

Residual phytotoxicity and persistence of chlorimuron and metsulfuron in soils of Argentina

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Abstract: Studies were conducted to determine the residual phytotoxicity and persistence of several rates of chlorimuron and metsulfuron in two soils of Buenos Aires Province, Argentina, using a sunflower bioassay procedure. Chlorimuron at 0, 3.6, 7.2, 14.4, and 21.6 ng g⁻¹ and metsulfuron at 0, 1.15, 2.3, 4.6, and 6.9 ng g⁻¹ were applied to a Balcarce loam soil (7.5% organic matter content, and pH 5.9), and a San Cayetano loam soil (4.0% organic matter, and pH 6.8). The residual phytotoxicity (time to achieve 90% of relative plant height to the untreated check:R₉₀) for both herbicides, was greater in San Cayetano soil than in Balcarce soil, likewise R₉₀ of metsulfuron was longer than chlorimuron in both soils. The residual phytotoxicity and persistence of chlorimuron and metsulfuron increased in the two soils with increasing rate of application. Depending on the rate, half-lives for chlorimuron ranged from 30 to 43 days at Balcarce and from 50 to 69 days at San Cayetano. In the case of metsulfuron, the half-lives were between 38 to 51 days in Balcarce soil and from 54 to 84 days in San Cayetano soil.

Key words: Soil persistence, Chlorimuron, Metsulfuron, Bioassay, Sunflower.

Introduction

Chlorimuron and metsulfuron are sulfonylurea herbicides registered in Argentina for broadleaf weed control in soybeans and wheat respectively. Application in these crops is recommended on early postemergence at rates of 10-15 g ai ha⁻¹ for chlorimuron and 4-6 g ai ha⁻¹ for metsulfuron (Pórfido, 2003). Even at low rates, these herbicides can persist in the soil throughout more than one growing season and may injure rotational crops (Kotoula-Syka *et al.*, 1993a, 1993b; Monks and Banks, 1991; Moyer, 1995; Junnila *et al.*, 1994; Vicari *et al.*, 1994). Sulfonylurea herbicides show a wide range of persistence in both laboratory and field conditions, depending upon soil pH, temperature, and soil moisture. Several authors reported that persistence of sulfonylurea herbicides increased with increasing rate of application (Eleftherohorinos, 1987; Eleftherohorinos and Kotoula-Syka, 1989; Kotoula-Syka *et al.*, 1993a; Kotoula-Syka *et al.*, 1993b), increasing soil pH and decreasing organic matter content (Smith and Hsiao, 1985; Goetz *et al.*, 1989; Castro *et al.*, 2002).

Half-life of chlorimuron in a non sterile silt loam soil with 1.1 to 1.4% organic matter content was 7 days at pH 6.3 and 18 days at pH 7.8 (Brown, 1990). Schroeder (1994) determined that half-life of chlorimuron in soils from 11 locations in southern United States was 12 to 50 days. Vencill and Banks (1994) reported that the time for 50% dissipation of bioavailable chlorimuron in nine soils in southern United States varied by year and soil between 5 and 20 days. Half-lives of metsulfuron in surface soils incubated under uniform temperature and moisture conditions from eight locations of United Kingdom ranged from 23 to 73 days (Walker *et al.*, 1989). James *et al.*, (1995) reported that half-life of metsulfuron ranged from 8 to 36 days in New Zealand. Cranmer *et al.*,

(1999) studied dissipation of metsulfuron in six soils in Colorado, United States and determined that half-life ranged from 11.8 to 27.7.

Although sulfonylureas are broadly used in Argentina, information regarding the biological persistence in soil of chlorimuron and metsulfuron is very limited or inexistent, especially on sunflower that is one of the main crops seeded after applications of these herbicides. For this reason the objective of the study was to determine the phytotoxic persistence on sunflower of chlorimuron and metsulfuron applied at different rates in two soils.

Materials and Methods

General procedures: The soils used in this study were a Balcarce loam (fine silty, mixed, thermic, mixed illitic-montmorillonitic, Typic Argiudoll) with 46% sand, 28% silt, 26% clay, 7.5% organic matter, and pH 5.9, and a San Cayetano loam (fine, thermic, mixed illitic-montmorillonitic, Petrocalcic Paleudoll) with 38% sand, 32% silt, 30% clay, 4.0% organic matter, and pH 6.8. Soil samples were collected from 0-15 cm depth at Balcarce and San Cayetano locations in southeast Buenos Aires Province, Argentina. Previous land use records of the sampling sites reported no history of sulfonylurea herbicide use. The samples were homogenized, air dried, and sieved to a particle size of 2 mm.

Aqueous solutions (150 ml) of chlorimuron at 0, 3.6, 7.2, 14.4, and 21.6 ng g⁻¹, or metsulfuron at 0, 1.15, 2.3, 4.6, and 6.9 ng g⁻¹ were prepared and applied to soils using a hand sprayer. Each sample was then thoroughly mixed to allow a homogeneous distribution of herbicides. A completely randomized design with five single-pot replicates for each concentration was used.

Table – 1: Sunflower response^a to chlorimuron and metsulfuron in two soils.

Soil	Herbicide	Intercept ± SE ln (RPH%)	Slope ± SE ln (RPH%) (ng g ⁻¹) ⁻¹	p ^b	r ²	GR ₁₀ ^c ng g ⁻¹	GR ₅₀ ^c ng g ⁻¹
Balcarce	chlorimuron	4.5372±0.0423	0.0126±0.0035	0.0363	0.81	8.4	55
	metsulfuron	4.5728±0.0198	0.0701±0.0051	0.0008	0.98	1.5	9.9
San Cayetano	chlorimuron	4.5558±0.0347	0.0272±0.0029	0.0025	0.97	3.9	25.5
	metsulfuron	4.5159±0.0530	0.1235±0.0136	0.0028	0.96	0.9	5.6

^aEquations were of the form $\ln(y) = \ln(a) - bx$ where x = chlorimuron or metsulfuron concentration and y = sunflower RPH.; ^bProbability level for slope; ^cGR₁₀ and GR₅₀ = concentration of chlorimuron or metsulfuron that reduced sunflower RPH by 10% or 50% respectively. Calculated from regression equation.

A sunflower bioassay was used to evaluate soils for herbicidal activity. Eight sunflower seeds (SPS 3130) were seeded in pots of 400 g dry weight at 0, 43, 85, 127 and 169 days after treatment (DAT), and then were thinned to four plants. Bioassays were conducted in a growth chamber at $20 \pm 2^\circ\text{C}$, and $14 \pm 2^\circ\text{C}$ of day and night temperature respectively, with a 14/10-hr light/dark cycle. The pots were irrigated with distilled water as necessary to maintain soil at 80% field capacity. The phytotoxicity of herbicides was measured when plants achieved the two-leaf stage in terms of relative plant height (RPH) to the untreated check expressed in percentage. Sunflower plant heights were measured from the soil line to the apex meristem. Seeding was made until neither external symptoms of phytotoxicity in the plants nor differences among concentrations were visible. For this reason seeding at 169 DAT in Balcarce soil was not made.

Data analysis: Chlorimuron and metsulfuron standard response curves were calculated for each soil. The natural logarithm (ln) of RPH for each herbicide and the average for each herbicide concentration was determined. Standard response equations were calculated for each herbicide and soil by regressing the ln (RPH) against concentration. The concentration of herbicide required in soils to reduce RPH for 10 (GR₁₀) and 50 (GR₅₀) percent were calculated for the first crop and each replication.

Individual equations for each herbicide/concentration/soil combination were used to determine the residual phytotoxicity of either chlorimuron or metsulfuron. Equations were calculated by regressing ln (RPH) against DAT. Residual phytotoxicity (R₉₀) was calculated as days to achieve 90% RPH.

Individual standard equations for each herbicide/concentration/soil combination were used to determine the concentration of either chlorimuron or metsulfuron in each soil sample from the persistence studies. The mean concentration of chlorimuron and metsulfuron was initially regressed against days for each herbicide, concentration, and soil. Half-life (time to obtain the 50% of initial rate) of each herbicide/concentration combination in each soil was calculated from the regression equations.

Results and Discussion

Initial activity: A linear equation provided the best fit of the data. Response of sunflower RPH to increasing concentrations

of chlorimuron and metsulfuron for all soils was significantly different considering a probability level of 6% ($p \leq 0.06$) (Table 1). Slopes of regression equations were higher for San Cayetano than for Balcarce soil. This indicates that response of sunflower RPH to increasing concentrations of both herbicides was greater at San Cayetano soil than at Balcarce soil. Likewise, the response of sunflower RPH in both soils was greater for metsulfuron than chlorimuron. Only the GR₁₀ value of metsulfuron at San Cayetano was out of range of concentrations applied. In the case of GR₅₀ only metsulfuron value in San Cayetano soil was within the range of concentrations applied. The GR₁₀ and GR₅₀ values were about 2.2 times for chlorimuron and 1.7 times greater for metsulfuron in Balcarce soil than in San Cayetano soil. These results could be explained by the high organic matter content and low pH of the Balcarce soil that has shown to reduce the phytotoxicity of the sulfonylurea herbicides (Günther *et al.*, 1989). The highest standard concentration of chlorimuron and metsulfuron used in the bioassay were 21.6 and 6.9 ng g⁻¹ of soil respectively. Therefore, GR₅₀ values (except for metsulfuron in Balcarce soil) indicated that higher standard concentrations should have been applied for these soils.

Minimum and maximum values of GR₅₀ for chlorimuron were in general out of the range of 6 to 36 ng g⁻¹ reported by Schroeder (1994) for 11 soils of southern United States using *Cassia obtusifolia* as bioassay species. In the case of metsulfuron, Walker and Welch (1989) determined an ED₅₀ of 0.35 µg kg⁻¹ using lettuce (*Lactuca sativa* L.) as test species. Streibig *et al.*, (1995) reported ED₅₀ values for metsulfuron within an interval of 0.1-1.0 g a.i. ha⁻¹ using *Brassica rapa* L. as test species. These values are substantially lower than the range of 9.8-17.3 g a.i. ha⁻¹ (5.6-9.9 ng g⁻¹) determined in our study. Differences could be mainly due to higher sensitivity of the test species used respect to sunflower and lower organic matter content of the soils (0.24 to 2.2 %) and not to pH, that is quite similar to soils in the southeast of Buenos Aires Province.

Residual phytotoxicity: A linear equation provided the best fit of the data.

Chlorimuron: Response of sunflower RPH was significantly different at a probability level of 6% ($p \leq 0.06$) for all concentrations except for 3.6 ng g⁻¹ in both soils and 14.4 ng g⁻¹ in Balcarce soil (Table 2). Residual activity (R₉₀) of chlorimuron and metsulfuron increased almost linearly in both soils as application rate increased. However, when the rate was

Table – 2: Residual phytotoxicity^a of different concentrations of chlorimuron in two soils.

Soil	Concentration ng g ⁻¹	Intercept ± SE ln(RPH%)	Slope ± SE ln(RPH%) day ⁻¹	p ^b	r ²	R ₉₀ ^c days
Balcarce	3.6	4.4715±0.0363	0.0010±0.0005	0.1606	0.71	28
	7.2	4.4488±0.0208	0.0015±0.0003	0.0278	0.95	34
	14.4	4.3311±0.0486	0.0024±0.0006	0.0606	0.88	70
	21.6	4.3116±0.0171	0.0029±0.0002	0.0055	0.99	65
San Cayetano	3.6	4.4496±0.0378	0.0011±0.0004	0.0614	0.74	46
	7.2	4.3757±0.0425	0.0016±0.0004	0.0279	0.84	78
	14.4	4.1663±0.0869	0.0033±0.0008	0.0302	0.83	101
	21.6	3.9958±0.1023	0.0043±0.0010	0.0218	0.87	117

^aEquations were of the form $\ln(y) = \ln(a) + bx$ where $x = \text{days}$ and $y = \text{sunflower RPH}$; ^bProbability level for slope; ^cR₉₀ = days to achieve 90% of RPH. Calculated from regression equation.

Table – 3: Residual phytotoxicity^a of different concentrations of metsulfuron in two soils.

Soil	Concentration ng g ⁻¹	Intercept ± SE ln(RPH%)	Slope ± SE ln(RPH%) day ⁻¹	p ^b	r ²	R ₉₀ ^c days
Balcarce	1.15	4.5004±0.0373	0.0010±0.0005	0.1591	0.71	≅1
	2.3	4.4216±0.0196	0.0017±0.0003	0.0204	0.96	46
	4.6	4.2206±0.0661	0.0034±0.0008	0.0550	0.89	82
	6.9	4.1188±0.0704	0.0045±0.0009	0.0371	0.93	85
San Cayetano	1.15	4.3042±0.0610	0.0020±0.0005	0.0615	0.88	98
	2.3	4.0281±0.0569	0.0034±0.0005	0.0203	0.96	139
	4.6	3.7008±0.0848	0.0053±0.0007	0.0185	0.96	151
	6.9	3.4453±0.0639	0.0069±0.0006	0.0063	0.99	153

^aEquations were of the form $\ln(y) = \ln(a) + bx$ where $x = \text{days}$ and $y = \text{sunflower RPH}$; ^bProbability level for slope; ^cR₉₀ = days to achieve 90% of RPH. Calculated from regression equation.

increased more than 14.4 ng g⁻¹ of chlorimuron and 4.6 ng g⁻¹ of metsulfuron the response was asymptotic. Similarly to our results, several authors found greater residual bioactivity with increasing concentrations of sulfonylurea herbicides (Kotoula-Syka *et al.*, 1993a; Kotoula-Syka *et al.*, 1993b; Junnila *et al.*, 1994). Likewise, residual phytotoxicity of metsulfuron lasted longer than chlorimuron in both soils.

Chlorimuron and metsulfuron at all concentrations had greater residual activity in San Cayetano soil than in Balcarce soil. These results could be related to certain soil characteristics such as pH and organic matter content. Herbicides were more retained and degraded in Balcarce soil possibly due to its higher organic matter content and smaller pH than in San Cayetano soil. These results are coincident with previous findings (Streibig *et al.*, 1995; Cranmer *et al.*, 1999).

Persistence: The data were best described by linear regression in all cases.

Chlorimuron: The slopes (dissipation rate) were significantly different, considering a probability level of 6% ($p \leq 0.06$), for all concentrations except for 3.6 ng g⁻¹ in Balcarce soil (Table 4). The rate of dissipation increased almost linearly with increasing concentrations in both soils. Moreover, the slopes for all concentrations were greater in Balcarce soil than in San

Cayetano soil. Comparison of half-lives between soils indicated that chlorimuron persistence was greater in San Cayetano soil for any concentration.

Metsulfuron: slopes were significantly different at a probability level of 6% ($p \leq 0.06$) for all concentrations and soils except for 1.15 ng g⁻¹ (Table 5).

The dissipation rate increased with increasing concentrations in both soils. The dissipation for the highest concentrations was greater in Balcarce soil than in San Cayetano soil. Metsulfuron showed greater persistence in San Cayetano soil for any concentration by comparison of half-lives between soils.

Persistence of both herbicides, as occurred with residual phytotoxicity, increased in the two soils with increasing rate of application. The response was almost asymptotic when the rate increased more than 14.4 ng g⁻¹ for chlorimuron and 4.6 ng g⁻¹ for metsulfuron. The findings in our study are in agreement with those of Menne and Berger (2001), who reported that half-lives of several sulfonylureas, including metsulfuron, were positively influenced by their initial concentration.

Half-lives obtained here for chlorimuron ranged from 30 to 43 days for Balcarce soil and from 50 to 69 days for San

Table – 4: Persistence^a of different concentrations of chlorimuron in two soils.

Soil	Concentration ng g ⁻¹	Intercept ± SE ng g ⁻¹	Slope ± SE (ng g ⁻¹) day ⁻¹	p ^b	r ²	T _{1/2} ^c days
Balcarce	3.6	5.3232±2.7169	0.0831±0.0342	0.1358	0.75	32
	7.2	6.6552±2.0438	0.1092±0.0258	0.0514	0.90	30
	14.4	16.4894±3.6612	0.1930±0.0461	0.0526	0.90	43
	21.6	17.7544±1.5784	0.2236±0.0199	0.0078	0.98	40
San Cayetano	3.6	4.0323±1.0332	0.0402±0.0100	0.0273	0.85	50
	7.2	6.7148±1.4498	0.0614±0.0140	0.0219	0.87	55
	14.4	14.0567±3.8400	0.1171±0.0370	0.0508	0.77	60
	21.6	20.4081±3.5431	0.1489±0.342	0.0223	0.86	69

^aEquations were of the form $y = a - bx$ where x = days and y = chlorimuron concentration; ^bProbability level for slope; ^cT_{1/2} = half life calculated from regression equation.

Table – 5: Persistence^a of different concentrations of metsulfuron in two soils.

Soil	Concentration ng g ⁻¹	Intercept ± SE ng g ⁻¹	Slope ± SE (ng g ⁻¹) day ⁻¹	p ^b	r ²	T _{1/2} ^c days
Balcarce	1.15	0.9882±0.5900	0.0131±0.0074	0.2196	0.61	38
	2.3	2.1262±0.3343	0.0232±0.0042	0.0315	0.94	46
	4.6	5.0521±0.9233	0.0494±0.0116	0.0513	0.90	51
	6.9	6.4968±0.9908	0.0643±0.0125	0.0356	0.93	51
San Cayetano	1.15	1.6996±0.4926	0.0158±0.0043	0.0655	0.87	54
	2.3	3.7884±0.4574	0.0254±0.0040	0.0234	0.95	75
	4.6	6.2592±0.5979	0.0380±0.0052	0.0180	0.96	82
	6.9	8.3387±0.7203	0.0499±0.0062	0.0152	0.97	84

^aEquations were of the form $y = a - bx$ where x = days and y = metsulfuron concentration; ^bProbability level for slope; ^cT_{1/2} = half life calculated from regression equation.

Cayetano soil. These results do not agree with the findings of Vencill and Banks (1994), who determined half-lives of chlorimuron by means of bioassays ranging between 5 and 20 days. However, we agree results reported by other researchers who determined chlorimuron half-lives ranging from 12 to 50 days (Schroeder, 1994) and from 13 to 44 days (Baughman *et al.*, 1996). Our findings are also coincident with those of other authors that using chemical determinations reported half-lives ranging between 30 days (Weber, 1994) to 40 days (Comfort *et al.*, 1994; Wauchope *et al.*, 1992).

Metsulfuron half-lives were between 38 to 51 days in Balcarce soil and from 54 to 84 days in San Cayetano soil. These values are also coincident with those determined by chemical methods and ranged from 23 to 79 days reported by Walker *et al.* (1989) with soils at 0-20 cm depth, and Weber (1994) with 63 days. Other researchers reported half-lives as short as 30 days (Wauchope *et al.*, 1992; Comfort *et al.*, 1994), 8-36 days (James *et al.*, 1995), or 12-28 days (Cranmer *et al.*, 1999).

Dissipation and consequently persistence of sulfonylurea herbicides depend on soil-related degradation and sorption characteristics. As was pointed out by several authors (Walker *et al.*, 1989; Streibig *et al.*, 1995; Cranmer *et al.*, 1999), sorption of sulfonylureas decreases when pH increases and

organic matter decreases. As consequence, greater bioavailability, and therefore residual phytotoxicity as well as persistence of herbicides at all concentrations, was achieved in San Cayetano soil due to greater pH and lower organic matter content than in Balcarce soil. Likewise, is well known that the most important pathways of sulfonylurea degradation in soil are chemical hydrolysis and microbial breakdown. The factors having the greatest influence in these processes include pH and organic matter. For this reason, the rate of degradation of chlorimuron and metsulfuron herbicides was in general fastest in Balcarce soil than in San Cayetano (Tables 3 and 4).

Finally, it must be noted that residual activity also depends on sensitivity of bioassay species. In our case, we used sunflower that is cited as one of the more susceptible bioassay species (Günther *et al.*, 1993; Castro *et al.*, 2002), and is usually sown in the region of this study. Taking into account only the normal rate applied of herbicides (7.2 ng g⁻¹ for chlorimuron, and 2.3 ng g⁻¹ for metsulfuron), the interval between application of herbicides and planting of sunflower should be of 34 and 78 DAT for chlorimuron in Balcarce and San Cayetano soil respectively, or 46 and 139 DAT for metsulfuron in Balcarce and San Cayetano soil respectively. Shorter intervals would be possible when more tolerant crop species were sown.

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