

Vertical Mobility, Soil Adsorption, and Cotton Tolerance to Three
Protoporphyrinogen Oxidase (PPO) Inhibiting Herbicides in Three West Texas
Soils

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ABSTRACT

Soil-residual herbicides are very important in global crop production and are the foundation of an effective cotton (*Gossypium hirsutum* L.) weed management program. Soil-residual herbicides may reduce the number of postemergence (POST) herbicide applications that are needed and may prevent or delay weed shifts or the development of glyphosate- or glufosinate-resistant weeds. Increasing infestations of glyphosate- and ALS-resistant Palmer amaranth (*Amaranthus palmeri* S. Wats) in cotton has forced producers to utilize herbicides with alternative modes of action in their management systems, including glufosinate and protoporphyrinogen oxidase inhibiting (PPO) herbicides. Trifludimoxazin [1,5-dimethyl-6-sulfanylidene-3-(2,2,7-trifluoro-3-oxo-4-prop-2-ynyl-1,4-benzoxazin-6-yl)-1,3,5-triazinane-2,4-dione] is a new PPO herbicide under development by BASF Corporation that is being evaluated for possible use as a soil-residual treatment in cotton for the control of annual small-seeded broadleaf weeds, including Palmer amaranth. Laboratory and greenhouse studies were conducted to compare vertical mobility, soil adsorption, and cotton tolerance of trifludimoxazin in three West Texas soils [Acuff (Cotton Center, TX) - loam, 1.5% organic matter (OM), 8.3 pH, 22.1 cation exchange capacity (CEC); Amarillo (Seagraves, TX) - loamy sand, 0.3% OM, 8.2 pH, 8.7 CEC; Olton (Halfway, TX) - loam, 1.0% OM, 7.6 pH, 23.1 CEC] to flumioxazin and saflufenacil, which are two currently registered PPO herbicides for use in cotton.

Vertical soil mobility of trifludimoxazin was similar to flumioxazin in the Acuff and Olton soils, but was more mobile in the Amarillo soil. The depth of

movement, resulting from the 2.54 cm irrigation event, of trifludimoxazin in all soils ranged from 2.5 to 5.0 cm, which would not allow for crop selectivity based on placement, as ideal cotton planting is from a depth of 0.6 to 2.54 cm deep. Soil adsorption studies estimated that trifludimoxazin was ~70% adsorbed in the Acuff soil, ~ 60% in the Olton soil, and ~50-60% in the Amarillo soil. Additional adsorption studies with four additional soils indicated that percent OM was the only soil parameter that was important for adsorption of trifludimoxazin. Greenhouse studies indicated that preemergence treatments were more injurious than the 14 d preplant (PP) treatment when summarized across soils for the three herbicides (43% and 14% injury, respectively). No differences in visual cotton response or dry-weight was observed following PP trifludimoxazin treatments as compared to the nontreated control within each of the three West Texas soils and was similar to the flumioxazin PP treatments across soils. Based on the results of these studies, a use pattern for trifludimoxazin in cotton may be established with the utilization of a ≥ 14 d PP application requirement prior to cotton planting.

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CHAPTER I

INTRODUCTION

Herbicides with soil-residual activity are very important in global crop production. Their ability to provide periods of extended weed control helps crops reach their maximum yield potential. In the 2014 Phillips McDougall AgriService report (McDougall 2014a), seven of the top fifteen global herbicides (by market value) have soil-residual activity (Table 1.1). In the production of the three major row crops in the US [corn (*Zea mays L.*), soybean (*Glycine max L.*), and cotton (*Gossypium hirsutum L.*)], a similar trend is seen with soil-residual active herbicides being some of the most commonly used (approximately 64%, 29%, and 37% of the market value, respectively) (McDougall 2014b) (Table 1.2).

Soil-residual herbicides are especially important for maintaining weed control during the critical period for weed control (CPWC), defined as a period in the crop growth-cycle when weeds must be controlled to prevent yield losses (Zimdahl 1988). In corn, a 4 to 5 week (wk) weed-free period after planting was required to avoid significant yield losses (Wilson and Westra 1991). In soybean, the CPWC ranged from approximately the second trifoliolate to the V1 to V4 growth stage, depending on row spacing (Knezevic et al. 2003). In cotton, the CPWC was 1 to 2 wk post crop emergence up to 11 to 12 wk past crop emergence (Bunkun 2004). The use of soil-residual herbicides can not only eliminate or reduce early-season competition from weeds to help secure crop yield, but also allow the grower

flexibility in timing of a postemergence (POST) application if needed (Ellis and Griffin 2002).

Since the introduction of glyphosate-resistant cotton in 1997, adoption has been rapid. In 2014 approximately 93% of US upland-cotton was planted to this technology (NASS 2014). Broad-spectrum weed control, conveniences of over-the-top (OTT) applications, simplicity of management systems, increased rotational options, reductions in labor, and time savings are reasons growers readily adopted this technology (Fernandez-Cornejo et al. 2014; Roberts et al. 2006; Culpepper and York, 1998, 1999b; Faircloth et al. 2001; Gianessi 2008; Young 2006).

Following the introduction of glyphosate-resistant cotton, many producers decreased the use of traditional soil-residual herbicides that were once common to cotton weed management programs (Givens et al. 2009; Young 2006).

Overreliance on glyphosate has led to the development of multiple glyphosate-resistant biotypes, and recently Palmer amaranth (*Amaranthus palmeri* S. Wats.) has developed resistance to glyphosate in a number of states (Culpepper et al. 2006; Heap 2015). In response to the development of these glyphosate-resistant weed populations, researchers are seeking herbicide options with an alternative mode of action to complement glyphosate in crop weed management systems (Keeling et al. 2011).

The use of protoporphyrinogen oxidase (PPO) inhibiting herbicides has increased 24.9% (by value) globally since 2009. The increase in usage has been in response to the occurrence of glyphosate-resistant weeds in the US, especially *Amaranthus* species (McDougall 2014a). Flumioxazin, fomesafen, and saflufenacil

are examples of PPO-inhibiting herbicides whose use has increased over the past several years.

Herbicide sorption, the binding of herbicides to soil colloids (clay and organic matter fraction of the soil), determines the herbicide availability to plant roots and also impacts leaching, volatilization and microbial degradation. Herbicide sorption occurs due to a variety of chemical and physical interactions with soil surfaces (Harper 1994). The intrinsic properties of both the herbicide and the soil determine the nature of this interaction. Therefore, herbicide sorption is the major contributing factor related to herbicide efficacy and environmental fate of soil-residual herbicides. Pesticide adsorption to soil colloids is greatly affected by the composition of the individual soil. Sorption is dependent on the soil clay content, type of clay present, presence of oxide complexes, and organic matter content (Harper 1994; Fissel and Bolt 1962; Sannino et al. 1997; Piccolo 1994).

West Texas cotton production soils range in texture from fine to coarse, generally have low soil organic matter (OM) content, and typically have high soil pH levels. Herbicide use, application timing, and rate may be affected by these properties. Many soil-residual herbicides have use restrictions/limitations that are related to soil properties that affect their behavior in the soil. The use of imazethapyr [5-ethyl-2-(4-methyl-5-oxo-4-propan-2-yl-1H-imidazol-2-yl)pyridine-3-carboxylic acid] in West Texas peanut (*Arachis hypogaea* L.) production is limited to POST applications only. Increased imazethapyr availability in the high pH soil systems of West Texas may lead to increased peanut phytotoxicity. Thus, preplant (PP) and preemergence (PRE) applications of imazethapyr are restricted in West

Texas peanut production (Anonymous 2015a; personal knowledge). The restriction against using dicamba [3,6-dichloro-2-methoxybenzoic acid] PP to cotton in areas west of the Mississippi river (Anonymous 2015b) is related to the increased herbicide persistence under semi-arid environments, which can lead to increased risk of crop injury (personal knowledge). As new soil-residual herbicides are developed for commercialization, testing under a wide range of soil conditions is required to determine the utility of using these herbicides in weed management programs in West Texas. With the development and spread of glyphosate-resistant Palmer amaranth, multiple control options including the use of soil-residual herbicides will be needed to effectively manage this weed in cotton.

Table 1.1 Top global herbicide active ingredients in 2014.^a

Rank	Active Ingredient ^b	Sales (\$m)	Chemical Family	Launch Year	Manufacture
1	glyphosate	5,720	glycine	1972	Monsanto
2	paraquat	850	bipyridylum	1962	Syngenta
3	2,4-D	680	phenoxy	1945	Nufarm, Dow
4	mesotrione*	670	triketone	2001	Syngenta
5	metolachlor*	585	chloroacetamide	1975	Syngenta
6	glufosinate	560	phosphinic acid	1986	Bayer
7	atrazine*	550	triazine	1957	Syngenta
8	acetochlor*	460	chloroacetamide	1985	Monsanto
9	pinoxaden	425	phenylpyrazoline	2006	Syngenta
10	pendimethalin*	380	dinitroaniline	1976	BASF
11	flumioxazin*	370	N-phenylphthalimide	1993	Sumitomo
12	clomazone	360	isoxazolidinone	1986	FMC
13	clethodim	330	cyclohexanedione	1987	Sumitomo, Arysta
14	picloram	290	pyridine carboxylic acid	1963	Dow
15	fluroxypyr	275	pyridine carboxylic acid	1985	Dow

^a Phillips McDougal, AgriService Market Summary (2014a).^b * Indicates herbicides with soil-residual activity.

Table 1.2. Top soil-residual herbicide active ingredients in the US by market value in 2014.^a

Crop	Rank	Active Ingredient	Market Share ----- % -----
corn	2	mesotrione	16.9
corn	3	acetochlor	12.9
corn	4	s-metolachlor	12.4
corn	5	atrazine	6.7
corn	6	thiencarbazone-methyl	6.2
corn	7	isoxaflutole	3.5
corn	8	clopyralid	3.3
corn	10	dimethenamid-P	2.4
total			64.3
soybean	2	flumioxazin	9.7
soybean	3	chlorimuron	6.9
soybean	4	sulfentrazone	5.2
soybean	6	fomesafen	4.0
soybean	7	imazethapyr	3.2
total			29.0
cotton	2	flumioxazin	10.6
cotton	3	pyrithiobac	6.5
cotton	4	s-metolachlor	6.0
cotton	5	trifluralin	5.9
cotton	6	fomesafen	4.7
cotton	9	pendimethalin	3.3
total			37.0

^a Phillips McDougal, AgriService Market Summary (2014b).

CHAPTER II

LITERATURE REVIEW

History of Weed Management in Cotton

Historical weed control in cotton was accomplished by hand-hoeing, which was often performed by slaves or later low-paid workers in large groups. By the early 1900s, this practice had become expensive but necessary to keep weeds from competing with cotton (Brown 1927). In the early 1960's, trifluralin [2,6-dinitro-N,N-dipropyl-4-(trifluoromethyl)aniline] and MSMA [sodium;hydroxy(methyl)arsenate] were introduced and significantly changed weed control in cotton (Reed 2012). Trifluralin could be applied preplant incorporated (PPI) with standard land preparation equipment to effectively control annual grasses and small-seeded annual broadleaf weeds. MSMA was used POST to control annual grasses, Johnsongrass (*Sorghum halepense* L. Pers.), and nutsedge (*Cyperus* spp.) with minimal crop injury. The selectivity and effectiveness of these two herbicides led to their widespread adoption in the 1960's (Reed 2012).

Other important herbicides that were developed and used in cotton in the 1960's include linuron [3-(3,4-dichlorophenyl)-1-methoxy-1-methylurea], fluometuron [1,1-dimethyl-3-[3-(trifluoromethyl)phenyl]urea], prometryn [6-methylsulfanyl-2-N,4-N-di(propan-2-yl)-1,3,5-triazine-2,4-diamine], and paraquat [1-methyl-4-(1-methylpyridin-1-i um-4-yl)pyridine-1-i um;methyl sulfate]. Paraquat is a non-selective herbicide used PP and PRE to control emerged weeds prior to crop

emergence. Linuron, fluometuron, and prometryn are selective herbicides that may be used PRE to control annual small-seeded broadleaf weeds as they emerged.

In the 1970's, glyphosate [2-(phosphonomethylamino)acetic acid] and pendimethalin [3,4-dimethyl-2,6-dinitro-N-pentan-3-ylaniline] were introduced. Glyphosate is used POST as a non-selective PP and spot-spray herbicide to control emerged weeds and pendimethalin may be used soil-applied (PPI or PRE) to selectively control annual grasses and small-seeded annual broadleaf weeds as they emerge.

In the 1980s, POST graminicides were introduced for the control of annual and perennial grasses. Herbicides such as sethoxydim [2-[1-(ethoxyamino)butylidene]-5(2-ethylsulfanylpropyl)cyclohexane-1,3-dione] and fluazifop [2-[4-[5-(trifluoromethyl)pyridine-2-yl]oxyphenoxy]propanoic acid] were very useful for controlling emerged grassy weeds in cotton that other herbicides did not control well. While most small-seeded annual grasses continued to be managed with soil-residual herbicides or burndown treatments, management of certain grasses such as Johnsongrass and bermudagrass (*Cynodon dactylon* L. Pers.) became easier with the additional selectivity of POST graminicides (McWhorter and Bryson 1992).

In the early 1990s, pyrithibac-sodium [2-chloro-6-(4,6-dimethoxypyrimidin-2-yl)sulfanylbenzoate] was introduced and significantly improved the control of broadleaf weeds POST with little to no injury to cotton (Jordan et al. 1993; Keeling et al. 1993).

Glyphosate-resistant cotton was first introduced in 1997, which caused a major change in the overall weed management. Glyphosate is a systemic, non-selective herbicide that inhibits 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS; EC 2.5.1.19), hindering the synthesis of aromatic amino acids tryptophan, tyrosine, and phenylalanine (Duke 1990; Askew and Wilcut 1999; Askew et al. 2002a; Bailey et al. 2003; Corbett et al. 2004; Culpepper and York 1999). Glyphosate-resistant cotton was achieved by expression of EPSPS gene derived from *Agrobacterium* spp. strain CP4 (CP4 EPSPS) that encodes for glyphosate-resistant EPSPS. Initial glyphosate-resistant cotton cultivars had a high level of vegetative tolerance, but reproductive tolerance was not as great because of reduced expression of *CP4 EPSPS* in male reproductive tissues (Nida et al. 1996; Pline et al. 2002). Thus, labeled glyphosate use was restricted to the four-leaf stage of cotton to prevent late maturity, boll abortion, and yield loss (Viator et al. 2004; Light et al. 2003). Enhanced glyphosate-resistant cultivars were subsequently developed and introduced in 2006 that allowed POST over-the-top (OTT) applications of glyphosate on cotton with more than four leaves without any detrimental effect on reproductive tissue and yield (Huff et al. 2010). Enhanced resistance in these cotton cultivars is due to the use of an altered promoter sequence in the same *CP4 EPSPS* gene (May et al. 2004).

Glufosinate-resistant cotton was introduced in 2004 and represented another option that could be used for effective weed management. Glufosinate [2-amino-4-[hydroxyl(methyl)phosphoryl]butanoic acid] is a non-selective POST-applied herbicide that inhibits glutamine synthetase (EC 6.3.1.2), an enzyme that

catalyzes the synthesis of glutamine from glutamate and ammonium (Bellinder et al. 1987). Glutamine synthetase inhibition leads to the accumulation of toxic levels of ammonia within the cell, and reduces the rate of photosynthesis and stomatal conductance (Coetzer and Al-Khatib 2001). Consequently, severe cell membrane disruption and necrosis leads to rapid plant death following an application to a sensitive species (Bellinder et al. 1987). Glufosinate-resistant cotton was accomplished by incorporation of the bialaphos resistance (*BAR*) gene from the fungus *Streptomyces viridochromogenes* that encodes for increased levels of phosphinothrin acetyltransferase (PAT). The PAT enzyme converts the active molecule of glufosinate (L-phosphinothrin) into nontoxic N-acetyl-L-phosphinothrin (Devine et al. 1993; Droege et al. 1992). Glufosinate-resistant cotton has excellent tolerance and glufosinate may be applied from cotton emergence until the early bloom stage (Anonymous 2006).

Weed Management System in Herbicide-Tolerant Cotton

Before glyphosate- or glufosinate-resistant cotton was available, producers used PPI and PRE herbicides for early-season soil-residual weed control. Postemergence control of annual grasses was achieved with POST-applied graminicides and POST control of annual broadleaf weeds was accomplished with postemergence-directed (PDIR) herbicides, multiple cultivations, or sometimes both (Culpepper and York 1998; Snipes and Mueller 1992a, 1992b; Wilcut et al. 1997). Directing these herbicides on small cotton required a height differential between the crop and the weeds, which at times could be difficult to achieve (Culpepper and

York 1998; Snipes and Muller 1992a). The inclusion of PDIR herbicide treatments and the demand of special equipment for application made this a slow and tedious process (Askew and Wilcut 1999; Culpepper and York 1998; Wilcut et al. 1997).

Since glyphosate lacks soil activity (Franz et al. 1997), and the POST application window on the initial glyphosate-resistant cotton cultivars was narrow, views on whether or not soil-residual herbicides should be used with glyphosate-resistant varieties varied (Asher et al. 1997; Askew and Wilcut 1999; Welch et al. 1997). Glyphosate-only herbicide programs have resulted in equivalent yields and greater net returns due to the reduction in hand-weeding in glyphosate-resistant cotton (Mills and Voth 1997; Vencill 1998; Webster et al. 1997; Wilcut and Hinton 1997). However, other research suggested that glyphosate-resistant cotton yields were best when soil-residual herbicides were used along with glyphosate to control weeds (Askew and Wilcut 1999; Brecke and Colvin 1997; Wilcut et al. 1998).

In enhanced glyphosate-resistant cotton, glyphosate may be applied POST OTT throughout most of the growing season without negative effect to the crop (Keeling et al. 2003; Martens et al. 2003). However, it is expected that timely glyphosate applications will be necessary to maintain consistent weed control and cotton yield (Croon et al. 2003). Optimum weed control from glyphosate is achieved when applications are made to small, actively growing weeds and, conversely, a reduction in weed control has been observed when glyphosate is applied to larger weeds (Shaw and Arnold 2002). Since glyphosate does not have soil-residual activity, multiple applications are required to control weeds that emerge over an extended period (Reddy and Norsworthy 2010). Glyphosate-only

weed management programs in enhanced glyphosate-resistant cotton have resulted in excellent weed control if the applications were timely (Askew et al. 2002a; Culpepper and York 1998, 1999b; Faircloth et al. 2001; Scott et al. 2002).

Glufosinate-resistant cotton has provided another POST weed management tool for cotton growers, especially those that are fighting glyphosate resistant weeds such as Palmer amaranth (Everman et al. 2009). Glufosinate controls many annual weeds when applied in a timely manner; however, control of annual grasses and *Amaranthus* spp. can be marginal, especially in less than ideal growing conditions (Beyers et al. 2002; Coetzer et al. 2002; Corbett et al. 2004; Culpepper et al. 2000; Hill et al. 1997; Steckel et al. 1997; York and Culpepper 2004). Since glufosinate does not provide residual activity, it has been shown that when PRE or POST applied soil-residuals were not utilized, a reduction in the overall control of several weeds, such as Palmer amaranth and annual grasses, has been observed (Everman et al. 2007).

Although multiple herbicide applications are possible in enhanced glyphosate-resistant and glufosinate-resistant cotton, use of residual herbicides as PRE, mid-postemergence (MPOST) and PDIR applications may reduce the number of needed glyphosate or glufosinate applications. Additionally, soil-residual herbicides with different modes of action may prevent or delay weed shifts or evolution of glyphosate- or glufosinate- resistant weeds by providing enhanced control of weeds that are usually marginally controlled by glyphosate or glufosinate alone (Martinez-Ghersa et al. 2003). Everman et al. (2009) and Scroggs et al. (2007) reported increased weed control with the addition of PRE soil-residual herbicides in weed

control programs for glufosinate- and enhanced glyphosate-resistant cotton, respectively.

Integration of soil-residual herbicides in glyphosate-resistant crops is commonly an academic recommendation to improve consistency of weed management and deter the evolution of glyphosate-resistant weed species (Bond et al. 2011; Norsworthy et al. 2012). Past research has shown that programs containing a soil-residual herbicide in glyphosate-resistant cotton maximizes weed control and cotton lint yield (Burke et al. 2005; Clewis et al. 2008; Culpepper 2006; Grichar et al. 2004; Price et al. 2008; Scroggs et al. 2007). Despite these results, many US producers have decreased the use of traditional soil-residual herbicides that were once common to cotton weed management programs (Givens et al. 2009; Young 2006). Excessive reliance on glyphosate in cotton and other crops, often in the absence of other herbicides, led to selection for glyphosate-resistant weed biotypes. The most problematic has been Palmer amaranth (Culpepper et al. 2010; Whitaker et al. 2011b). The first glyphosate-resistant Palmer amaranth biotype was confirmed in Georgia in 2005 (Culpepper et al. 2006) and North Carolina in 2006 (Culpepper et al. 2008). Glyphosate-resistant Palmer amaranth has expanded to the cotton growing states of Alabama (2008), Arkansas (2006), Georgia (2005), Mississippi (2008), North Carolina (2005), South Carolina (2006), Tennessee (2006), Texas (2011) and Virginia (2011) (WSSA 2016).

Protoporphyrinogen Oxidase (PPO) Herbicide Use in Cotton

Protoporphyrinogen oxidase (PPO) inhibiting herbicides control weeds by inhibiting protoporphyrinogen oxidase in the chlorophyll biosynthetic pathway, accumulating porphyrins, and enhancing peroxidation of membrane lipids, which leads to irreversible damage of the membrane function and structure (Duke et al. 1991; Grossman et al. 2010, 2011). Increasing infestations of glyphosate- and acetolactate synthase- (ALS) resistant Palmer amaranth in cotton has forced producers to utilize herbicides with alternative modes of action in their management systems (Sosnoskie and Culpepper 2014). Of particular note is the increased use of PPO herbicides and glufosinate for the management of glyphosate-resistant Palmer amaranth. Palmer amaranth can be controlled in systems utilizing glufosinate and PPO herbicides such as flumioxazin and fomesafen (Everman et al. 2009; Gardner et al. 2006; Whitaker et al. 2011a,b). Of the PPO herbicides typically applied PRE or PP, both fomesafen and flumioxazin have been two of the most effective, providing 74 to 100% control 20 d after planting (Whitaker et al. 2011b).

Saflufenacil [2-chloro-4-fluro-5[3-methyl-2,6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]-N-[methyl(propan-2-yl)sulfamoyl]benzamide] is registered for PP, PRE, and POST for control of annual broadleaf weeds before or at planting in numerous row crops and noncropland areas (Grossman et al. 2010, 2011). Currently, the greatest use rates ($\geq 50 \text{ g ai ha}^{-1}$) of saflufenacil are for PRE broadleaf weed control in field corn (*Zea mays* L.). Such rates provide residual control of troublesome and herbicide-resistant broadleaf weeds such as morningglory (*Ipomoea* spp.), tall waterhemp (*Amaranthus tuberculatus* Moq.

Sauer), giant ragweed (*Ambrosia trifida* L.), and velvetleaf (*Abutilon theophrasti* Medik.). Applications of saflufenacil in soybean (*Glycine max* L. Merr.), cereal grains, sorghum (*Sorghum bicolor* L. Moench), and field pea (*Pisum sativum* L.) may be applied from preplant-burndown (PPB) through PRE at rates ≤ 30.3 g ai ha $^{-1}$ (Gannon et al. 2014, Anonymous 2015c). In cotton, saflufenacil may be applied as a PPB application at ≥ 42 days prior to cotton planting (Anonymous 2015c). This application would be targeted for the control of annual broadleaf weeds prior to planting and would not be expected to provide early-season residual control of annual broadleaf weeds such as Palmer amaranth.

Flumioxazin [2-(7-fluoro-3-oxo-4-prop-2-ynl-1,4-benzoxazin-6-yl)-4,5,6,7-tetrahydroisoindole-1,3-dione] is a N-phenylphthalimide herbicide registered for PP and PRE treatments in soybean, peanut (*Arachis hypogaea* L.), corn, and various other crops (Askew et al. 1999; Clewis et al. 2002; Grichar and Colburn 1996; Anonymous 2015d). In cotton, flumioxazin also may be applied PDIR. For PP treatments in cotton, applications are limited to non-till or strip-till fields only, and must be applied 14 to 21 d prior to planting at a rate of 35.5 to 71.0 g ai ha $^{-1}$ (Anonymous 2015d). Flumioxazin PRE controls common lambsquarter (*Chenopodium album* L.), common ragweed (*Ambrosia artemisiifolia* L.), entireleaf morningglory (*Ipomoea hederacea* var. *intergruiscula* L.), ivyleaf morningglory (*Ipomoea hederacea* L. Jacq.), Palmer amaranth, pitted morningglory (*Ipomoea lacunose* L.), prickly sida (*Sida spinosa* L.), smooth pigweed (*Amaranthus hybridus* L.), and tall morningglory (*Ipomoea purpurea* L. Roth) (Askew et al. 2002b; Clewis et al. 2002; Niekamp et al. 1999). Flumioxazin PRE effectively controlled Palmer

amaranth; however, efficacy may be affected by adverse environmental conditions, shortening the duration of control (Dobrow et al. 2011). With reported residual lengths of 16 to 35 d of control of Palmer amaranth (Dobrow et al. 2011), it is beneficial to make applications as close to cotton planting to maximize the residual control benefit into the season. Recent studies (Berger et al. 2012) indicated that flumioxazin application intervals may be shortened with little crop impact at rates of $\leq 30.0 \text{ g ai ha}^{-1}$.

Fomesafen [5-[2-chloro-4-(trifluoromethyl)phenoxy]-N-methylsulfonyl-2-nitrobenzamide] is a diphenyl ether herbicide registered for use PRE and PDIR in cotton at rates of 280 to 420 g ai ha $^{-1}$, and in soybean for POST applications at similar rates (Everman et al. 2009; Stephenson et al. 2004). Fomesafen PRE controlled common cocklebur (*Xanthium strumarium* L.), *Amaranthus* spp., yellow nutsedge (*Cyperus esculentus* L.), prickly sida, and *Ipomoea* spp. (Lunsford et al. 1998). In West Texas, fomesafen may be used PP at 280 g ai ha $^{-1}$ 14 to 21 d prior to planting. A minimum of a 14 d interval must be maintained, and a minimum of 1.27 cm of rainfall or overhead sprinkler irrigation must occur before planting of cotton. Fomesafen also may be applied PRE at 280 g ai ha $^{-1}$ immediately after cotton planting in West Texas provided that 1.27 cm of overhead irrigation is applied prior to cotton cracking the soil surface (Anonymous 2015e)

Soil-Residual Herbicide Behavior

Herbicide efficacy and availability in soil depends much on the soil sorption capacity and affinity between the herbicide molecule and exchange sites on the soil

particle surface (Bailey and White 1970; Calvert 1980; Harper 1994; Peter and Weber 1985; Weber 1970). Most studies that relate herbicide activity to soil organic matter (SOM) (Corbin et al. 1971), humic matter (HM), clay (Weber et al. 1993), pH (Wolcott 1970) suggest that SOM was consistently the factor with the most significant effect on herbicide bioactivity (Parochetti 1973; Rahman and Matthews 1979; Sheets et al. 1962; Stevenson 1972; Weber et al. 1987). Herbicide bioactivity also has been inversely correlated with HM and soil mineral content (Blumhorst et al. 1990; Harper 1994; Peter and Weber 1985; Weber 1970). Soil pH indirectly affected sorption of ionizable herbicides through its effect on the properties of soil particle surfaces and the herbicide (Corbin et al. 1971). Additionally, the level of SOM and clay mineral content may affect cation exchange capacity (CEC), which is the sum of positive charges of absorbed cations for a particular soil type. Therefore, CEC may often be inversely correlated with herbicide bioactivity (Kerr et al. 2004).

Soil bioactivity of other acidic herbicides such as 2,4-D [2-(2,4-dichlorophenoxy)acetic acid], oryzalin [4-(dipropylamino)-3,5-dinitrobenzenesulfonamide], chlorsulfuron [1-(2-chlorophenyl)sulfonyl-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea], metsulfuron-methyl [2-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)carbamoylsulfamoyl]benzoate], and sulfentrazone [N-[2,4-dichloro-5[-4-difluoromethyl]-3-methyl-5-oxo-1,2,4-triazol-1-yl]phenyl]methanesulfonamide] were inversely related to SOM and directly related to pH (Anderson 1985; Anderson and Barrett 1985, Grey et al. 1997; Jorgensen and Hamner 1948; Kerr et al. 2004; Weber et al. 1974). Acid herbicides are

repelled by clays under neutral conditions but sorbed through physical bonding mechanisms under acidic conditions when the compounds are in the molecular form (Bailey and White 1970; Fissel and Bolt 1962; Weber et al. 1993). Therefore, increasing soil pH causes more acidic herbicide anions to remain in soil solution and available for uptake by plants.

Soils differ across cropping regions in the US, often resulting in herbicide rate adjustment to obtain desired efficacy and crop selectivity. Evaluating application rates and timings of soil-applied residual herbicides as influenced by various soil parameters is critical for determining effective rates for weed control, crop selectivity, and recropping intervals (Gannon et al. 2014).

Reasons for Research

Trifludimoxazin [1,5-dimethyl-6-sulfanylidene-3-(2,2,7-trifluoro-3-oxo-4-prop-2-ynyl-1,4-benzoxazin-6-yl)-1,3,5-triazinane-2,4-dione] is a new PPO herbicide under development by BASF Corporation. It has shown excellent residual control of annual broadleaf weeds, including Palmer amaranth when applied PP or PRE. Primary means of crop selectivity is by placement in time and space. Trifludimoxazin has a water solubility of 1.78 mg L^{-1} at 20 degree C with no apparent relationship to pH. Trifludimoxazin has a Koc in the range of 207 to 610 (personal knowledge, interview with BASF).

Flumioxazin has a water solubility of 1.79 mg L^{-1} with no apparent relationship to pH (Mueller et al. 2014), and a Koc of 889 (Anonymous 2016a). Flumioxazin adsorption to soil was most highly correlated with OM, although it can

become readily available in solution with an increase in soil water content (Ferrell et al. 2005). In cotton, with the required 14 to 21 d before planting restriction, and the lower application rate, selectivity is expected to be mostly driven by placement in time and space (personal opinion).

Saflufenacil has a water solubility of 210 mg L⁻¹ at pH 7 and is directly related to pH (greater solubility with an increase in pH). Saflufenacil has a K_{oc} value of 27 (Anonymous 2016b). Observed low sorption to soil and rapid dissipation suggested that saflufenacil would be readily available for degradation or plant uptake in the plant root zone (Mueller et al. 2014). In cotton, with the required 42 d before planting restriction, cotton selectivity is expected to be driven by placement in time and space (personal opinion).

To develop a use pattern for the use of trifludimoxazin in West Texas cotton, an understanding of its behavior in West Texas soils, and an understanding of cotton tolerance is needed. Vertical mobility, soil adsorption, and greenhouse cotton tolerance trials were performed using three West Texas soils, and the results were compared with two commercially used PPO's in cotton, flumioxazin and saflufenacil.

Hypothesis

Trifludimoxazin will behave similar to flumioxazin in the three West Texas soils, and thus could have a similar use pattern in cotton (≥ 14 d PP before cotton planting).

Objective

To develop a use pattern for trifludimoxazin in West Texas cotton by comparing its vertical soil mobility, soil adsorption characteristics, and cotton tolerance to flumioxazin and saflufenacil, two commercially available PPO herbicides for use in West Texas cotton production.

CHAPTER III

MATERIALS AND METHODS

Bulk Soils

Bulk samples were collected from the top 0 to 15 cm of the soil profile for the soils used in these studies, except for the North Carolina sub-soil, which was collected from a depth of 15 to 20 cm. To prepare the soils, each was air dried at room temperature and passed through a 2 mm sieve. Samples were sent to Midwest Laboratories Inc., Omaha, Nebraska 68144-3693 for soil property analysis (Table 3.1).

Herbicide Vertical Mobility in Three West Texas Soils

Comparative vertical mobility of trifludimoxazin, saflufenacil, and flumioxazin was evaluated within each of the three West Texas soils [Acuff (Cotton Center, TX), Olton (Halfway, TX), and Amarillo (Seagraves, TX)] using a bioassay soil column technique. A 15.24 cm-height by 7.62 cm-diam polyvinyl chloride (PVC) soil column was filled with 626.6 cm³ of each soil, weights of each soil listed in Table 3.2. Soil was placed into each column, 1/3 of the total amount at a time and hand-packed between fillings. After filling and packing, a 15 mm headspace remained at the top of each PVC soil column. The packed soil columns were irrigated with the rainfall simulator bringing each soil to field capacity. Total amount of water applied to each soil to bring to field capacity is listed in Table 3.2. Columns were allowed to drain/dry for 48 h. Five ml of stock herbicide solution using herbicide rates shown

in Table 3.3 was applied using a bulb-pipet to the surface of each soil column. Rates represent the current commercial use rate for flumioxazin and saflufenacil and the targeted use rate for trifludimoxazin. After 2 h, columns were placed into the rainfall simulator and 2.54 cm of rainfall (116 ml of water applied to each soil column) was applied over a 40 min duration. Columns were set-aside for 24 h to dry before being split vertically into two halves (Figure 3.1). Two rows of canola (*Brassica napus* L.) was seeded lengthwise into the soil column as the indicator species. Columns were placed into the greenhouse (constant temperature of 28° C, 14 h day-length with supplemental lighting triggered when ambient light reached less than 2,000 watts/m², constant 50% relative humidity) and watered twice daily. Vertical mobility from the soil surface, indicated by the depth to where the indicator species exhibited a visual response (Figure 3.2.), was measured at 7 and 10 d after treatment (DAT).

Comparative Cotton Root Response and Overall Plant Response in Three West Texas Soils

Comparative cotton root response and overall plant response in each of the three West Texas soils was evaluated using a modified slurry technique (Talbert and Fletchall 1965). Ten g of each soil was added to a 50 ml plastic centrifuge tube. Thirty ml of 0.01 M CaCl₂ solution containing herbicide rates defined in Table 3.4 was added to each tube. Tubes were capped and placed onto a horizontal bed shaker (IKA Labortechnik, model HS 500 S1), which operated at 230 motions/min for 10 h. Previous research indicated that flumioxazin sorption was near

instantaneous with approximately 72% being adsorbed to a Greenville clay loam soil after 1 h and reaching a max of 78% at about 71 h (Ferrell et al. 2005). Tubes were centrifuged at 10,000 rpm for 15 min. Twelve ml of the supernatant was transferred into two separate 15 ml test tubes (two subsamples per treatment). Three ml of de-ionized (DI) water was added to each test tube and tubes were capped. A 2 to 3 d old cotton seedling, which had been germinated in vermiculite in the greenhouse, was placed through a hole in the cap with the radical placed in the supernatant and the cotyledons resting on top of the cap. The tubes were placed into growth boxes, which blocked light from all sides except from the top, and placed into a growth chamber (Conviron model E7/2, 14 h day-length, 29° C during the day and 21° C at night, constant relative humidity of 50%, lighting was both fluorescent and incandescent at 375 $\mu\text{mol m}^2/\text{s}$ measured using a LiCor photometer). Supplemental DI water was added to the tubes on an as-needed basis starting at 3 DAT. Cotton root response (comparative tap root and lateral root development and growth versus the nontreated control; 0 to 100% scale with the greater number indicating more inhibition of growth) and overall plant response (comparative tap root and lateral root development and growth, and cotyledon and first true-leaf development and growth versus the nontreated control; 0 to 100% scale with the greater number indicating more inhibition of growth) was recorded at 3, 7, and 10 DAT (Figure 3.3 – 3.4).

Estimated Herbicide Adsorption in Three West Texas Soils

To estimate the amount of each herbicide adsorbed by each of the three West Texas soils, an additional adsorption experiment was conducted with four additional herbicide treatments added to the modified slurry experiment previously described (standard rates listed in Table 3.3). These four additional herbicide treatments represented 20%, 30%, 40%, and 50% of the applied herbicide freely available in the supernatant (80% adsorbed, 70% adsorbed, 60% adsorbed, and 50% adsorbed to the soil, respectively). For these additional soil treatments, supernatant was generated in the same fashion described previously utilizing the modified slurry technique, but with only a stock solution 0.01 M CaCl₂. Twelve ml of supernatant was transferred to the test tubes as before (Figure 3.5). The 3 ml of additional DI solution added to each test tube contained one of the herbicide rates outlined above. This process ensured similar amounts of dissolved nutrients from the soil in the supernatant across all treatments and the only treatment variable was the amount of herbicide in the supernatant. Root response was recorded at 10 DAT.

Comparative Cotton Root Response and Overall Plant Response as Influenced by Differences in Soil Properties

Four additional soils (Table 3.1) were selected along with the three West Texas soils to be included in an additional modified slurry adsorption test. The four additional soils were selected in order to broaden the range in OM, texture, CEC, and pH. Information generated in this experiment would be used to build a step-wise

regression model for the various soil components impacting herbicide adsorption. Root response and overall plant response was recorded at 3, 7, and 10 DAT. Data was pooled across observation dates for analysis in the model.

Greenhouse Cotton Tolerance in Three West Texas Soils

Plastic pots, 10.16 cm by 10.16 cm, were filled with each of the three West Texas soils. Pots were transferred to the greenhouse (same conditions as described in the vertical mobility study), fully watered, and allowed to drain to field capacity. A single cottonseed of Stoneville 4946 GlyTol® LibertyLink® Genuity® Bollgard II® was planted to a depth of 2.54 cm in each plot, immediately before treatment for the PRE treatments and 14 d after application for the PP treatments. To optimize emergence, a planting depth of 0.6 to 3.8 cm was recommended for cotton (Cotton Foundation 2016). Each of the herbicides (trifludimoxazin at 25.0 g ai ha⁻¹, saflufenacil at 25.0 g ai ha⁻¹, and flumioxazin at 35.5 g ai ha⁻¹) were applied using a spray chamber (TeeJet® XR 80015, 140 L ha⁻¹, 275 kPa, 4.8 km h⁻¹). Pots were transferred to the greenhouse and allowed to dry for 2 h prior to receiving an overhead watering of 2.54 cm to activate the herbicide. Pots were watered twice daily for the duration of the experiment. Plant response was recorded at 14 and 28 DAP. Above ground cotton fresh weight was recorded 28 DAP (data not shown) by cutting each plant at the soil surface. Plants were placed into an oven dryer at 32° C for 12 h and dry weights recorded. Dry weight as compared to the nontreated control (NTC) within each soil type was calculated for each plant.

Statistical Analysis

The herbicide vertical mobility in three West Texas soils experiment used a completely randomized design with three replications for each soil by herbicide combination and the experiment was conducted twice. Data were subjected to ANOVA and means separated by Fisher's Protected LSD at the 5% level (SAS 2014).

The comparative soil adsorption in three West Texas soils experiment used a completely randomized design with two sub-samples (pooled for analysis) for each soil by herbicide combination and the experiment was conducted four times. Data were subjected to ANOVA and means separated by Fisher's Protected LSD at the 5% level (SAS 2014).

The estimated adsorption in three West Texas soils experiment used a completely randomized design with two sub-samples (pooled for analysis) for each soil by herbicide rate combination and the experiment was conducted three times. Data were subjected to ANOVA and means separated by Fisher's Protected LSD at the 5% level (SAS 2014).

The multiple soil comparative adsorption experiment used a completely randomized design with two sub-samples (pooled for analysis) for each soil by herbicide combination and the experiment was conducted twice. Data were subjected to forward-selection linear regression (SAS 2014).

The greenhouse cotton tolerance experiment was conducted in a completely randomized design with 3 pots per rep with 3 reps per run. The experiment was

conducted twice. Data were subjected to ANOVA and means separated by Fisher's Protected LSD at the 5% level (SAS 2014).

Table 3.1. Properties of 0 to 15 cm-deep soil samples from each trial site.^a

Series	State	Tex ^b	OM ^c	Sand ^d	Silt	Clay	CEC ^e	pH ^f
				%			cmol kg ⁻¹	1:1
Acuff	TX	L	1.5	45	43	12	22.1	8.3
Amarillo	TX	LS	0.3	86	6	8	8.7	8.2
Candor (sub-soil) ^g	NC	S	1.1	90	4	6	7.7	4.3
Clarion	IA	L	3.2	42	40	18	19.3	7.2
Hastings	NE	SiL	3.0	14	60	26	20.8	5.1
Olton	TX	L	1.0	51	29	20	19.1	8.1
Richfield	OK	SiL	2.1	23	50	27	23.1	7.6

^a Abbreviations: Tex, soil texture; OM, organic matter; CEC, cation exchange capacity; C, clay; L, loam; LS, loamy sand; S, sand; SiL, silt loam.^b Soil textural classification.^c Organic matter was determined by a loss on ignition method (Dean 1974).^d Particle analysis was determined for sand, silt, and clay using the hydrometer method (Gee and Orr 2002).^e Cation exchange capacity was determined using the summation of exchangeable cations procedure (Mehlich 1984).^f pH was determined using a 1 : 1 soil : distilled water ration (Peech 1965).^g Sample taken from the sub-soil at 15 to 25 cm.

Table 3.2. Weights of West Texas soils and volume of water added to each soil column.

Series	Soil weight ^a	Water volume ^b
	----- g -----	----- ml -----
Acuff	755	198
Amarillo	845	125
Olton	741	211

^a Weight of soil added to each soil column to reach a of volume of 626.6 cm³.

^b Volume of water added to each soil column to reach field capacity.

Table 3.3. Herbicide rate used in soil column studies.

Herbicide	1X rate ---- g ai ha ⁻¹ ----	Stock solution ---- g ai L ⁻¹ ----
flumioxazin ^a	35.5	0.0032
saflufenacil ^b	25.0	0.0022
trifludimoxazin ^c	25.0	0.0022

^a 51% ai water dispersible granule formulation, Valent U.S.A. Corporation, Walnut Creek, CA.^b 342 g ai L⁻¹ soluble concentrate (SC) formulation, BASF Corporation, Research Triangle Park, NC.^c 500 g ai L⁻¹ SC formulation, BASF Corporation, Research Triangle Park, NC.

Table 3.4. Herbicide rate used in soil adsorption studies.

Herbicide	1X rate ---- g ai ha ⁻¹ ----	Stock solution ---- µg ai L ⁻¹ ----
flumioxazin ^a	17.8	5.3
saflufenacil ^b	12.5	3.7
trifludimoxazin ^c	12.5	3.7

^a 51% ai water dispersible granule formulation, Valent U.S.A. Corporation, Walnut Creek, CA.^b 342 g ai L⁻¹ soluble concentrate (SC) formulation, BASF Corporation, Research Triangle Park, NC.^c 500 g ai L⁻¹ SC formulation, BASF Corporation, Research Triangle Park, NC.

Figure 3.1. Soil columns split into two vertical halves after the herbicide treatments were applied, watered in, and allowed to dry for 24 hours.



Figure 3.2. Canola injury as a result of protoporphyrinogen oxidase inhibiting herbicide vertical mobility in the Olton soil.

A – nontreated control, B – trifludimoxazin, C – flumioxazin, D – saflufenacil.

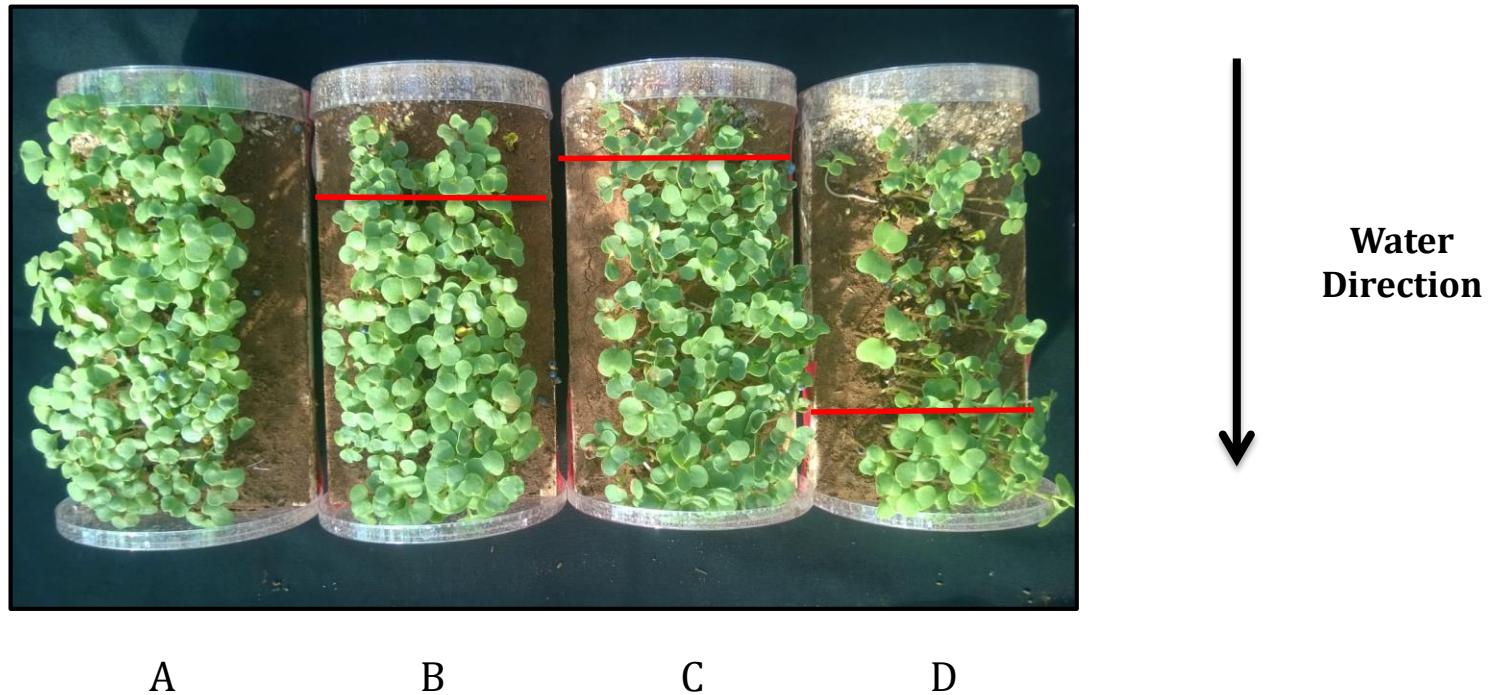


Figure 3.3. Comparative tap root and lateral root development and growth in the Amarillo soil. A – nontreated control, B – trifludimoxazin, C – saflufenacil, D – flumioxazin.

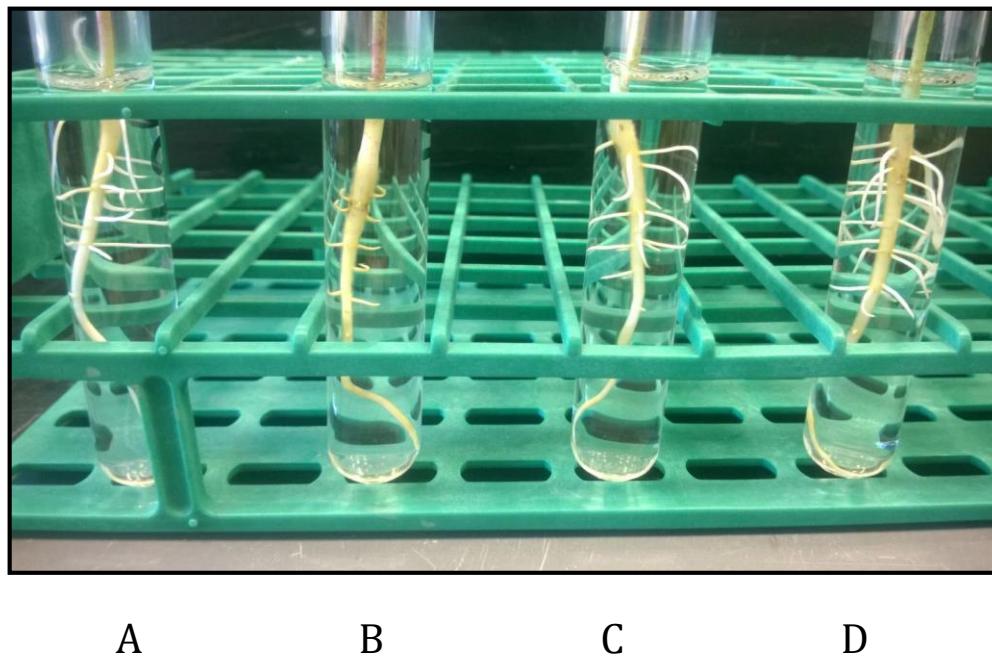


Figure 3.4. Comparative cotyledon and first true-leaf development and growth in the Clarion soil. A – nontreated control, B – trifludimoxazin, C – saflufenacil, D – flumioxazin.



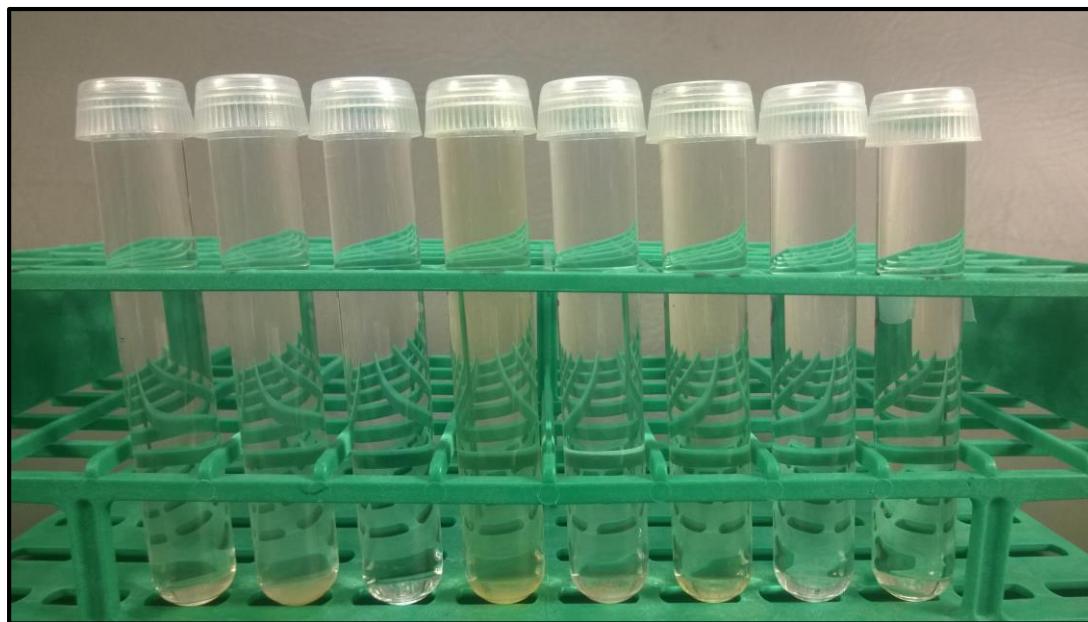
A

B

C

D

Figure 3.5. Visual differences in supernatant between the soils.



CHAPTER IV

RESULTS AND DISCUSSION

Herbicide Vertical Mobility in Three West Texas Soils

Differences in herbicide vertical mobility were observed among herbicide by soil combinations, herbicides, and soils at 7 and 10 DAT. All herbicide by soil combinations that included flumioxazin, regardless of soil type, had similar vertical mobility at 7 and 10 DAT (Table 4.1). Trifludimoxazin mobility in the Acuff and Olton soils (2.5 cm each) was similar to all flumioxazin by soil combinations (1.7 to 2.8 cm) at 10 DAT and were different than all saflufenacil by soil combinations (6.2 to 13.5 cm). At 10 DAT, trifludimoxazin vertical mobility in the Amarillo soil (5.0 cm) was greater than any flumioxazin by soil combination (1.7 to 2.8 cm), trifludimoxazin in the Acuff soil (2.5 cm), or trifludimoxazin in the Olton soil (2.5 cm) (Table 4.1). Saflufenacil vertical mobility was the greatest in the Amarillo soil at 7 (13.4 cm) and 10 DAT (13.5 cm). Vertical mobility of saflufenacil across soils (6.2 to 13.5 cm) was greater than all other herbicide by soil combinations except for the vertical mobility of trifludimoxazin in the Amarillo soil (5.0 cm) at 10 DAT (Table 4.1).

Means for herbicide vertical mobility, summarized across soils, indicated that saflufenacil was the most vertical mobile herbicide (8.6 cm), followed-by trifludimoxazin (2.4 cm), and flumioxazin (1.7 cm) was least mobile at 7 DAT (Table 4.2) (Figure 4.1). Similar results were observed at 10 DAT, with saflufenacil being

the most mobile (9.0 cm), followed by trifludimoxazin (3.3 cm), and flumioxazin (2.1 cm).

Means for herbicide vertical mobility, summarized across herbicides, indicated that the Amarillo soil allowed for the most vertical mobility at 7 (4.8 cm) and 10 (5.3 cm) DAT. No differences in vertical mobility was observed for the Acuff and Olton soils at 7 (2.4 cm) or 10 (2.6 to 2.9 cm) DAT (Table 4.3) (Figure 4.2). The Amarillo soil was the most coarse-textured soil (loamy sand) and contained the least amount of OM (0.3%) of the three West Texas soils tested. These findings are consistent with the documented reports that indicated OM content was consistently the factor with the most significant effect on herbicide bioactivity (Parochetti 1973; Rahman and Matthews 1979; Sheets et al. 1962; Stevenson 1972; Weber et al. 1987). Since trifludimoxazin and flumioxazin are both non-ionic herbicides with similar solubility in water (Table 4.4), they would be expected to have similar vertical mobility profiles. The differences observed (Table 4.3) may be explained by the slight differences in Koc (soil adsorption coefficient) values of the two compounds (Table 4.4).

The water solubility of saflufenacil is pH dependent (greater in higher pH solutions) and it has a lower Koc value; therefore, it should be more mobile in the three soils compared to either trifludimoxazin or flumioxazin. This was confirmed in this study. All three West Texas soils have medium- to fine-soil textures, soil pH values of ≥ 8.1 , and an OM content of $\leq 1.5\%$, which would favor vertical soil mobility of ionic herbicides or those whose mobility is strongly related to OM.

Differences in vertical mobility of the three herbicides are likely due to herbicide chemical composition differences and/or their adsorption to OM.

Comparative Cotton Root Response and Overall Plant Response in Three West Texas Soils

Differences in cotton root response (comparative tap root and lateral root development and growth versus the nontreated control; 0 to 100% scale with the greater number indicating more inhibition of growth) and overall plant response (comparative tap root and lateral root development and growth, and cotyledon and first true-leaf development and growth versus the nontreated control; 0 to 100% scale with the greater number indicating more inhibition of growth) were observed among herbicide by soil combinations, herbicides, and soils at 3, 7, and 10 DAT. These results suggest that there may be differences in intrinsic cotton tolerance among the three herbicides and also differences in herbicide adsorption among the three West Texas soils.

The greatest level of root response was observed with trifludimoxazin across all three soils and was greater than any other herbicide by soil combination at 3, 7, and 10 DAT (Table 4.5). Similar root response (9 to 23%) were observed in the Olton and Acuff soils for both saflufenacil and flumioxazin across all rating dates. Root response was similar in all three soils with saflufenacil (14 to 36%) at all rating dates. Root response was similar in all three soils with flumioxazin (9 to 24%) at all rating dates. Root response was similar in the saflufenacil (14 to 36%) and flumioxazin (14 to 24%) treated Amarillo soil at 3 DAT.

Overall plant response was greater with trifludimoxazin in all three soils (42 to 56%) except for saflufenacil in the Amarillo soil (21%) at 3 DAT, which was similar to trifludimoxazin in the Acuff soil (42%) and the Olton soil (45%) (Table 4.5). At 3 DAT, similar overall plant response was observed with all saflufenacil and flumioxazin by soil combinations (12 to 31%). At 7 DAT, overall plant response was the least in the flumioxazin by Amarillo soil (7%), which was similar to all other flumioxazin and saflufenacil soil combinations except for the overall plant response observed in the saflufenacil by Amarillo combination (23%). At 10 DAT, observed overall plant responses for all flumioxazin and saflufenacil by soil combination treatments were the same (6 to 13%) except for the saflufenacil by Amarillo combination (18%).

Visual plant response across soils by herbicide indicated that the greatest root response and overall plant response was seen with trifludimoxazin at all rating dates (Table 4.6). At 3 DAT, root response and overall plant response was similar for saflufenacil (22% and 19%) and flumioxazin (18% and 14%). At 7 and 10 DAT, flumioxazin caused the least root response (13% and 11%) and overall plant response (9% and 7%) among the three herbicides tested.

When plant response was examined across herbicides by soil, the greatest root response was observed in the Amarillo soil at 3, 7, and 10 DAT (49%, 42%, and 42%, respectively) (Table 4.7). The Amarillo soil has the least OM (0.3%), clay content (8%), and highest pH (8.3) among the three soils. For soil-residual herbicides, these factors are very important for soil adsorption and would explain why the Amarillo soil allowed the greatest root response. This suggests that the

Amarillo soil has the least adsorption capacity of the three West Texas soils tested. Similar root responses were observed in the Acuff (31 to 32%) and Olton soils (34 to 35%) across all rating dates. These soils have similar properties that are important for herbicide adsorption. Overall plant response at 3 DAT was greatest in the Amarillo soil (34%), but was similar to the Olton soil at 7 (23 to 28%) and 10 DAT (26% to 21%). Overall plant response in the Acuff soil was less than the overall plant response in the Amarillo soil, but was similar to the overall plant response observed in the Olton soil across all rating dates.

Similar root response (64 to 72%) and overall plant response (41 to 45%) was observed by trifludimoxazin in both the Acuff and the Olton soils across all rating dates (Table 4.8). The greatest root response (81 to 86%) and overall plant response (52 to 56%) caused by trifludimoxazin occurred in the Amarillo soil across all rating dates. This is an indication that trifludimoxazin was adsorbed similar in the Acuff and Olton soils, but is less adsorbed in the Amarillo soil.

Similar to what was observed with trifludimoxazin, similar root response (14 to 23%) and overall plant response (11 to 15%) was observed following saflufenacil in both the Acuff and the Olton soil across all rating dates (Table 4.9). Initial root and overall plant response at 3 DAT was greatest with saflufenacil in the Amarillo soil (36% and 31%, respectively). At 7 and 10 DAT, root response and overall plant response was similar among the three West Texas soils and ranged from 20 to 30% and 11 to 23%, respectively. The initial difference in root response and overall plant response could indicate that there are differences in adsorption of saflufenacil by the three West Texas soils. Over time, these differences were not detected, which

might indicate that there was rapid breakdown of the herbicide in solution and/or partial cotton tolerance to saflufenacil.

For flumioxazin, similar root response (8 to 24%) and overall plant response (6 to 14%) was observed across all soils at all rating dates (Table 4.10). These results indicate similarities in soil adsorption exist for flumioxazin among the three West Texas soils. A high level of cotton tolerance to flumioxazin could mask any differences in soil adsorption between the three soils.

At 10 DAT, trifludimoxazin was the only herbicide where differences were observed among the three soils (Table 4.8, 4.9, and 4.10). This would indicate there are differences, at least for trifludimoxazin, in adsorption among the three West Texas soils. The initial differences seen in visual plant response, both in root and overall plant response (Table 4.5), suggests there was a lower level of intrinsic cotton tolerance to trifludimoxazin when compared to saflufenacil or flumioxazin. This was further supported by the summary shown in Table 4.10, where by 10 DAT all trifludimoxazin by soil combinations caused greater root response and overall plant response than all other herbicide by soil combinations.

Estimated Adsorption in Three West Texas Soils

Herbicides were evaluated independently for the amount adsorbed in each of the three West Texas soils as earlier experiments indicated there were differences among the three soils based on visual plant responses. Comparing the 1X rate of trifludimoxazin, which is the standard rate applied to the soil and subjected to the modified slurry technique, to the four additional treatments, adsorption estimates

for each of the three West Texas soil based on visual root response between treatments were 70%, 60%, and 50 to 60% for the Acuff, Olton, and Amarillo soils, respectively (Table 4.11, 4.12 and 4.13). When comparing saflufenacil in the same way, estimated soil adsorption was 70% in the Acuff and Olton soils and 60% in the Amarillo soil (Table 4.14, 4.15, and 4.16). Flumioxazin estimated soil adsorption was 70% in the Acuff and Olton soils and 70 to 80% in the Amarillo soil (Table 4.17, 4.18 and 4.19). All three herbicides had similar adsorption in the Acuff soil (70%) (Table 4.11, 4.14, and 4.17). In the Olton soil, trifludimoxazin was adsorbed the least (60%) and saflufenacil and flumioxazin were adsorbed similarly (70%) (Table 4.12, 4.15, and 4.18). In the Amarillo soil, flumioxazin had the greatest adsorption (70 to 80%) and adsorption of trifludimoxazin and saflufenacil were similar (50 to 60% and 60%, respectively) (Table 4.13, 4.16, and 4.19).

Cotton had a good level of intrinsic tolerance to flumioxazin and differences in the amount adsorbed might not be able to be seen visually. Quantitative adsorption amounts need to be measured to definitively determine the adsorption difference among the soils and herbicides. Based on the visual plant responses, it appeared that trifludimoxazin and flumioxazin behaved similar across soils except in the Amarillo soil where trifludimoxazin appeared to be less absorbed.

Comparative Cotton Root Response and Overall Plant Response as Influenced by Differences in Soil Properties

Differences in visual plant response, both for root response and overall response, were observed among herbicide by soil combinations, soils, and herbicides at 3, 7, and 10 DAT. These observed differences suggest that there might be differences in intrinsic cotton tolerance among the herbicides and also differences in herbicide adsorption among the seven soils.

Differences in root response and overall response were observed between the herbicide by soil treatments (Table 4.20). By 10 DAT, all flumioxazin treatments regardless of soil had low levels of root response (0 to 10%) and overall response (1 to 6%) and were similar. At 10 DAT, all saflufenacil treatments caused similar root and overall response to the flumioxazin treatments except for in the Amarillo soil, the Acuff soil, and the Candor sub-soil (NC), which all had greater levels of injury. All trifludimoxazin treatments had greater levels of root response (30 to 85%) and overall response (19 to 56%) than treatments with the lowest level of root response and overall response, such as flumioxazin in the Clarion (IA) soil (0% and 1%, respectively) and saflufenacil in the Richfield (OK) soil (4% and 2%, respectively). Differences in levels of root response and overall response between soils with the same herbicide suggests there are differences in herbicide adsorption among the seven soils (Figure 4.3 – 4.4).

When visual plant response was averaged across soils by herbicide, the greatest root response (57%) and overall response (35 to 39%) was observed with trifludimoxazin on all rating dates (Table 4.21). At 3 DAT, root response (14 to 19%) and overall response (12 to 17%) was similar for saflufenacil and flumioxazin. At 7 and 10 DAT, flumioxazin caused the least amount of root response (7%) and

overall response (12%) among the three herbicides tested. This suggests that there could possibly be differences in intrinsic cotton tolerance among the three herbicides.

When visual plant response was averaged across herbicides by soils, the greatest root response was observed in the Amarillo soil at 3, 7, and 10 DAT (55%, 43% and 42%, respectively) (Table 4.22). The Amarillo soil also had the greatest level of overall response across all ratings dates (28 to 38%) and was greater than all other soils except for the Candor sub-soil (22%) at 10 DAT.

Root response and overall response was lowest in the Clarion soil (12 to 13% and 8 to 11%), the Hastings (NE) soil (16 to 20% and 14 to 15%) and the Richfield soil (15 to 19% and 9 to 15%) across all rating dates. These are the three soils with the greatest OM among the seven soils, ranging from 2.1 to 3.0% (Table 3.1). This agrees with previous studies that showed OM was consistently the factor with the most significant effect on herbicide bioactivity (Parochetti 1973; Rahman and Matthews 1979; Sheets et al. 1962; Stevenson 1972; Weber et al. 1987). The three West Texas soils, which have lower levels of OM (0.3 to 1.5%), all had a greater root response (30 to 50%) and overall response (21 to 38%) than the Candor sub-soil, Clarion soil, and Richfield soil.

Pooling data across rating dates and running forward-selection linear regression analysis over the different soil parameters (Table 3.1), the following relationships were identified for each herbicide. For trifludimoxazin, forward-selection linear regression suggested that OM was the only soil parameter that was

significant for predicting root response ($\text{Pr}>\text{F} = 6.31\text{E}-15$) (Table 4.23). The equation for predicting root response for trifludimoxazin was:

Root response predictive equation: R-Square = 0.7849

% root response = 86.2045 - 16.7430 (% OM)

For saflufenacil, forward-selection linear regression suggested that OM, sand (%), and CEC were soil parameters that were significant for predicting root response ($\text{Pr}>\text{F}=5.52\text{E}-10$) (Table 4.24). The equation for predicting root response for saflufenacil was:

Root response predictive equation: R-Square = 0.7370

% root response = 95.1634 - 7.4011 (% OM) - 1.4219 (CEC) - 1.1195 (% clay) - 0.4231 (% sand)

In a previous study conducted by Gannon et al. (2014), multiple regression analysis for different soil properties suggested that a model with OM, pH, and sand (%) was a good fit for predicting plant phytotoxicity to saflufenacil. Further analysis using stepwise linear-regression ultimately showed that OM was the key measure for creating a predictive model for bioactivity.

For flumioxazin, forward-selection linear regression suggested that OM was the only significant soil parameter for predicting root response ($\text{PrF}>0.0006$) (Table 4.25). The equation for predicting root response for flumioxazin was:

Root response predictive equation: R-Square = 0.2559

% root response = 18.5434 - 5.7558 (% OM)

This is similar to what was reported by Ferrell et al. (2005), who found that flumioxazin adsorption was dependent on OM in the soil. The low R-Square value

could be due to the low level of root response and overall response caused by flumioxazin, which would make it difficult to separate treatments and soil variables.

For both of the non-ionic herbicides trifludimoxazin and flumioxazin, OM was the most important factor in predicting the amount of response (an indicator of amount of herbicide adsorbed) each herbicide would cause. For saflufenacil, an ionic herbicide, the additional soil factors of sand (%) and CEC are important for predicting the amount of response that would be seen in different soils.

Greenhouse Cotton Tolerance in Three West Texas Soils

Differences in plant response were observed among herbicide by soil combinations, application timings, herbicides, and soils at 14 and 28 DAP. When herbicides were averaged within application timing, the PRE application was more injurious than the 14 d PP application at both 14 and 28 DAP (40% vs 13% and 43% vs 13%, respectively) (Table 4.26). When application timings were averaged within herbicides, flumioxazin caused the least amount of visual cotton response 14 (14%) and 28 (12%) DAP. Visual cotton response was similar for trifludimoxazin (44%) and saflufenacil (49%) at 14 DAP. At 28 DAP, visual response was greater with saflufenacil (59%) than it was with trifludimoxazin (42%) (Table 4.27). Cotton injury was similar in the three West Texas soil when means were combined across application timings and ranged from 25 to 30% (Table 4.28).

Cotton response at 28 DAP was similar for all PP trifludimoxazin by soil and flumioxazin by soil combination treatments when compared to the nontreated control (NTC) and ranged from 0 to 7% (Table 4.29). The only PRE herbicide by

soil treatment with similar cotton response to the NTC at 28 DAP was flumioxazin in the Olton soil (8%).

All PRE herbicide by soil combination treatments adversely affected cotton when compared to the NTC except flumioxazin in the Acuff soil at 14 DAP (7%) and flumioxazin in the Olton soil at 28 DAP (7%) (Table 4.30, Figure 4.7).

Trifludimoxazin and saflufenacil had similar levels of cotton response across all soil combinations at 28 DAP (65 to 79%) and were greater than any flumioxazin by soil treatment (7 to 36%).

The PRE application of flumioxazin resulted in the least amount of visual cotton response among the three herbicides (24%) at both 14 and 28 DAP (Table 4.31). Trifludimoxazin had the greatest level of response (77%) at 14 DAP, but was similar to saflufenacil at 28 DAT (78% to 72%, respectively). The level of response caused by trifludimoxazin resulted in plant death in some pots (Figure 4.3). The saflufenacil-induced response was leaf necrosis (Figure 4.6-4.7). Visual cotton response was similar across soils at 14 and 28 DAT and ranged from 39 to 46% (Table 4.32).

All PP trifludimoxazin and flumioxazin by soil combinations resulted in similar responses at 14 and 28 DAP (3 to 12%) and were similar to the NTC (Table 4.33). All PP saflufenacil by soil treatments were similar at 14 DAP (33 to 42%) and had greater levels of visual cotton response than any trifludimoxazin or flumioxazin by soil treatment. At 28 DAP, all saflufenacil by soil treatments had greater levels of cotton response (31 to 54%) than any trifludimoxazin or flumioxazin by soil combination and the NTC. The PP saflufenacil treatment in the Acuff and the Olton

soils caused the greatest level of response (54% and 55%, respectively), which was greater than the saflufenacil PP treatment in the Amarillo soil (31%) at 28 DAP.

The PP application of flumioxazin and trifludimoxazin showed similar low levels of visual cotton responses at 14 and 28 DAP (0 to 10%) (Table 4.34). Saflufenacil caused the greatest level of response and the response was 38% at 14 DAP and 47% at 28 DAP. Cotton response was similar across soils at 14 DAP (11 to 13%) for the PP applications (Table 4.35). Response in the Acuff and the Olton soils were similar at 28 DAT (14% and 15%, respectively) and both had greater visual cotton responses than the Amarillo soil (9%).

Above ground cotton dry weight was determined after 28 DAP for all herbicide by soil combination treatments. Across soils, cotton dry weights for trifludimoxazin and saflufenacil PRE were similar and were lower than the NTC (29 to 37% and 27 to 47%, respectively) (Table 4.36). Flumioxazin-induced cotton dry weights were similar to the NTC for the PRE treatments across all three West Texas soils (80 to 105%).

Within each soil, cotton dry weights for trifludimoxazin and flumioxazin PP treatments were similar to the NTC and ranged from 96 to 107% and 103 to 105%, respectively (Table 4.37). This corresponds to the visual cotton response recorded at 28 DAP, which indicated that these treatments were similar (Table 4.29).

Saflufenacil-induced cotton dry weight within each soil were less than the NTC and lower than trifludimoxazin and flumioxazin (Table 4.37). This also corresponded to the cotton response observed at 28 DAP (Table 4.29).

Table 4.1. Vertical mobility of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Rate g ai ha ⁻¹	7 DAT ^b ----- cm -----	10 DAT
flumioxazin	Acuff	35.5	1.2 a	1.7 a
flumioxazin	Amarillo	35.5	2.3 ab	2.8 a
flumioxazin	Olton	35.5	1.6 a	1.9 a
saflufenacil	Acuff	25.0	6.6 c	7.2 c
saflufenacil	Amarillo	25.0	13.4 d	13.5 d
saflufenacil	Olton	25.0	5.7 c	6.2 bc
trifludimoxazin	Acuff	25.0	1.6 a	2.5 a
trifludimoxazin	Amarillo	25.0	3.4 b	5.0 b
trifludimoxazin	Olton	25.0	2.2 ab	2.5 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.^b Abbreviation: DAT, days after treatment.Table 4.2. Vertical mobility of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils averaged across herbicides.^{a,b}

Soil	Rate g ai ha ⁻¹	7 DAT ^c ----- cm -----	10 DAT
Acuff	25.0	2.4 a	2.9 a
Amarillo	35.5	4.8 b	5.3 b
Olton	25.0	2.4 a	2.6 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).^c Abbreviation: DAT, days after treatment.

Table 4.3. Vertical mobility of three protoporphyrinogen oxidase inhibiting herbicides averaged across three West Texas soils.^{a,b}

Treatment	Rate	7 DAT ^c	10 DAT
	g ai ha ⁻¹	----- cm -----	
flumioxazin	35.5	1.7 a	2.1 a
saflufenacil	25.0	8.6 c	9.0 c
trifludimoxazin	25.0	2.4 b	3.3 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviation: DAT, days after treatment.

Table 4.4. Active ingredient properties of three protoporphyrinogen oxidase inhibiting herbicides.

Name	Water Solubility	Adsorption Coefficient	Ionic
	mg L ⁻¹	Koc ^a	
flumioxazin	1.79	889	No
saflufenacil	201 @ pH7	27	Yes
trifludimoxazin	1.78	207 - 610	No

^a Abbreviations: Koc, adsorption coefficient.

Table 4.5. Cotton root and overall plant response with three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Rate	3 DAT ^b		7 DAT		10 DAT	
			Root	Overall	Root	Overall	Root	Overall
		g ai ha ⁻¹			%			
flumioxazin	Acuff	17.8	14 a	13 a	11 a	8 ab	9 a	6 a
flumioxazin	Amarillo	17.8	24 a	14 a	14 a	7 a	12 a	7 ab
flumioxazin	Olton	17.8	16 a	13 a	16 ab	13 ab	11 a	8 ab
saflufenacil	Acuff	12.5	16 a	15 a	22 ab	14 ab	23 ab	13 ab
saflufenacil	Amarillo	12.5	36 a	31 ab	31 b	23 b	30 b	18 b
saflufenacil	Olton	12.5	14 a	12 a	21 ab	11 ab	20 ab	11 ab
trifludimoxazin	Acuff	12.5	65 b	42 bc	64 c	43 c	64 c	41 c
trifludimoxazin	Amarillo	12.5	86 b	56 c	81 d	54 c	83 d	52 c
trifludimoxazin	Olton	12.5	72 b	45 bc	69 cd	44 c	70 c	44 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviation: DAT, days after treatment.

Table 4.6. Cotton root and overall plant response averaged across three West Texas soils for three protoporphyrinogen oxidase inhibiting herbicides.^{a,b}

Treatment	Rate	3 DAT ^c		7 DAT		10 DAT	
		Root	Overall	Root	Overall	Root	Overall
	g ai ha ⁻¹			%			
flumioxazin	17.8	18 a	14 a	13 a	9 a	11 a	7 a
saflufenacil	12.5	22 a	19 a	25 b	16 b	24 b	14 b
trifludimoxazin	12.5	74 b	48 b	72 c	47 c	73 c	46 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviation: DAT, days after treatment.

Table 4.7. Cotton root and overall plant response averaged across three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^{a,b}

Soil	3 DAT ^c		7 DAT		10 DAT	
	Root	Overall	Root	Overall	Root	Overall
			%			
Acuff	31 a	24 a	32 a	22 a	32 a	20 a
Amarillo	49 b	34 b	42 b	28 b	42 b	26 b
Olton	34 a	23 a	35 a	23 ab	34 a	21 ab

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).

^c Abbreviation: DAT, days after treatment.

Table 4.8. Cotton root and overall plant response with trifludimoxazin in three West Texas soils.^a

Treatment	Rate	3 DAT ^b		7 DAT		10 DAT	
		Root	Overall	Root	Overall	Root	Overall
	g ai ha ⁻¹				%		
Acuff	12.5	65 a	42 a	64 a	43 a	64 a	41 a
Amarillo	12.5	86 b	56 b	81 b	54 b	83 b	52 b
Olton	12.5	72 a	45 a	69 a	44 a	70 a	44 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviation: DAT, days after treatment.

Table 4.9. Cotton root and overall plant response with saflufenacil in three West Texas soils.^a

Treatment	Rate	3 DAT ^b		7 DAT		10 DAT	
		Root	Overall	Root	Overall	Root	Overall
	g ai ha ⁻¹				%		
Acuff	12.5	16 a	15 a	23 a	14 a	23 a	13 a
Amarillo	12.5	36 b	31 b	30 a	23 a	30 a	18 a
Olton	12.5	14 a	12 a	20 a	11 a	20 a	11 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviation: DAT, days after treatment.

Table 4.10. Cotton root and overall plant response with flumioxazin in three West Texas soils.^a

Treatment	Rate	3 DAT ^b		7 DAT		10 DAT	
		Root	Overall	Root	Overall	Root	Overall
	g ai ha ⁻¹	-----	-----	% -----			
Acuff	17.8	14 a	13 a	11 a	8 a	9 a	6 a
Amarillo	17.8	24 a	14 a	14 a	8 a	12 a	7 a
Olton	17.8	16 a	13 a	16 a	13 a	11 a	8 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviation: DAT, days after treatment.

Table 4.11. Cotton root response with trifludimoxazin in the Acuff soil at 10 days after treatment.^a

Treatment	Rate	Root	
		g ai ha ⁻¹	% -----
50% adsorbed	6.3		77 c
60% adsorbed	5.0		73 c
70% adsorbed	3.8		63 b
80% adsorbed	2.5		43 a
1X rate to soil	12.5		63 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.12. Cotton root response with trifludimoxazin in the Olton soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	6.3	77 d
60% adsorbed	5.0	71 cd
70% adsorbed	3.8	57 b
80% adsorbed	2.5	42 a
1X rate to soil	12.5	67 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.13. Cotton root response with trifludimoxazin in the Amarillo soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	6.3	88 d
60% adsorbed	5.0	79 c
70% adsorbed	3.8	64 b
80% adsorbed	2.5	48 a
1X rate to soil	12.5	83 cd

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.14. Cotton root response with saflufenacil in the Acuff soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	6.3	43 d
60% adsorbed	5.0	37 cd
70% adsorbed	3.8	29 bc
80% adsorbed	2.5	11 a
1X rate to soil	12.5	27 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.15. Cotton root response with saflufenacil in the Olton soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	6.3	50 d
60% adsorbed	5.0	38 c
70% adsorbed	3.8	32 bc
80% adsorbed	2.5	14 a
1X rate to soil	12.5	28 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.16. Cotton root response with saflufenacil in the Amarillo soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	6.3	56 d
60% adsorbed	5.0	30 c
70% adsorbed	3.8	16 b
80% adsorbed	2.5	7 a
1X rate to soil	12.5	27 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.17. Cotton root response with flumioxazin in the Acuff soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ---- % ----
50% adsorbed	8.9	37 c
60% adsorbed	7.1	32 c
70% adsorbed	5.3	14 ab
80% adsorbed	3.6	8 a
1X rate to soil	17.8	16 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.18. Cotton root response with flumioxazin in the Olton soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ----- % -----
50% adsorbed	8.9	32 d
60% adsorbed	7.1	22 c
70% adsorbed	5.3	13 b
80% adsorbed	3.6	6 a
1X rate to soil	17.8	14 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.19. Cotton root response with flumioxazin in the Amarillo soil at 10 days after treatment.^a

Treatment	Rate g ai ha ⁻¹	Root ----- % -----
50% adsorbed	8.9	35 d
60% adsorbed	7.1	22 c
70% adsorbed	5.3	14 b
80% adsorbed	3.6	7 a
1X rate to soil	17.8	13 ab

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

Table 4.20. Cotton root and overall plant response with three protoporphyrinogen oxidase inhibiting herbicides in seven different soils.^a

Treatment	Soil	Rate	3 DAT ^b		7 DAT		10 DAT	
			Root	Overall	Root	Overall	Root	Overall
flumioxazin	Acuff	17.8	11 abc	8 abc	5 ab	5 ab	3 ab	3 a
flumioxazin	Amarillo	17.8	35 d	21 bcde	13 abc	7 ab	10 abc	6 ab
flumioxazin	Candor	17.8	20 abcd	8 abc	9 abc	5 ab	6 ab	4 ab
flumioxazin	Clarion	17.8	1 a	5 a	0 a	1 a	0 a	1 a
flumioxazin	Hastings	17.8	3 a	8 abc	3 ab	3 ab	3 ab	1 a
flumioxazin	Olton	17.8	20 abcd	17 abcde	14 abc	12 abc	8 abc	6 ab
flumioxazin	Richfield	17.8	4 a	8 abc	8 abc	4 ab	3 ab	1 a
saflufenacil	Acuff	12.5	19 abcd	22 cde	20 bcd	14 bc	21 cd	14 bcd
saflufenacil	Amarillo	12.5	45 de	40 ghi	34 de	29 e	33 de	21 d
saflufenacil	Candor	12.5	28 bcd	17 abcde	30 cde	5 ab	31 de	19 cd
saflufenacil	Clarion	12.5	9 ab	9 abc	6 ab	4 ab	6 ab	4 ab
saflufenacil	Hastings	12.5	3 a	7 ab	13 abc	10 abc	9 abc	6 ab
saflufenacil	Olton	12.5	16 abcd	12 abcd	16 bc	9 abc	15 abc	9 abc
saflufenacil	Richfield	12.5	11 abc	11 abc	6 ab	3 ab	4 ab	2 a
trifludimoxazin	Acuff	12.5	69 ef	44 hi	65 f	45 g	66 g	46 gh
trifludimoxazin	Amarillo	12.5	85 f	53 i	81 g	59 h	84 h	56 h
trifludimoxazin	Candor	12.5	60 e	37 fgh	61 f	43 fg	63 g	43 fg
trifludimoxazin	Clarion	12.5	30 cd	18 abcde	30 cde	18 cd	30 de	19 cd
trifludimoxazin	Hastings	12.5	40 d	28 efg	45 e	33 ef	48 f	33 ef
trifludimoxazin	Olton	12.5	75 ef	44 hi	71 fg	47 g	73 gh	48 gh
trifludimoxazin	Richfield	12.5	43 de	26 def	43 e	28 de	38 ef	24 de

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviation: DAT, days after treatment.

Table 4.21. Cotton root and overall plant response averaged across seven soils for three protoporphyrinogen oxidase inhibiting herbicides.^{a,b}

Treatment	Rate g ai ha ⁻¹	<u>3 DAT^c</u>		<u>7 DAT</u>		<u>10 DAT</u>	
		Root	Overall	Root	Overall	Root	Overall
flumioxazin	17.8	14 a	12 a	7 a	5 a	4 a	3 a
saflufenacil	12.5	19 a	17 a	18 b	13 b	17 b	11 b
trifludimoxazin	12.5	57 b	35 b	57 c	39 c	57 c	38 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Soils included Acuff, Amarillo, Candor, Clarion, Hastings, Olton, Richfield.

^c Abbreviation: DAT, days after treatment.

Table 4.22. Cotton root and overall plant response averaged across three protoporphyrinogen oxidase inhibiting herbicides in seven different soils.^{a,b}

Soil	3 DAT ^c		7 DAT		10 DAT	
	Root	Overall	Root	Overall	Root	Overall
-----%-----						
Acuff	33 b	25 b	30 b	21 bc	30 b	21 b
Amarillo	55 c	38 c	43 c	32 d	42 c	28 c
Candor	36 b	24 b	33 b	22 c	33 b	22 bc
Clarion	13 a	11 a	12 a	8 a	12 a	8 a
Hastings	16 a	14 a	20 a	15 ab	20 a	14 a
Olton	37 b	24 b	34 b	23 c	32 b	21 b
Richfield	19 a	15 a	19 a	12 a	15 a	9 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).

^c Abbreviation: DAT, days after treatment.

Table 4.23. Summary of forward selection regression analysis for trifludimoxazin for cotton root response across seven soils.^a

Step	Variable ^b	Partial R-Square	Pr>F
1	% OM	0.7849	6.31E-15
2	% Silt	0.0156	0.0883
3	% Clay	0.0162	0.0750
4	CEC	0.0113	0.1274
5	pH	0.0042	0.3492

^a Soils included Acuff, Amarillo, Candor, Clarion, Hastings, Olton, Richfield.

^b Abbreviations: CEC, cation exchange capacity; OM, organic matter.

Table 4.24. Summary of forward selection regression analysis for saflufenacil for cotton root response across seven soils.^a

Step	Variable ^b	Partial R-Square	Pr>F
1	% Sand	0.6221	5.52E-10
2	% OM	0.0473	0.0232
3	% Clay	0.0212	0.1144
4	CEC	0.0462	0.0150
5	pH	0.0069	0.3300

^a Soils included Acuff, Amarillo, Candor, Clarion, Hastings, Olton, Richfield.

^b Abbreviations: CEC, cation exchange capacity; OM, organic matter.

Table 4.25. Summary of forward selection regression analysis for flumioxazin for cotton root response across seven soils.^a

Step	Variable ^b	Partial R-Square	Pr>F
1	% OM	0.2559	0.0006

^a Soils included Acuff, Amarillo, Candor, Clarion, Hastings, Olton, Richfield.

^b Abbreviation: OM, organic matter.

Table 4.26. Cotton response averaged across three West Texas soils for three protoporphyrinogen oxidase inhibiting herbicides applied preplant and preemergence.^{a,b}

Application Timing	14 DAT ^c	28 DAT
----- % -----		
preplant	13 a	13 a
preemergence	40 b	43 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviation: DAT, days after treatment.

Table 4.27. Cotton response averaged across application timings for three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^{a,b}

Treatment	Rate	<u>14 DAT^c</u>	<u>28 DAT</u>
		PRE + PP	PRE + PP
	g ai ha ⁻¹	-----%	-----%
flumioxazin	35.5	14 a	12 a
saflufenacil	25.0	49 b	59 c
trifludimoxazin	25.0	44 b	42 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviations: DAT, days after treatment; PP, preplant; PRE, preemergence.

Table 4.28. Cotton response averaged across application timings in three West Texas soils for three protoporphyrinogen oxidase inhibiting herbicides.^{a,b}

Soil	<u>14 DAT^c</u>	<u>28 DAT</u>
	PRE + PP	PRE + PP
	-----%	-----%
Acuff	25 a	29 a
Amarillo	30 a	27 a
Olton	25 a	28 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).

^c Abbreviations: DAT, days after treatment; PP, preplant; PRE, preemergence.

Table 4.29. Cotton response from preemergence and preplant applications of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Application Timing ^b	Rate	14 DAT	28 DAT
			g ai ha ⁻¹	----- % -----	
flumioxazin	Acuff	PRE	35.5	33 b	28 b
flumioxazin	Acuff	PP	35.5	0 a	0 a
flumioxazin	Amarillo	PRE	35.5	34 b	36 b
flumioxazin	Amarillo	PP	35.5	8 a	0 a
flumioxazin	Olton	PRE	35.5	7 a	8 a
flumioxazin	Olton	PP	35.5	1 a	0 a
saflufenacil	Acuff	PRE	25.0	48 b	65 cd
saflufenacil	Acuff	PP	25.0	38 b	54 c
saflufenacil	Amarillo	PRE	25.0	70 cd	71 cd
saflufenacil	Amarillo	PP	25.0	13 a	31 b
saflufenacil	Olton	PRE	25.0	61 c	79 d
saflufenacil	Olton	PP	25.0	42 b	55 c
trifludimoxazin	Acuff	PRE	25.0	74 cd	79 d
trifludimoxazin	Acuff	PP	25.0	5 a	3 a
trifludimoxazin	Amarillo	PRE	25.0	80 d	76 d
trifludimoxazin	Amarillo	PP	25.0	8 a	5 a
trifludimoxazin	Olton	PRE	25.0	77 cd	79 d
trifludimoxazin	Olton	PP	25.0	13 a	7 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviations: DAT, days after treatment; PP, preplant; PRE, preemergence.

Table 4.30. Cotton response from preemergence applications of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Rate	<u>14 DAT^b</u>	<u>28 DAT</u>
			PRE	PRE
		g ai ha ⁻¹	----- % -----	
flumioxazin	Acuff	35.5	33 b	28 b
flumioxazin	Amarillo	35.5	34 b	36 b
flumioxazin	Olton	35.5	7 a	8 a
saflufenacil	Acuff	25.0	48 bc	65 c
saflufenacil	Amarillo	25.0	70 d	71 c
saflufenacil	Olton	25.0	61 cd	79 c
trifludimoxazin	Acuff	25.0	74 d	79 c
trifludimoxazin	Amarillo	25.0	80 d	76 c
trifludimoxazin	Olton	25.0	77 d	79 c

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviations: DAT, days after treatment; PRE, preemergence.

Table 4.31. Cotton response from preemergence applications averaged across three West Texas soils for three protoporphyrinogen oxidase inhibiting herbicides.^{a,b}

Treatment	Rate	<u>14 DAT^c</u>		<u>28 DAT</u>	
		PRE	%	PRE	%
	g ai ha ⁻¹		-----		-----
flumioxazin	35.5	24 a		24 a	
saflufenacil	25.0	59 b		72 b	
trifludimoxazin	25.0	77 c		78 b	

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviations: DAT, days after treatment; PRE, preemergence.

Table 4.32. Cotton response from preemergence applications averaged across three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^{a,b}

Treatment		<u>14 DAT^c</u>		<u>28 DAT</u>	
		PRE	%	PRE	%
			-----		-----
Acuff		39 a		43 a	
Amarillo		46 a		46 a	
Olton		36 a		41 a	

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).

^c Abbreviations: DAT, days after treatment; PRE, preemergence.

Table 4.33. Cotton response from preplant applications of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Rate	<u>14 DAT^b</u>		<u>28 DAT</u>	
			PP	%	PP	%
		g ai ha ⁻¹		----- % -----		
flumioxazin	Acuff	35.5	0 a	10 a		
flumioxazin	Amarillo	35.5	1 a	12 a		
flumioxazin	Olton	35.5	1 a	11 a		
saflufenacil	Acuff	25.0	38 b	54 c		
saflufenacil	Amarillo	25.0	33 b	31 b		
saflufenacil	Olton	25.0	42 b	55 c		
trifludimoxazin	Acuff	25.0	5 a	3 a		
trifludimoxazin	Amarillo	25.0	13 a	5 a		
trifludimoxazin	Olton	25.0	13 a	7 a		

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviations: DAT, days after treatment; PP, preplant.

Table 4.34. Cotton response from preplant applications averaged across three West Texas soils for three protoporphyrinogen oxidase inhibiting herbicides.^{a,b}

Treatment	Rate	<u>14 DAT^c</u>		<u>28 DAT</u>	
		PP	%	PP	%
	g ai ha ⁻¹		----- % -----		
flumioxazin	35.5	3 a		0 a	
saflufenacil	25.0	38 b		47 b	
trifludimoxazin	25.0	10 a		5 a	

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three West Texas soils (Acuff, Amarillo, Olton).

^c Abbreviations: DAT, days after treatment; PP, preplant.

Table 4.35. Cotton response from preplant applications averaged across three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^{a,b}

Treatment	<u>14 DAT^c</u>		<u>28 DAT</u>	
	PP	%	PP	%
		----- % -----		
Acuff	11 a		14 b	
Amarillo	13 a		9 a	
Olton	14 a		15 b	

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Three protoporphyrinogen oxidase inhibiting herbicides (flumioxazin, saflufenacil, trifludimoxazin).

^c Abbreviations: DAT, days after treatment; PP, preplant.

Table 4.36. Percent of the nontreated control dry weight from preemergence applications of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Application Timing ^b	Rate g ai ha ⁻¹	% NTC
flumioxazin	Acuff	PRE	35.5	80 a
saflufenacil	Acuff	PRE	25.0	47 b
trifludimoxazin	Acuff	PRE	25.0	37 b
flumioxazin	Olton	PRE	35.5	105 a
saflufenacil	Olton	PRE	25.0	27 b
trifludimoxazin	Olton	PRE	25.0	34 b
flumioxazin	Amarillo	PRE	35.5	94 a
saflufenacil	Amarillo	PRE	25.0	29 b
trifludimoxazin	Amarillo	PRE	25.0	29 b

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviations: NTC, nontreated control; PRE, preemergence.

Table 4.37. Percent of the nontreated control dry weight from preplant applications of three protoporphyrinogen oxidase inhibiting herbicides in three West Texas soils.^a

Treatment	Soil	Application Timing ^b	Rate	
			g ai ha ⁻¹	% NTC
flumioxazin	Acuff	PP	35.5	104 a
saflufenacil	Acuff	PP	25.0	45 b
trifludimoxazin	Acuff	PP	25.0	107 a
flumioxazin	Olton	PP	35.5	103 a
saflufenacil	Olton	PP	25.0	39 b
trifludimoxazin	Olton	PP	25.0	97 a
flumioxazin	Amarillo	PP	35.5	105 a
saflufenacil	Amarillo	PP	25.0	57 b
trifludimoxazin	Amarillo	PP	25.0	96 a

^a Means within a column followed by the same letter are not different according to Fisher's Protected LSD test at P = 0.05.

^b Abbreviations: NTC, nontreated control; PP, preplant.

Figure 4.1. Canola injury as a result of protoporphyrinogen oxidase inhibiting herbicide vertical mobility in the Amarillo soil. A – nontreated control, B – trifludimoxazin, C – flumioxazin, D – saflufenacil.

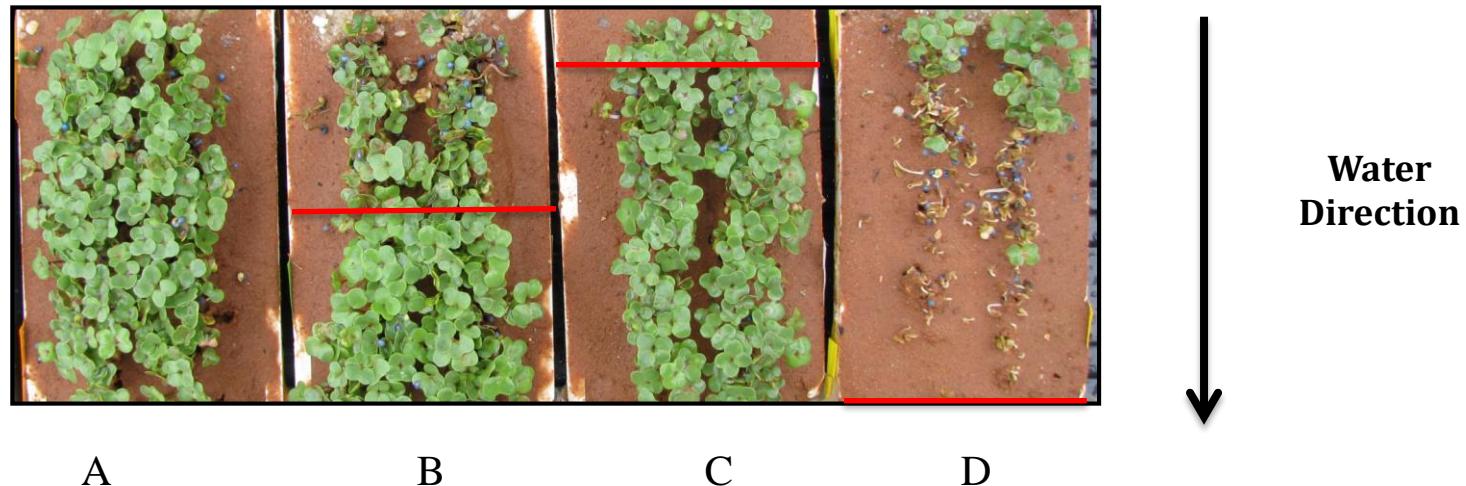
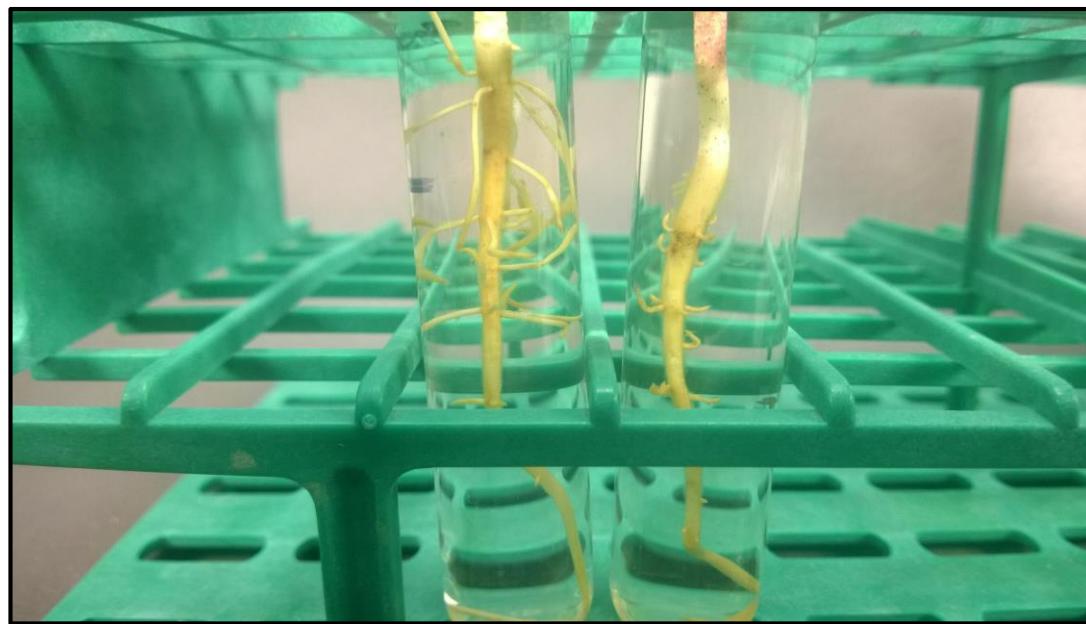


Figure 4.2. Canola injury following trifludimoxazin vertical mobility in three West Texas soils.

A – Acuff, B – Olton, C – Amarillo.



Figure 4.3. Cotton root response to trifludimoxazin in the Amarillo soil. A – nontreated control, B – trifludimoxazin.



A B

Figure 4.4. Cotton root response to trifludimoxazin in the Clarion soil. A – nontreated control, B – trifludimoxazin.

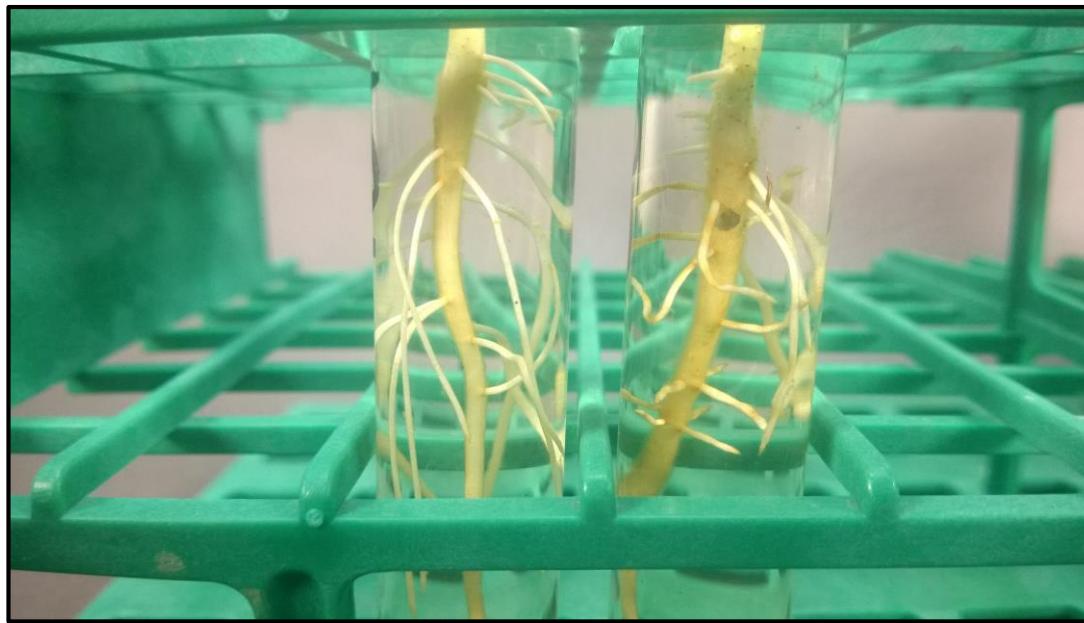


Figure 4.5. Cotton seedling response to three protoporphyrinogen oxidase inhibiting herbicides in the Amarillo soil 14 days after planting. A – trifludimoxazin, B – saflufenacil, C – flumioxazin, D – nontreated control.

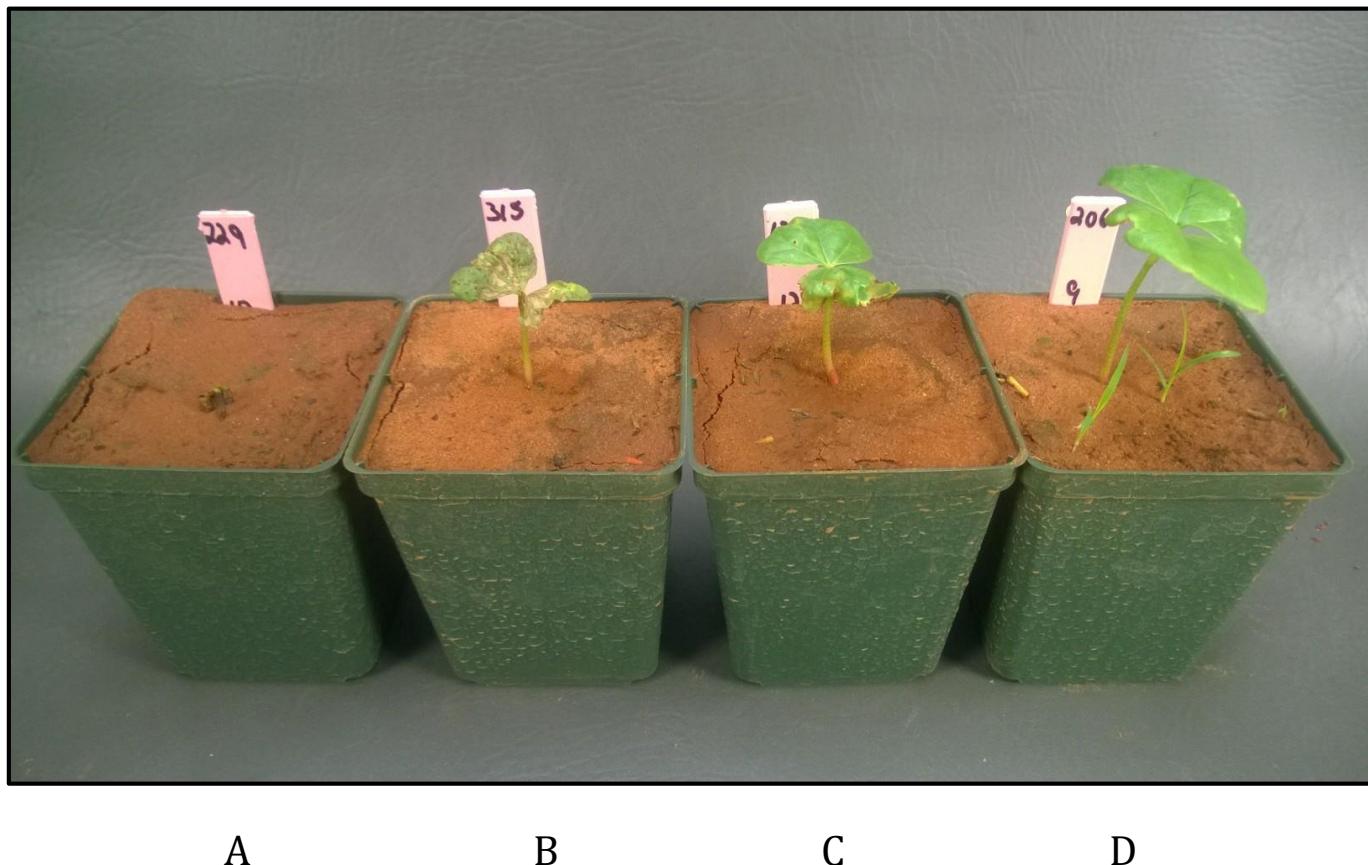


Figure 4.6. Cotton seedling response to saflufenacil in the Acuff soil 14 days after planting. A – nontreated control, B – saflufenacil.



A



B

Figure 4.7. Cotton seedling response to saflufenacil and flumioxzin in Acuff and Olton soils 28 days after planting.

A – nontreated control (Acuff), B – saflufenacil (Acuff), C – nontreated control (Olton), D – flumioxazin (Olton).



A



B



C



D

CHAPTER V

SUMMARY AND CONCLUSION

Differences in vertical soil mobility were observed among three PPO-inhibiting herbicides (flumioxazin, saflufenacil and trifludimoxazin) in three West Texas soils (Acuff, Amarillo, and Olton). Herbicides were most mobile in the Amarillo soil and saflufenacil was the most mobile herbicide across all soils. Individual herbicide by soil combinations indicated that trifludimoxazin and flumioxazin had similar vertical movement profiles in all cases except in the Amarillo soil, where trifludimoxazin was more mobile. Being highly mobile in these soils, saflufenacil moved to a depth where exposure would occur to the germinating cotton seed and radical development during early growth and development. This would prolong the exposure of the cotton seedling to saflufenacil, which might increase the likelihood of cotton response. For flumioxazin, planting cotton at a depth of 2 cm or deeper would create an opportunity for selectivity based on placement, at least in the Acuff and Olton soils, as the herbicide only moved to a depth of 1.7 to 1.9 cm. For these soils, there does not seem to be an opportunity for selectivity based on placement for either trifludimoxazin or saflufenacil as an ideal cotton planting depth of 0.6 to 2.54 cm (Cotton Foundation 2016) would expose the germinating seed to both herbicides.

Trifludimoxazin caused greater levels of root response and overall response across the soils at all observation dates. Flumioxazin had the least amount of root response and overall response across the soils at 7 DAT. The Acuff and Olton soils

allowed similar root responses across the rating dates and allowed less than the root response rating in the Amarillo soil. At 7 and 10 DAT, overall response was less in the Acuff soil than in the Amarillo soil, but response in the Olton soil was similar to all soils. For individual herbicides treatments, overall response differences were not observed among soils for saflufenacil or flumioxazin. Root response and overall plant response was greatest for trifludimoxazin in the Amarillo soil and least in both the Acuff and Olton soils, where results were similar. This strongly indicated that there are differences in the amount of trifludimoxazin being adsorbed by the soils, with the Amarillo soil adsorbing the least amount. When taking into account all herbicide by soil combination treatments, all trifludimoxazin by soil combinations had greater levels of root response and overall response at 10 DAT.

When comparing the root response following the 1X rate applied to the soil to the root response in the adsorbed treatments (which simulated different herbicide amounts being free in the soil solution), an estimate of the amount of each herbicide being adsorbed in the three West Texas soils can be determined. For trifludimoxazin, it is estimated that the Acuff soil adsorbed 70% of the herbicide, the Olton soil absorbed 60% of the herbicide, and the Amarillo soil adsorbed 50 to 60% of the herbicide. For saflufenacil, the estimated adsorption in the Acuff, Olton, and Amarillo soils was 70%, 70%, and 60%, respectively. For flumioxazin, it is estimated that the Acuff and the Olton soils adsorbed 70% of the herbicide and the Amarillo soil adsorbed 70 to 80% of the herbicide.

Trifludimoxazin caused the greatest levels of root response and overall response across the expanded soils tested, and the same trend was observed in the

three West Texas soils. Flumioxazin had the least amount of root response and overall response. The three soils with 2.0% OM or more (Clarion, Hastings, and Richfield) resulted in the least amount of root response and overall plant response. Forward-selection linear regression identified OM as the only significant soil parameter for predicting root response for both trifludimoxazin and flumioxazin. For saflufenacil, additional soil parameters of percent sand and CEC also were significant for predicting root response. This would indicate the amount and type of clay present, which directly impacts CEC, is a factor for the possible adsorption of saflufenacil.

Preemergence herbicide treatments resulted in greater levels of cotton response than did the PP treatments across soils. Preemergence treatments of trifludimoxazin and saflufenacil resulted in similar high levels of cotton response across soils at 28 DAT (78 and 72%, respectively), which were much greater than flumioxazin (24%). The only PRE treatment that had similar cotton response to the nontreated control at 28 DAT was flumioxazin in the Olton soil. Plant dry weights, as a percent of the nontreated control within each soil, were only the same for the flumioxazin PRE treatments. All PP treatments of trifludimoxazin and flumioxazin when averaged across soils had similar levels of cotton response to the control at 14 and 28 DAT. Plant dry weights, as a percent of the check within each soil, were the same for all trifludimoxazin and flumioxazin PP treatments. All saflufenacil PP treatments resulted in greater cotton response and reduced dry weights when compared to the control.

This research confirmed the need for the current ≥ 42 d PP cotton plant back restriction following saflufenacil. The soil vertical mobility of saflufenacil does not offer the opportunity for selectivity based on placement for a PRE or 14 d PP applications in cotton. This work supports the work reported by Berfe et al. (2012), where possibly the current PP application window of 14 to 21 d could be shortened with little crop impact for flumioxazin. Along with a PP planting interval, placement selectivity with flumioxazin with planting depths of 2 cm or greater might be achieved.

To achieve selectivity in cotton with trifludimoxazin, a PP application window must be implemented. Selectivity based on placement will not be possible as the vertical mobility of trifludimoxazin was ≥ 2.5 cm in all three West Texas soils. To refine the possible use pattern even further for trifludimoxazin in cotton, more work is needed to define the PP application window and if a use rate-range can be created for different levels of OM in the soil. As the current commercial label stands for flumioxazin use in cotton, trifludimoxazin may be used in a similar manner without adverse impact to cotton.

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