

# Spray carrier pH effect on absorption and translocation of trifloxysulfuron in Palmer amaranth (*Amaranthus palmeri*) and Texasweed (*Caperonia palustris*)

M. A. Matocha

Corresponding author. Agricultural and Environmental Safety, Texas Cooperative Extension, 115 Agronomy Field Lab, College Station, TX 77843-2488; ma-matocha@tamu.edu

L. J. Krutz

Southern Weed Science Research Unit, Agricultural Research Service, U.S. Department of Agriculture, P.O. Box 350, Stoneville, MS 38776

S. A. Senseman

Department of Soil and Crop Sciences, Texas Agricultural Experiment Station, Texas A&M University, College Station, TX 77843

C. H. Koger

Crop Genetics and Production Research Unit, Agricultural Research Service, U.S. Department of Agriculture, P.O. Box 345, Stoneville, MS 38776

K. N. Reddy

Southern Weed Science Research Unit, Agricultural Research Service, U.S. Department of Agriculture, P.O. Box 350, Stoneville, MS 38776

E. W. Palmer

Syngenta Crop Protection, 118 Kennedy Flat Road, Leland, MS 38756

Trifloxysulfuron is a sulfonylurea (SU) herbicide developed for POST weed control in cotton (*Gossypium hirsutum* L.), sugarcane (*Saccharum* spp.) and turfgrass (Holloway et al. 2000; Hudetz et al. 2000). It is a weak acid with a  $pK_a$  of 4.81, and its water solubility is related to the  $pK_a$  of the hydrogen atom on the urea bridge and the pH of the solution. The water solubility of trifloxysulfuron at pH 5 and 7 is 63 mg L<sup>-1</sup> and 5,016 mg L<sup>-1</sup>, respectively (Vencill 2002). Although several studies have reported on the effect of pH on efficacy of SUs, few studies have evaluated the effect of carrier pH on the absorption and translocation of SUs in general and trifloxysulfuron specifically when applied to foliage of susceptible plants (Green and Cahill 2003; Green and Hale 2005a; Nalewaja et al. 1997).

Generally, the uptake of weak acids by plant tissues is greater at lower carrier pH because of a higher proportion of the molecules being present in an undissociated form. Yet, Liu et al. (2002) reported greater uptake of bentazon by white mustard (*Sinapis alba* L.) and wheat (*Triticum aestivum* L.) plant leaves at pH 7 and 9 compared with pH 5. Absorption of bentazon by field bean (*Vicia faba* L.) was minimal regardless of carrier pH. Similarly, researchers have indicated that the addition of adjuvants that increase pH of the spray mixture increase the biological activity of nicosul-

Sulfuron under specific conditions (Green and Cahill 2003). These adjuvants called “basic blends” intentionally increase the pH of the spray solution to solubilize SU herbicides (Green and Hale 2005a). Collectively, these studies indicate that increasing the pH of the carrier solution may enhance absorption and translocation of trifloxysulfuron in some species.

Trifloxysulfuron has activity on numerous broadleaf, grass, and sedge species (Koger et al. 2005; Porterfield et al. 2003; Richardson et al. 2004) that are troublesome in row crop production systems. Palmer amaranth and Texasweed are annual broadleaf weeds that are troublesome in cotton production. Morgan et al. (2001) reported that cotton yields decreased linearly 13 to 54% because of 1 to 10 Palmer amaranth plants per 9.1 m of row. Moreover, Palmer amaranth in cotton can increase mechanical harvest time at densities greater than 650 plants ha<sup>-1</sup> (Smith et al. 2000). In recent years, Texasweed has become a problem weed in soybean [*Glycine max* (L.) Merr.] and rice (*Oryza sativa* L.) production in the midsouth United States (Koger et al. 2004) and in cotton along the Texas Upper Gulf Coast (Matocha and Baumann 2004). Texasweed seed germinates and survives under a broad range of climatic and edaphic conditions (Koger et al. 2004) and is difficult to control

**Nomenclature:** Trifloxysulfuron; Palmer amaranth, *Amaranthus palmeri* S. Wats. AMAPA; Texasweed, *Caperonia palustris* (L.) St. Hil. CNPPA.

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with glyphosate and other cotton herbicides including pyri-thiobac, MSMA, DSMA, and fluometuron (Matocha and Baumann 2004). The effect of pH has been shown to vary with weed species and herbicide (Green and Hale 2005b). Adjusting the pH of the spray carrier may enhance absorption and subsequent translocation of trifloxysulfuron by these species; therefore, studies were conducted to evaluate the absorption and translocation of foliar-applied trifloxysulfuron in Palmer amaranth and Texasweed as influenced by pH of the spray carrier.

## Materials and Methods

### Plant Material

Palmer amaranth and Texasweed plants were propagated from seed in a greenhouse. Texasweed seeds were planted in 26- by 52- by 6-cm trays containing a mixture of Bosket sandy loam (fine-loamy, mixed thermic Mollic Hapludalfs) and Jiffy Mix potting soil<sup>1</sup> (1 : 1, v/v). Seeds were spread on top of the soil mixture and subirrigated with distilled water. After emergence, seedlings in the cotyledon growth stage were transplanted into 11-cm-diam pots containing potting soil. Palmer amaranth seeds were planted directly into 11-cm-diam pots containing a mixture of Bosket sandy loam and Jiffy Mix potting soil (1 : 1, v/v). Each treatment unit consisted of 1 plant per pot. Plants were grown at 32/ 25 C ( $\pm$  3 C) day/night temperatures and were subirrigated as needed. Palmer amaranth plants were at the six- to eight-leaf stage and Texasweed plants were at the three- to four-leaf stage when <sup>14</sup>C-trifloxysulfuron was applied. Plants were not presprayed with formulated trifloxysulfuron before application of <sup>14</sup>C-trifloxysulfuron to minimize stress during the exposure period. Pretreated (Camacho and Moshier 1991) and nontreated (Gillespie 1994) plants have produced similar absorption and translocation trends when sulfonyl-urea herbicides were spotted on the leaves.

### Absorption and Translocation

Technical grade [pyridinyl-2-<sup>14</sup>C] trifloxysulfuron with 815.7 kBq  $\mu$ mol<sup>-1</sup> specific activity and 95.9% radiochemical purity was used in these experiments. <sup>14</sup>C-trifloxysulfuron was dissolved in high-performance liquid chromatography (HPLC)-grade methanol and deionized water. Radioactive solutions were a mixture of KH<sub>2</sub>PO<sub>4</sub> : NaOH-buff-

ered deionized water at pH 5, 7, and 9 and 0.25% v/v nonionic surfactant.<sup>2</sup> Final treatment solution contained 5% (v/v) methanol. A 5- $\mu$ l volume of each treatment solution containing 2.8 kBq radioactivity was placed on the adaxial surface of the fourth true leaf of a Palmer amaranth or the second true leaf of a Texasweed plant as 15 droplets.

Plants were harvested at 4, 24, 48, and 72 h after treatment (HAT) and separated into treated leaf, aerial sections above and below treated leaf, and roots. The treated leaf, including the petiole, was rinsed by gently shaking for 15 s with 15 ml methanol : water (1 : 1, v/v) to remove nonabsorbed herbicide. Two 1-ml aliquots of the leaf rinse were added to 10 ml scintillation fluid,<sup>3</sup> and radioactivity was quantified via liquid scintillation spectrometry<sup>4</sup> (LSS). Plant sections were wrapped in tissue paper,<sup>5</sup> placed in glass scintillation vials and oven-dried for 48 h at 45 C. Oven-dried plant samples were combusted with a biological sample oxidizer.<sup>6</sup> Sample radioactivity was quantified by LSS.

The amount of <sup>14</sup>C present in the leaf washes and plant sections was considered as total <sup>14</sup>C recovered, which averaged 91% of applied <sup>14</sup>C-trifloxysulfuron. Sum of the radioactivity present in all plant parts was considered as absorption and expressed as percentage of the <sup>14</sup>C recovered. Radioactivity in all plant parts except the treated leaf was considered as translocated and expressed as a percentage of the <sup>14</sup>C recovered.

Treatments were replicated three times and the experiment was repeated. Data were subjected to ANOVA with sums of squares partitioned to reflect a split-split-plot treatment structure and trial effects. The four harvest timings were considered as main plots, the two species were considered as subplots, and the three pH levels were considered as sub-subplots. Trial effects were considered random, and mean squares were tested appropriately based on the treatment design (McIntosh 1983). Where main plot effects were significant, regressions were used to explain the relationship of measured responses over time. Where appropriate, significant main effects were averaged over harvest interval, species, and pH and separated by Fisher's protected least significant difference at P < 0.05.

## Results and Discussion

Trial effects were not significant at any harvest interval, plant species, or pH. Statistical significance levels for ab-

TABLE 1. Statistical significance levels for absorption, translocation, and partitioning to selected plant sections of trifloxysulfuron in Palmer amaranth and Texas weed at pH levels 5, 7, and 9.<sup>a</sup>

Effect	df <sup>b</sup>	P > F					
		Absorption	Translocation	Treated leaf	Above treated leaf	Below treated leaf	Roots
Block	5	0.5822	0.4089	0.4457	0.7757	0.0944	0.1014
Time	3	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*	0.0006*
Species	1	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*	< 0.0001*	0.2753
pH	2	0.0011*	0.0189*	0.0041*	0.3958	0.0197*	0.0468*
Time $\times$ species	3	0.1855	0.0132*	0.4520	0.0024*	0.1072	0.0872
Time $\times$ pH	6	0.2188	0.0754	0.1360	0.2888	0.0705	0.0306*
Species $\times$ pH	2	0.5308	0.0222*	0.9269	0.2786	0.0516	0.0168*
Time $\times$ species $\times$ pH	6	0.2204	0.0094*	0.6701	0.0959	0.1913	0.0711

<sup>a</sup> Asterisks show factors that were significant at the 0.05 level of probability.

<sup>b</sup> Degrees of freedom were the same for all parameters.

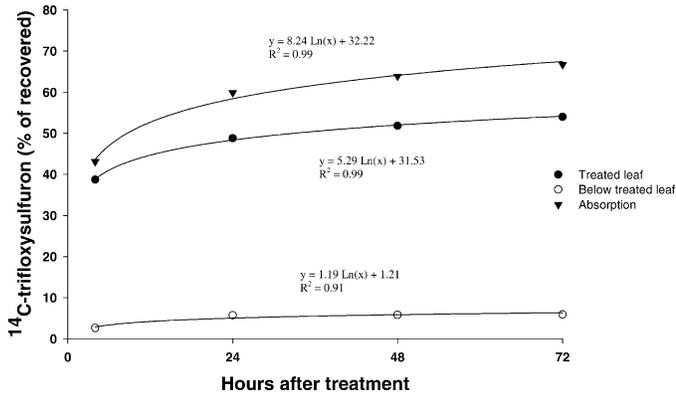


FIGURE 1. Absorption and accumulation of  $^{14}\text{C}$ -trifloxysulfuron in treated leaf and below treated leaf sections averaged over pH (5, 7, and 9) and species (Palmer amaranth and Texasweed).

sorption, translocation, and partitioning to selected plant sections of trifloxysulfuron are shown in Table 1. Main effects for the absorption of  $^{14}\text{C}$ -trifloxysulfuron were significant. Absorption of  $^{14}\text{C}$ -trifloxysulfuron followed a logarithmic trend with the majority of the herbicide absorbed by 24 HAT (Figure 1). A logarithmic increase in the foliar absorption of trifloxysulfuron has been reported for various species including cotton, jimsonweed (*Datura stramonium* L.), peanut (*Arachis hypogaea* L.), sicklepod [*Senna obtusifolia* (L.) Irwin and Barneby], purple nutsedge (*Cyperus rotundus* L.), yellow nutsedge (*Cyperus esculentus* L.), green kyllinga (*Kyllinga brevifolia* Rottb.), and false green kyllinga (*Kyllinga gracillima* L.) (Askew and Wilcut 2002; McElroy et al. 2004; Troxler et al. 2003). Generally, for the aforementioned studies, absorption of trifloxysulfuron was rapid and occurred by 4 HAT.

Absorption of  $^{14}\text{C}$ -trifloxysulfuron by Palmer amaranth was greater than that by Texasweed (Table 2). Askew and Wilcut (2002) reported that absorption of  $^{14}\text{C}$ -trifloxysulfuron decreased in the order of jimsonweed > sicklepod > peanut > cotton. Wilcut et al. (2000) reported > 95% early season control of Palmer amaranth with  $^{14}\text{C}$ -trifloxysulfuron. Low absorption of  $^{14}\text{C}$ -trifloxysulfuron by Texasweed may contribute to poor weed control observed along Texas Upper Gulf Coast (P. Baumann, personal communication).

Absorption of trifloxysulfuron decreased in the order of pH 9 = pH 7 > pH 5 (Table 3). Greater absorption of

TABLE 2. Absorption and distribution of  $^{14}\text{C}$ -trifloxysulfuron into treated leaf and below treated leaf plant sections in Palmer amaranth and Texasweed averaged over time and pH (5, 7, and 9).<sup>a</sup>

Species	$^{14}\text{C}$ -trifloxysulfuron distribution <sup>c</sup>		
	Absorption <sup>b</sup>	Treated leaf	Below treated leaf
	————— % of recovered —————		
Palmer amaranth	88.0	73.7	7.1
Texasweed	28.8	23.0	2.9
LSD (0.05)	1.2	3.3	0.8

<sup>a</sup> Plants were maintained in the greenhouse at 32/25 C day/night temperatures.

<sup>b</sup>  $^{14}\text{C}$  present in all plant parts was considered as absorption and expressed as percentage of the  $^{14}\text{C}$  recovered.

<sup>c</sup>  $^{14}\text{C}$ -trifloxysulfuron distribution throughout the plant is based on the percentage of  $^{14}\text{C}$ -trifloxysulfuron recovered.

TABLE 3. Effect of pH (5, 7, and 9) on absorption and partitioning of  $^{14}\text{C}$ -trifloxysulfuron to treated leaf and below treated leaf plant sections averaged over time and species.<sup>a</sup>

pH	$^{14}\text{C}$ -trifloxysulfuron distribution <sup>c</sup>		
	Absorption <sup>b</sup>	Treated leaf	Below treated leaf
	————— % of recovered —————		
5	53.8	44.9	4.4
7	60.4	50.1	5.5
9	61.0	50.0	5.2
LSD (0.05)	1.4	3.5	0.8

<sup>a</sup> Plants were maintained in the greenhouse at 32/25 C day/night temperatures.

<sup>b</sup>  $^{14}\text{C}$  present in all plant parts was considered as absorption and expressed as percentage of the  $^{14}\text{C}$  recovered.

<sup>c</sup>  $^{14}\text{C}$ -trifloxysulfuron distribution throughout the plant is based on the percentage of  $^{14}\text{C}$ -trifloxysulfuron recovered.

trifloxysulfuron at high pH is in contrast to most weak acid herbicides. Liu (2002) noted that the absorption of most weak acid herbicides into plant tissues is greater at a low carrier pH because of a greater proportion of the molecules being present in an undissociated form. However, most of the reviewed studies were conducted with plant tissues immersed or cultured cells incubated in a solution of nonformulated weak acid herbicides (Liu 2002). Liu (2002) did observe greater absorption of bentazon by white mustard and wheat with spray carrier pH of 7 and 9 as compared with pH 5. Another study found that the addition of adjuvants that increased pH of the spray mixture increased the biological activity of high nicosulfuron rates at low spray volumes on difficult-to-control weeds (Green and Cahill 2003).

Decreased absorption of trifloxysulfuron at pH 5 may be because of reduced solubility at low pH. Liu (2002) noted that solubility in droplet residue can affect the uptake of weakly acidic molecules, perhaps by crystallization on the leaf surface. As water evaporates from the spray droplet, herbicide uptake will be reduced if the herbicide crystallizes or precipitates on the leaf (Nalewaja 2000). At pH 7 and 9, spray carriers produce more dissociated anions that encounter greater difficulty in diffusing through the cuticle, this effect may be offset by greater water solubility.

The main effects and interaction of pH, species, and har-

TABLE 4. Translocation of  $^{14}\text{C}$ -trifloxysulfuron in Palmer amaranth and Texasweed as related to spray carrier pH and harvest interval.<sup>a</sup>

Species	pH	Harvest interval				LSD
		4	24	48	72	
		————— % of recovered —————				
Palmer amaranth	5	7.8 <sup>b</sup>	13.9	18.7	16.6	4.4
	7	6.3	14.7	18.5	18.1	5.1
	9	5.8	19.4	14.9	16.3	3.4
LSD		NS	NS	NS	NS	
Texasweed	5	2.4	3.0	4.7	4.6	NS
	7	1.7	10.2	7.0	10.4	4.2
	9	2.1	5.5	9.0	10.1	4.5
LSD		NS	3.6	NS	5.1	

<sup>a</sup> Plants were maintained in the greenhouse at 32/25 C day/night temperatures.

<sup>b</sup> LSD may be used for comparison of mean values within species.

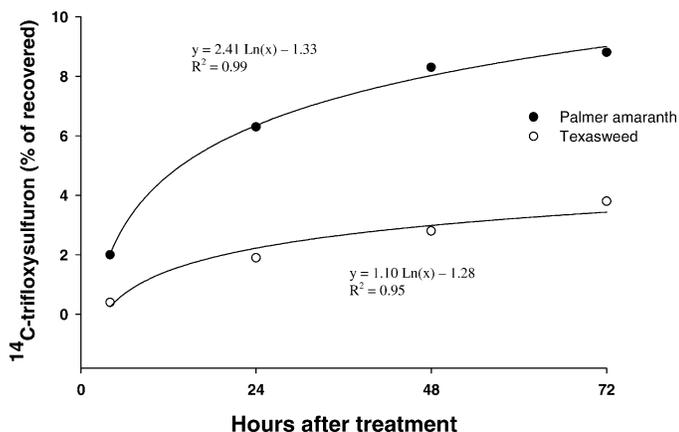


FIGURE 2. Partitioning of  $^{14}\text{C}$ -trifloxysulfuron in the above treated leaf sections of Palmer amaranth and Texasweed averaged over pH (5, 7, and 9).

vest intervals were significant ( $P < 0.05$ ) for the translocation of trifloxysulfuron. Therefore, translocation data were analyzed by weed species. Absorption of trifloxysulfuron in Palmer amaranth did not translate into differences in translocation (Table 4). However, translocation of trifloxysulfuron in Texasweed followed the pattern of trifloxysulfuron absorption and increased in the order of pH 5 (5%) < pH 7 (10%) = pH 9 (10%). These differences in translocation are because of reduced absorption of trifloxysulfuron at acidic pH (Table 3). Less than 11 and 20% of recovered  $^{14}\text{C}$ -trifloxysulfuron moved out of the treated leaves of Texasweed and Palmer amaranth, respectively. Wilcut et al. (1989) found that although two crop species and four weed species varied in amounts of  $^{14}\text{C}$ -chlorimuron that moved out of the treated leaf, no species translocated > 25% of applied radioactivity.

For recovery of  $^{14}\text{C}$ -trifloxysulfuron in the treated leaf and below treated leaf, main effects for harvest interval, species, and pH were significant. Averaged over species and pH, the recovery of trifloxysulfuron increased logarithmically for both treated leaf and below treated leaf (Figure 1). Previous research also showed logarithmic trends for  $^{14}\text{C}$ -trifloxysulfuron recovered from plant parts (Troxler et al. 2003). Averaged over time and pH, the recovery of trifloxysulfuron was greater in the treated leaf and below treated leaf for Palmer amaranth compared with Texasweed (Table 2). Another study found that jimsonweed absorbed and translocated more  $^{14}\text{C}$ -trifloxysulfuron than other weed and crop species (Askew and Wilcut 2002). However, the majority of recovered  $^{14}\text{C}$ -trifloxysulfuron remained in the treated leaf. Troxler et al. (2003) also reported that the majority of applied  $^{14}\text{C}$ -trifloxysulfuron remained in treated leaves of yellow and purple nutsedge. Less than 6% of  $^{14}\text{C}$ -nicosulfuron and primisulfuron moved out of treated leaves of eastern black nightshade (*Solanum ptycanthum* Dun.) (Carey et al. 1997). Averaged over species and harvest interval, recovery of  $^{14}\text{C}$ -trifloxysulfuron increased in the order of pH 5 < pH 7 = pH 9. Greater translocation of  $^{14}\text{C}$ -trifloxysulfuron at higher pH was likely because of greater absorption of trifloxysulfuron at these pH levels.

Trifloxysulfuron accumulation in above-treated leaf was significant for harvest interval by species interaction. At all harvest intervals, Palmer amaranth translocated more  $^{14}\text{C}$ -trifloxysulfuron into above-treated leaf sections than Texas-

weed (Figure 2). Askew and Wilcut (2002) showed that jimsonweed translocated significantly more  $^{14}\text{C}$ -trifloxysulfuron to meristems than cotton, peanut, or sicklepod.  $^{14}\text{C}$ -trifloxysulfuron partitioning into roots accounted for less than 2% of recovered  $^{14}\text{C}$  for all pH and species combinations (data not shown). Manley et al. (1999) reported very limited translocation of nicosulfuron and chlorimuron to roots of smooth pigweed (*Amaranthus hybridus* L.), indicating reduced basipetal translocation.

In summary, acidic spray carrier pH led to decreased absorption of trifloxysulfuron when averaged over harvest interval and species. Consequently, reduced absorption of trifloxysulfuron resulted in decreased translocation in Texasweed at acidic spray carrier pH. These data suggest that increasing the pH of the spray carrier by approximately 2 pH units above the  $\text{pK}_a$  can enhance absorption and subsequent translocation of trifloxysulfuron in some weed species as evident in Palmer amaranth and Texasweed.

## Sources of Materials

<sup>1</sup> Jiffy mix, Jiffy Products of America Inc., 951 Swanson Drive, Batavia, IL 60510.

<sup>2</sup> Induce® Nonionic low foam wetter-spreader adjuvant contains 90% nonionic surfactant (alkylarylpoloxyalkane ether isopropanol, free fatty acids), and 10% water, Helena Chemical Company, Suite 500, 6075 Poplar Avenue, Memphis, TN 38137.

<sup>3</sup> Ecolume, ICN, 330 Hyland Avenue, Costa Mesa, CA 92626.

<sup>4</sup> Tri-carb 2500TR Liquid Scintillation Analyzer, Packard Bio-Science Company, 800 Research Parkway, Downers Grove, IL 60515.

<sup>5</sup> Kimwipes EX-L, Kimberly-Clark Corporation, 1400 Holcomb Bridge Road, Roswell, GA 30076.

<sup>6</sup> Packard Oxidizer 306, Packard Instruments Company, 2200 Warrenville Road, Downers Grove, IL 60515.

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