

**Investigations on the Interactions of Acetolactate Synthase (ALS)-Inhibiting Herbicides with  
Growth Regulator and non ALS-Inhibiting Herbicides in Corn (*Zea mays*) and Selected  
Weeds**

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Mark A. Isaacs

(ABSTRACT)

Herbicide combinations are common in corn production in the United States to control broadleaf and grass weed species. Studies were conducted in 1995 and 1996 to: (1) investigate the interactions of 2,4-D and dicamba with halosulfuron-methyl on common lambsquarters and common ragweed control in corn, (2) determine the effect of 2,4-D on the foliar absorption, translocation, and metabolism of <sup>14</sup>C halosulfuron-methyl in common lambsquarters, (3) examine the interactions of 2,4-D, dicamba, and ALS-inhibitor herbicides with rimsulfuron plus thifensulfuron-methyl (RT) and with sethoxydim on giant foxtail, common ragweed, and common lambsquarters control in corn. Combinations of halosulfuron-methyl with 2,4-D or dicamba were generally additive in their effects on common lambsquarters and common ragweed control, and were occasionally synergistic on common lambsquarters. Synergistic herbicide interactions in the greenhouse were observed with 2,4-D (17 g/ha) and halosulfuron-methyl (18 g/ha) and 2,4-D (70 g/ha) in combination with halosulfuron-methyl at 4.5 and 36 g/ha, respectively. Absorption and translocation of <sup>14</sup>C-halosulfuron-methyl were not influenced by the addition of 2,4-D, with absorption increasing with time. Three unknown halosulfuron-methyl metabolites (M1, M2, and M3) with R<sub>f</sub> values of 0.0, 0.97, and 0.94, respectively, were isolated. The addition of 2,4-D increased the level of M3 at the 18 g/ha halosulfuron-methyl rate, which may contribute to common lambsquarters phytotoxicity. Antagonism on giant foxtail control was observed with all combinations of RT and 2,4-D. Tank mixtures of RT with flumetsulam plus clopyralid plus 2,4-D, atrazine, 2,4-D, and dicamba plus atrazine controlled giant foxtail  $\leq 78\%$  65 (DAT). RT mixed with flumetsulam plus clopyralid plus 2,4-D injured corn 26%, and yields were reduced 34% when compared to RT alone. Giant foxtail control from sethoxydim tank-mixed with bentazon plus atrazine with urea ammonium nitrate (UAN), or with ALS-inhibiting herbicides except halosulfuron-methyl in combination with 2,4-D was 24% lower when averaged over treatments. Yields of sethoxydim-resistant (SR) corn treated with sethoxydim mixed with combinations of

sulfonylurea herbicides plus 2,4-D were low, with the exception of the combination halosulfuron-methyl with sethoxydim and 2,4-D. These studies indicate that thoroughly understanding postemergence (POST) corn herbicide tank mixtures is crucial for effective weed management.

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# Chapter I

## Introduction and Review of Literature

### Herbicide Combinations

Herbicide combinations are commonly used in corn production in the United States to control difficult broadleaf and grass weed species. Hatzios and Penner (1985) list several advantages of tank-mix combinations which include improved spectrum of weed control and reductions in crop production costs, soil compaction, and herbicide residues. New active ingredients are occasionally introduced and are being mixed with a growing number of low-cost, post-patent herbicides for broad-spectrum, season-long weed control. Previous research has demonstrated the benefit of utilizing reduced rates of selected herbicides in mixtures to enhance weed control and to diminish the development of resistant weed biotypes (Baldwin et al. 1985; Defelice et al. 1989; Feldick and Kapusta 1986; Gressel 1990; Starke and Oliver 1998).

### Herbicide Interactions

When two or more herbicides are tank-mixed and applied together and the resulting control is more than the expected control of the individual herbicides applied alone, the combination is said to be synergistic; when less than expected, it is antagonistic (Colby 1967). If the observed and expected responses are equal, the combination is additive. Zang et al. (1995) examined 479 examples of herbicide interactions involving 126 different herbicides and 76 different plant species from 24 families. Synergistic interactions were reported in all (12) studies involving the Chenopodiaceae family and in 25% of the Compositae family. Antagonistic interactions occurred more frequently when the target plants were monocots, and were reported in 80 and 73% of studies involving the Gramineae and Compositae families, respectively. Hart and Penner (1993) observed that the efficacy of primisulfuron {2-[[[[4,6-bis(difluoromethoxy)-2-pyrimidinyl]amino]carbonyl]amino]sulfonyl]benzoic acid } was reduced by 15 and 16% when applied at 30 or 60 g ai/ha, respectively, in combination with 1700 g ai/ha atrazine { 6-chloro-N-ethyl-N'-(1-methylethyl)-1,3,5-triazine-2,4-diamine } to giant foxtail. Young and Hart (1997) reported 48% reduction in giant foxtail control with combinations of sethoxydim { 2-[1-

(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one } with primisulfuron plus CGA 152005 { 1-(4-methoxy-6-methyl-triazin-2-yl)-3-[2-(3,3,3,-trifluoropropyl)-phenylsulfonyl]urea } in sethoxydim-resistant corn.

### **Halosulfuron-methyl**

Halosulfuron-methyl is a sulfonylurea herbicide that controls broadleaf weeds and sedges postemergence (POST) in field corn, grain sorghum [*Sorghum bicolor* (L.) Moench], turf, and sugarcane (*Saccharum officinarum* L.) (Anonymous 1994). Application rates ranging from 18 to 70 g/ha control common cocklebur (*Xanthium strumarium* L.), velvetleaf (*Abutilon theophrasti* Medicus), common ragweed (*Ambrosia artemisiifolia* L.), giant ragweed (*Ambrosia trifida* L.), pigweeds (*Amaranthus* spp.), and yellow nutsedge (*Cyperus esculentus* L.) (Ackley et al. 1994; Dubelman et al. 1997; Dutt and Riego 1994; Hart 1997; Majek 1994; Mayonado et al. 1994). Halosulfuron-methyl inhibits acetolactate synthase (ALS) (also called acetohydroxyacid synthase), the first common enzyme in the biosynthesis of the branched-chain amino acids valine, leucine, and isoleucine. (Schloss 1990; Wittenbach and Abell 1999).

### **Rimsulfuron plus Thifensulfuron-methyl (RT)**

Rimsulfuron (12 g/ha) plus thifensulfuron-methyl (6 g/ha) (RT), commercially formulated as Basis<sup>®</sup>, is a pre-packaged sulfonylurea herbicide that controls selected annual broadleaf and grass weeds POST in field corn. (Anonymous 1997). An application rate of 18 g/ha controls barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.], foxtails (*Setaria* spp.), and fall panicum (*Panicum dichotomiflorum* Michx.) at 3- to 5-cm, and common lambsquarters (*Chenopodium album* L.), pigweeds, smartweed (*Polygonum* spp.), velvetleaf, and wild mustard [*Brassica kaber* (DC.) Wheeler] at 3- to 8-cm (Anonymous 1997; Ganske et al. 1996; Kalnay and Glen 1997) by also inhibiting the enzyme ALS.

### **Weed Prevalence and Competition**

Common lambsquarters, common ragweed, and giant foxtail are annual weeds and

members of the Chenopodiaceae, Compositae, and Gramineae families, respectively. These species are prevalent and competitive in 40 crops throughout the world (Holm et al. 1977; Knake 1977). Despite their susceptibility to many herbicides, these weeds persist in corn fields due to prolific seed production and dormancy, the presence of large seed reserves in soil, genetic diversity, and, in the case of common lambsquarters, resistance to triazine herbicides (Barbour et al. 1994; Chu et al. 1978; Darmency 1994; Fausey and Renner 1997; Mester and Buhler 1986; Saini et al. 1986). Successful corn production relies heavily on weed control, and it is estimated that competition from uncontrolled weeds may cause 30 to 90% yield losses (Hall et al. 1992). Beckett et al. (1988) reported a 12% corn yield loss at 49 common lambsquarters/10 m of row. In a Canadian study, corn yield decreased when common lambsquarters density was greater than 46 and 109 plants/m<sup>2</sup> in 1976 and 1977, respectively (Sibuga et al. 1985). Scientists have reported 25 to 52% reductions in corn yield with 180 and 200 giant foxtail plants/m, respectively (Knake and Slife 1962; Lambert et al. 1994). Fausey et al. (1997) observed corn yields were reduced 14% from only 10 giant foxtail plants/m.

### **ALS and non ALS-inhibitor Herbicide Mixtures**

Atrazine, 2,4-D, and dicamba are non ALS-inhibiting herbicides used extensively in corn production to control broadleaf weeds. Tank mixtures of halosulfuron-methyl or RT with these herbicides may enhance control of several weed species, and may assist in controlling ALS-resistant biotypes. Growth regulator herbicides like 2,4-D and dicamba have complimented broadleaf weed control when mixed with POST ALS-inhibitor herbicides (Hart 1997; Himmelstein and Durgy 1996; Kalnay et al. 1995; Menbere and Ritter 1995; Parks et al. 1995; VanGessel et al. 1997a). However, many herbicides with POST grass activity in corn often provide reduced control when mixed with broadleaf herbicides (Corkern et al. 1999; Hart and Wax 1996; Kalnay and Glen 1997; Young et al. 1996). Hahn and Stachowski (2000) reported reduced control (76%) of green foxtail [*Setaria viridis* (L.) Beauv.] when ALS-inhibitor herbicides were mixed together. However, these researchers also observed excellent control (97%) of triazine-resistant common lambsquarters and yellow foxtail [*Setaria glauca* (L.) Beauv.] with combinations of nicosulfuron (ALS-inhibitor) plus rimsulfuron plus clopyralid (growth regulator)

plus flumetsulam (ALS-inhibitor) in corn. Understanding interactions among herbicides with POST grass activity and broadleaf herbicides can aid in the development of more effective weed-management strategies in corn.

### **Sethoxydim Use in SR Corn**

Sethoxydim, a cyclohexanedione herbicide, is a POST graminicide that controls annual and perennial grasses, and previously could be safely applied only to certain dicotyledonous crops. Sethoxydim inhibits the enzyme acetyl-coenzyme A carboxylase (ACCase) and disrupts fatty acid biosynthesis in susceptible grasses and monocotyledonous crops (Marshall et al. 1992). However, recent development of sethoxydim-resistant (SR) corn has allowed the use of sethoxydim in SR corn with excellent crop safety (Dotray et al. 1993; Parker et al. 1990; VanGessel et al. 1997b). SR corn was developed by an alteration in the enzyme ACCase through tissue culture and mutation breeding (Parker et al. 1990). Weed control systems utilizing SR corn with sethoxydim have been equally effective as other herbicides for grass control. Young and Hart (1997) reported sethoxydim applied alone controlled giant foxtail 8% better than nicosulfuron. Dotray et al. (1993) observed equal or greater control of foxtail species with POST applications of sethoxydim compared to preemergence (PRE) treatments of atrazine plus alachlor [2-chloro-*N*-(2,6-diethylphenyl)-*N*-(methoxymethyl)acetamide].

### **Sethoxydim Tank-mixed with non ALS-inhibitor Herbicides**

Sethoxydim activity is limited to grasses, thus an effective broad spectrum weed management program must include a broadleaf herbicide. However, tank mixtures applied POST to control broadleaf and grass weeds often result in reduced grass control (Corkern et al. 1998; Hart and Wax 1996; Hatzios and Penner 1985; Holshouser and Coble 1990; Jordan 1995; Snipes and Allen 1996). Hart and Penner (1993) observed that primisulfuron's efficacy was reduced by 15 and 16% when applied at 30 or 60 g/ha, respectively, in combination with 1700 g/ha atrazine to giant foxtail. Research has shown that sethoxydim's efficacy on grasses is reduced when tank-mixed with non ALS-inhibitor herbicides such as bentazon (Campbell and Penner 1982),



bromoxynil (Corkern et al. 1998; Jordan et al. 1993), pyridate [*O*-(6-chloro-3-phenyl-4-pyridazinyl) S-octyl carbonothioate] (Grichar 1991), and 2,4-D (Mueller et al. 1989; Young et al. 1996). However, Young et al. (1996) reported no reduction of giant foxtail, large crabgrass [*Digitaria sanguinalis* (L.) Scop.], or shattercane [*Sorghum bicolor* (L.) Moench] control when sethoxydim (50 g/ha) was tank-mixed with 1120 g/ha atrazine. Corkern et al. (1999) also observed no reduction in barnyardgrass or broadleaf signalgrass [*Brachiaria platyphylla* (Griseb.) Nash] control when sethoxydim was mixed with atrazine or 2,4-D. Understanding interactions with herbicide combinations becomes more complex when multiple weed species comprise the infestation.

### **Sethoxydim Tank-mixed with ALS-inhibitor Herbicides**

Grass control with sethoxydim can also be antagonized when tank-mixed with ALS-inhibitor herbicides (Holshouser and Coble 1990; Young et al. 1996; Young and Hart 1997). Young et al. (1996) evaluated twenty-two POST corn broadleaf herbicide combinations with sethoxydim under greenhouse conditions, and found a reduction in sethoxydim efficacy on at least one grass species in eighteen combinations. Young and Hart (1997) also reported 48% reduction in giant foxtail control with combinations of sethoxydim with primisulfuron plus CGA 152005 in SR corn. The importance of managing herbicide interactions is growing as the use of mixtures, number of potential mixtures, and the number of components in mixtures grows. Developing an effective weed management system in SR and non-herbicide resistant corn requires a more thorough understanding about how sethoxydim, halosulfuron-methyl, and RT interact with corn broadleaf herbicides.

### **Research Objectives**

The objectives of this dissertation research were to: (1) investigate the interactions of 2,4-D or dicamba with halosulfuron-methyl on common lambsquarters and common ragweed control in corn, (2) determine the effect of 2,4-D on the foliar absorption, translocation, and metabolism of <sup>14</sup>C halosulfuron-methyl in common lambsquarters, (3) examine the interactions of 2,4-D, dicamba and ALS-inhibitor herbicides with rimsulfuron plus thifensulfuron-methyl on giant foxtail,

common ragweed, and common lambsquarters control in corn, and (4) investigate sethoxydim tank mixtures with selected ALS and non ALS-inhibitor herbicides on giant foxtail, common ragweed, and common lambsquarters control in SR corn.

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## Chapter II

### Interactions of Halosulfuron-methyl and Growth Regulator Herbicides in Corn (*Zea mays*)

**Abstract.** Field studies were conducted in 1995 and 1996 to investigate postemergence (POST) tank mixtures of halosulfuron-methyl with 2,4-D and dicamba for control of common lambsquarters and common ragweed in field corn. In one study, halosulfuron-methyl was applied at 0, 18, 27, and 36 g ai/ha alone and in combination with 2,4-D at 0, 70, 105, and 140 g ai/ha. Halosulfuron-methyl controlled common ragweed but did not control common lambsquarters. Halosulfuron-methyl at 18 g/ha in combination with 2,4-D at 140 g/ha was synergistic for common lambsquarters control ( $\geq 93\%$ ) at 11 and 25 days after treatment (DAT). In a second study, halosulfuron-methyl at 0, 18, and 36 g/ha was applied alone and in combination with dicamba at 0, 70, 105, and 140 g ai/ha. Averaged across rates 60 DAT, common lambsquarters control with 2,4-D and with dicamba alone was 90%. Common lambsquarters control was synergized with tank mixtures of dicamba and halosulfuron-methyl only at the high rates 11 DAT. Four out of nine herbicide combinations involving 2,4-D and two out of six combinations with dicamba exhibited synergistic responses on common lambsquarters control 25 DAT. All herbicide combinations were additive for control of both weed species 60 DAT, and were equally effective in reducing weed biomass 70 DAT. Corn yields were increased 100 to 170 % by all herbicide combinations when compared to the untreated checks.

**Nomenclature:** 2,4-D, [(2,4-dichlorophenoxy)acetic acid]; dicamba, 3,6-dichloro-2-methoxybenzoic acid; halosulfuron-methyl, methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonylamino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate; common lambsquarters, *Chenopodium album* L. #<sup>1</sup> CHEAL; common ragweed, *Ambrosia artemisiifolia* L. # AMBEL; corn, *Zea mays* L., ‘Pioneer 3394.’

**Additional index words:** Herbicide interaction, reduced rate application, synergism, 2,4-D, dicamba, halosulfuron-methyl, *Ambrosia artemisiifolia*, *Chenopodium album*, AMBEL, CHEAL.

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<sup>1</sup> Letters following this symbol are a WSSA-approved computer code from *Composite List of Weeds*, Revised 1989. Available from WSSA, 810 East 10th Street, Lawrence, KS 66044-8897.



**Abbreviations:** ALS, acetolactate synthase (EC 4.1.3.18); NIS, non-ionic surfactant; POST, postemergence; DAT, days after treatment.

## Introduction

Herbicide combinations are common in corn production in the United States to control broadleaf and grass weed species. Hatzios and Penner (1985) list several advantages of tank-mix combinations which include improved spectrum of weed control and reductions in crop production costs, soil compaction, and herbicide residues. New active ingredients are occasionally introduced and are being mixed with a growing number of low-cost, post-patent herbicides for broad-spectrum, season-long weed control. Previous research has demonstrated the benefit of utilizing reduced rates of selected herbicides in mixtures to enhance weed control and diminish the development of resistant weed biotypes (Baldwin et al. 1985; Defelice et al. 1989; Feldick and Kapusta 1986; Gressel 1990; Starke and Oliver 1998).

When two or more herbicides are tank-mixed and applied together and the resulting control is greater than the expected control of the individual herbicides applied alone, the combination is said to be synergistic (Colby 1967). Zang et al. (1995) examined 479 examples of herbicide interactions involving 126 different herbicides and 76 different plant species from 24 families. Synergistic interactions were reported in all (12) studies involving the Chenopodiaceae family and in 25% of the Compositae family. Sorensen et al. (1987) observed that a mixture of acifluorfen {5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoic acid} and bentazon [3-(1-methylethyl)-(1*H*)-2,1,3-benzothiadiazin-4(3*H*)-one 2,2-dioxide] had a synergistic effect on common lambsquarters.

Common lambsquarters and common ragweed are annual weeds and members of the Chenopodiaceae and Compositae families, respectively. These species are prevalent and competitive in 40 crops throughout the world (Holm et al. 1977). Despite their susceptibility to many herbicides, these weeds persist in corn fields due to the presence of large seed reserves in soil, seed dormancy, genetic diversity, and, in the case of common lambsquarters, evolved resistance to triazine herbicides (Chu et al. 1978; Darmency 1994; Lewis 1973; Parks et al. 1995; Saini et al. 1986). Successful corn production relies heavily on weed control, and it is estimated

that competition from uncontrolled weeds may cause 30 to 90% yield losses (Hall et al. 1992). Beckett et al. (1988) reported a 12% corn yield loss at 49 common lambsquarters/10 m of row. In a Canadian study, corn yield decreased when common lambsquarters density was greater than 46 and 109 plants/m<sup>2</sup> in 1976 and 1977, respectively (Sibuga et al. 1985).

Halosulfuron-methyl is a sulfonylurea herbicide that controls broadleaf weeds and sedges postemergence (POST) in field corn, grain sorghum [*Sorghum bicolor* (L.) Moench], turf, and sugarcane (*Saccharum officinarum* L.) (Anonymous 1994). Application rates ranging from 18 to 70 g/ha control common cocklebur (*Xanthium strumarium* L.), velvetleaf (*Abutilon theophrasti* Medicus), common ragweed (*Ambrosia artemisiifolia* L.), giant ragweed (*Ambrosia trifida* L.), pigweeds (*Amaranthus* spp.), and yellow nutsedge (*Cyperus esculentus* L.) (Ackley et al. 1994; Dubelman et al. 1997; Dutt and Riego 1994; Hart 1997; Majek 1994; Mayonado et al. 1994). Halosulfuron-methyl inhibits acetolactate synthase (ALS) (also called acetohydroxyacid synthase), the first common enzyme in the biosynthesis of the branched-chain amino acids valine, leucine, and isoleucine. (Schloss 1990; Wittenbach and Abell 1999).

Dicamba and 2,4-D are growth regulator herbicides used extensively in corn production to control broadleaf weeds. Tank mixtures of halosulfuron-methyl with these herbicides may enhance control of several weed species, especially common lambsquarters, and may assist in controlling ALS-resistant biotypes. Herbicides like 2,4-D and dicamba have complimented broadleaf weed control when mixed with POST ALS-inhibitor herbicides (Hart 1997; Himmelstein and Durgy 1996; Kalnay et al. 1995; Menbere and Ritter 1995; Parks et al. 1995; VanGessel et al. 1997). Halosulfuron-methyl applied POST (36-70 g/ha) provided poor control of common lambsquarters and excellent control of common ragweed (Hart 1995; Kalnay et al. 1995; M.A. Isaacs, unpublished data; Majek 1994). Incomplete control of common lambsquarters substantiates the need for a tank-mix partner to obtain effective control. Preliminary evaluations of early POST tank mixtures of halosulfuron-methyl with growth regulator herbicides indicated improved control of common lambsquarters (D. J. Mayonado and H. P. Wilson, personal communication). Limited research has been done on evaluation of reduced rates of halosulfuron-methyl in combination with reduced rates of 2,4-D and dicamba for broadleaf weed control in corn. Therefore, field studies were conducted to examine the interactions of 2,4-D and dicamba

with halosulfuron-methyl on common lambsquarters and common ragweed control in corn.

### Materials and Methods

Field experiments were conducted at the University of Delaware's Research and Education Center at Georgetown, DE, in 1995 and 1996. The soil was a Woodstown sandy loam (Aquic Hapludults; fine-loamy, siliceous, mesic; 78% sand, 10% silt, and 12% clay) with an organic matter content of 1.5% and pH 6.0. Cultural practices were typical of non-irrigated corn on the Delmarva peninsula. These practices included fertilizing according to soil tests, and chisel plowing followed with a tandem disc two times. 'Pioneer 3394' corn was planted on May 17, 1995, and May 15, 1996 at 44,460 seeds/ha. Plots were 8 m long and consisted of four rows planted 76 cm apart. The experimental area was treated immediately after planting with metolachlor [2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-(2-methoxy-1-methylethyl) acetamide] at 1.1 kg ai/ha to suppress annual grasses.

POST herbicide treatments were randomly assigned to each plot as a randomized complete block with four replications. In one study, halosulfuron-methyl at 0, 18, 27, and 36 g/ha was applied alone and in combination with 2,4-D at 0, 70, 105, and 140 g/ha. In another study, halosulfuron-methyl at 0, 18, and 36 g/ha was applied alone and in combination with dicamba at 0, 70, 105, and 140 g/ha. All herbicide treatments included a nonionic surfactant<sup>2</sup> at 0.25% v/v. In 1995, the corn was in the fifth visible leaf stage at application time and sixth leaf stage in 1996. Common lambsquarters and common ragweed seedlings were 5- to 10-cm tall at POST application. Treatments were applied with a tractor-mounted compressed-air sprayer in 234 L/ha of water at 207 kPa through flat fan spray nozzles.<sup>3</sup>

Weed control and corn injury were rated visually 11, 25, and 60 d after treatments (DAT)

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<sup>2</sup> Ortho X-77 nonionic surfactant with 80% principal functioning agents as: alkylaryl/polyoxy ethylene glycols, free fatty acids, and isopropanol. Valent USA Corp., 1333 North California Boulevard, Walnut Creek, CA 94596-8025.

<sup>3</sup> Teejet 8003 flat fan spray tips. Spraying Systems Co., North Avenue, Wheaton, IL 60188.

based on a scale of 0 to 100 (0 = no control or crop injury and 100 = complete weed control or crop death). No significant corn injury occurred from any of the herbicide treatments and therefore, data are not presented. Weed biomass was determined 70 DAT by hand-harvesting weeds in an area 98 by 47 cm (0.5 m<sup>2</sup>) from the center two rows of each plot. Samples were divided into common lambsquarters and common ragweed and dried for a minimum of 72 h at 50 C before determining dry weights. Corn yields were determined by mechanically harvesting three rows of each plot with a small-plot combine, and grain weight was adjusted to 15.5% moisture.

Visual ratings, dry weight, and grain yield data were subjected to a factorial analysis of variance (ANOVA). Homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Control estimates were subjected to arcsine transformation and weed dry weight data were transformed using a logarithmic transformation, yet this did not change the ANOVA. Expected responses for combination treatments were calculated by Colby's method, which is based on a multiplicative survival model (Colby 1967). The equation used for calculating the expected response was:

$$E = 100 - [((100 - x) \times (100 - y))/100] \quad [1]$$

where  $E$  is the expected growth reduction as a percent of the control, and  $x$  and  $y$  represent the growth reduction as a percent of the control from the two herbicides applied alone. When the observed response was greater than the expected response, according to Fisher's protected least significant difference (LSD) at the 0.05 significance level, the interactions were synergistic. When the expected and observed values were not significantly different, the herbicide combination was declared additive.

## Results and Discussion

**General.** Combinations of halosulfuron-methyl with 2,4-D or dicamba were generally additive in their effects on common lambsquarters and common ragweed control, and were occasionally synergistic on common lambsquarters. Synergistic effects with these herbicide combinations occurred most frequently when control was rated 11 and 25 DAT, and were not evident 60 DAT. High rates of halosulfuron-methyl tank-mixed with high rates of 2,4-D or dicamba controlled common lambsquarters more than low rate combinations 60 DAT; yet common ragweed control

did not differ between low and high rate herbicide combinations. However, no differences were observed in biomass reduction of either weed species or in corn grain yields when comparing low to high rates of all herbicide mixtures.

**Halosulfuron-methyl Applied with 2,4-D.** Common lambsquarters control was low ( $\leq 27\%$ ) when halosulfuron-methyl was applied alone regardless of rating date (Tables 1, 2, and 3), but this herbicide provided good to excellent (89 to 97%) common ragweed control. Kalnay et al. (1995) reported halosulfuron-methyl applications of 27 to 70 g/ha alone did not control common lambsquarters (0 to 23%) 56 DAT. Mayonado et al. (1994) reported similar common ragweed control with halosulfuron-methyl applications of 18 and 36 g/ha. Common lambsquarters and common ragweed control 11 DAT with 2,4-D was 73 to 78 and 49 to 60%, respectively (Table 1). Increasing the rate of 2,4-D had no effect on common lambsquarters control. However, 140 g/ha 2,4-D provided better common ragweed control than 70 g/ha. Tank mixtures of 18 g/ha halosulfuron-methyl with 140 g/ha 2,4-D were synergistic, providing 93% common lambsquarters control. Mayonado et al. (1994) also observed  $\geq 90\%$  control of this weed with the same rates and herbicide combinations. Synergism on common lambsquarters also occurred with halosulfuron-methyl at 27 g/ha mixed with 105 g/ha 2,4-D, providing 91% control of this weed. All other herbicide combinations were additive for control of common lambsquarters and common ragweed at this rating date.

Common lambsquarters and common ragweed control 25 DAT with 2,4-D applied alone was 80 to 86 and 38 to 59%, respectively (Table 2). Increasing the 2,4-D rate from 70 to 105 g/ha improved control of common lambsquarters but did not impact common ragweed control. When the rate of 2,4-D was increased to 140 g/ha, common lambsquarters control did not increase while common ragweed control improved compared to the 105 g/ha rate. 2,4-D applied at 140 g/ha in combination with halosulfuron-methyl at all rates was synergistic for the control of common lambsquarters, providing  $\geq 95\%$  control. These herbicide combinations improved common lambsquarters control 10% compared to the two higher rates of 2,4-D applied alone. Kalnay et al. (1995) reported 90% common lambsquarters control 56 DAT with 2,4-D at 140 g/ha tank-mixed with 36 g/ha halosulfuron-methyl. Synergism on common lambsquarters control was also observed when 2,4-D at 105 g/ha was tank-mixed with 36 g/ha halosulfuron-methyl, providing

95% control of this weed. Additivity for control of both weed species was observed with all other herbicide combinations at this rating date.

Common lambsquarters and common ragweed control 60 DAT with 2,4-D applied alone was 86 to 93 and 23 to 45%, respectively (Table 3). Averaged across all rates of 2,4-D, common lambsquarters and common ragweed biomass 70 DAT was reduced by 100 and 59%, respectively (Table 4). Common lambsquarters biomass was not reduced with halosulfuron-methyl; however, this herbicide reduced common ragweed biomass  $\geq 98\%$ . All herbicide combinations were additive, providing  $\geq 83\%$  control of both weed species (Table 3), and were equally effective in reducing weed biomass by  $\geq 94\%$  for both weeds (Table 4). Across all rates of halosulfuron-methyl, increasing the rate of 2,4-D from 70 to 140 g/ha generally improved common lambsquarters control (Table 3)

Corn yield in the untreated check was 2120 kg/ha, and all herbicide treatments increased yield  $\geq 88\%$  compared to the weedy check (Table 5). No differences in yield were observed with halosulfuron-methyl or 2,4-D applied alone regardless of rate. Halosulfuron-methyl at 27 g/ha mixed with 105 g/ha 2,4-D resulted in a yield of 5730 kg/ha, which was 170% above the weedy check. This tank mixture also improved yields 20% more than the same rate of halosulfuron-methyl when combined with 70 g/ha 2,4-D. Halosulfuron-methyl at 18 g/ha combined with 2,4-D at 140 g/ha, along with halosulfuron-methyl at 27 and 36 g/ha tank-mixed with 105 g/ha 2,4-D, provided an average yield increase of 19% compared to 140 g/ha 2,4-D applied alone. All other herbicide combinations were equally effective in increasing corn yields.

**Halosulfuron-methyl Applied with Dicamba.** Common lambsquarters control was low ( $\leq 22\%$ ) when halosulfuron-methyl was applied alone regardless of rating date (Tables 6, 7, and 8). Averaged across 18 and 36 g/ha and rating dates, this herbicide controlled common ragweed 96%. Mayonado et al (1994) reported similar results with halosulfuron-methyl averaged across 18 and 36 g/ha for control ( $\geq 90\%$ ) of this weed. Common lambsquarters and common ragweed control with dicamba was 64 to 73 and 55 to 68%, respectively, 11 DAT (Table 6). Increasing the rate of dicamba applied from 70 to 140 g/ha improved control of both weed species. Halosulfuron-methyl at 36 g/ha tank-mixed with 140 g/ha dicamba was synergistic for common lambsquarters control (84%). All other herbicide combinations were additive for control of common

lambsquarters and common ragweed at this rating date.

Common lambsquarters and common ragweed control 25 DAT with dicamba was 75 to 84 and 66 to 81%, respectively (Table 7). Increasing dicamba from 70 to 140 g/ha improved control of both weed species, but increasing the rate from 105 to 140 g/ha did not affect common lambsquarters control. Synergism was observed on common lambsquarters control when dicamba at 140 g/ha was mixed with both rates of halosulfuron-methyl, providing an average control of 93%. Other researchers have reported similar common lambsquarters control with these herbicide combinations (Himmelstein and Durgy 1996; Mayonado et al. 1994). Additivity for control of both weed species was observed with all other herbicide combinations.

Common lambsquarters and common ragweed control 60 DAT with dicamba was 87 to 93 and 80 to 90%, respectively (Table 8). Averaged across all rates of dicamba, common lambsquarters and common ragweed biomass 70 DAT was reduced by 98 and 93%, respectively (Table 9). Reductions in common lambsquarters biomass were minimal when halosulfuron-methyl was applied alone, but this herbicide reduced common ragweed biomass  $\geq 99\%$ . Increasing the rate of dicamba from 70 to 105 g/ha in combination with 36 g/ha halosulfuron-methyl improved common lambsquarters control (Table 8). Additivity for control of both weed species was observed with all other herbicide combinations, providing  $\geq 89\%$  control, and these tank mixtures were equally effective in reducing weed biomass by  $\geq 97\%$  for both weeds (Table 9).

Corn yield in the untreated check was 2550 kg/ha, and all herbicide treatments increased yield  $\geq 61\%$  compared to the weedy check (Table 10). No differences in yield were observed with halosulfuron-methyl and dicamba applied alone regardless of rates. Halosulfuron-methyl at 18 g/ha in combination with dicamba at 140 g/ha provided a yield of 5980 kg/ha, resulting in 134% yield increase compared to the weedy check. Halosulfuron-methyl at 18 g/ha tank-mixed with 70 and 140 g/ha dicamba and halosulfuron-methyl at 36 g/ha combined with 105 g/ha dicamba increased corn yields by an average of 25% compared to 140 g/ha dicamba applied alone. Kalnay et al. (1995) reported 26 to 52% higher corn yields from plots treated with halosulfuron-methyl at 27 to 70 g/ha combined with 140 g/ha dicamba. Corn yields were similar for all other herbicide combinations.

According to these studies, tank mixtures of halosulfuron-methyl with 2,4-D or dicamba

controlled common lambsquarters and common ragweed at low rates of each herbicide. Producers can utilize POST herbicide combinations like these to obtain effective broadleaf weed control, reduce the amount of active ingredient applied, assist in controlling ALS-inhibitor-resistant weeds, and lower corn production costs.



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Table 1. Common lambsquarters and common ragweed control 11 d after postemergence application of halosulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	2,4-D rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	73	78	78	0	49	53	60
18	26	74(80)	78(84)	93(84)+	89	88(94)	88(95)	92(96)
27	26	82(80)	91(84)+	84(84)	92	89(96)	89(96)	88(97)
36	27	74(80)	85(84)	85(84)	91	90(95)	91(96)	88(96)
LSD (0.05)	_____ 6 _____				_____ 10 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 2. Common lambsquarters and common ragweed control 25 d after postemergence application of halosulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	2,4-D rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	80	86	86	0	38	44	59
18	25	84(85)	92(90)	96(90)+	94	93(96)	94(97)	96(98)
27	19	89(84)	94(89)	95(89)+	94	93(96)	96(97)	96(98)
36	19	82(84)	95(89)+	96(89)+	97	96(98)	97(98)	98(99)
LSD (0.05)	_____ 5 _____				_____ 7 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parenthesis are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 3. Common lambsquarters and common ragweed control 60 d after postemergence application of halosulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	2,4-D rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	86	91	93	0	23	28	45
18	21	86(89)	90(93)	97(94)	92	90(94)	90(94)	94(96)
27	13	91(88)	95(92)	97(94)	92	89(94)	92(94)	94(96)
36	13	83(88)	95(92)	96(94)	96	94(97)	94(97)	96(98)
LSD (0.05)	_____ 6 _____				_____ 9 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parenthesis are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 4. Common lambsquarters and common ragweed dry weights 70 d after postemergence application of halosulfuron-methyl and 2,4-D.<sup>a</sup>

Halosulfuron - methyl rate g ai/ha	2,4-D rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	g/0.5m <sup>2</sup>							
0	156	0	0	0	213	89	64	46
18	150	0	7	0	0	0	2	0
27	144	0	0	0	5	2	0	0
36	150	9	0	0	0	0	0	0
LSD (0.05)	9				14			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v), and means were separated using Fisher's protected LSD at 0.05 significance level.



Table 5. Grain yield of corn treated postemergence with halosulfuron-methyl and 2,4-D.<sup>a,b</sup>

Halosulfuron - methyl rate g ai/ha	2,4-D rate (g ai/ha)			
	0	70	105	140
0	2120 f	4110 de	4050 de	4480 cde
18	4110 de	5040 abc	4980 abc	5480 ab
27	3980 e	4790 bcd	5730 a	4930 abc
36	4050 de	5100 abc	5480 ab	5100 abc
LSD (0.05)	810			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

Table 6. Common lambsquarters and common ragweed control 11 d after postemergence application of halosulfuron-methyl and dicamba.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	Dicamba rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	64	72	73	0	55	63	68
18	22	68(72)	77(78)	74(79)	92	91(96)	93(97)	91(97)
36	18	68(70)	74(77)	84(78)+	95	93(98)	94(98)	93(98)
LSD (0.05)	_____ 5 _____				_____ 6 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 7. Common lambsquarters and common ragweed control 25 d after postemergence application of halosulfuron-methyl and dicamba.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	Dicamba rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	75	84	84	0	66	73	81
18	13	77(78)	88(86)	92(86)+	98	98(99)	98(99)	96(99)
36	12	83(78)	89(86)	94(86)+	98	98(99)	97(99)	98(99)
LSD (0.05)	_____ 5 _____				_____ 4 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 8. Common lambsquarters and common ragweed control 60 d after postemergence application of halosulfuron-methyl and dicamba.<sup>a,b,c</sup>

Halosulfuron - methyl rate g ai/ha	Dicamba rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	_____ % control _____				_____			
0	0	87	91	93	0	80	85	90
18	7	89(88)	94(92)	96(93)	96	96(99)	96(99)	96(100)
36	2	89(87)	95(91)	97(93)	97	98(99)	98(99)	98(100)
LSD (0.05)	_____ 5 _____				_____ 6 _____			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A positive sign following the expected value indicates synergism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 9. Common lambsquarters and common ragweed dry weights 70 d after postemergence application of halosulfuron-methyl and dicamba.<sup>a</sup>

Halosulfuron - methyl rate g ai/ha	Dicamba rate (g ai/ha)							
	Common lambsquarters				Common ragweed			
	0	70	105	140	0	70	105	140
	g/0.5m <sup>2</sup>							
0	175	5	2	1	327	46	19	2
18	126	4	5	1	1	0	1	1
36	116	7	1	0	0	0	1	0
LSD (0.05)	10				13			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v), and means were separated using Fisher's protected LSD at 0.05 significance level..

Table 10. Grain yield of corn treated postemergence with halosulfuron-methyl and dicamba.<sup>a,b</sup>

Halosulfuron - methyl rate g ai/ha	Dicamba rate (g ai/ha)			
	0	70	105	140
0	2550 e	4360 d	4730 bcd	4670 cd
18	4170 d	5730 ab	5660 abc	5980 a
36	4110 d	5420 abc	5850 a	5100 abcd
LSD (0.05)	1060			

<sup>a</sup>All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

## Chapter III

### Interactions of Halosulfuron-methyl and 2,4-D on *Chenopodium album*

**Abstract.** Greenhouse and laboratory studies were conducted to evaluate the efficacy, foliar absorption, translocation, and metabolism of halosulfuron-methyl in combination with 2,4-D on *Chenopodium album* L. In the greenhouse, halosulfuron-methyl at 0, 4.5, 9, 18, and 36 g ai ha<sup>-1</sup> was applied alone and in combination with 2,4-D at 0, 17, 35, and 70 g ai ha<sup>-1</sup> postemergence (POST) to 7.6-cm seedlings. Control and fresh weight reduction of *C. album* with halosulfuron-methyl applied alone was low. Increasing the rate of 2,4-D applied alone from 17 to 70 g ha<sup>-1</sup> improved weed control 12%. Mixtures of 2,4-D at 17 g ha<sup>-1</sup> with halosulfuron-methyl at 4.5, 9, and 36 g ha<sup>-1</sup> exhibited an additive herbicide response for control and fresh weight reduction. Synergistic herbicide interactions were observed with 2,4-D (17 g ha<sup>-1</sup>) and halosulfuron-methyl (18 g ha<sup>-1</sup>) and 2,4-D (70 g ha<sup>-1</sup>) in combination with halosulfuron-methyl at 4.5 and 36 g ha<sup>-1</sup>, respectively. All other herbicide combinations were additive. In the greenhouse, 7.6-cm seedlings were treated POST with commercially formulated halosulfuron-methyl at 9 and 18 g ha<sup>-1</sup> and 2,4-D at 0, 70, and 140 g ha<sup>-1</sup>, respectively. Plants were then treated with foliar-applied <sup>14</sup>C-halosulfuron-methyl and harvested at 6, 24, and 72 h after treatment (HAT). Absorption and translocation of <sup>14</sup>C-halosulfuron-methyl were not influenced by the addition of 2,4-D, with absorption increasing with time. Analysis of treated-leaf extracts by thin layer chromatography revealed three unknown halosulfuron-methyl metabolites (M1, M2, and M3) with R<sub>f</sub> values of 0.0, 0.97, and 0.94, respectively. Metabolites M2 and M3 were less polar than the parent halosulfuron-methyl, while M1 was more polar. The addition of 2,4-D increased the level of M3 at the 18 g ha<sup>-1</sup> halosulfuron-methyl rate, which may contribute to *C. album* phytotoxicity.

**Nomenclature:** 2,4-D [(2,4-dichlorophenoxy)acetic acid]; Halosulfuron-methyl, methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonylamino]sulfonyl]-3-chloro-1-methyl-1-H-pyrazole-4-carboxylate; *Chenopodium album* L., CHEAL, common lambsquarters.

**Key words:** Absorption; herbicide interactions; translocation; metabolism; herbicide mixtures;

reduced rate application;  $^{14}\text{C}$ -halosulfuron-methyl; CHEAL; acetolactate synthase (ALS); thin-layer chromatography (TLC); liquid scintillation spectrometry (LSS).

## Introduction

Halosulfuron-methyl is a sulfonylurea herbicide that controls broadleaf weeds and sedges POST in *Zea mays* L., *Sorghum bicolor* (L.) Moench, turf, and *Saccharum officinarum* L. (Anonymous 1994). Application rates ranging from 18 to 70 g ha<sup>-1</sup> control *Xanthium strumarium* L., *Abutilon theophrasti* Medicus, *Ambrosia artemisiifolia* L., *Ambrosia trifida* L., *Amaranthus* spp., and *Cyperus esculentus* L. (Ackley et al. 1994; Dubelman et al. 1997; Dutt and Riego 1994; Hart 1997; Majek 1994; Mayonado et al. 1994). Halosulfuron-methyl inhibits acetolactate synthase (ALS), an enzyme responsible for branched-chain amino acid biosynthesis (Schloss 1990; Wittenbach and Abell 1999).

*Chenopodium album* is an annual weed which is prevalent and competitive in 40 crops throughout the world (Holm et al. 1977). Control of this species varies with many POST herbicides. Halosulfuron-methyl applied POST (36-70 g ha<sup>-1</sup>) provided poor control of *C. album* (Hart 1995; Kalnay et al. 1995; M.A. Isaacs, Chapter II, Ph.D dissertation; Majek 1994). If this weed is present in a field and halosulfuron-methyl is applied for control of other species, a tank-mix partner will be required.

Growth regulator herbicides like 2,4-D have complimented broadleaf weed control when mixed with POST ALS-inhibitors (Hart 1997; Himmelstein and Durgy 1996; Kalnay et al. 1995; Menbere and Ritter 1995; Parks et al. 1995; VanGessel et al. 1997). The development of ALS-resistant weeds (Barrentine and Kendig 1995; Ohmes and Kendig 1999; Manley et al. 1998) also substantiates the need for a non-ALS-inhibiting herbicide as a component of a mixture for broadleaf weed control. Interactions of herbicide mixtures have been extensively investigated (Hatzios and Penner 1985). Previous research has demonstrated the benefit of utilizing reduced rates of selected herbicides in mixtures to enhance weed control and diminish the development of resistant weed biotypes (Baldwin et al. 1985; Defelice et al. 1989; Feldick and Kapusta 1986; Gressel 1990; Starke and Oliver 1998).

The following greenhouse and laboratory studies were conducted to examine the



interactions of 2,4-D and halosulfuron-methyl on *C. album*, and to determine the potential effects of 2,4-D on the foliar absorption, translocation, and metabolism of <sup>14</sup>C halosulfuron-methyl.

## Materials and Methods

### Plant Material

*Chenopodium album* seeds<sup>1</sup> were planted in a commercial potting medium<sup>2</sup> in 0.5 L styrofoam cups. Cups were placed in the greenhouse and seed were germinated and grown with temperatures maintained at 31 ± 5 C. Natural sunlight was supplemented with metal halide lamps producing 14 h illumination, with a minimum light intensity of 300 μmol m<sup>-2</sup> s<sup>-1</sup> photosynthetic photon flux (PPF) density. After emergence, plants were thinned to four and one plant(s) per cup for greenhouse and laboratory studies, respectively. Cups were watered daily, and a complete fertilizer<sup>3</sup> was applied weekly.

### Greenhouse

In whole plant growth response studies, halosulfuron-methyl at 0, 4.5, 9, 18, and 36 g ha<sup>-1</sup> was applied alone and in combinations with 2,4-D at 0, 17, 35, and 70 g ha<sup>-1</sup> POST to 7.6-cm *C. album*. Herbicides were applied with a greenhouse bench sprayer delivering 234 L ha<sup>-1</sup> at 221 kPa utilizing a flat fan spray tip<sup>4</sup>. A nonionic surfactant<sup>5</sup> (NIS) at 0.25% (v/v) was included with all herbicide treatments.

*Chenopodium album* plants were evaluated visually 4 wk after treatment (WAT) utilizing a scale with 0% representing no visual effects on plant growth and a 100% representing plant death. Aboveground fresh weights were measured 4 WAT.

### Foliar Absorption and Translocation

*Chenopodium album* seedlings were treated at the four- to six-leaf stage of development (7.6-cm). Prior to exposure to radiolabeled halosulfuron-methyl, plants were treated POST with commercially formulated halosulfuron-methyl at 9 and 18 g ha<sup>-1</sup> and 2,4-D at 0, 70 and 140 g ha<sup>-1</sup>, respectively. Herbicides were applied using the same methods and surfactant as described in the

previous section. These rates were selected since preliminary data from field (unpublished data) and greenhouse studies appeared to demonstrate a synergism on this weed species. Radiolabeled halosulfuron-methyl, with a specific activity of 2554 kBq mg<sup>-1</sup> and 98% radiochemical purity, was foliarly applied by the method described by Wilcut et al. (1989). Immediately following application of formulated spray solutions, 10  $\mu$ l of <sup>14</sup>C-labeled halosulfuron-methyl were applied with a micropipette in four 2.5- $\mu$ l drops to the adaxial leaf surface of the third-youngest leaf of each plant. The 10  $\mu$ l solution contained 3.77 kBq in aqueous ethanol (Et:H<sub>2</sub>O, 80:20 v/v) with a NIS at 0.25% (v/v).

Six, 24, and 72 HAT, treated leaves were excised and rinsed in 20 ml of water : methanol (9:1, v/v) solution for 30 s to remove unabsorbed radioactivity. A 1-ml aliquot of the leaf rinse was added to 10 ml of scintillation cocktail and radioactivity was quantified by liquid scintillation spectrometry <sup>6</sup> (LSS) . Soil was washed from the roots of the remainder of the plant and the plant sectioned into three other parts: above the treated leaf, below the treated leaf, and roots. Plant sections were dried 48 h at 60 C, combusted with a biological sample oxidizer <sup>7</sup>, and radioactivity trapped as <sup>14</sup>CO<sub>2</sub> was quantified by LSS. Percent absorption was calculated from the fraction of total applied <sup>14</sup>C recovered in the leaf wash. All other data are expressed as a percentage of total applied radioactivity.

## **Metabolism**

Metabolism of halosulfuron-methyl in *C. album* seedlings was determined by foliar absorption using the same plant growth and herbicide treatment methods. Plants were treated at the four- to six-leaf stage (7.6-cm) with commercially formulated halosulfuron-methyl in combination with 2,4-D and a NIS, immediately followed by radiolabeled halosulfuron-methyl (3.77 kBq in aqueous ethanol with a NIS at 0.25%, v/v), using the rates and techniques as stated in the absorption and translocation studies. Absorption and translocation studies showed the majority of radioactivity in the treated leaves, thus metabolite and parent herbicide extraction was performed on this plant part. Treated leaves were excised and washed at 6, 24, and 72 HAT using techniques described in the previous section. Following excision, treated leaves were frozen in liquid nitrogen, pulverized with a mortar and pestle, then homogenized in 3 ml of 80% methanol.

The homogenates were centrifuged (2,000 × g for 30 min), and the supernatant was saved. The pellets were extracted two more times with 3 ml of 80% methanol. The combined supernatants were concentrated to 0.5 ml by evaporation. The concentrated supernatant was filtered through 2- $\mu$ m nylon disposable filters <sup>8</sup>.

A 30- $\mu$ l aliquot of concentrated supernatant from each sample was spotted on 20 by 20-cm silica gel thin-layer chromatography (TLC) plates <sup>9</sup>. Thirty microliters of <sup>14</sup>C-halosulfuron-methyl (3.46 kBq) was also spotted as a standard on the TLC plates. Prior to sample loading, TLC plates were activated by baking at 65 C for 10 h. TLC plates were developed to 16 cm in a solvent solution of methanol : acetic acid : distilled water (4:1:4, v/v/v). Developed TLC plates were exposed to X-ray film <sup>10</sup> for 21 d. After development, metabolite spots were detected on the TLC plates from the film. Radioactive bands were then scraped from the plate and the amount of radioactivity was determined by LSS. Metabolites were separated by their ratio of front (Rf) values and the radioactivity recovered during the TLC analysis.

## Statistical Analysis

### *Greenhouse*

The experiment was a completely randomized design with a two-factor factorial treatment arrangement consisting of halosulfuron-methyl and 2,4-D. Each experiment had three replications and was repeated in time. Visual rating and fresh weight data were subjected to a factorial analysis of variance (ANOVA). Homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Plant fresh weight data were subjected to a logarithmic transformation, and this did not change the ANOVA. Percent reduction in fresh weights are presented, and were determined by comparing fresh weights to the untreated check. Expected responses for combination treatments were calculated by Colby's method, which is based on a multiplicative survival model (Colby 1967). The equation used for calculating the expected response was:

$$E = 100 - [((100 - x) \times (100 - y))/100] \quad [1]$$

where  $E$  is the expected growth reduction as a percent of the control, and  $x$  and  $y$  represent the growth reduction as a percent of the control from the two herbicides applied alone. When the observed response was greater than the expected response, according to Fisher's protected least significant difference (LSD) at the 0.05 significance level, the interactions were synergistic. When the expected and observed values were not significantly different, the herbicide combination was declared additive.

#### *Absorption, translocation, and metabolism*

The experimental design of the absorption, translocation, and metabolism studies was a completely randomized design with two replications of each treatment. Each experiment was repeated in time. An ANOVA was performed, and homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Treatment means were separated using Fischer's protected LSD test at the 0.05 significance level.

## **Results and Discussion**

### **Greenhouse**

Control and fresh weight reduction of *C. album* with halosulfuron-methyl applied alone was  $\leq 17$  and 9%, respectively (Tables 1 and 2). Increasing the rate of 2,4-D applied alone from 17 to 70 g ha<sup>-1</sup> increased control from 69 to 81% (Table 1). Reductions in fresh weight were greater when the rate of 2,4-D applied alone was increased from 17 (21%) to 35 (40%) g ha<sup>-1</sup> (Table 2). When the rate of 2,4-D applied alone was increased from 35 to 70 g ha<sup>-1</sup>, fresh weight was not reduced. 2,4-D applied at 17 g ha<sup>-1</sup> in combination with halosulfuron-methyl at 4.5, 9, and 36 g ha<sup>-1</sup> exhibited additivity, with an average control and fresh weight reduction of 73 and 32%, respectively (Tables 1 and 2). The same rate of 2,4-D when applied with halosulfuron-methyl at 18 g ha<sup>-1</sup> was synergistic, providing 82% control and a fresh weight reduction of 48%. All rates of halosulfuron-methyl when tank-mixed with 2,4-D at 35 g ha<sup>-1</sup> were additive, providing an average control and fresh weight reduction of 83 and 44%, respectively. 2,4-D applied at 70 g ha<sup>-1</sup> in combination with halosulfuron-methyl at 4.5 and 36 g ha<sup>-1</sup> was synergistic, providing 89 and 92% control with fresh weight reductions of 52 and 51%, respectively. When tank-mixed with

halosulfuron-methyl at 9 and 18 g ha<sup>-1</sup>, this same rate of 2,4-D exhibited additivity for control and fresh weight reduction in *C. album*. Additive herbicide responses relate to higher control than either herbicide applied alone, and are therefore beneficial in crop production. The synergistic effects from herbicide combinations observed in these studies support field observations (M. A. Isaacs, Chapter II, Ph.D. dissertation) when halosulfuron-methyl and 2,4-D are mixed for control of *C. album* in field corn.

### **Foliar Absorption and Translocation**

Averaged over all levels of 2,4-D and halosulfuron-methyl, absorption of <sup>14</sup>C-halosulfuron-methyl increased over time (Figure 1). *Chenopodium album* absorbed 54, 76, and 88% of the <sup>14</sup>C-halosulfuron-methyl applied at 6, 24, and 72 h, respectively. 2,4-D had no effect on the absorption of <sup>14</sup>C-halosulfuron-methyl, regardless of the rate of 2,4-D or halosulfuron-methyl applied. These results are in agreement with those reported by Hart (1997) who showed that absorption of <sup>14</sup>C-halosulfuron-methyl increased over time in *Abutilon theophrasti*, and the addition of dicamba (3,6-dichloro-2-methoxybenzoic acid) had no influence on foliar absorption.

Translocation of <sup>14</sup>C-halosulfuron-methyl out of the treated leaf was ≤ 6% (Figure 2). Averaged across all levels of 2,4-D, halosulfuron-methyl, and harvest times, the amount of <sup>14</sup>C-halosulfuron-methyl detected in the treated leaves was 96%. The addition of 2,4-D had no effect on the translocation pattern of <sup>14</sup>C-halosulfuron-methyl, regardless of the application rate of formulated halosulfuron-methyl. These results agree with Hart (1997), who reported that mixing dicamba with halosulfuron-methyl had no effect on translocation patterns of <sup>14</sup>C from halosulfuron-methyl. Greater translocation of <sup>14</sup>C-halosulfuron-methyl out of the treated leaf was observed 72 HAT as compared to 6 and 24 h when halosulfuron-methyl was applied at 9 g ha<sup>-1</sup> (Figures 2 and 3). At the 18 g ha<sup>-1</sup> halosulfuron-methyl rate, 97% of <sup>14</sup>C-halosulfuron-methyl was detected in the treated leaf 6 HAT as compared to 24 (95%) and 72 (94%) h, respectively (Figure 2). Thus, from 6 to 72 HAT more translocation was observed out of the treated leaves at the higher formulated halosulfuron-methyl rate (Figures 2 and 3).

In comparing the 9 and 18 g ha<sup>-1</sup> rate of formulated halosulfuron-methyl 6 HAT, <sup>14</sup>C-

halosulfuron-methyl in the treated leaves was 96 and 97%, respectively (Figure 2). Yet, 24 HAT more  $^{14}\text{C}$ -halosulfuron-methyl (97%) was present in the treated leaf at the 9 g ha<sup>-1</sup> rate as compared to 18 g ha<sup>-1</sup> (95%). Similar translocation patterns were observed 72 HAT. According to these data more translocation of  $^{14}\text{C}$ -halosulfuron-methyl out of the treated leaf occurred with the higher rate of halosulfuron-methyl 24 and 72 HAT (Figures 2 and 3).

The majority of  $^{14}\text{C}$  from halosulfuron-methyl that translocated out of the treated leaf was found in the shoot above (Figure 3). Averaged across all levels of halosulfuron-methyl, 2,4-D, and harvest times, only 3% of applied  $^{14}\text{C}$ -halosulfuron-methyl was detected in the above foliage. Less than 1%  $^{14}\text{C}$ -halosulfuron-methyl was found in the shoot below and roots, respectively (data not shown). Similar results were reported by Hart (1997), who found 5, 4, and less than 1%  $^{14}\text{C}$ -halosulfuron-methyl in *Abutilon theophrasti* shoots above, shoots below, and roots when combined with dicamba and crop oil concentrate, respectively. Gallaher et al. (1999) reported < 10% translocation of nicosulfuron and primisulfuron out of the treated leaf of *Brachiaria platyphylla* L. 72 HAT. These translocation patterns of sulfonyleurea herbicides, including  $^{14}\text{C}$ -halosulfuron-methyl in *C. album*, may be attributed to the physicochemical properties of the herbicide. Halosulfuron-methyl has a pKa value of 3.5, and is classified as a weak acid (Ahrens 1994). Kleier (1988) developed a model demonstrating that weak acids which exhibit intermediate permeability are more likely to be translocated in the phloem with photosynthate. Devine et al. (1990) found that translocation of sulfonyleureas from treated leaves of *Fagopyrum tataricum* L. is limited by the availability of the branched-chain amino acids. A deficiency of these amino acids will cause the meristems to cease division, resulting in a loss of sink strength. Because the meristems are no longer sinks, phloem translocation of photosynthates to the meristems ceases. Based on these results it is likely that translocation of foliar-absorbed  $^{14}\text{C}$ -halosulfuron-methyl is largely via the phloem (symplastic) and is limited by the herbicide's own mechanism of action.

### **Halosulfuron-methyl Metabolism**

Since the majority of the absorbed radioactivity at all exposure times remained in the treated leaf, metabolism of  $^{14}\text{C}$ -halosulfuron-methyl was conducted by extracting the treated

leaves of *C. Album* seedlings at 6, 24, and 72 HAT.

Halosulfuron-methyl migrated to an R<sub>f</sub> value of 0.91, and averaged across all harvest times, 2,4-D and halosulfuron-methyl rates, 56% of the radioactivity recovered was the non-metabolized parent herbicide (Figures 4 and 5). Three additional unknown metabolites (M1, M2, M3) with R<sub>f</sub> values of 0.0, 0.97, and 0.94 were detected, respectively (Figures 4 and 5). Metabolites M2 and M3 were less polar than the parent halosulfuron-methyl, while M1 exhibited greater polarity remaining at the origin of the TLC plates. Studies investigating the metabolism of halosulfuron-methyl in weeds and crops are limited. Dubelman et al. (1997) reported that halosulfuron-methyl metabolism in corn and wheat involved de-esterification, oxidative O-demethylation, and hydroxylation of the pyrimidine ring followed by rapid conjugation with glucose. The most significant early metabolites identified by these researchers were 5-hydroxyhalosulfuron and its glycosyl conjugate, halosulfuron acid, and halosulfuron desmethyl. The highly polar nature demonstrated by M1 may indicate a conjugation reaction, resulting in the formation of the glycosyl conjugate 5-Hexosehalosulfuron likely catalyzed by the enzyme UDP-glucose glucosyltransferase (Dubelman et al. 1997; Lamoureux and Rusness 1986). Based on the studies conducted by Dubelman et al. (1997), we speculate that M2 and M3 metabolites in our study may well be 5-hydroxyhalosulfuron and halosulfuron acid and/or halosulfuron desmethyl, respectively. The 5-hydroxyhalosulfuron metabolite (M2) is formed by the oxidation of carbon-5 of the pyrimidine ring catalyzed by the mixed function oxidase cytochrome P450 monooxygenase (Diehl et al. 1995; Fonne-Pfister et al. 1990; Koeppe and Brown 1995). The metabolite halosulfuron acid is formed by de-esterification while halosulfuron desmethyl involves oxidative demethylation of the methoxy substituent of the pyrimidine ring (Dubelman et al. 1997). Formation of these metabolites is also catalyzed by cytochrome P450 monooxygenase (Diehl et al. 1995; Fonne-Pfister et al. 1990).

Examination of metabolite levels of <sup>14</sup>C-halosulfuron-methyl at the 9 g ha<sup>-1</sup> rate revealed no differences with M1 and M2, regardless of the rate of 2,4-D (Figure 4). The percent recovery of M3 was 32, 33, and 26% at the 0, 70, and 140 g ha<sup>-1</sup> rates of 2,4-D, respectively. There were no differences in M2 levels of <sup>14</sup>C-halosulfuron-methyl at the 18 g ha<sup>-1</sup> rate, regardless of the rate of 2,4-D (Figure 5). The percent recovery of M1 was 1, 1, and 2% at the 0, 70, 140 g ha<sup>-1</sup> rates of

2,4-D, respectively. Levels of M3 were 26, 32, and 33 % at these same 2,4-D rates, yet a different recovery pattern was observed compared to the 9 g ha<sup>-1</sup> rate of halosulfuron-methyl (Figures 4 and 5). The addition of 2,4-D at 70 and 140 g ha<sup>-1</sup> increased the level of M3 at the higher halosulfuron-methyl rate (Figure 5).

In conclusion, 2,4-D does not seem to affect the absorption and translocation of <sup>14</sup>C-halosulfuron-methyl in *C. album* regardless of the rate of commercially formulated halosulfuron-methyl applied. The addition of 2,4-D to halosulfuron-methyl applied at 18 g ha<sup>-1</sup> increased the levels of the major metabolite M3, and thus may contribute to *C. album* phytotoxicity. Ma et al. (1997) reported that metabolite accumulation from a sulfonylurea enhanced its phytotoxicity on *Xanthium strumarium*. The synergistic response observed in our greenhouse studies with 2,4-D applied at 70 g ha<sup>-1</sup> in combination with halosulfuron-methyl at 36 g ha<sup>-1</sup>, may be explained by the increased levels of M3 with increasing rates of halosulfuron-methyl. Future research should examine the effect of 2,4-D on the ALS enzyme site, focusing on ALS activity in protein extracts from *C. album* as affected by halosulfuron-methyl concentrations.



### Sources of Materials

- <sup>1</sup> Lambsquarters seed, F & J Seed Service, Inc., P.O. Box 82, Woodstock, IL 60098-0082.
- <sup>2</sup> Metro Mix 360, Wetsel Seed Co. Inc., 1345 Diamond Springs Rd., Virginia Beach, VA 23455.
- <sup>3</sup> Peters 20-20-20 general purpose fertilizer, Wetsel Seed Co. Inc., 1345 Diamond Springs Rd., Virginia Beach, VA 23455.
- <sup>4</sup> Teejet 8001E flat fan tip, Spraying Systems Co., North Avenue, Wheaton, IL 60188.
- <sup>5</sup> X-77 Spreader, Loveland Industries Inc., P.O. Box 1289, Greely, CO 80632-1289.
- <sup>6</sup> LS-5800TA, Beckman Instrument Co., Fullerton, CA 92634.
- <sup>7</sup> B0306, Biological Sample Oxidizer, Packard Instrument Co., Downers Grove, IL 60515.
- <sup>8</sup> Acrodisc Disposable Filters, Gelman Sciences, Ann Arbor, MI 48106.
- <sup>9</sup> Silica Gel 60 F<sub>254</sub> precoated TLC plates, EM Science, 480 Democrat Road, Gibbstown, NJ 08027.
- <sup>10</sup> Kodak X-OMAT AR film, Eastman Kodak Co., Rochester, NY 14650.

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Table 1. Visual control ratings of *C. album* in the greenhouse 4 wk after postemergence herbicide application.<sup>a,b,c</sup>

Halosulfuron- methyl rate g ha <sup>-1</sup>	2,4-D rate (g ha <sup>-1</sup> )			
	0	17	35	70
0	0	69	80	81
4.5	7	75 (71)	83 (81)	89 (82) +
9	16	74 (74)	83 (83)	88 (84)
18	15	82 (74) +	81 (83)	87 (84)
36	17	71 (74)	84 (83)	92 (84) +
LSD <sub>0.05</sub>	6			

<sup>a</sup> All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup> Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup> A positive sign following the expected value indicates a synergistic interaction, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 2. *Chenopodium album* fresh weight reduction in the greenhouse 4 wk after postemergence herbicide application.<sup>a,b,c</sup>

Halosulfuron- methyl rate g ha <sup>-1</sup>	2,4-D rate (g ha <sup>-1</sup> )			
	0	17	35	70
0	0	21	40	38
4.5	9	35 (28)	56 (45)	52 (44)
9	0	33 (21)	47 (40)	48 (38)
18	5	48 (25) +	35 (43)	47 (41)
36	0	28 (21)	38 (40)	51 (38) +
LSD <sub>0.05</sub>			13	

<sup>a</sup> All treatments included a nonionic surfactant at 0.25% (v/v).

<sup>b</sup> Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup> A positive sign following the expected value indicates a synergistic interaction, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

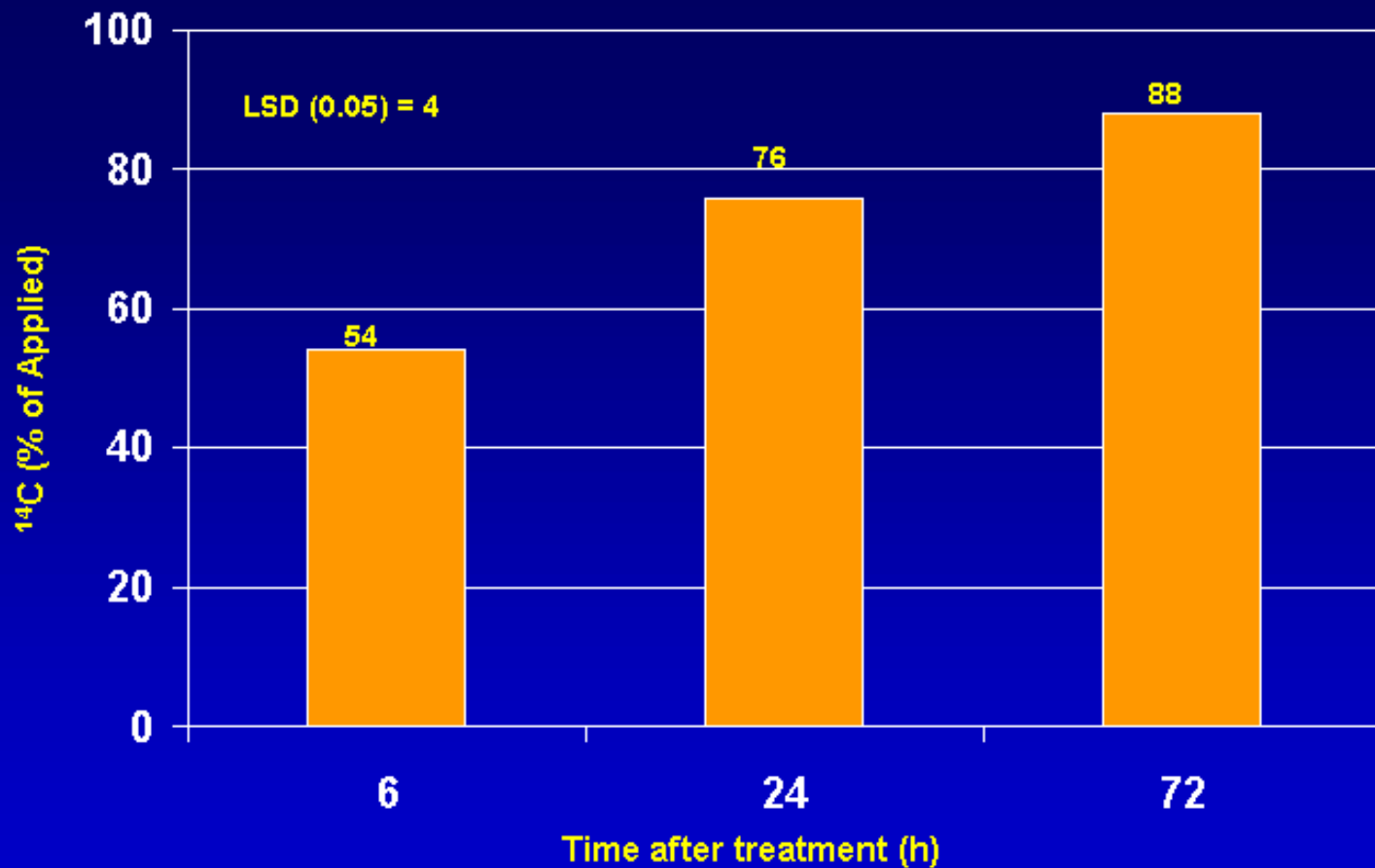


Figure 1. Absorption of <sup>14</sup>C-halosulfuron-methyl. Means are the average from two studies and both herbicides across all levels.

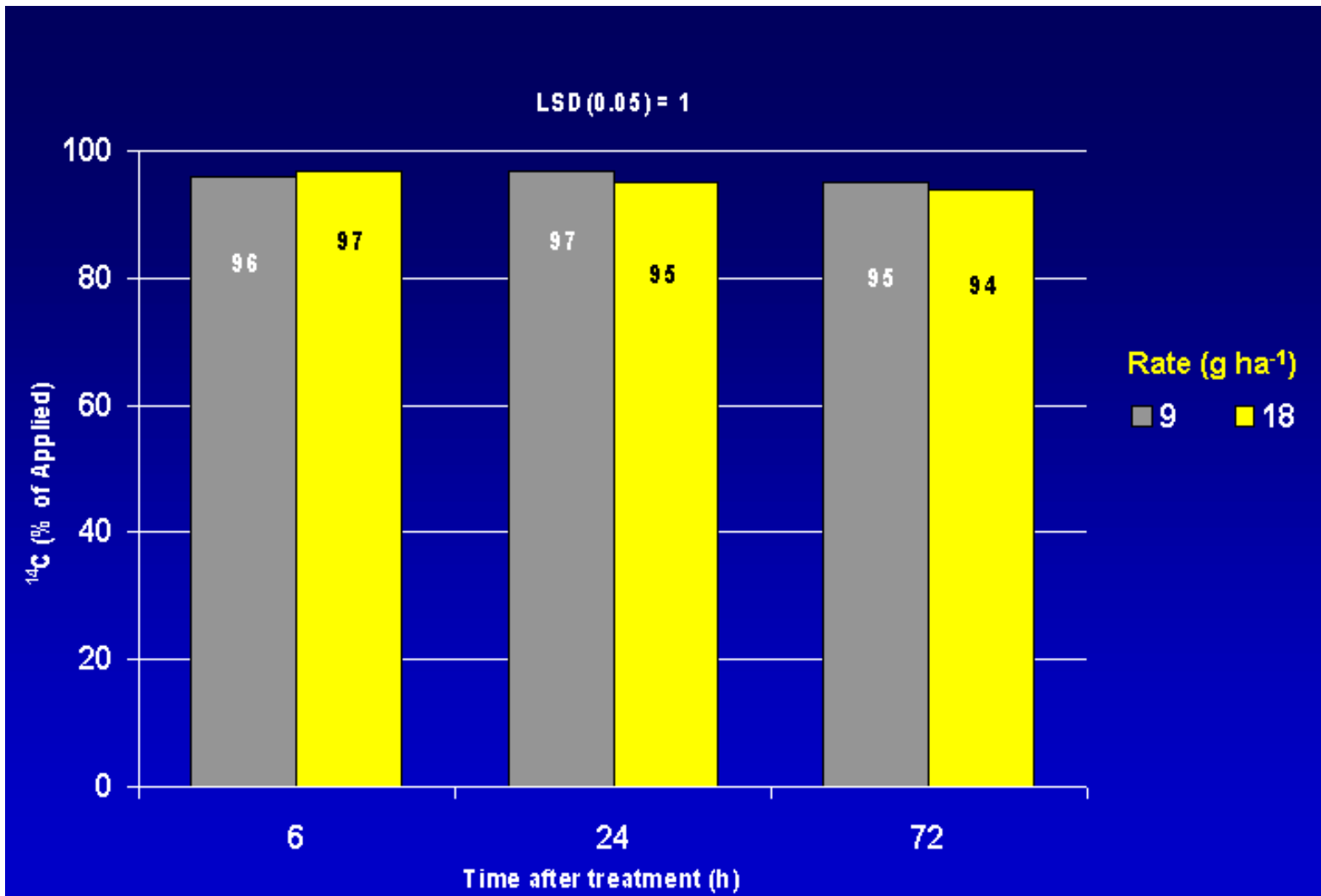
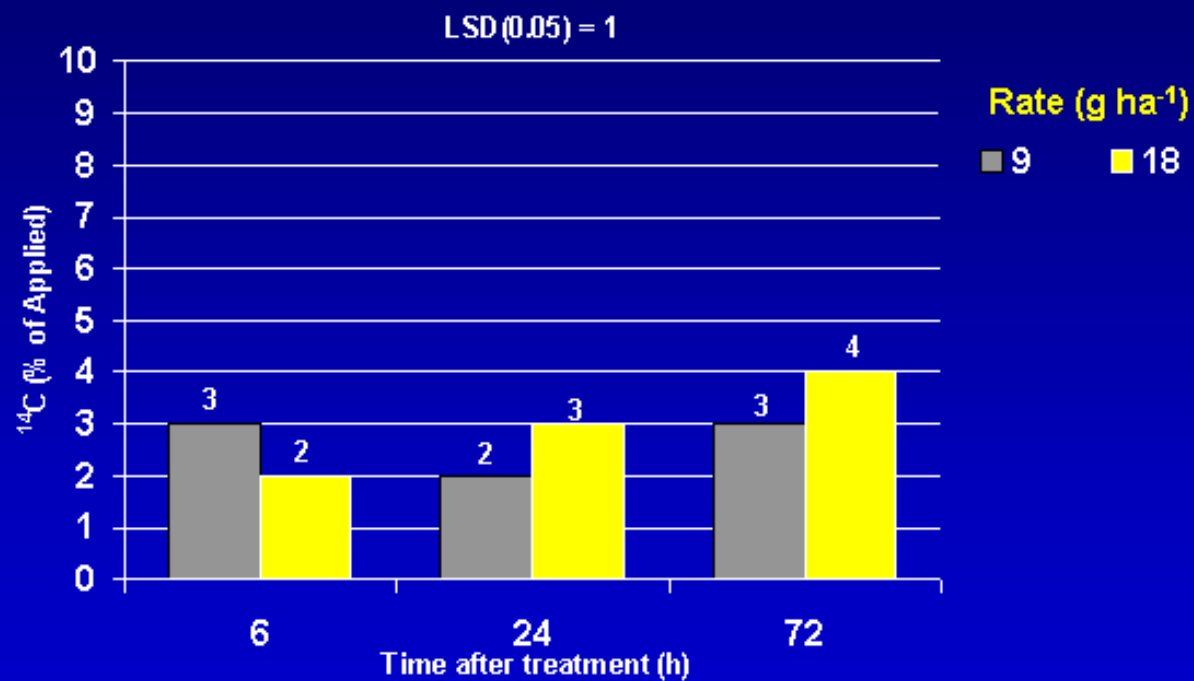


Figure 2. Translocation of <sup>14</sup>C-halosulfuron-methyl (treated leaf). Means are the average from two studies across all levels of 2,4-D.





**Figure 3. Translocation of <sup>14</sup>C-halosulfuron-methyl (shoot above). Means are the average from two studies across all levels of 2,4-D.**

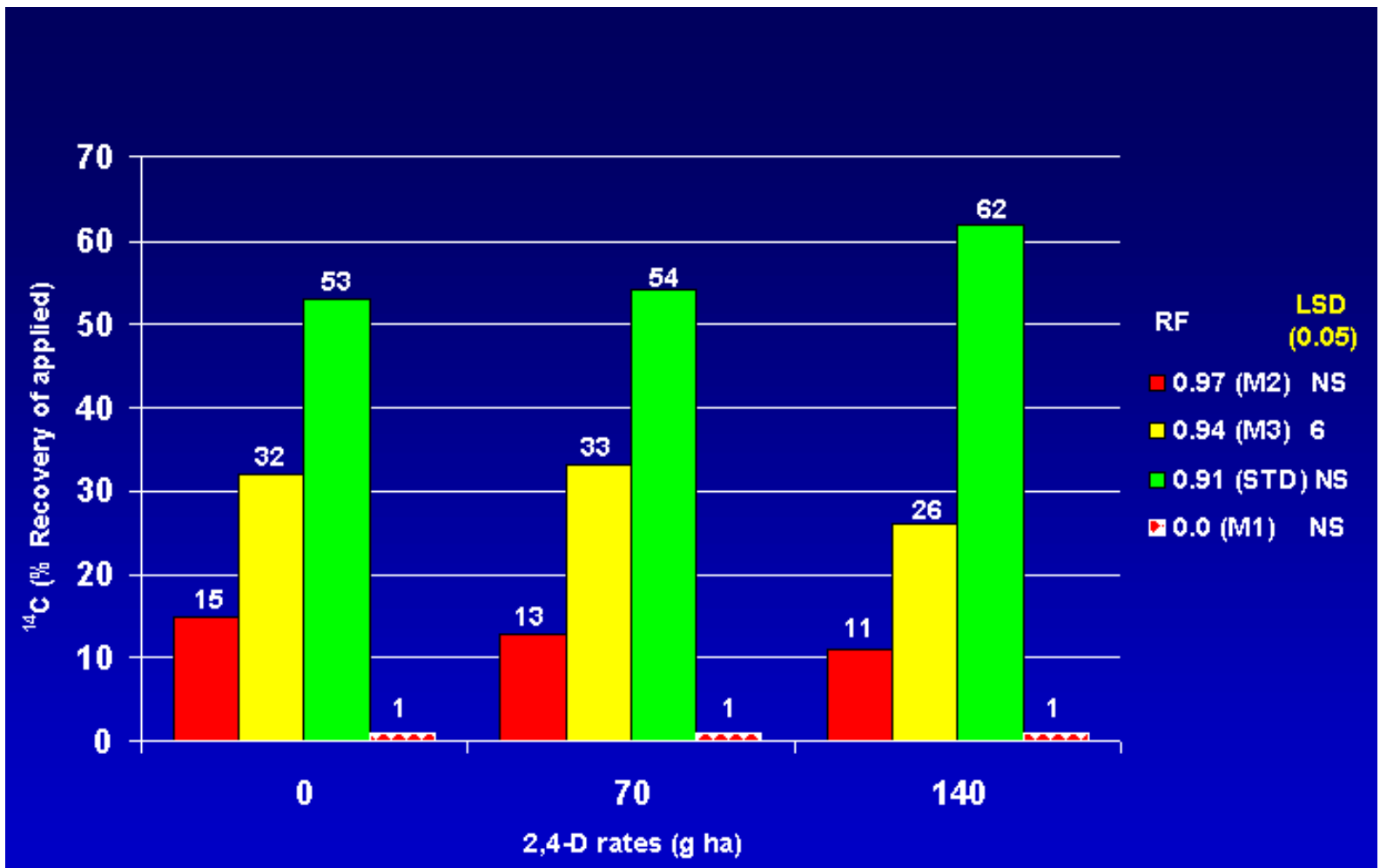


Figure 4. Metabolism of <sup>14</sup>C-halosulfuron-methyl at 9 g ha<sup>-1</sup> (treated leaf). Means are the average from two studies across all harvest times. Standard halosulfuron-methyl migrated to an Rf value of 0.91.

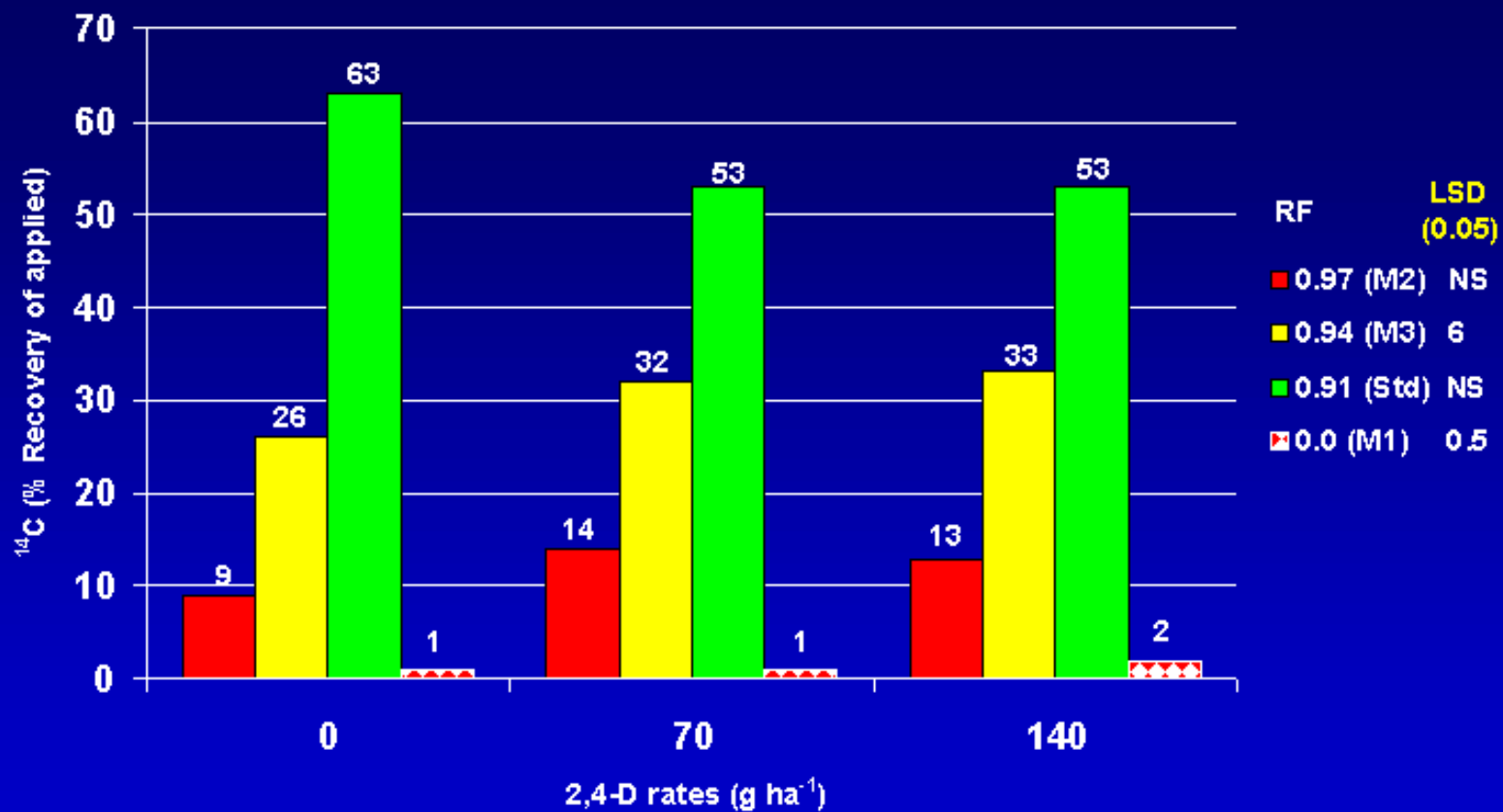


Figure 5. Metabolism of <sup>14</sup>C-halosulfuron-methyl at 18 g ha<sup>-1</sup> (treated leaf). Means are the average from two studies across all harvest times. Standard (Std) halosulfuron-methyl migrated to an Rf value of 0.91.

## Chapter IV

### Characterization of Rimsulfuron plus Thifensulfuron-methyl Interactions with Growth Regulator Herbicides in Corn (*Zea mays*)

**Abstract.** Field studies were conducted in 1995 and 1996 to investigate postemergence (POST) tank mixtures of rimsulfuron plus thifensulfuron-methyl (RT) with 2,4-D and dicamba for weed control in field corn. RT at 0, 13, and 18 g ai/ha was applied alone and in combination with 2,4-D or with dicamba at 0, 140, and 280 g ai/ha in separate studies. Averaged over rates and rating dates, RT controlled giant foxtail 87% but controlled common ragweed only 42%. Antagonism on giant foxtail control at all ratings was observed with all combinations of RT and 2,4-D; these tank mixtures reduced control of this weed an average of 18% and increased biomass 88% compared to RT applied alone. Common ragweed control was synergistic when RT at 13 g/ha was mixed with 140 g/ha 2,4-D at 25 and 60 DAT; other combinations were additive for control of this weed. All RT combinations with 2,4-D and with dicamba were equally effective in reducing common ragweed biomass, providing an average reduction of 99%. Averaged over rates and rating dates, RT combinations with dicamba controlled giant foxtail 91% and these tank mixtures exhibited additivity. Common ragweed control was antagonized by all tank mixtures of RT with dicamba 11 DAT, but these mixtures were additive at 25 and 60 DAT. When compared to untreated checks, corn yields were increased an average of 122 and 249% with RT combinations with 2,4-D and dicamba, respectively. Low rates of RT tank-mixed with low rates of these growth regulator herbicides provided equal control and biomass reduction of these weeds, and similar corn grain yields compared to higher rate combinations.

**Nomenclature:** 2,4-D, [(2,4-dichlorophenoxy)acetic acid]; dicamba, 3,6-dichloro-2-methoxybenzoic acid; rimsulfuron, N-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(ethylsulfonyl)-2-pyridinesulfonamide; thifensulfuron-methyl, 3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-thiophenecarboxylic acid; common ragweed, *Ambrosia artemisiifolia* L. #<sup>4</sup> AMBEL; giant foxtail, *Setaria faberi* Herrm. # SETFA; corn, *Zea*

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<sup>4</sup> Letters following this symbol are a WSSA-approved computer code from Composite List of Weeds, Revised 1989. Available from WSSA, 810 East 10th Street, Lawrence, KS 66044-8897

*mays* L., ‘Pioneer 3394.’

**Additional index words:** Antagonism, herbicide interaction, reduced rate application, 2,4-D, dicamba, rimsulfuron, thifensulfuron-methyl, *Ambrosia artemisiifolia*, *Setaria faberi*, AMBEL, SETFA.

**Abbreviations:** ALS, acetolactate synthase (EC 4.1.3.18); DAT, days after treatment; MSO, methylated seed oil; NIS, non-ionic surfactant; POST, postemergence; RT, rimsulfuron plus thifensulfuron-methyl.

## Introduction

Herbicide combinations are common in corn production in the United States to control difficult broadleaf and grass weed species. Hatzios and Penner (1985) list several advantages of tank-mix combinations which include improved spectrum of weed control and reductions in crop production costs, soil compaction, and herbicide residues. New active ingredients are occasionally introduced and are being mixed with a growing number of low-cost, post-patent herbicides for broad-spectrum, season-long weed control. However, tank mixtures applied postemergence (POST) to control broadleaf and grass weeds often result in reduced grass control (Corkern et al. 1998; Hart and Wax 1996; Hatzios and Penner 1985; Holshouser and Coble 1990; Jordan 1995; Snipes and Allen 1996).

When two or more herbicides are tank-mixed and applied together and the resulting control is more than the expected control of the individual herbicides applied alone, the combination is said to be synergistic; when less than expected, it is antagonistic (Colby 1967). If the observed and expected responses are statistically equal, the combination is additive. Zang et al. (1995) examined 479 examples of herbicide interactions involving 126 different herbicides and 76 different plant species from 24 families. Antagonistic interactions occurred more frequently when the target plants were monocots, and were reported in 80 and 73% of studies involving the Gramineae and Compositae families, respectively. Hart and Penner (1993) observed that the efficacy of primisulfuron {2-[[[[[4,6-bis(difluoromethoxy)-2-pyrimidinyl]amino]carbonyl]amino]sulfonyl]benzoic acid } was reduced by 15 and 16% when

applied at 30 or 60 g ai/ha, respectively, in combination with 1700 g ai/ha atrazine { 6-chloro-N-ethyl-N'-(1-methylethyl)-1,3,5-triazine-2,4-diamine } to giant foxtail. Young and Hart (1997) reported 48% reduction in giant foxtail control with combinations of sethoxydim { 2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one } with primisulfuron plus prosulfuron { 1-(4-methoxy-6-methyl-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl]urea } in sethoxydim-resistant corn. The importance of managing herbicide interactions is growing as the use of mixtures, number of potential mixtures, and the number of components in mixtures grows.

Giant foxtail and common ragweed are annual weeds and members of the Gramineae and Compositae families, respectively. These species are prevalent and competitive in 40 crops throughout the world (Holm et al. 1977; Knake 1977). Despite their susceptibility to many herbicides, these weeds persist in corn fields due to prolific seed production and dormancy, the presence of large seed reserves in soil, and genetic diversity (Barbour et al. 1994; Fausey and Renner 1997; Mester and Buhler 1986). Successful corn production relies heavily on weed control, and it is estimated that competition from uncontrolled weeds may cause 30 to 90% yield losses (Hall et al. 1992). Scientists have reported 25 to 52% reductions in corn yield with 180 and 200 giant foxtail plants/m, respectively (Knake and Slife 1962; Lambert et al. 1994). Fausey et al. (1997) observed corn yields were reduced 14% from only 10 giant foxtail plants/m.

Rimsulfuron plus thifensulfuron-methyl (RT), trade name Basis<sup>®</sup>, is a pre-packaged sulfonylurea herbicide in a 2:1 ratio that controls selected annual broadleaf and grass weeds POST in field corn. (Anonymous 1997). An application rate of 18 g/ha controls barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.], foxtails (*Setaria* spp.), and fall panicum (*Panicum dichotomiflorum* Michx.) at 3- to 5-cm, and common lambsquarters (*Chenopodium album* L.), pigweeds (*Amaranthus* spp.), smartweed (*Polygonum* spp.), velvetleaf (*Abutilon theophrasti* Medicus), and wild mustard [*Brassica kaber* (DC.) Wheeler] at 3- to 8-cm stage (Anonymous 1997; Ganske et al. 1996; Kalnay and Glen 1997). RT inhibits acetolactate synthase (ALS) (also called acetohydroxyacid synthase), the first common enzyme in the biosynthesis of the branched-chain amino acids valine, leucine, and isoleucine. (Schloss 1990; Wittenbach and Abell 1999).

Dicamba and 2,4-D are growth regulator herbicides used extensively in corn production to

control broadleaf weeds. Tank mixtures of RT with these herbicides may enhance control of several weed species, and may assist in controlling ALS-resistant biotypes. Herbicides like 2,4-D and dicamba have complimented broadleaf weed control when mixed with POST ALS-inhibitor herbicides (Hart 1997; Himmelstein and Durgy 1996; Kalnay et al. 1995; Menbere and Ritter 1995; Parks et al. 1995; VanGessel et al. 1997). However, many herbicides with POST grass activity in corn often have reduced control when mixed with broadleaf herbicides (Corkern et al. 1999; Hart and Wax 1996; Kalnay and Glen 1997; Young et al. 1996). Preliminary evaluations of early POST tank mixtures of RT with growth regulator herbicides indicated decreased control of giant foxtail (D. D. Ganske and H. P. Wilson, personal communication). Understanding interactions among herbicides with POST grass activity and broadleaf herbicides can aid in the development of more effective weed-management strategies in corn. Little research has been done on the evaluation of reduced rates of RT in combination with 2,4-D and dicamba for grass and broadleaf weed control in corn. Therefore, field studies were conducted to examine the interactions of 2,4-D and dicamba with RT on giant foxtail and common ragweed control in corn.

### **Materials and Methods**

Field experiments were conducted at the University of Delaware's Research and Education Center at Georgetown, DE, in 1995 and 1996. The soil was a Woodstown sandy loam (Aquic Hapludults; fine-loamy, siliceous, mesic; 78% sand, 10% silt, and 12% clay) with an organic matter content of 1.5% and pH 6.0. Cultural practices were typical of non-irrigated corn on the Delmarva peninsula. These practices included fertilizing according to soil tests, and chisel plowing followed with a tandem disc two times. 'Pioneer 3394' corn was planted on May 17, 1995, and May 15, 1996 at 44,460 seeds/ha. Plots were 8 m long and consisted of four rows planted 76 cm apart.

POST herbicide treatments were randomly assigned to each plot as a randomized complete block with four replications. RT at 0, 13, and 18 g/ha was applied alone and in combination with 2,4-D and with dicamba each at 0, 140, and 280 g/ha in separate studies. All

herbicide treatments included methylated seed oil<sup>5</sup> at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v. In 1995 and 1996, the corn was in the fourth visible leaf stage (2-collar) at application time. Giant foxtail and common ragweed seedlings were 3- to 5-cm and 3- to 8-cm tall at POST application, respectively. Treatments were applied with a tractor-mounted compressed-air sprayer in 234 L/ha of water at 207 kPa through flat fan spray nozzles.<sup>6</sup>

Weed control and corn injury were rated visually 11, 25, and 60 days after treatment (DAT) based on a scale of 0 to 100 (0 = no control or crop injury and 100 = complete weed control or crop death). No significant corn injury was observed from any of the herbicide treatments (data not shown). Weed biomass was determined 70 DAT by hand-harvesting weeds in an area 98 by 47 cm (0.5 m<sup>2</sup>) from the center two rows of each plot. Samples were divided into giant foxtail and common ragweed and dried for a minimum of 72 h at 50 C before determining dry weights. Corn yields were determined by mechanically harvesting three rows of each plot with a small-plot combine, and grain weight was adjusted to 15.5% moisture.

Visual ratings, dry weight, and grain yield data were subjected to a factorial analysis of variance (ANOVA). Homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Control estimates were subjected to arcsine transformation and weed dry weight data were transformed using a logarithmic transformation, yet this did not change the ANOVA. Expected responses for combination treatments were calculated by Colby's method, which is based on a multiplicative survival model (Colby 1967). The equation used for calculating the expected response was:

$$E = 100 - [((100 - x) \times (100 - y))/100] \quad [1]$$

where  $E$  is the expected growth reduction as a percent of the control, and  $x$  and  $y$  represent the growth reduction as a percent of the control from the two herbicides applied alone. Antagonistic interactions were determined when the observed response was less than the expected response

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<sup>5</sup> Methylated seed oil was Sunit-II marketed by American Cyanamid Co., One Cyanamid Plaza, Wayne, NJ 07470.

<sup>6</sup> Teejet 8003 flat fan spray tips. Spraying Systems Co., North Avenue, Wheaton, IL 60188.



according to Fisher's protected least significant difference (LSD) at the 0.05 significance level; when more than expected, the combination was synergistic. When the expected and observed values were not significantly different, the herbicide combination was declared additive.

## Results and Discussion

**General.** Giant foxtail and common ragweed responses to combinations of RT with 2,4-D and dicamba differed with the specific growth regulator herbicide, and with evaluation date.

Antagonism on giant foxtail control was observed with all combinations of RT and 2,4-D. Low rates of RT tank-mixed with low rates of 2,4-D and dicamba provided equal control and biomass reduction of both weed species, and similar corn grain yields compared to higher rate combinations.

**RT Applied with 2,4-D.** All herbicide combinations were antagonistic for control of giant foxtail 11 DAT (Table 1). RT applied alone at 13 and 18 g/ha provided 76 and 79% control of giant foxtail, and 49 and 54% control of common ragweed, respectively. 2,4-D applied alone at 140 and 280 g/ha provided no control of giant foxtail, and 60 and 91% control of common ragweed, respectively. 2,4-D at 140 and 280 g/ha mixed with 13 g/ha RT reduced giant foxtail control by 29 and 22%, respectively, when compared to this same rate of RT alone. RT at 18 g/ha applied with 2,4-D at 140 and 280 g/ha reduced giant foxtail control 27 and 20%, respectively, when compared to 18 g/ha of RT applied alone. Averaged across all herbicide combinations, control of giant foxtail was reduced 25% when compared to RT alone at 13 and 18 g/ha. Common ragweed control was additive when 2,4-D at 140 g/ha was mixed with RT at 13 and 18 g/ha. However, common ragweed control was antagonized when these RT rates were mixed with 280 g/ha 2,4-D, providing 81 and 87% control.

Giant foxtail control 25 and 60 DAT was antagonized with all herbicide combinations, and RT applied alone at 13 and 18 g/ha provided 83 and 86% control of this weed, respectively (Tables 2 and 3). Similar giant foxtail control (85%) was reported by Kalnay and Glenn (1997) with RT applied alone at 18 and 35 g/ha 56 DAT. Averaged across both rates of RT at 25 and 60 DAT evaluations, combinations with 2,4-D at 140 and 280 g/ha provided 70 and 74% control of giant foxtail, respectively. Control of this weed was reduced by an average of 15%, compared to

RT alone at 13 and 18 g/ha (Tables 2 and 3). Common ragweed control was low ( $\leq 44\%$ ) with RT alone, regardless of rate or DAT. Averaged across 25 and 60 DAT, 2,4-D applied at 140 g/ha provided fair control (66%) of common ragweed, yet at 280 g/ha control of this weed was excellent (95%). The tank mixture of low rates of RT (13 g/ha) and 2,4-D (140 g/ha) was synergistic for control of common ragweed at 25 and 60 DAT, providing an average control of 91% (Tables 2 and 3). The antagonism seen with this weed 11 DAT (Table 1) with the combination of 2,4-D at 280 g/ha with both rates of RT did not exist at 25 and 60 DAT (Tables 2 and 3); rather these RT plus 2,4-D combinations were additive at the later rating dates.

Giant foxtail biomass was reduced with RT applied alone at 13 and 18 g/ha 70 DAT, providing an average reduction of 88% (Table 4). 2,4-D applied alone at both rates had no effect on giant foxtail biomass. The antagonism observed on giant foxtail control with all herbicide combinations (Tables 1, 2, and 3) was further substantiated by an average weed biomass increase of 88% (Table 4) compared to both rates of RT applied alone. 2,4-D at 280 g/ha combined with either rate of RT resulted in more giant foxtail biomass than the combination of RT (13 g/ha) and the low rate of 2,4-D (140 g/ha). RT was not effective in reducing common ragweed biomass, but 2,4-D at 140 and 280 g/ha reduced weed biomass by 67 and 100%, respectively. All herbicide combinations were equally effective in reducing common ragweed biomass, providing an average reduction of 99%.

Corn yield in the untreated check was 1990 kg/ha, and all herbicide combinations increased yield an average of 122% compared to the check (Table 5). 2,4-D applied alone, regardless of rate, did not improve yield compared to the weedy check. RT at 18 g/ha applied with 280g/ha 2,4-D increased yield 27% compared to the low rate of RT combined with this same 2,4-D rate. RT applied alone at 13 and 18 g/ha was equally effective in increasing corn yields compared to all herbicide combinations, and provided an average increase of 116%.

**RT Applied with Dicamba.** Across all rating dates, no antagonism on giant foxtail control occurred from any herbicide mixtures regardless of rate, and all provided equal control to RT applied alone (Tables 6, 7, and 8). However, Kalnay and Glenn (1997) reported reduced giant foxtail control (63%) when RT at 18 g/ha was mixed with a higher rate of dicamba (560 g/ha). RT at 13 and 18 g/ha provided  $\geq 85\%$  control of giant foxtail 11 DAT (Table 6), and control

improved to  $\geq 92\%$  at 25 and 60 DAT (Tables 7 and 8). Common ragweed control was low ( $\leq 53\%$ ) with RT applied alone, regardless of rating dates (Tables 6, 7, and 8), and control of this weed was excellent ( $\geq 93\%$ ) with all rates of dicamba applied alone. Common ragweed control was antagonized by all combinations of RT and dicamba 11 DAT (Table 6). These same herbicide combinations were additive at 25 and 60 DAT for control of this weed species (Tables 7 and 8).

All herbicide combinations reduced giant foxtail and common ragweed biomass  $\geq 97\%$  70 DAT compared to the untreated checks (Table 9). RT at both rates was equally effective in biomass reduction for giant foxtail compared to all herbicide combinations, and did not reduce common ragweed biomass. Dicamba applied alone at 140 and 280 g/ha had no effect on giant foxtail biomass, yet was equally effective in reducing common ragweed biomass compared to all herbicide mixtures.

Corn yield in the untreated check was 1490 kg/ha, and all herbicide combinations increased yield an average of 249% compared to the check (Table 10). Increasing the rate of dicamba applied alone and in combination with RT had no effect on grain yield. RT applied alone at 13 and 18 g/ha was equally effective in increasing corn yields compared to all herbicide combinations, and provided an average increase of 251%.

According to these studies, RT and 2,4-D are not complimentary when tank-mixed, and exhibit significant antagonism on giant foxtail control. Even though low rates of these two herbicides in mixture provided a synergistic response for common ragweed control, this could be of little importance commercially. Growers might select RT for POST control of annual grass and broadleaf weeds and the combination with 2,4-D would negatively affect grass control. Conversely, RT combinations with dicamba would provide excellent control of giant foxtail while enhancing control of common ragweed and other broadleaf weeds. Application of reduced rates of RT and dicamba can provide excellent grass and broadleaf weed control, reduce the amount of active ingredient applied, assist in controlling ALS-inhibitor-resistant weed biotypes, and lower corn production costs.

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Table 1. Giant foxtail and common ragweed control 11 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	2,4-D rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	60	91
13	76	54(76)-	59(76)-	49	76(80)	81(95)-
18	79	58(79)-	63(79)-	54	79(82)	87(96)-
LSD (0.05)	6			4		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level .



Table 2. Giant foxtail and common ragweed control 25 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	2,4-D rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	65	95
13	83	68(83)-	73(83)-	40	92(79)+	95(97)
18	86	74(86)-	76(86)-	44	86(80)	94(97)
LSD (0.05)	6			7		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while a positive sign indicates synergism, and no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 3. Giant foxtail and common ragweed control 60 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and 2,4-D.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	2,4-D rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	67	95
13	82	68(82)-	73(82)-	28	90(76)+	94(96)
18	86	68(86)-	73(86)-	38	87(80)	96(97)
LSD (0.05)	8			7		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while a positive sign indicates synergism, and no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 4. Giant foxtail and common ragweed dry weights 70 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and 2,4-D.<sup>a</sup>

Rimsulfuron plus thifensulfuron- methyl rate	2,4-D rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	g/0.5m <sup>2</sup>			g/0.5m <sup>2</sup>		
0	316	309	300	120	40	0
13	45	64	80	109	3	0
18	36	73	82	117	0	0
LSD (0.05)	11			14		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v, and means were separated using Fisher's protected LSD at 0.05 significance level.

Table 5. Grain yield of corn treated postemergence with rimsulfuron plus thifensulfuron-methyl and 2,4-D.<sup>a,b</sup>

Rimsulfuron plus thifensulfuron-methyl rate	2,4-D rate (g ai/ha)		
	0	140	280
g ai/ha	kg/ha		
0	1990 c	2050 c	1680 c
13	4050 ab	4230 ab	3740 b
18	4540 ab	4610 ab	5100 a
LSD (0.05)	1120		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

Table 6. Giant foxtail and common ragweed control 11 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and dicamba.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	Dicamba rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	93	96
13	85	85(85)	88(85)	49	90(96)-	91(98)-
18	86	84(86)	83(86)	53	85(97)-	90(98)-
LSD (0.05)	5			4		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 7. Giant foxtail and common ragweed control 25 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and dicamba.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	Dicamba rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	95	96
13	92	90(92)	93(92)	39	93(97)	95(98)
18	95	95(95)	90(95)	41	94(97)	95(98)
LSD (0.05)	6			4		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 8. Giant foxtail and common ragweed control 60 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and dicamba.<sup>a,b,c</sup>

Rimsulfuron plus thifensulfuron- methyl rate	Dicamba rate (g ai/ha)					
	Giant foxtail			Common ragweed		
	0	140	280	0	140	280
g ai/ha	% control					
0	0	0	0	0	94	96
13	94	94(94)	94(94)	32	92(96)	95(97)
18	97	95(97)	96(97)	35	91(96)	96(97)
LSD (0.05)	4			6		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Values in parentheses are the expected values as calculated by Colby's (1967) method.

<sup>c</sup>A negative sign following the expected value indicates antagonism, while no sign indicates additivity according to Fisher's protected LSD at 0.05 significance level.

Table 9. Giant foxtail and common ragweed dry weights 70 d after postemergence application of rimsulfuron plus thifensulfuron-methyl and dicamba.<sup>a</sup>

Rimsulfuron plus thifensulfuron- methyl rate	Dicamba rate (g ai/ha)						
	Giant foxtail			Common ragweed			
	0	140	280	0	140	280	
g ai/ha	g ai/ha			g/0.5m <sup>2</sup>	g/0.5m <sup>2</sup>		
0	286	273	283	70	0	0	
13	0	3	2	56	1	0	
18	2	3	5	60	2	1	
LSD (0.05)	14			17			

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v, and means were separated using Fisher's protected LSD at 0.05 significance level.



Table 10. Grain yield of corn treated postemergence with rimsulfuron plus thifensulfuron-methyl and dicamba.<sup>a,b</sup>

Rimsulfuron plus thifensulfuron-methyl rate	Dicamba rate (g ai/ha)		
	0	140	280
g ai/ha	kg/ha		
0	1490 b	1740 b	1930 b
13	5350 a	5040 a	5350 a
18	5100 a	5230 a	5170 a
LSD (0.05)	870		

<sup>a</sup>All treatments included methylated seed oil (MSO) at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v.

<sup>b</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

## Chapter V

### Rimsulfuron plus Thifensulfuron-methyl Combinations with Selected Broadleaf Herbicides in Corn (*Zea mays*)

**Abstract.** Field studies were conducted in 1995 and 1996 to investigate postemergence (POST) tank mixtures of rimsulfuron plus thifensulfuron-methyl (RT) with various acetolactate synthase (ALS) and non ALS-inhibitor herbicides for weed control in field corn. RT alone controlled giant foxtail and common lambsquarters  $\geq 92\%$ , but did not control common ragweed. Averaged across rating dates, RT at 18 g ai/ha tank-mixed with 20 g ai/ha primisulfuron, 35 g ai/ha CGA 152005 plus primisulfuron, halosulfuron-methyl at 18 and 36 g ai/ha, and 280 g ai/ha dicamba provided 86% control of giant foxtail, common ragweed, and common lambsquarters. Tank mixtures of RT with flumetsulam plus clopyralid plus 2,4-D at 235 g ai/ha, atrazine (560 g ai/ha), 2,4-D (280 g ai/ha), and dicamba plus atrazine at 896 g ai/ha controlled giant foxtail  $\leq 78\%$  65 DAT. RT mixed with flumetsulam plus clopyralid plus 2,4-D injured corn 26%, and yields were reduced 34% when compared to RT alone. RT tank-mixed with 36 g/ha halosulfuron-methyl injured corn 11%, and corn yield was 5040 kg/ha. All other tank mixtures with RT provided minimal corn injury ( $\leq 11\%$ ) and equal yields.

**Nomenclature:** Atrazine, 6-chloro-*N*-ethyl-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine; CGA 152005, 1-(4-methoxy-6-methyl-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl] urea, proposed common name prosulfuron; clopyralid, 3,6-dichloro-2-pyridinecarboxylic acid; 2,4-D, [(2,4-dichlorophenoxy)acetic acid]; dicamba, 3,6-dichloro-2-methoxybenzoic acid; flumetsulam, *N*-(2,6-difluorophenyl)-5-methyl[1,2,4]triazolo[1,5- $\alpha$ ]pyrimidine-2-sulfonamide; halosulfuron-methyl, methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonylamino]sulfonyl]-3-chloro-1-methyl-1-*H*-pyrazole-4-carboxylate; nicosulfuron, 2-[[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-*N,N*-dimethyl-3-pyridinecarboxamide]; primisulfuron-methyl, 2-[[[[[4,6-bis(difluoromethoxy)-2-pyrimidinyl]amino]carbonyl]amino]sulfonyl]benzoic acid; rimsulfuron, *N*-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(ethylsulfonyl)-2-pyridinesulfonamide]; thifensulfuron-methyl, 3-[[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-

thiophenecarboxylic acid; common lambsquarters, *Chenopodium album* L. #<sup>7</sup> CHEAL; common ragweed, *Ambrosia artemisiifolia* L. # AMBEL; giant foxtail, *Setaria faberi* Herrm. # SETFA; corn, *Zea mays* L., ‘ Pioneer 3394.’

**Additional index words:** Herbicide interaction, atrazine, CGA 152005, clopyralid, 2,4-D, dicamba, flumetsulam, halosulfuron-methyl, nicosulfuron, primisulfuron-methyl, rimsulfuron, thifensulfuron-methyl, *Ambrosia artemisiifolia*, *Chenopodium album*, *Setaria faberi*, AMBEL, CHEAL, SETFA.

**Abbreviations:** ALS, acetolactate synthase (EC 4.1.3.18); DAT, days after treatment; MSO, methylated seed oil; NIS, nonionic surfactant; POST, postemergence; RT, rimsulfuron plus thifensulfuron-methyl.

## Introduction

Rimsulfuron plus thifensulfuron-methyl (RT), trade name Basis<sup>®</sup>, is a pre-packaged sulfonylurea herbicide in a 2:1 ratio that controls selected annual broadleaf and grass weeds POST in field corn. (Anonymous 1997). An application rate of 18 g/ha controls barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.], foxtails (*Setaria* spp.), and fall panicum (*Panicum dichotomiflorum* Michx.) at 3- to 5-cm, and common lambsquarters (*Chenopodium album* L.), pigweed (*Amaranthus* spp.), smartweed (*Polygonum* sp.), velvetleaf (*Abutilon theophrasti* Medicus), and wild mustard [*Brassica kaber* (DC.) Wheeler] at 3- to 8-cm stage (Anonymous 1997; Ganske et al. 1996; Kalnay and Glen 1997). RT inhibits acetolactate synthase (ALS) (also called acetohydroxyacid synthase), the first common enzyme in the biosynthesis of the branched-chain amino acids valine, leucine, and isoleucine. (Schloss 1990; Wittenbach and Abell 1999).

Herbicide combinations are common in corn production in the United States to control difficult broadleaf and grass weed species. Hatzios and Penner (1985) list several advantages of tank-mix combinations which include improved spectrum of weed control and reductions in crop

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<sup>7</sup> Letters following this symbol are a WSSA-approved computer code from *Composite List of Weeds*, Revised 1989. Available from WSSA, 810 East 10th Street, Lawrence, KS 66044-8897.

production costs, soil compaction, and herbicide residues. New active ingredients are occasionally introduced and are being mixed with a growing number of low-cost, post-patent herbicides for broad-spectrum, season-long weed control. However, tank mixtures applied POST to control broadleaf and grass weeds often result in reduced grass control (Corkern et al. 1998; Hart and Wax 1996; Hatzios and Penner 1985; Holshouser and Coble 1990; Jordan 1995; Snipes and Allen 1996).

Zang et al. (1995) examined 479 examples of herbicide interactions involving 126 different herbicides and 76 different plant species from 24 families. Synergistic interactions were reported in all (12) studies involving the Chenopodiaceae family and in 25% of the Compositae family. Antagonistic interactions occurred more frequently when the target plants were monocots, and were reported in 80 and 73% of studies involving the Gramineae and Compositae families, respectively. Hart and Penner (1993) observed that the efficacy of primisulfuron was reduced by 15 and 16% when applied at 30 or 60 g/ha, respectively, in combination with 1700 g/ha atrazine to giant foxtail. Young and Hart (1997) reported 48% reduction in giant foxtail control with combinations of sethoxydim { 2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one } with primisulfuron plus CGA 152005 in sethoxydim-resistant corn. The importance of managing herbicide interactions is growing as the use of mixtures, number of potential mixtures, and the number of components in mixtures grows.

Common lambsquarters, common ragweed, and giant foxtail are annual weeds and members of the Chenopodiaceae, Compositae, and Gramineae families, respectively. These species are prevalent and competitive in 40 crops throughout the world (Holm et al. 1977; Knake 1977). Despite their susceptibility to many herbicides, these weeds persist in corn fields due to prolific seed production and dormancy, the presence of large seed reserves in soil, genetic diversity, and, in the case of common lambsquarters, resistance to triazine herbicides (Barbour et al. 1994; Chu et al. 1978; Darmency 1994; Fausey and Renner 1997; Mester and Buhler 1986; Saini et al. 1986). Successful corn production relies heavily on weed control, and it is estimated that competition from uncontrolled weeds may cause 30 to 90% yield losses (Hall et al. 1992). Beckett et al. (1988) reported a 12% corn yield loss at 49 common lambsquarters/10 m of row. In a Canadian study, corn yield decreased when common lambsquarters density was greater than 46

and 109 plants/m<sup>2</sup> in 1976 and 1977, respectively (Sibuga et al. 1985). Scientists have reported 25 to 52% reductions in corn yield with 180 and 200 giant foxtail plants/m, respectively (Knake and Slife 1962; Lambert et al. 1994). Fausey et al. (1997) observed corn yields were reduced 14% from only 10 giant foxtail plants/m.

Atrazine, 2,4-D, and dicamba are non ALS-inhibiting herbicides used extensively in corn production to control broadleaf weeds. Tank mixtures of RT with these herbicides may enhance control of several weed species, and may assist in controlling ALS-resistant biotypes. Growth regulator herbicides like 2,4-D and dicamba have complimented broadleaf weed control when mixed with POST ALS-inhibitor herbicides (Hart 1997; Himmelstein and Durgy 1996; Kalnay et al. 1995; Menbere and Ritter 1995; Parks et al. 1995; VanGessel et al. 1997). However, many herbicides with POST grass activity in corn often have reduced control when mixed with broadleaf herbicides (Corkern et al. 1999; Hart and Wax 1996; Kalnay and Glen 1997; Young et al. 1996). Hahn and Stachowski (2000) reported reduced control (76%) of green foxtail [*Setaria viridis* (L.) Beauv.] when ALS-inhibitor herbicides were mixed together. However, these researchers also observed excellent control (97%) of triazine-resistant common lambsquarters and yellow foxtail [*Setaria glauca* (L.) Beauv.] with combinations of nicosulfuron (ALS-inhibitor) plus rimsulfuron plus clopyralid (growth regulator) plus flumetsulam (ALS-inhibitor) in corn. Understanding interactions among herbicides with POST grass activity and broadleaf herbicides can aid in the development of more effective weed-management strategies in corn. Therefore, field studies were conducted to examine RT in combination with selected POST broadleaf corn herbicides for control of common lambsquarters, common ragweed, and giant foxtail.

### **Materials and Methods**

Field experiments were conducted at the University of Delaware's Research and Education Center at Georgetown, DE, in 1995 and 1996. The soil was a Woodstown sandy loam (Aquic Hapludults; fine-loamy, siliceous, mesic; 78% sand, 10% silt, and 12% clay) with an organic matter content of 1.5% and pH 6.0. Cultural practices were typical of non-irrigated corn on the Delmarva peninsula. These practices included fertilizing according to soil tests, and chisel

plowing followed with a tandem disc two times. ' Pioneer 3394 ' corn was planted on May 17, 1995, and May 15, 1996 at 44,460 seeds/ha. Plots were 8 m long and consisted of four rows planted 76 cm apart.

POST herbicide treatments were randomly assigned to each plot as a randomized complete block with four replications. RT at 18 g/ha was applied alone and in combination with primisulfuron at 20 g/ha, CGA 152005 plus primisulfuron at 35 g/ha, halosulfuron-methyl at 18 and 36 g/ha, flumetsulam plus clopyralid plus 2,4-D at 235 g/ha, nicosulfuron at 18 g/ha, dicamba plus atrazine at 896 g/ha, atrazine at 560 g/ha, 2,4-D at 280 g/ha, and dicamba at 280 g/ha. Additional treatments included halosulfuron-methyl at 36 g/ha, primisulfuron at 20 g/ha plus 2,4-D at 280 g/ha, and CGA 152005 plus primisulfuron at 35 g/ha plus 2,4-D at 280 g/ha. All herbicide treatments included methylated seed oil<sup>8</sup> at 1% v/v and 30% urea ammonium nitrate (UAN) at 2% v/v except those including growth regulator herbicides; these treatments included a nonionic surfactant<sup>9</sup> at 0.25% v/v. In 1995 and 1996, the corn was in the fourth visible leaf stage (2-collar) at application time. Common lambsquarters and common ragweed seedlings were 5- to 10-cm and giant foxtail were 3- to 5-cm tall at POST application, respectively. Treatments were applied with a tractor-mounted compressed-air sprayer in 234 L/ha of water at 207 kPa through flat fan spray nozzles.<sup>10</sup>

Visual estimates of crop injury and weed control were determined using a scale of 0 to 100 (0 = no control or crop injury and 100 = complete weed control or crop death), and evaluated 12, 26, and 65 DAT. Injury ratings are reported 26 DAT, and are reflective of the other rating dates. Corn yields were determined by mechanically harvesting three rows of each plot with a small-plot

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<sup>8</sup> Methylated seed oil was Sunit-II marketed by American Cyanamid Co., One Cyanamid Plaza, Wayne, NJ 07470.

<sup>9</sup> Ortho X-77 nonionic surfactant with 80% principal functioning agents as: alkylaryl/polyoxy ethylene glycols, free fatty acids, and isopropanol. Valent USA Corp., 1333 North California Boulevard, Walnut Creek, CA 94596-8025.

<sup>10</sup> Teejet 8003 flat fan spray tips. Spraying Systems Co., North Avenue, Wheaton, IL 60188.

combine, and grain weight was adjusted to 15.5% moisture. Control, injury, and grain yield data were subjected to analysis of variance (ANOVA). Homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Control and injury estimates were subjected to arcsine transformation, yet this did not change the ANOVA. Means of nontransformed data were separated using Fisher's protected LSD test at  $P = 0.05$ .

## Results and Discussion

**Giant Foxtail Control.** RT applied alone at 18 g/ha provided excellent control ( $\geq 92\%$ ) of giant foxtail at all ratings (Table 1). Control of this weed species was significantly reduced when RT was tank-mixed with four herbicide treatments. Averaged across rating dates, RT combined with flumetsulam plus clopyralid plus 2,4-D, atrazine, 2,4-D, and dicamba plus atrazine provided 68, 76, 72, and 75% control of giant foxtail, respectively. All other tank mixtures provided good to excellent control ( $\geq 89\%$ ) of this weed when averaged across ratings.

Halosulfuron-methyl applied alone at 36 g/ha did not control giant foxtail (Table 1). Averaged across rating dates, CGA 152005 plus primisulfuron combined with 2,4-D controlled this weed  $\leq 16\%$ . However, primisulfuron tank-mixed with 2,4-D controlled giant foxtail 84%.

**Common Ragweed Control.** Averaged across rating dates, RT applied alone controlled common ragweed 32% (Table 1). RT tank-mixed with nicosulfuron also provided poor control ( $\leq 25\%$ ) of common ragweed averaged over ratings. The same rate of RT combined with atrazine provided 73% control of this weed. All other tank mixtures with RT provided good to excellent control ( $\geq 86\%$ ) of this weed when averaged over ratings.

Halosulfuron-methyl applied alone at 36 g/ha, primisulfuron tank-mixed with 2,4-D, and the mixture of CGA 152005 plus primisulfuron combined with 2,4-D provided excellent control ( $\geq 90$ ) of common ragweed (Table 1).

**Common Lambsquarters Control.** RT applied alone and in combination with all treatments provided excellent control ( $\geq 93\%$ ) of common lambsquarters (Table 1). Averaged over rating dates, primisulfuron mixed with 2,4-D and CGA 152005 plus primisulfuron combined with 2,4-D provided  $\geq 92\%$  control of this weed. Halosulfuron-methyl was the only treatment that did not

control common lambsquarters.

**Corn Injury and Yield.** RT applied alone injured corn 6% at 26 DAT (Table 2), and injury symptoms consisted of a yellowish cast with slight leaf chlorosis. Several treatments, other than halosulfuron-methyl, caused injury which was still evident at the later rating dates. Primisulfuron mixed with 2,4-D and CGA 152005 plus primisulfuron combined with 2,4-D injured corn  $\leq$  8%, and was equal to injury caused by RT applied alone. Tank mixtures of RT with the herbicides evaluated provided equal injury (5 to 11%) compared to RT alone, except when combined with flumetsulam plus clopyralid plus 2,4-D. Corn treated with this tank mixture was injured 26% and yielded 2380 kg/ha (Table 2). This injury translated into a 34% reduction in corn yield compared to RT applied alone which yielded 3610 kg/ha. Corn yield was 5040 kg/ha from the tank mixture of RT and 36 g/ha halosulfuron-methyl, and was higher than injury caused by RT alone and the tank mixture of RT with dicamba plus atrazine. All other seven herbicide combinations tank-mixed with RT provided equal corn yields, averaging 4470 kg/ha.

Yields from plots treated with halosulfuron-methyl alone was reduced 48% compared to corn yields from plots treated with RT alone (Table 2). This yield reduction was due to the poor control of giant foxtail and common lambsquarters (Table 1), and was equal to CGA 152005 plus primisulfuron mixed with 2,4-D and RT tank-mixed with flumetsulam plus clopyralid plus 2,4-D (Table 2). CGA 152005 plus primisulfuron plus 2,4-D did not control giant foxtail (Table 1) and provided minimal corn injury (Table 2). However, RT tank-mixed with flumetsulam plus clopyralid plus 2,4-D controlled giant foxtail 71% 65 DAT (Table 1), and significantly injured corn (Table 2). Corn treated with primisulfuron mixed with 2,4-D yielded 3240 kg/ha, and yield was equal to RT alone and RT tank-mixed with dicamba plus atrazine.

Understanding potential interactions between herbicides when tank-mixed is important when formulating weed management systems. Developing an effective POST weed control program in corn using RT is possible by tank-mixing with selected broadleaf herbicides. However, according to these studies tank-mixing some ALS and non ALS-inhibiting herbicides with RT can reduce giant foxtail and common ragweed control as well as injure corn. Multiple weed species in corn fields present a challenge to producers. For example, in these experiments RT tank-mixed with nicosulfuron controlled giant foxtail  $\geq$  96%; Hahn and Stachowski (2000)



reported 76% control of green foxtail with this same tank mixture. With the introduction of new herbicides and more components in mixtures, it is critical that weed scientists thoroughly evaluate mixture performance.

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Table 1. Giant foxtail, common ragweed, and common lambsquarters control with tank mixtures of rimsulfuron plus thifensulfuron-methyl with selected POST broadleaf herbicides in corn.<sup>a,b,c</sup>

Herbicide	Rate	Control <sup>d</sup>								
		Giant Foxtail			Common ragweed			Common lambsquarters		
		12	26	65	12	26	65	12	26	65
	g ai/ha	%								
Rimsulfuron + thifensulfuron-methyl <sup>e</sup>	12 + 6	92	95	95	38	33	25	93	96	96
+ primisulfuron <sup>e</sup>	20	90	95	95	80	88	91	94	97	97
+ CGA 152005 + primisulfuron <sup>e</sup>	17 + 18	91	93	92	87	94	94	95	97	97
+ halosulfuron-methyl <sup>e</sup>	18	90	96	94	90	91	91	95	97	96
+ halosulfuron-methyl <sup>e</sup>	36	92	95	95	91	98	97	96	96	94
+ flumetsulam + clopyralid + 2,4-D <sup>f</sup>	26 + 69 + 140	64	70	71	90	94	93	95	97	96
+ atrazine <sup>e</sup>	560	73	77	78	75	73	70	97	97	96
+ 2,4-D <sup>f</sup>	280	66	75	76	83	93	93	93	95	95
+ dicamba <sup>f</sup>	280	87	89	90	87	94	92	96	97	97
+ dicamba + atrazine <sup>f</sup>	308 + 588	75	75	74	98	97	97	98	98	98
+ nicosulfuron <sup>e</sup>	18	96	98	98	38	23	13	95	97	96
Halosulfuron-methyl <sup>e</sup>	36	0	0	0	92	95	96	39	38	38
Primisulfuron + 2,4-D <sup>f</sup>	20 + 280	83	83	85	90	93	94	93	94	93
CGA 152005 + primisulfuron + 2,4-D <sup>f</sup>	17 + 18 + 280	21	13	13	92	93	93	92	93	92
Untreated check		0	0	0	0	0	0	0	0	0
LSD (0.05)		5	4	5	5	7	7	6	4	4

<sup>a</sup>Data presented are the means combined over years.

<sup>b</sup>Percent control from 0-100; 0 (no control), 100 (complete control).

<sup>c</sup>Urea ammonium nitrate (30% UAN) was applied at 2% v/v in all treatments.

<sup>d</sup>Control ratings recorded 12, 26, 65, DAT.

<sup>e</sup>Methylated seed oil (MSO) applied at 1% v/v.

<sup>f</sup>Nonionic surfactant (NIS) applied at 0.25% v/v.

Table 2. Corn injury and yield with tank mixtures of rimsulfuron plus thifensulfuron-methyl with selected POST broadleaf herbicides.<sup>a,b</sup>

Herbicide	Rate	Corn injury <sup>c</sup>	Corn yield <sup>d</sup>
	g ai/A	%	kg/ha
Rimsulfuron + thifensulfuron-methyl <sup>e</sup>	12 + 6	6	3610 bcd
+ primisulfuron <sup>e</sup>	20	10	4420 abc
+ CGA 152005 + primisulfuron <sup>e</sup>	17 + 18	8	4050 abc
+ halosulfuron-methyl <sup>e</sup>	18	10	4420 abc
+ halosulfuron-methyl <sup>e</sup>	36	11	5040 a
+ flumetsulam + clopyralid +2,4-D <sup>f</sup>	26 + 69 +140	26	2380 e
+ atrazine <sup>e</sup>	560	5	4480 abc
+ 2,4-D <sup>f</sup>	280	6	4670 ab
+ dicamba <sup>f</sup>	280	7	4420 abc
+ dicamba + atrazine <sup>f</sup>	308 + 588	11	3550 bcd
+ nicosulfuron <sup>e</sup>	18	3	4850 a
Halosulfuron-methyl <sup>e</sup>	36	0	1870 ef
Primisulfuron + 2,4-D <sup>f</sup>	20 + 280	6	3240 d
CGA 152005 + primisulfuron + 2,4-D <sup>f</sup>	17 + 18+ 280	8	1000 fg
Untreated check		0	440 g
LSD (0.05)		6	1120

<sup>a</sup>Data presented are the means combined over years.

<sup>b</sup>Urea ammonium nitrate (30% UAN) was applied at 2% v/v in all treatments.

<sup>c</sup>Percent injury from 0-100 recorded 26 DAT; 0 (no injury), 100 (plant death).

<sup>d</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

<sup>e</sup>Methylated seed oil (MSO) applied at 1% v.v.

<sup>f</sup>Nonionic surfactant (NIS) applied at 0.25% v/v.

## Chapter VI

### Interactions of Sethoxydim with Postemergence Broadleaf Herbicides in Sethoxydim-Resistant Corn (*Zea mays*)

**Abstract.** Field studies were conducted in 1995 and 1996 to investigate postemergence (POST) tank mixtures of sethoxydim with various acetolactate synthase (ALS) and non ALS-inhibitor herbicides for weed control in sethoxydim-resistant (SR) corn. Sethoxydim alone controlled giant foxtail  $\geq 92\%$  and was equal in control to nicosulfuron plus bromoxynil. However, giant foxtail control from sethoxydim tank-mixed with bentazon plus atrazine with urea ammonium nitrate (UAN), or with ALS-inhibiting herbicides plus 2,4-D except halosulfuron-methyl was 24% lower when averaged over treatments. Most herbicide combinations controlled common ragweed and common lambsquarters 90 days after treatment (DAT), except when bentazon and bromoxynil were included. Averaged across all sethoxydim tank mixtures and nicosulfuron plus bromoxynil, corn yields were increased 168% compared to yields from plots treated with sethoxydim alone. Sethoxydim mixed with atrazine provided a corn yield of 5790 kg/ha. Yield of corn treated with sethoxydim or sethoxydim mixed with combinations of sulfonyleurea herbicides plus 2,4-D were low, with the exception of halosulfuron-methyl. According to these studies, understanding tank mixtures with sethoxydim and selected POST broadleaf corn herbicides is crucial for effective weed management.

**Nomenclature:** Atrazine, 6-chloro-*N*-ethyl-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine; bentazon, 3-(1-methylethyl)-(1*H*)-2,1,3-benzothiadiazin-4(3*H*)-one 2,2-dioxide; bromoxynil, 3,5-dibromo-4-hydroxybenzotrile; CGA 152005, 1-(4-methoxy-6-methyl-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl] urea, proposed common name prosulfuron; clopyralid, 3,6-dichloro-2-pyridinecarboxylic acid; 2,4-D, [(2,4-dichlorophenoxy)acetic acid]; dicamba, 3,6-dichloro-2-methoxybenzoic acid; flumetsulam, *N*-(2,6-difluorophenyl)-5-methyl[1,2,4]triazolo[1,5- $\alpha$ ]pyrimidine-2-sulfonamide; halosulfuron-methyl, methyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonylamino]sulfonyl]-3-chloro-1-methyl-1*H*-pyrazole-4-carboxylate; nicosulfuron, 2-[[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]-*N*, *N*-dimethyl-3-pyridinecarboxamide; primisulfuron-methyl, 2-[[[[[4,6-bis(difluoromethoxy)-2-

pyrimidinyl]amino]carbonyl]amino]sulfonyl]benzoic acid; rimsulfuron, N-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(ethylsulfonyl)-2-pyridinesulfonamide; sethoxydim, 2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one; thifensulfuron-methyl, 3-[[[[[4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-thiophenecarboxylic acid; common lambsquarters, *Chenopodium album* L. #<sup>11</sup> CHEAL; common ragweed, *Ambrosia artemisiifolia* L. # AMBEL; giant foxtail, *Setaria faberi* Herrm. # SETFA; corn, *Zea mays* L., ‘ Dekalb 592SR2.’

**Additional index words:** Herbicide interaction, herbicide-resistant crops, atrazine, bentazon, bromoxynil, CGA 152005, clopyralid, 2,4-D, dicamba, flumetsulam, halosulfuron-methyl, nicosulfuron, primisulfuron-methyl, rimsulfuron, sethoxydim, thifensulfuron-methyl, *Ambrosia artemisiifolia*, *Chenopodium album*, *Setaria faberi*, AMBEL, CHEAL, SETFA.

**Abbreviations:** ALS, acetolactate synthase (EC 4.1.3.18); DAT, days after treatment; NIS, nonionic surfactant; POST, postemergence; RT, rimsulfuron plus thifensulfuron-methyl; SR, sethoxydim-resistant; UAN, 30% urea ammonium nitrate.

## Introduction

Sethoxydim, a cyclohexanedione herbicide, is a postemergence (POST) graminicide that controls annual and perennial grasses, and previously could be safely applied only to certain dicotyledonous crops. Sethoxydim inhibits the enzyme acetyl-coenzyme A carboxylase (ACCase) and disrupts fatty acid biosynthesis in susceptible grasses and monocotyledonous crops (Marshall et al. 1992). However, the development of sethoxydim-resistant (SR) corn has allowed the use of sethoxydim in SR corn with excellent crop safety (Dotray et al. 1993; Parker et al. 1990; VanGessel et al. 1997). SR corn was developed by an alteration in the enzyme ACCase through tissue culture and mutation breeding (Parker et al. 1990). Weed control systems utilizing SR corn with sethoxydim have been equally effective as other herbicides for grass control. Young and Hart (1997) reported sethoxydim applied alone controlled giant foxtail 8% better than nicosulfuron.

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<sup>11</sup> Letters following this symbol are a WSSA-approved computer code from *Composite List of Weeds*, Revised 1989. Available from WSSA, 810 East 10th Street, Lawrence, KS 66044-8897.



Dotray et al. (1993) observed equal or greater control of foxtail species with POST applications of sethoxydim compared to preemergence (PRE) treatments of atrazine plus alachlor [2-chloro-*N*-(2,6-diethylphenyl)-*N*-(methoxymethyl)acetamide].

Common lambsquarters, common ragweed, and giant foxtail are annual weeds and members of the Chenopodiaceae, Compositae, and Gramineae families, respectively. These species are prevalent and competitive in 40 crops throughout the world (Holm et al. 1977; Knake 1977). Despite their susceptibility to many herbicides, these weeds persist in corn fields due to prolific seed production and dormancy, the presence of large seed reserves in soil, genetic diversity, and, in the case of common lambsquarters, evolved resistance to triazine herbicides (Barbour et al. 1994; Chu et al. 1978; Darmency 1994; Fausey and Renner 1997; Mester and Buhler 1986; Saini et al. 1986). Successful corn production relies heavily on weed control, and it is estimated that competition from uncontrolled weeds may cause 30 to 90% yield losses (Hall et al. 1992). Beckett et al. (1988) reported a 12% corn yield loss at 49 common lambsquarters/10 m of row. In a Canadian study, corn yield decreased when common lambsquarters density was greater than 46 and 109 plants/m<sup>2</sup> in 1976 and 1977, respectively (Sibuga et al. 1985). Scientists have reported 25 to 52% reductions in corn yield with 180 and 200 giant foxtail plants/m, respectively (Knake and Slife 1962; Lambert et al. 1994). Fausey et al. (1997) observed corn yields were reduced 14% from only 10 giant foxtail plants/m.

Sethoxydim activity is limited to grasses, thus an effective broad spectrum weed management program must include a broadleaf herbicide mixture. Herbicide combinations are common in corn production in the United States to control broadleaf and grass weed species. Hatzios and Penner (1985) list several advantages of tank-mix combinations which include improved spectrum of weed control and reductions in crop production costs, soil compaction, and herbicide residues. However, tank mixtures applied POST to control broadleaf and grass weeds often result in reduced grass control (Corkern et al. 1998; Hart and Wax 1996; Hatzios and Penner 1985; Holshouser and Coble 1990; Jordan 1995; Snipes and Allen 1996). Hart and Penner (1993) observed that primisulfuron's efficacy was reduced by 15 and 16% when applied at 30 or 60 g/ha, respectively, in combination with 1700 g/ha atrazine to giant foxtail. Research has shown that sethoxydim's efficacy on grasses is reduced when tank-mixed with non ALS-inhibitor herbicides

such as bentazon (Campbell and Penner 1982), bromoxynil (Corkern et al. 1998; Jordan et al. 1993), pyridate [*O*-(6-chloro-3-phenyl-4-pyridazinyl) S-octyl carbonothioate] (Grichar 1991), and 2,4-D (Mueller et al. 1989; Young et al. 1996). However, Young et al. (1996) reported no reduction of giant foxtail, large crabgrass [*Digitaria sanguinalis* (L.) Scop.], or shattercane [*Sorghum bicolor* (L.) Moench] control when sethoxydim (50 g/ha) was tank-mixed with 1120 g/ha atrazine. Corkern et al. (1999) also observed no reduction in barnyardgrass [*Echinochloa crus-galli* (L.) Beauv.] or broadleaf signalgrass [*Brachiaria platyphylla* (Griseb.) Nash] control when sethoxydim was mixed with atrazine or 2,4-D. Thus, understanding interactions with herbicide combinations becomes more complex with multiple weed species in fields.

Grass control with sethoxydim can also be antagonized when tank-mixed with ALS-inhibitor herbicides (Holshouser and Coble 1990; Young et al. 1996; Young and Hart 1997). Young et al. (1996) evaluated twenty-two POST corn broadleaf herbicide combinations with sethoxydim under greenhouse conditions, and found a reduction in sethoxydim efficacy on at least one grass species in eighteen combinations. Young and Hart (1997) also reported 48% reduction in giant foxtail control with combinations of sethoxydim with primisulfuron plus CGA 152005 in SR corn. The importance of managing herbicide interactions is growing as the use of mixtures, number of potential mixtures, and the number of components in mixtures grows. Developing an effective weed management system in SR corn requires a more thorough understanding about sethoxydim interactions with corn broadleaf herbicides. Therefore, the objective of this research was to evaluate sethoxydim tank-mixed with selected ALS and non ALS-inhibiting herbicides in SR corn.

### **Materials and Methods**

Field experiments were conducted at the University of Delaware's Research and Education Center at Georgetown, DE, in 1995 and 1996. The soil was a Woodstown sandy loam (Aquic Hapludults; fine-loamy, siliceous, mesic; 78% sand, 10% silt, and 12% clay) with an organic matter content of 1.5% and pH 6.0. Cultural practices were typical of non-irrigated corn on the Delmarva peninsula. These practices included fertilizing according to soil tests, and chisel

plowing followed with a tandem disc two times. ‘Dekalb 592 SR’ corn was planted on May 17, 1995, and May 15, 1996 at 44,460 seeds/ha. Plots were 8 m long and consisted of four rows planted 76 cm apart.

POST herbicide treatments were randomly assigned to each plot as a randomized complete block with four replications. Sethoxydim at 213 g/ha was applied alone and in combination with bentazon (1120 g/ha), bentazon plus atrazine (1166 g/ha), dicamba (280 g/ha), atrazine (1120), dicamba plus atrazine (896 g/ha), bromoxynil (140 g/ha), halosulfuron-methyl (35 g/ha) plus 2,4-D (140 g/ha), CGA 152005 plus primisulfuron at 35 g/ha plus 2,4-D (140 g/ha), rimsulfuron plus thifensulfuron-methyl (RT) at 18 g/ha plus 2,4-D (140 g/ha), RT (18 g/ha) plus 280 g/ha 2,4-D, and flumetsulam plus clopyralid plus 2,4-D at 235 g/ha. An additional treatment included nicosulfuron (35 g/ha) plus bromoxynil (140 g/ha). All herbicide treatments included DASH HC<sup>®</sup> spray adjuvant<sup>12</sup> at 1% v/v except one sethoxydim plus bentazon plus atrazine treatment which included 30% urea ammonium nitrate (UAN) at 2% v/v. Also, sethoxydim tank-mixed with flumetsulam plus clopyralid plus 2,4-D and nicosulfuron plus bromoxynil were applied with only a nonionic surfactant<sup>13</sup> at 0.25% v/v. In 1995 and 1996, the corn was in the fourth visible leaf stage (2-collar) at application time. Common lambsquarters and common ragweed seedlings were 5- to 10-cm and giant foxtail were 3- to 5-cm tall at POST application, respectively. Treatments were applied with a tractor-mounted compressed-air sprayer in 234 L/ha of water at 207 kPa through flat fan spray nozzles.<sup>14</sup>

Visual estimates of crop injury and weed control were determined using a scale of 0 to 100

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<sup>12</sup> Dash HC<sup>®</sup> spray adjuvant, 45% petroleum hydrocarbons, 5% naphthalene, 1.5% phosphoric acid, and 48.5% inert ingredients. BASF Corp., 100 Cherry Hill Road, Parsippany, NJ 07054.

<sup>13</sup> Ortho X-77 nonionic surfactant with 80% principal functioning agents as: alkylaryl/polyoxy ethylene glycols, free fatty acids, and isopropanol. Valent USA Corp., 1333 North California Boulevard, Walnut Creek, CA 94596-8025.

<sup>14</sup> Teejet 8003 flat fan spray tips. Spraying Systems Co., North Avenue, Wheaton, IL 60188.

(0 = no control or crop injury and 100 = complete weed control or crop death), and evaluated 13, 26, and 90 DAT. Corn injury was minimal and not significant with any treatments (data not shown). Corn yields were determined by mechanically harvesting three rows of each plot with a small-plot combine, and grain weight was adjusted to 15.5% moisture. Control and grain yield data were subjected to analysis of variance (ANOVA). Homogeneity of variance analysis revealed no significant interactions between repetitions; therefore, data were pooled over experiments. Control estimates were subjected to arcsine transformation, yet this did not change the ANOVA. Means of nontransformed data were separated using Fisher's protected LSD test at  $P = 0.05$ .

## Results and Discussion

**Giant Foxtail Control.** Sethoxydim applied alone at 213 g/ha controlled giant foxtail  $\geq 92\%$  at all rating dates, and was equal in control to nicosulfuron plus bromoxynil (Table 1). Control of this weed was significantly reduced when sethoxydim was tank-mixed with five herbicide treatments. Averaged across rating dates, sethoxydim combined with bentazon plus atrazine plus UAN, CGA 152005 plus primisulfuron plus 2,4-D, RT plus 2,4-D at 140 and 280 g/ha, and flumetsulam plus clopyralid plus 2,4-D provided 86, 59, 60, 66, and 73% control of giant foxtail, respectively. Young et al. (1996) reported similar giant foxtail control in greenhouse studies with sethoxydim tank-mixed with bentazon plus atrazine (86%), CGA 152005 plus primisulfuron (58%), and flumetsulam plus clopyralid plus 2,4-D (82%). No differences were observed in giant foxtail control when sethoxydim was tank-mixed with bentazon plus atrazine plus either UAN or DASH. However, the mixture without DASH provided significantly less control of this weed when compared to sethoxydim alone. The addition of DASH to tank mixtures of sethoxydim with bentazon has been shown to negate antagonism of annual grass control seen by these herbicide combinations (Campbell and Penner 1982; Jordan and York 1989; Wanamarta et al. 1989; Young et al. 1996). All other tank mixtures provided excellent giant foxtail control (90%) when averaged across treatments and rating dates. These results are also similar to those reported by Young et al. (1996), who found no antagonism on giant foxtail control when sethoxydim was mixed with dicamba, atrazine, dicamba plus atrazine, bromoxynil, and halosulfuron-methyl alone or with dicamba.

**Common Ragweed and Common Lambsquarters Control.** As anticipated, sethoxydim alone did not control either weed species (Table 1). Sethoxydim tank-mixed with bentazon provided  $\leq$  58% control of common ragweed and common lambsquarters, regardless of rating date. This common ragweed control with bentazon is similar to that generally obtained under grower conditions on the Delmarva peninsula. Averaged across ratings, sethoxydim mixed with bromoxynil controlled common ragweed and common lambsquarters 80 and 91%, respectively. All other tank mixtures provided excellent control (93%) of these weed species 90 DAT when averaged over treatments.

**Corn Yield.** Corn yield was 1810 kg/ha with sethoxydim applied alone compared to the untreated check (620 kg/ha) (Table 1). Averaged across all sethoxydim tank mixtures and nicosulfuron plus bromoxynil, yields were increased 168% compared to yields from sethoxydim alone. Corn yield was 4730 kg/ha with nicosulfuron plus bromoxynil, and all sethoxydim tank mixtures provided equal yield compared to this treatment except sethoxydim mixed with atrazine (5790 kg/ha), CGA 152005 plus primisulfuron plus 2,4-D (3760 kg/ha), and RT plus 140 g/ha 2,4-D (3730 kg/ha). The latter two tank mixtures with sethoxydim reduced corn yields 26% compared to nicosulfuron plus bromoxynil. Young and Hart (1997) reported similar corn yield reductions (32%) when sethoxydim was tank-mixed with CGA 152005 plus primisulfuron. Sethoxydim tank-mixed with bentazon plus atrazine with either UAN or DASH provided equal yields. However, these mixtures provided higher corn yields compared to yields from sethoxydim mixed with bromoxynil or with RT plus 280 g/ha 2,4-D.

Understanding potential interactions between herbicides when tank-mixed is important when formulating weed management systems. Developing an effective POST weed control program in SR corn is possible by tank-mixing with selected broadleaf herbicides. Sethoxydim can also provide growers an alternative mode of action for controlling perennial grasses in corn, thereby managing herbicide resistance to sulfonyleurea herbicides. According to these studies, tank-mixing some ALS and non ALS-inhibiting herbicides with sethoxydim can reduce giant foxtail control, resulting in lower corn yields. Research from field studies showed significant antagonism on giant foxtail control when RT was tank-mixed with 2,4-D (Isaacs 2000, Ph.D. dissertation). One may speculate that adding sethoxydim to this mixture would enhance grass

control. However, these results show that when sethoxydim is tank-mixed with another herbicide which has POST grass activity in corn (RT), the antagonism on grass control cannot be overcome when the mixture contains growth regulator herbicides like 2,4-D. With the introduction of new herbicides and higher number of components in mixtures, it is critical that weed scientists thoroughly evaluate mixture performance.

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Table 1. Control of giant foxtail, common ragweed, and common lambsquarters with tank mixtures of sethoxydim with selected POST broadleaf herbicides in sethoxydim-resistant corn.<sup>a,b</sup>

Herbicide	Rate g ai/ha	Control <sup>c</sup>									Corn yield <sup>d</sup> kg/ha
		Giant Foxtail			Common ragweed			Common lambsquarters			
		13	26	90	13	26	90	13	26	90	
Sethoxydim <sup>e</sup>	213	92	96	96	0	0	0	0	0	0	1810 g
+ bentazon <sup>e</sup>	1120	84	93	94	57	57	58	58	56	52	4670 bcd
+ bentazon + atrazine <sup>e</sup>	583 + 583	86	88	90	96	95	93	97	95	94	5230 abc
+ bentazon + atrazine <sup>f</sup>	583 + 583	86	87	84	98	96	95	98	97	96	5540 ab
+ dicamba <sup>e</sup>	280	87	93	94	79	91	91	86	88	89	5230 abc
+ dicamba + atrazine <sup>e</sup>	308 + 588	89	91	91	96	97	97	97	97	96	5290 abc
+ bromoxynil <sup>e</sup>	140	77	90	96	83	80	76	91	92	90	4480 cde
+ atrazine <sup>e</sup>	1120	90	89	96	93	89	88	96	94	93	5790 a
+ halosulfuron-methyl + 2,4-D <sup>e</sup>	35 + 140	83	88	96	88	93	95	89	90	90	5540 ab
+ CGA 152005 + primisulfuron + 2,4-D <sup>e</sup>	17 + 18 + 140	55	60	61	93	95	95	92	94	91	3760 e
+ rimsulfuron + thifensulfuron + 2,4-D <sup>e</sup>	12 + 6 + 140	54	61	64	83	87	87	93	95	97	3730 ef
+ rimsulfuron + thifensulfuron + 2,4-D <sup>e</sup>	12 + 6 + 280	58	68	73	96	94	94	93	95	95	4050 de
+ flumetsulam + clopyralid + 2,4-D <sup>e</sup>	26 + 69 + 140	60	74	84	74	85	90	92	92	90	5040 abc
Nicosulfuron + bromoxynil <sup>e</sup>	35 + 140	86	92	95	92	94	91	93	93	92	4730 bcd
Untreated check		0	0	0	0	0	0	0	0	0	620 h
LSD (0.05)		6	5	6	4	5	5	4	4	5	870

<sup>a</sup>Data presented are the means combined over years.

<sup>b</sup>Percent control from 0-100; 0 (no control), 100 (complete control).

<sup>c</sup>Control ratings recorded 13, 26, 90, DAT.

<sup>d</sup>Means followed by the same letter are not significantly different according to Fishers Protected LSD test at P = 0.05.

<sup>e</sup>DASH HC® spray adjuvant was applied at 1% v/v.

<sup>f</sup>Urea ammonium nitrate (30% UAN) was applied at 2% v/v.

<sup>g</sup>Nonionic surfactant (NIS) applied at 0.25% v/v.

## Chapter VII

### Summary and Conclusions

Corn field studies investigating halosulfuron-methyl tank-mixed with 2,4-D or dicamba indicated good to excellent control ( $\geq 83\%$ ) of common lambsquarters and common ragweed 60 DAT, regardless of the growth regulator used. Halosulfuron-methyl alone controlled common ragweed but did not control common lambsquarters. 2,4-D or dicamba alone at 140 g/ha controlled common lambsquarters, but only this rate of dicamba controlled common ragweed. Combinations of halosulfuron-methyl with these growth regulators were generally additive for control of these weed species, and occasionally synergistic on common lambsquarters control at early ratings. High rates of halosulfuron-methyl tank-mixed with high rates of 2,4-D and dicamba controlled common lambsquarters more than low rate combinations 60 DAT. However, common ragweed control did not differ between low and high rate herbicide combinations. No differences were observed in biomass reduction of either weed species or in corn grain yields when comparing low to higher rates of all herbicide mixtures. Therefore, tank mixtures of these herbicides even at reduced rates effectively control these weed species without decreasing corn yields.

Synergistic control of common lambsquarters was observed in the greenhouse with halosulfuron-methyl rates as low as 4.5 g/ha tank-mixed with 70 g/ha 2,4-D. Mixtures of these herbicides generally exhibited additivity for control of this weed. The addition of 2,4-D to halosulfuron-methyl did not affect absorption and translocation of  $^{14}\text{C}$ -halosulfuron-methyl. However, the addition of 2,4-D increased the level of metabolite M3 at the 18 g/ha halosulfuron-methyl rate, which may contribute to common lambsquarters phytotoxicity. Future research should examine the effect of 2,4-D on the ALS enzyme site, focusing on ALS activity in protein extracts from common lambsquarters as affected by halosulfuron-methyl concentrations.

Field studies showed that giant foxtail control was antagonized when rimsulfuron plus thifensulfuron-methyl (RT) was combined with 2,4-D. Common ragweed control was synergistic when RT at 13 g/ha was mixed with 140 g/ha 2,4-D, while other combinations exhibited additivity. All RT combinations with 2,4-D and with dicamba were equally effective in reducing common ragweed biomass, providing an average reduction of 99%. Averaged over rates and

rating dates, RT combinations with dicamba controlled giant foxtail 91% and mixtures were additive. Low rates of RT tank-mixed with low rates of 2,4-D and dicamba provided equal control and biomass reduction of both weed species, and similar corn grain yields compared to higher rate combinations. Therefore, tank mixtures of RT with dicamba provide effective POST weed control in corn even at low rates.

RT tank-mixed with primisulfuron, CGA 152005 plus primisulfuron, halosulfuron-methyl, and dicamba controlled giant foxtail, common ragweed, and common lambsquarters in the field. Tank mixtures of RT with flumetsulam plus clopyralid plus 2,4-D, atrazine, 2,4-D, and dicamba plus atrazine antagonized giant foxtail control as much as 25%. RT mixed with flumetsulam plus clopyralid plus 2,4-D injured corn 26%, and yields were reduced 34% when compared to RT alone. RT tank-mixed with halosulfuron-methyl injured corn 11%, and corn yield was 5040 kg/ha. All other tank mixtures with RT provided minimal corn injury ( $\leq 11\%$ ) and equal yields. Therefore, caution must be observed when tank mixing RT with selected POST broadleaf herbicides, especially when multiple weed species are present in a field.

POST tank mixtures of sethoxydim with various ALS and non ALS-inhibitor herbicides in sethoxydim-resistant (SR) corn antagonized giant foxtail control. Sethoxydim tank-mixed with bentazon plus atrazine with urea ammonium nitrate (UAN), or with ALS-inhibiting herbicides plus 2,4-D except halosulfuron-methyl reduced giant foxtail control. Antagonism on giant foxtail control when RT was tank-mixed with 2,4-D was not overcome when sethoxydim was added to the mixture. Most herbicide combinations controlled common ragweed and common lambsquarters 90 DAT, except when bentazon and bromoxynil were included. Averaged across all sethoxydim tank mixtures and nicosulfuron plus bromoxynil, corn yields were increased 168% compared to sethoxydim alone. Yields of corn treated with sethoxydim or sethoxydim mixed with combinations of sulfonyleurea herbicides plus 2,4-D were low, with the exception of halosulfuron-methyl. According to these studies, thoroughly understanding tank mixtures with sethoxydim and ALS and non ALS-inhibiting POST broadleaf corn herbicides is crucial for effective weed management.

## VITA

Mark A. Isaacs was born the second child to Mr. and Mrs. George S. Isaacs on February 22, 1962 in Milford, Delaware. He grew up on a family swine, poultry, equine, and grain farm in Georgetown, Delaware and graduated from Sussex Central High School in 1980. He received a Bachelor of Science degree in Agronomy (Plant and Soil Science) with a minor in business and a Master of Science degree in Agronomy (Weed Science) with a minor in experimental statistics from Clemson University in 1984 and 1986, respectively. Mark accepted a position as an Extension Assistant in nutrient management and water quality with the University of Delaware in the summer of 1986. In 1987, Mark became the Crops Research Coordinator for the College of Agriculture and Natural Resources' Research and Education Center (REC) in Georgetown, Delaware. In 1991, he became the Director of the REC where his responsibilities included overseeing the operations of the Experiment Station. In the fall of 1994, Mark took a sabbatical from the University for one year to attend Virginia Polytechnic Institute and State University to pursue a Ph.D. in Weed Science. After completing appropriate course and laboratory research, Mark returned to Delaware to conduct his field research and return to his job as Director of the REC. Mark completed his requirements for a Doctor of Philosophy degree in April of 2000.