

Sorption–Desorption of Rimsulfuron,
Nicosulfuron, and Metabolites in Soils
from Argentina and the USAMariela P. Azcarate, Sharon K. Papiernik,* Jorgelina C. Montoya,
and William C. Koskinen

Core Ideas

- Sorption of each sulfonylurea compound varied by site, landscape position, and depth.
- Sorption trends were generally predictable based on soil organic C.
- Metabolite K_d values differed greatly from the parent compound's K_d .
- All compounds exhibited resistance to desorption.

Abstract: Sulfonylurea herbicides provide broad-spectrum weed control in a variety of crops. To better understand the potential mobility of these compounds and their metabolites in soil, sorption–desorption studies were conducted using ^{14}C -labeled compounds: rimsulfuron and its metabolites IN-E9260, IN-70941, and IN-70942; nicosulfuron and its metabolites IN-HYY21 and IN-GDC42. Soils were collected from two depths in the upper slope, backslope, and lower slope of landforms in La Pampa, Argentina, and Minnesota, USA. Soil-water partition coefficients (K_d) ranged from 0 to 21 mL g $^{-1}$ and were usually ≤ 2 mL g $^{-1}$. Metabolite K_d values differed from the parent compound K_d by up to an order of magnitude. For all compounds, desorption was hysteretic. For most compounds tested, K_d values were greater in the US soils, which were higher in organic carbon and pH than the Argentinian soils. At both sites, K_d values were lowest in the upper slope and highest in the lower slope.

M.P. Azcarate and J.C. Montoya, Instituto Nacional de Tecnología Agropecuaria (INTA), EEA Anguil, Anguil, La Pampa, Argentina; S.K. Papiernik, USDA–ARS, North Central Agricultural Research Laboratory, Brookings, 57006 SD; W.C. Koskinen, USDA–ARS, Soil and Water Management Unit, St. Paul, MN.

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*Corresponding author (Sharon.Papiernik@ars.usda.gov).

SULFONYLUREA HERBICIDES (SUs) provide broad-spectrum weed control in a variety of crops, including wheat (*Triticum aestivum* L.), rice (*Oryza sativa* L.), corn (*Zea mays* L.), soybean [*Glycine max* (L.) Merr.], and potato (*Solanum tuberosum* L.). Although these herbicides have low application rates of grams per hectare (Grey and McCullough, 2012), they can carry over, causing crop injury under some conditions; this may be more likely for winter wheat and cover crops (Cornelius and Bradley, 2017). They have also been detected in groundwater (Battaglin et al., 2000). The SUs are degraded by soil microbes and chemical hydrolysis, depending on soil conditions (Sarmah and Sabadie, 2002). The primary degradation pathways of SUs are chemical hydrolysis of the SU bridge and contraction/rearrangement of the SU bridge.

For rimsulfuron [1-(4,6-dimethoxypyrimidin-2-yl)-3-(3-ethylsulfonyl)-2-pyridylsulfonyl]urea], the primary transformation pathway in soil is bridge contraction to form N-(4,6-dimethoxy-2-pyrimidinyl)-N-((3-ethylsulfonyl)-2-pyridinyl)urea, designated IN-70941, further transformed to N-((3-ethylsulfonyl)-2-pyridinyl)-4,6-dimethoxy-2-pyrimidinamine, designated IN-70942 (Sarmah and Sabadie, 2002). Nicosulfuron (2-[4,6-dimethoxypyrimidin-2-yl]carbamoysulfamoyl]-N,N-dimethylnicotinamide) is transformed in soil by a demethylation followed by cleavage of the pyrimidinyl ring to form the metabolites 2-[[[(aminoiminomethyl)amino]carbonyl]amino]sulfonyl]-N,N-dimethyl-3-pyridinecarboxamide, designated IN-HYY21 or AUSN and 2-[[[(aminocarbonyl)amino]carbonyl]amino]sulfonyl]-N,N-dimethyl-3-pyridinecarboxamide, designated IN-GDC42 or UCSN (EFSA, 2007) (Fig. 1).

The environmental fate of SUs is dependent on soil and environmental conditions: mobility is greater in sandy or clay-poor soils, degradation is greater under warm, wet conditions, and leaching and degradation are pH-dependent under some conditions (Oliveira et al., 2001; Poppell et al., 2002). The half-lives of SUs and metabolites range from days to weeks (EFSA, 2007, 2018; Martins

Abbreviations: SU, sulfonylurea herbicide.

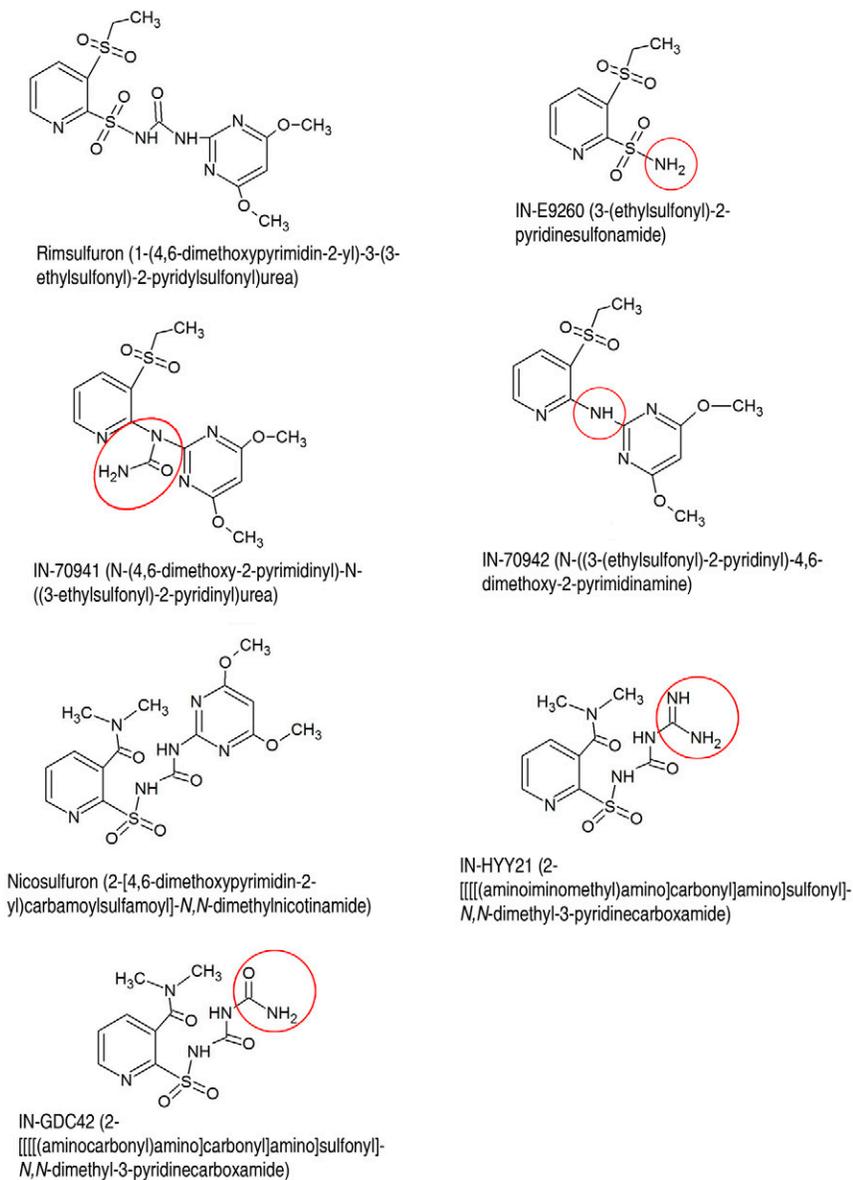


Fig. 1. Structures of herbicides and metabolites used in this study. Structural differences from the parent compound are highlighted.

et al., 2001; Martins and Mermoud, 1999; Schneiders et al., 1993). Some SU metabolites have been found to contaminate subsurface water resources (Rosenbom et al., 2010), demonstrating their potential mobility.

Few reports have been published in the peer-reviewed literature regarding interactions between agricultural soils and the metabolites of nicosulfuron and rimsulfuron. To better understand and predict the potential mobility of these compounds in a varied landscape, we characterized the sorption-desorption of rimsulfuron, nicosulfuron, and some of their metabolites that retain at least one sulfonyl functional group.

Materials and Methods

Soils were collected from two depths in the upper slope, backslope, and lower slope of agricultural fields in Intendente Alvear, La Pampa, Argentina (35.40° S, 63.69° W) and west-central Minnesota, USA (45.65° N, 95.83° W). The soils in

Argentina were collected by horizon from the Ap and AC horizons in the shoulder, backslope, and footslope positions. Soils from the United States were collected by depth in the shoulder, lower backslope, and toeslope positions of an eroded landform. Soils were air-dried and passed through a 2-mm sieve. Soil properties varied with site, landscape position, and depth (Tables 1 and 2). The Minnesota site is affected by long-term tillage and exhibits calcareous topsoil and shallow subsoil in the upper slope, an inverted soil profile in the midslope, and deep accumulation of topsoil in the lower slope (see Papiernik et al. [2009] for more information). The Argentina landform has >90% sand in the shoulder, >80% sand in the midslope, and >70% sand in the footslope and has ≤1.1% organic C throughout the landform (Azcarate et al., 2015).

Standard batch equilibration sorption-desorption studies were conducted using ¹⁴C-labeled compounds: rimsulfuron and its metabolites IN-70942, IN-70941, IN-E9260 [3-(ethylsulfonyl)-2-pyridine-sulfonamide], and nicosulfuron and its metabolites IN-HYY21 and IN-GDC42. Duplicate 8-g soil samples were equilibrated in glass centrifuge tubes with 8 mL of 0.005 M CaCl₂ aqueous solution containing 0.2 mg L⁻¹ of one compound. This concentration represents the usual field application rate for sulfonylurea herbicides diluted in the top 5 cm of soil. Only one concentration was used. Leaching indices and models typically use only one K_d value even if the isotherm is nonlinear (Oliveira et al., 2001). All solutions contained ¹⁴C-radiolabeled chemicals to between 160 and 470 Bq L⁻¹. Slurries were shaken for 24 h at 20°C, then centrifuged at 2500 rpm for 10

min. After centrifugation, no particulates in solution were observed using a Tyndall beam. A 2-mL aliquot of equilibrated solution was removed for analysis by liquid scintillation counting. Preliminary studies showed no sorption to the glass tubes or Teflon-lined caps.

For desorption, immediately after removing the 2-mL aliquot described above, 2 mL of 0.005 M CaCl₂ (with no test chemical) was added to each glass tube. Samples were reequilibrated for 24 h and processed as described above. This desorption was repeated twice more, for a total of three desorption equilibrations.

For each sample, the soil-water partition (sorption) coefficient K_d was calculated as C_s/C_w, where C_s is the concentration of chemical sorbed to the soil (mg kg⁻¹) determined from the difference in concentration before and after equilibration and C_w is the concentration of chemical in solution (mg L⁻¹) after equilibration. Sorption coefficients were

normalized to the fraction of organic C as $K_{oc} = K_d f_{oc}$, where f_{oc} is the fraction of organic C in a soil sample. Desorption coefficients (K_{d-des}) were calculated as C_{s-des}/C_{w-des} , where C_{s-des} is the concentration of chemical remaining sorbed to the soil (mg kg^{-1}), determined from the difference in the amount of chemical remaining in the centrifuge tube (accounting for the amount removed with the 2-mL aliquot withdrawn for analysis) and the amount in solution after equilibration, and C_{w-des} is the concentration of chemical in solution (mg L^{-1}) after reequilibration.

We used ^{14}C -labeled compounds to facilitate analysis. Preliminary analysis of the 24-h sorption equilibration solutions by high performance liquid chromatography showed no degradation of nicosulfuron or any SU metabolite. We did observe very small amounts of rimsulfuron degradation after 24 h, but it did not significantly affect the sorption calculations. These observations are consistent with the half-life (DT50) values of at least 7 d reported for nicosulfuron and all SU metabolites included in this study and shorter DT50 values reported for rimsulfuron in some soils (EFSA, 2007, 2018).

Table 1. La Pampa, Argentina, soil properties and soil-water partitioning coefficients for rimsulfuron and metabolites and nicosulfuron and metabolites to soil collected from three landscape positions and two depths. Sorption K_d (mL g^{-1}) was determined in a 24-h equilibration, and desorption K_{d-des} (mL g^{-1}) was measured following 1 (D1), 2 (D2), and 3 (D3) desorption steps.

	Shoulder		Backslope		Footslope	
Depth (cm)	0–7 cm	25–55 cm	0–7 cm	23–40 cm	0–13 cm	20–60 cm
OC (g kg^{-1})	4.6 b†	2.1 bc	9.6 a	2.6 bc	11.3 a	0.6 c
pH	5.7 d	6.4 c	6.0 cd	6.4 c	7.0 b	8.6 a
Texture	Sand	Sand	Loamy sand	Loamy sand	Loamy sand	Loamy sand
Rimsulfuron K_d ‡	1.5 b	1.1 c	1.9 a	1.5 b	2.0 a	1.9 a
D1/D2/D3 K_{d-des}	1.8/2.3/2.8	1.5/2.0/2.7	2.4/3.0/3.8	2.0/2.4/3.3	2.5/3.1/3.6	2.6/3.9/4.5
IN-E9260 K_d	0.2 bc	0.2 c	0.4 a	0.3 ab	0.4 a	0.3 a
D1/D2/D3 K_{d-des}	0.2/0.2/0.3	0.2/0.2/0.3	0.4/0.3/0.4	0.3/0.3/0.5	0.4/0.5/0.5	0.3/0.4/0.4
IN-70942 K_d	15.7 b	11.3 cd	13.0 bc	21.0 a	8.4 de	5.3 e
D1/D2/D3 K_{d-des}	15.5/20.4/19.1	9.8/8.8/8.4	15.2/19.6/17.8	20.0/20.6/22.5	9.3/11.9/11.8	5.6/7.6/8.0
IN-70941 K_d	0.6 cd	0.4 d	0.9 b	0.6 c	1.1 a	1.2 a
D1/D2/D3 K_{d-des}	0.6/0.6/0.8	0.5/0.5/0.8	0.9/1.0/1.2	0.7/0.8/0.9	1.2/1.3/1.4	2.0/2.9/4.5
Nicosulfuron K_d ‡	0.3 b	0.3 b	0.4 ab	0.4 ab	0.4 a	0.1 c
D1/D2/D3 K_{d-des}	0.4/0.5/0.6	0.3/0.5/0.6	0.4/0.6/0.7	0.5/0.5/0.6	0.4/0.5/0.6	0.2/0.3/0.3
IN-HYY21 K_d	0.4 c	0.4 c	0.6 b	0.6 b	0.7 a	0.6 ab
D1/D2/D3 K_{d-des}	0.6/0.7/1.0	0.6/0.8/1.2	0.7/0.9/1.2	0.7/0.9/1.3	0.8/1.0/1.4	0.7/0.9/1.1
IN-GDC42 K_d	0.1 a	0.1 ab	0.1 a	0.1 ab	0.2 a	0.0 b
D1/D2/D3 K_{d-des}	0.1/0.3/0.5	0.1/0.3/0.5	0.1/0.2/0.3	0.1/0.2/0.2	0.1/0.2/0.4	0.0/0.1/0.2

† Values within a row followed by different letters are significantly different (Tukey test, $p \leq 0.05$).

‡ Data from Azcarate et al. (2015).

Table 2. Minnesota, USA, soil properties and soil-water partitioning coefficients for rimsulfuron and metabolites and nicosulfuron and metabolites to soil collected from three landscape positions and two depths. Sorption K_d (mL g^{-1}) was determined in a 24-h equilibration, and desorption K_{d-des} (mL g^{-1}) was measured following 1 (D1), 2 (D2), and 3 (D3) desorption steps.

	Shoulder		Backslope		Footslope	
Depth (cm)	0–15 cm	46–61 cm	0–15 cm	46–61 cm	0–15 cm	46–61 cm
OC (g kg^{-1})	15.5 b†	6.5 cd	16.8 b	13.9 bc	37.6 a	4.3 d
pH	8.1 c	8.8 a	8.0 c	8.3 bc	8.1 c	8.5 ab
Texture	Sandy clay loam	Sandy loam	Sandy loam	Sandy loam	Sandy clay loam	Sandy loam
Rimsulfuron K_d †	1.5 d	0.9 e	2.0 c	2.6 b	5.4 a	2.8 b
D1/D2/D3 K_{d-des}	1.8/2.1/2.4	1.1/1.4/1.7	2.5/2.8/3.2	3.0/3.6/3.9	6.3/7.3/8.3	3.3/3.7/4.3
IN-E9260 K_d	0.9 de	0.7 e	1.0 c	0.9 cd	2.4 a	1.3 b
D1/D2/D3 K_{d-des}	0.9/1.0/1.0	1.0/0.9/0.9	1.1/1.1/1.2	1.1/1.1/1.2	2.8/2.7/2.8	1.4/1.4/1.4
IN-70942 K_d	3.1 e	1.8 f	5.3 d	6.8 c	18.6 a	10.2 b
D1/D2/D3 K_{d-des}	5.0/4.4/4.5	2.1/2.2/2.3	7.4/7.5/7.8	8.8/9.2/10.1	27.9/28.1/32.0	13.5/13.3/14.8
IN-70941 K_d	2.3 e	1.5 f	2.9 d	3.2 c	6.3 a	3.7 b
D1/D2/D3 K_{d-des}	2.8/2.7/2.9	2.3/1.9/2.0	4.1/3.6/3.8	4.3/4.5/5.0	7.8/8.0/8.6	4.7/4.9/5.4
Nicosulfuron K_d †	1.2 d	0.9 e	1.4 c	1.4 c	2.3 a	1.7 b
D1/D2/D3 K_{d-des}	1.4/1.5/1.6	1.0/1.1/1.2	1.6/1.7/1.9	1.6/1.9/2.1	2.8/3.2/3.7	2.0/2.2/2.4
IN-HYY21 K_d	1.3 e	1.0 f	1.5 d	1.8 c	2.7 a	1.9 b
D1/D2/D3 K_{d-des}	1.5/1.4/1.5	1.1/1.1/0.5	1.7/1.3/1.5	2.0/1.9/1.5	2.9/1.5/1.2	2.0/1.7/0.9
IN-GDC42 K_d	0.2 bc	0.1 c	0.2 bc	0.2 b	0.2 a	0.2 bc
D1/D2/D3 K_{d-des}	0.2/0.3/0.3	0.2/0.3/0.3	0.2/0.3/0.3	0.3/0.3/0.4	0.3/0.4/0.4	0.2/0.2/0.3

† Values within a row followed by different letters are significantly different (Tukey test, $p \leq 0.05$).

Results and Discussion

Sorption

For each compound, K_d values ranged by an order of magnitude (Tables 1 and 2). Values are generally consistent with those previously reported for nicosulfuron (Gonzalez and Ukrainczyk, 1996; Regitano and Koskinen, 2008) and rimsulfuron (Vicari et al., 1996) sorption to Mollisols. Except for rimsulfuron metabolite IN-70942, K_d values tended to be greater in soils from the United States than soils from Argentina, likely owing to their higher organic C and pH. In US soils, sorption of most compounds was correlated with soil organic C concentrations but not with clay content or pH. Correlation coefficients relating US soil OC to K_d were significant for rimsulfuron ($r = 0.80$), IN-E9260 ($r = 0.82$), IN-70941 ($r = 0.80$), and IN-GDC42 ($r = 0.91$). In Argentinian soils, K_d values of IN-70941 were significantly correlated with clay content ($r = 0.80$) and K_d values of IN-GDC42 were correlated with OC ($r = 0.88$), with no significant correlation between K_d and soil pH. At these pH levels, all compounds are predominantly in molecular or anionic form, so sorption would not be expected to be highly dependent on pH.

In general, sorption coefficients were consistent with trends of increasing organic C. Sorption K_d tended to increase in the order upper slope < middle slope < lower slope and was higher in surface soils than in subsurface soils within the same landscape position (Tables 1 and 2). However, normalizing the K_d to the fraction of organic C did not reduce the variation in sorption coefficients for any compound in either the Argentinian or US soils: K_{oc} values (not shown) were at least as variable as K_d values. Similar depth and landscape position effects were observed for the sorption of the weak acid saflufenacil to soils from the Minnesota site (Papiernik et al., 2012); these authors also reported a nonconstant K_{oc} .

Depending on the SU, small structural differences translated into large differences in sorption. In each Argentinian and US soil, K_d values followed the trend IN-HYY21 > nicosulfuron > IN-GDC42 (Tables 1 and 2), despite differing only in an imino group (IN-HYY21) versus a carbonyl group (IN-GDC42) (Fig. 1). Regulatory studies in the EU also reported that IN-GDC42 was less sorbed than IN-HYY21 (EFSA, 2007). In each US soil, K_d values followed the trend IN-70942 > IN-70941 > rimsulfuron > IN-E9260 and all were generally of the same magnitude (Table 2). In each Argentinian soil, K_d values followed the trend IN-70942 >> rimsulfuron > IN-70941 > IN-E9260 (Table 1). The greater sorption of IN-70942 is presumably due to the greater reactivity of the $-NH_2$ group attached to the carbonyl group. Previous studies (Martins and Mermoud, 1999; Schneiders et al., 1993) also reported that IN-70942 was less mobile than IN-70941.

Desorption

Sorption of all compounds exhibited hysteresis. Each of the three successive desorption steps exhibited an increase in K_{d-des} relative to the sorption K_d (Tables 1 and 2). Proportional increases in K_{d-des} were fairly consistent across sites and across compounds. After the first, second, and third desorption steps, K_{d-des} values were on average 1.2, 1.5, and 2.0 times the initial sorption K_d for the Argentinian

soils and 1.2, 1.3, and 1.4 times the initial sorption K_d for the US soils. For both sites, the maximum hysteresis was observed for IN-GDC42 in the shoulder slope position: K_{d-des} increased by a factor of 5.5 for the Argentinian site and 2.8 for the US site relative to the initial sorption K_d . Resistance to desorption was reported for sulfonylurea herbicides in these Argentinian soils (Azcarate et al., 2015) and other soils (e.g., Gonzalez and Ukrainczyk, 1996); resistance to nicosulfuron desorption was also observed by an increase in K_d with aging (Regitano and Koskinen, 2008). Hysteresis may affect plant availability, carryover, and leaching of herbicides and their degradation products.

Herbicide transport models and leaching indices often use a single sorption coefficient to estimate potential offsite movement for a chemical. Our results suggest that use of a single value could greatly under- or overestimate potential transport of SU herbicides and their metabolites. Quantitative data such as the values presented here are required for more accurate prediction of herbicide fate in soils for the following reasons:

- The K_d determined for the sulfonylurea parent compound does not necessarily represent the K_d for similarly structured metabolites. Metabolite K_d values were up to an order of magnitude greater or less than that of the parent compound.
- Results from one geographical region cannot be reliably generalized to another region. For some compounds, there were order-of-magnitude differences in K_d values measured in soils from the United States and Argentina.
- The K_d value determined for surface soils cannot be reliably extrapolated to nearby soils. Large differences were observed in K_d measured in surface and subsurface soils and in soils collected from different landscape positions in the same landform.
- Desorption results showed that sorption K_d values may overestimate the mobility of these chemicals. All compounds exhibited desorption hysteresis; on average, K_{d-des} values following three desorption steps were 1.4 to 2 times larger than the sorption K_d .

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