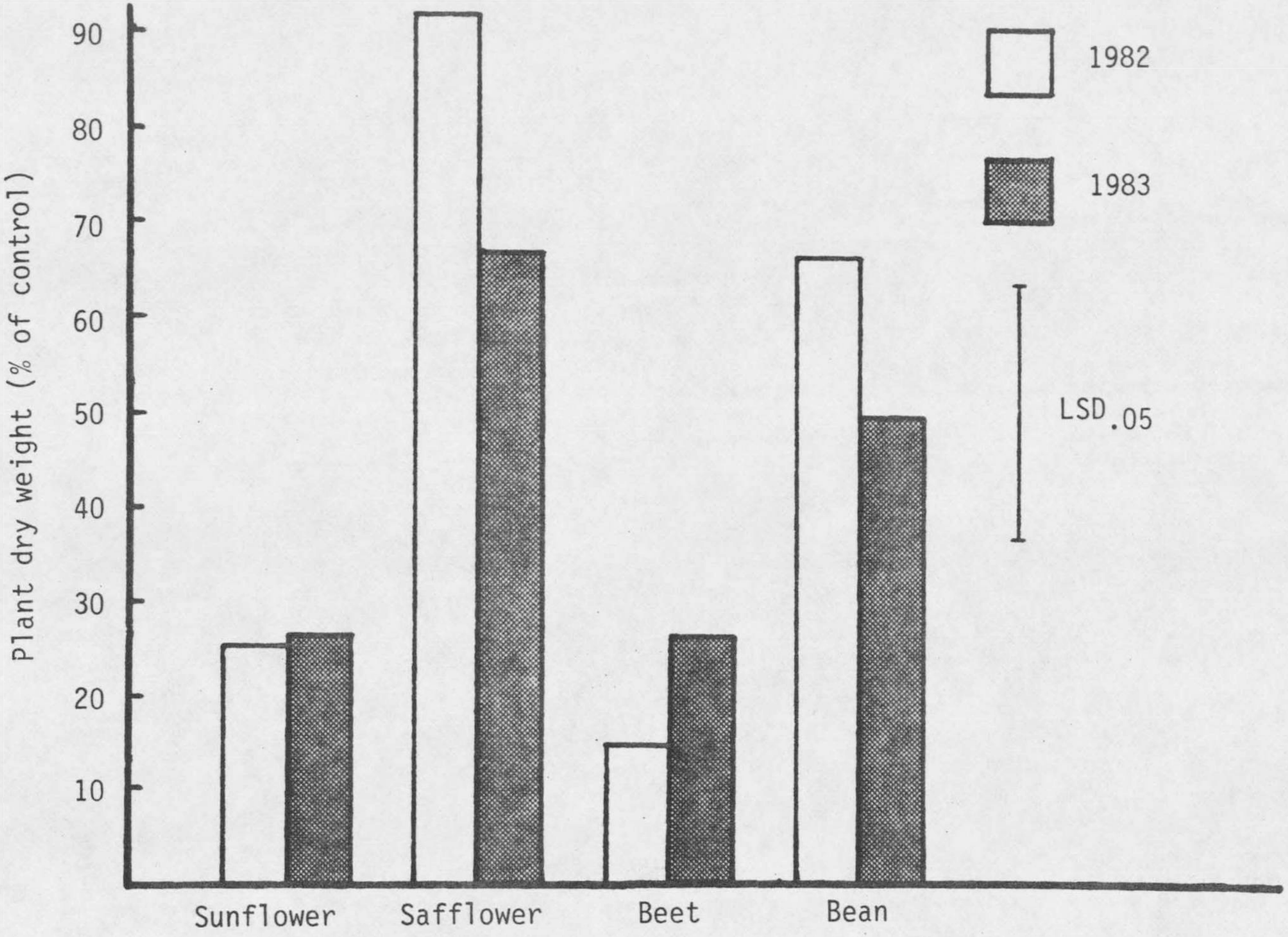

Figure 13. Plant dry weight (% of control) of 4 crops in 1982 and 1983 that were grown in soil treated with 35 g/ha chlorsulfuron on September 10, 1980 at Great Falls, Montana.

CHAPTER 4

THE EFFECT OF CHLORSULFURON ON CANADA THISTLE REGROWTH

Abstract

Canada thistle is the most troublesome perennial weed problem facing Montana grain growers. Chlorsulfuron was evaluated for its potential for control of Canada thistle regrowth. Chlorsulfuron, applied at 18, 35, and 70 g/ha, reduced Canada thistle stems/m^2 1 year after application. Applications at the 5 leaf stage of Canada thistle appeared to be slightly more effective than treatments applied at the bud stage. There was no reduction of Canada thistle stems/m^2 by any rate 2 years after application. Two applications of 18 g/ha chlorsulfuron in successive years were less effective than 2 successive applications of 1121 g/ha 2,4-D in reducing Canada thistle regrowth.

Introduction

Canada thistle is one of the most troublesome perennial weeds in Canada and the northern half of the United States (Friesen, 1968; Hodgson, 1977). The plant can persist and flourish under cultivation, due to its vegetative reproduction from adventitious buds on root segments. It is a troublesome weed in Montana, Idaho, Oregon, and Washington, and causes heavier crop yield losses than any other perennial weed in this region (Hodgson, 1958).

Chlorsulfuron has shown promise as a herbicide for use in small grains. Present use rates for control of broadleaf weeds in small grains range from 9 to 18 g/ha. Chlorsulfuron's residual nature and broad spectrum, which includes some 2,4-D resistant weeds and Canada thistle, are advantages over presently used herbicides.

In the following studies, the objectives were to determine the most effective time, method, and application rate of chlorsulfuron for controlling Canada thistle regrowth.

Materials and Methods

A preliminary study was established on July 3, 1979 to determine if chlorsulfuron had potential for controlling Canada thistle. The treatments were applied to a dense infestation of Canada thistle in 'Pondera' spring wheat, which was in the boot stage of growth. The Canada thistle was in the mid-bud stage of growth at the time of spraying. Chlorsulfuron was applied at 35, 70, and 140 g/ha and 2,4-D ester at 670 g/ha. The herbicides were applied using a CO_2-pressurized backpack sprayer to 2.0 by 7.6 m plots, in 57 l/ha of water. Surfactant was not added to the chlorsulfuron treatments. The plots were arranged in a randomized complete block design, with 3 replications. The plots were visually evaluated for percent control of Canada thistle on September 12, 1979 and June 13, 1980 (0 = no control; 100 = complete kill). Plant counts were taken on June 17, 1980 by determining the number of Canada thistle stems/m^2 in 3 random locations in each plot. All chlorsulfuron treatments subsequent to this study were applied with .25% v/v nonionic surfactant [[octoxynol [a(p-Nonylphenyl)-whydroxypoly (oxyethylene)]]].

Chlorsulfuron was applied to Canada thistle growing on fallow ground on August 27, 1982 to compare the effect of foliar and soil applications of the herbicide in controlling Canada thistle regrowth. Plots were mowed or left intact prior to herbicide application. Chlorsulfuron was applied at 35 and 70 g/ha to Canada thistle in the 20 cm diameter rosette or bloom stage, or to bare ground using a CO_2-pressurized backpack sprayer in 168 l/ha of water. The 3.3 by 4.0 m plots were arranged in a randomized complete block design with 3 replications. Canada thistle plant counts were taken on June 7, 1983 by determining the number of stems/m^2 in 3 random areas in each plot.

A 2-year study was initiated in the spring of 1981 to determine the most effective time and rate of application of chlorsulfuron for controlling Canada thistle regrowth. The plots were established in a uniform infestation of Canada thistle in a field of 'Otana' oats. Early treatments of chlorsulfuron at 18, 35, and 70 g/ha and 2,4-D amine at 1121 g/ha were applied on July 3, 1981 when the oats were in the 4 to 5 leaf stage and the Canada thistle was 15 to 20 cm tall. The treatments were applied to 3.3 by 39.0 m plots using a CO_2-pressurized backpack sprayer in 168 l/ha of water. Chlorsulfuron was also applied on August 21, 1981, at 18, 35, and 70 g/ha when the oats were in the milk stage and the Canada thistle was in the mid-bud stage. One year later on July 15, 1982, when Canada thistle was in the bud stage, retreatments of 18, 35, and 70 g/ha chlorsulfuron and 1121 g/ha 2,4-D amine were applied at right angles to the original treatments. The experiment was arranged in a split-plot design, with the 1981 treatments as the main plots and the 1982 treatments as the subplots. There were 4 replications. Canada thistle stand counts were taken on July 15, 1982 and June 9, 1983 by determining stems/m^2 in 4 random locations in each plot.

Results and Discussion

On September 12, 1979, the experiment comparing chlorsulfuron and 2,4-D ester was rated for percent control of Canada thistle (Table 16). All rates of chlorsulfuron were satisfactorily controlling Canada thistle, 2 months after application. Regrowth was occurring in the plots treated with 2,4-D ester. When the plots were rated the following June, control of regrowth was being maintained by the chlorsulfuron treatments, although at slightly lower levels than during the previous fall. All rates of chlorsulfuron reduced the

Table 16. The Effect of Chlorsulfuron and 2,4-D Ester on Canada Thistle Regrowth 3 and 12 Months After Treatment.*

Herbicide	Rate g/ha	Canada Thistle Control 9-12-79 %	Canada Thistle Control 6-13-80 %	Stems/m² 6-17-80
2,4-D ester	670	50	47	14.0
chlorsulfuron	35	83	57	27.3
chlorsulfuron	70	88	75	12.0
chlorsulfuron	140	100	90	3.0
control		0	0	65.0
	CV	22.5	20.2	7.4
	LSD .05	17.8	17.1	6.3

*The herbicides were applied on July 3, 1979.

number of Canada thistle stems/m² one year after application. Control of regrowth by the 2,4-D ester treatment was similar to the treatment of 70 g/ha of chlorsulfuron.

The study was designed to imitate a "crop salvage" situation, in which a producer must remove large weeds with herbicide treatments in order to harvest. Although the treatments were not applied at the usual time of year (early spring), the 2 highest rates of chlorsulfuron provided excellent control of Canada thistle regrowth 12 months after application. However, the rates of application tested were substantially greater than the recommended use rate of 18 g/ha.

Chlorsulfuron was applied to mowed or intact Canada thistle plants to determine whether foliar or soil applications were more effective in controlling regrowth. The only significant reduction in Canada thistle stand was from the 70 g/ha rate of chlorsulfuron applied to young, mowed plants (Table 17). The lack of control from all other treatments could be due to the absence of crop competition (Hodgson, 1958; Zimdahl, 1980), which was present in other trials. Soil applications of chlorsulfuron appeared to be slightly more effective than foliar applications in controlling Canada thistle regrowth, although differences were not significant. This trend is in agreement with O'Sullivan (1982), who suggested that the soil residue of chlorsulfuron was more important than herbicide translocation into the

Table 17. Canada Thistle Stems/m² 9 Months After Herbicide Treatment.*

		Canada Thistle Stems/m²			
		5 Leaf Stage		Bud Stage	
Herbicide	Rate g/ha	Mowed	Intact	Mowed	Intact
chlorsulfuron	35	12.3	12.0	13.7	18.3
chlorsulfuron	70	2.3	9.3	9.0	13.0
control		12.7	14.3	13.0	18.7
	CV	34.6	46.0	28.2	20.9
	LSD .05	7.1	12.4	7.6	7.9

*Herbicides were applied to 2 growth stages of Canada thistle that were mowed or left intact prior to treatment.

roots in controlling Canada thistle regrowth the year after application. Applications made at the 5 leaf stage of the Canada thistle appeared to be slightly more effective in controlling regrowth than applications made at the bud stage.

One year after treatment, Canada thistle regrowth was reduced by all treatments except chlorsulfuron at 18 g/ha, applied at the bud stage of growth (Table 18). There was a trend toward slightly better control of regrowth following application at the 5 leaf stage than at the bud stage, although the differences were not significant. From a cultural standpoint, applications at the 5 leaf stage of Canada thistle would be preferable over bud stage treatments, since normal weed control practices are usually carried out at this time of the cropping season.

Retreatments of chlorsulfuron and 2,4-D amine were applied in 1982 at right angles to the 1981 treatments. On June 9, 1983, Canada thistle stand counts were taken for the 1982 treatments which were applied to the control plots from 1981 (Table 19). The results were similar to the 1982 results of the 1981 treatments.

Chlorsulfuron at 18 g/ha and 2,4-D amine at 1121 g/ha gave similar control of Canada thistle regrowth. The rate of 2,4-D amine tested was twice the recommended use rate for this herbicide in small grains. Chlorsulfuron at 18 g/ha would appear, then, to provide

Table 18. Canada Thistle Stems/m² on July 15, 1982 After Herbicide Application at the 5 Leaf and Bud Stage in 1981.

Herbicide	Rate g/ha	Canada Thistle Growth Stage at Application	Canada Thistle Stems/m²
chlorsulfuron	18	5 leaf	34.9
chlorsulfuron	35	5 leaf	21.7
chlorsulfuron	70	5 leaf	4.8
chlorsulfuron	18	bud	37.9
chlorsulfuron	35	bud	24.6
chlorsulfuron	70	bud	9.6
2,4-D amine	1121	5 leaf	27.7
control			45.4
	CV		26.2
	LSD .05		9.9

Table 19. Canada Thistle Stems/m² on June 9, 1983 After Herbicides Were Applied on July 15, 1982 to 1981 Control Plots.

Herbicide	Rate g/ha	Canada Thistle Stems/m²
chlorsulfuron	18	24.8
chlorsulfuron	35	12.6
chlorsulfuron	70	.2
2,4-D amine	1121	27.9
control		47.3
	CV	33.9
	LSD .05	11.8

better control of Canada thistle regrowth at 18 g/ha than would 2,4-D amine at its normal use rate, when applied at the 5 leaf stage.

No reductions in Canada thistle stems/m² were observed in 1983 from any treatment applied in 1981 (Table 20). The 1981 2,4-D amine treatment, which injured the Canada thistle sufficiently to reduce regrowth in 1982, did not maintain its control in 1983. This lack of continued control would be expected from a non-residual herbicide, such as 2,4-D amine.

Table 20. Canada Thistle Stems/m^2 on June 9, 1983 After Herbicide Applications in July, 1981.*

Herbicide	Rate g/ha	Canada Thistle Stems/m^2
chlorsulfuron	18	40.9
chlorsulfuron	35	45.9
chlorsulfuron	70	34.7-
2,4-D amine	1121	38.5
control		35.1
	LSD .05	4.7

*The plots were not treated in 1982.

The residual control of Canada thistle regrowth did not continue for 2 cropping seasons. Chlorsulfuron residues from applications of 35 and 70 g/ha persist for more than 1 year in Bozeman silt loam soil (see Chapter 3). These residues are sufficient to injure several broadleaf crops after 1 and even 2 years, but are apparently below the levels necessary to control Canada thistle regrowth.

In 1983, 2 treatments of the same rate of chlorsulfuron in 1981 and 1982 provided slightly more control of Canada thistle regrowth than 1 application made in 1982 (Table 21). Chlorsulfuron treatments of 18 g/ha, applied in 1981 and 1982, increased control by 23% over the single 18 g/ha treatment in 1982. The rate of 18 g/ha applied in 1981 did not control regrowth 2 years after application (Table 20). The increased control of regrowth by successive treatments at this rate could indicate a gradual weakening of the Canada thistle plants, or a recharging of the chlorsulfuron residue in the soil. Yearly applications of chlorsulfuron could perhaps provide sustained control of Canada thistle regrowth, if applied for 2 or more successive years.

Chlorsulfuron treatments applied in 2 successive years at any combination of rates reduced Canada thistle stems/m^2 more than any single treatment, applied 2 years previously (Table 22). The highest rate of chlorsulfuron tested in 1981 (70 g/ha) failed to control Canada thistle regrowth 2 years after application. However, when this rate was

Table 21. Canada Thistle Stems/m² on June 9, 1983 After 1 or 2 Years of the Same Rate of Application of Chlorsulfuron at the Bud Stage.

g/ha		
chlorsulfuron 1981	chlorsulfuron 1982	Canada Thistle Stems/m²
–	18	24.7
18	18	18.9
–	35	8.5
35	35	5.2
–	70	1.6
70	70	.3
–	–	35.1
	LSD .05	4.8

Table 22. Canada Thistle Stems/m² on June 9, 1983 After 1 or 2 Years of Various Rate Combinations of Chlorsulfuron Applied at the Bud Stage.

g/ha		
chlorsulfuron 1981	chlorsulfuron 1982	Canada Thistle Stems/m²
18	–	40.9
–	18	24.7
18	18	18.9
35	18	18.4
70	18	6.3
35	–	45.9
–	35	8.5
18	35	5.4
35	35	5.2
70	35	.9
70	–	34.7
–	70	1.6
18	70	.5
35	70	.5
70	70	.3
–	–	35.1
	LSD .05	7.3

followed by 18 g/ha in 1982, control of Canada thistle regrowth was increased by 75% over the treatment of 18 g/ha alone in 1982. This increase in control indicates an additive effect from the herbicide, when it was applied in 2 successive years. Similar trends of increased control by combination treatments over single treatments applied 2 years previously were observed for the other rate combinations.

The increased control of Canada thistle regrowth from successive treatments could have been due to the rate of breakdown of chlorsulfuron. By 1983, chlorsulfuron residues in the soil from the 1981 applications had fallen below the level necessary to control Canada thistle regrowth. The addition of retreatments in 1982 increased chlorsulfuron levels so that Canada thistle regrowth was again controlled during 1983. While all treatments were continuously subject to degradation in the soil, the amount of chlorsulfuron present from 2 successive applications remained at or above the herbicidal level for a longer period. Thus, control of Canada thistle regrowth was increased by successive applications of chlorsulfuron over single applications.

The advantage of residual weed control in controlling Canada thistle regrowth was not extended for more than 1 year. It appears then, that a single application of chlorsulfuron (18 g/ha) will provide control of Canada thistle regrowth similar to a bud stage application of 1121 g/ha 2,4-D amine. Even though chlorsulfuron has the advantage of providing residual weed control of several annual weed species, a single application does not provide acceptable control of Canada thistle regrowth, 2 years after treatment. The prospect of eradicating Canada thistle from application of a single, high rate of chlorsulfuron has not been realized.

The rate of 2,4-D amine tested has been shown to be injurious to small grain yields (Swan, 1975). The chlorsulfuron rates used in these studies, however, have not injured spring wheat when applied at 7 wheat growth stages, from pre emergence to jointing (see Chapter 3). Canada thistle regrowth during the year of treatment with 2,4-D amine was apparent by August (data not shown). Regrowth from the plots treated with chlorsulfuron was not observed through the post harvest period and fall tillage. Canada thistle shoots treated at the 5 leaf stage remained green for up to 2 weeks following chlorsulfuron application, but had ceased growing. Weeds treated with chlorsulfuron can exhibit up to a 66% reduction in transpiration 2 to 4 days after application (DuPont, 1980). The shoots slowly became chlorotic and died. A typical herbicidal rate response (more rapid or more pronounced injury from higher rates of application) was not observed in these studies. The lowest rate of chlorsulfuron tested (18 g/ha) controlled regrowth during the cropping season as effectively as did the 70 g/ha rate of application. Thus, the tendency to apply higher rates of chlorsulfuron in hopes of obtaining increased control of Canada thistle is not justified.

The extended control of regrowth during the cropping season may be the most effective advantage of chlorsulfuron over 2,4-D. During combining, Canada thistle stems interfere with the harvesting process by adhering to straw and grain in the combine. Chlorsulfuron's residual control of Canada thistle regrowth during the cropping season could prevent this objectionable feature of harvesting infested grain. The prospect of sustained control of Canada thistle regrowth from successive applications at normal use rates in a continuous wheat situation is also a very desirable possibility.

SUMMARY

Chlorsulfuron has good potential for use in small grain agriculture in Montana. The broad spectrum and residual weed control obtained with this herbicide are desirable advantages over other presently used herbicides.

Chlorsulfuron was mobile in soil columns and on soil thin-layer chromatography (TLC) plates. In soil columns, ^{14}C-labeled chlorsulfuron was detected in decreasing amounts with increasing depth. ^{14}C-labeled chlorsulfuron moved as a concentrated front on soil TLC plates. The pattern of movement in soil columns was judged to most closely represent chlorsulfuron's actual behavior in field situations. The possibility for chlorsulfuron to be leached into groundwater was considered remote. Additional research on the chlorsulfuron-soil-water equilibrium is needed.

Soil residues of chlorsulfuron persisted for at least 36 months at 2 locations in Montana. Crops other than wheat, barley, or oats should not be planted into suspected chlorsulfuron residues in soil. Further studies on the rate of degradation of chlorsulfuron in different soils and under various climatic conditions should be carried out.

Canada thistle regrowth was reduced by applications of chlorsulfuron, the year after treatment. Successive yearly treatments provided better regrowth control than single applications of high rates. Chlorsulfuron's control of Canada thistle regrowth during the cropping season was considered to be a primary advantage over other presently used herbicides.

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APPENDICES

APPENDIX A

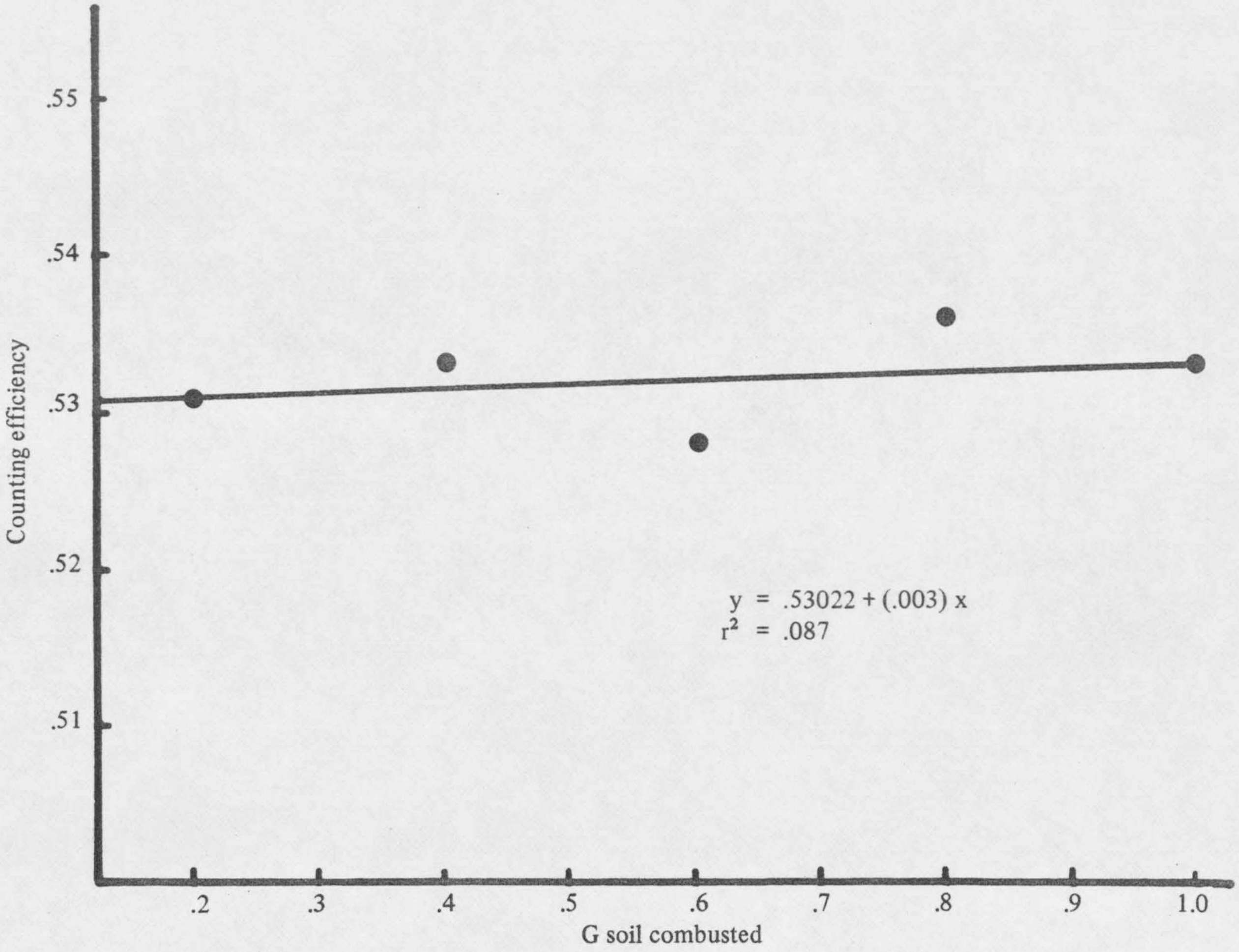

Figure 14. Soil combustion efficiency curve. Ten μl ^{14}C-labeled hexadecane (.868×10^6 dpm/ml) were added to .2, .4, .6, .8, and 1.0 g air-dried, screened Bozeman silt loam soil. The soil was combusted for 1.5 minutes and the evolved $^{14}CO_2$ was trapped in 15 ml carbon 14 cocktail (R. J. Harvey Instr. Co., Hillsdale, NJ).

APPENDIX B

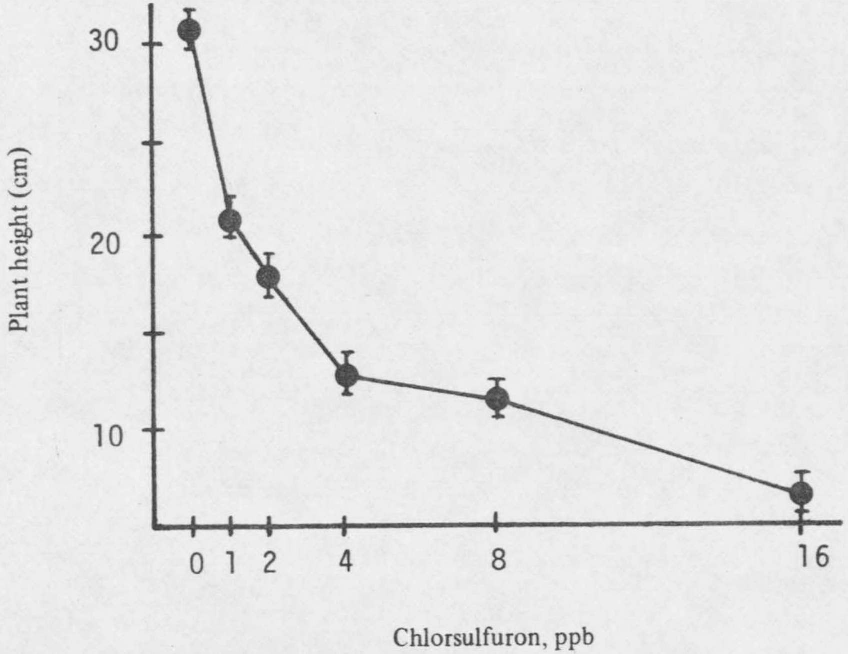

Figure 15. Plant height (cm) of foxtail millet [*Setaria italica* (L.) Beauv. 'Golden German'] grown in 6 concentrations of chlorsulfuron-treated Bozeman silt loam soil.

APPENDIX C

APPENDIX C

Table 23. Percent Weed Control (0 = no control; 100 = complete kill) of Field Pennycress and Annual Sowthistle on August 2, 1982 at Great Falls, Montana.*

Rate g/ha	Weed Control (%)	
	Field Pennycress	Annual Sowthistle
35	13	15
70	80	90
140	87	93
control	0	0
CV	7.6	7.9
LSD .05	6.8	7.8

*The weeds were growing in soil treated with chlorsulfuron on September 10, 1980.

APPENDIX D

APPENDIX D

Table 24. Percent Crop Injury (0 = no injury; 100 = complete kill) of Spring Wheat and Barley on July 12, 1983 at Bozeman.*

Rate g/ha	Crop Injury (%)	
	Spring Wheat	Barley
35	0	0
70	0	0
140	0	2.2
control	0	0
CV	0.0	131.4
LSD .05	***	7.3

*The crops were growing in soil treated with chlorsulfuron on September 20, 1980.