

# Behavior of Four Sulfonylurea Herbicides in the Presence of Hydroxy Compounds

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The behavior of four sulfonylurea herbicides (metsulfuron methyl, chlorsulfuron, chlorimuron ethyl, and bensulfuron methyl) was studied in the presence of various hydroxy compounds. When dissolved at 30 °C in simple primary, secondary, or tertiary alcohols (methanol, ethanol, isopropyl alcohol, and *tert*-butyl alcohol) and in glycerol or in poly(ethylene glycol), most of these herbicides underwent rapid alcoholysis involving the breakdown of the urea part of the molecule. The corresponding sulfonyl carbamate is recovered in high yields, along with a small amount of sulfonamide formed in the concomitant hydrolysis. Degradation rate constants and the selectivity of conversion were established. The addition of buffered water (pH 7.0) inhibited the alcoholysis reaction, leaving only hydrolysis, as already observed with concentrated saccharide solutions. In phenol solution, slight herbicide hydrolysis was primarily observed. It appeared that alcoholysis reactions only occurred under very particular conditions when sulfonylurea herbicides are dissolved in pure alcohols, without buffered water. These results led to the conclusion that in soil, similar alcoholysis reactions seem unlikely.

**Keywords:** *Chlorsulfuron; metsulfuron; chlorimuron; bensulfuron; degradation; alcoholysis*

## INTRODUCTION

The sulfonylurea herbicides are a relatively new class of compounds used worldwide for the control of broad-leaved weeds and some grasses in cereal crops (Beyer et al., 1988; Blair and Martin, 1988; Sarmah et al., 1998). Sulfonylureas undergo hydrolysis in aqueous media at a rate dependent upon both temperature and pH. Pathways of chemical hydrolysis for four of these herbicides were previously described (Figure 1): metsulfuron methyl ( $n = 0$ ; A = N; X = COOCH<sub>3</sub>, Y = CH<sub>3</sub>) (Sabadie, 1990), chlorsulfuron ( $n = 0$ ; A = N; X = Cl, Y = CH<sub>3</sub>) (Sabadie, 1991), chlorimuron ethyl ( $n = 0$ ; A = CH; X = COOC<sub>2</sub>H<sub>5</sub>, Y = Cl) (Sabadie, 1995) and bensulfuron methyl ( $n = 1$ ; A = CH; X = COOCH<sub>3</sub>, Y = OCH<sub>3</sub>) (Sabadie, 1996). Alcoholysis of these four herbicides was also reported in solutions of pure methyl or ethyl alcohol. The rates of these reactions could be described satisfactorily with first-order kinetics, and the stable sulfonyl carbamates (methyl, R = CH<sub>3</sub>; ethyl, R = C<sub>2</sub>H<sub>5</sub>), obtained quantitatively, involve only the breakdown of the urea part of the sulfonylurea molecule (Figure 1). Formation of an undetected isocyanate intermediate was also suggested.

Degradation of seven sulfonylurea herbicides dissolved in methanol was later described by Matchett et al. (1997) using capillary electrophoresis as the analytical technique. The methanolysis rate constant measured for bensulfuron was in good agreement with our earlier results (Sabadie, 1996), and mass spectrometry studies confirmed that the products of methanolysis are sulfonyl carbamates (R = CH<sub>3</sub>) as shown in Figure 1.

Relatively high rates of conversion were systematically observed for methanolysis or ethanolysis of all the sulfonylureas studied (half-lives were between 1.5 and 14 days at 30 °C). The alcoholysis reaction appears to be a characteristic of the phenyl (or benzyl) sulfonylurea bridge, and it is only slightly influenced by other parts of the molecule (aryl portion or heterocycle). However,

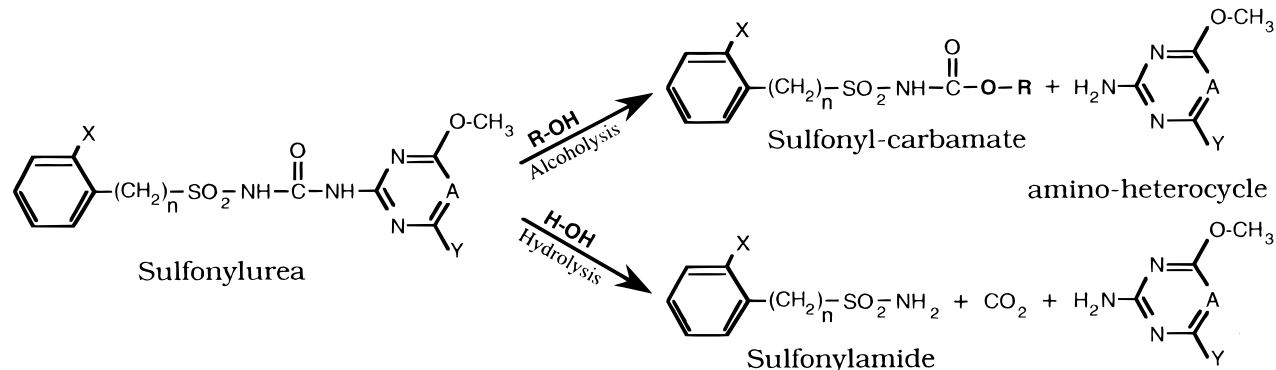
only methanol and ethanol, simple primary alcohols, have been studied.

Chemical hydrolysis may well be one of the predominant factors in the breakdown of sulfonylurea herbicides in soils (Beyer et al., 1988). However, a great many organic residues liberated into soil consists of complex macromolecules such as polysaccharides, which can be broken down in various ways (Lowe, 1978; Ruggiero et al., 1996). The saccharides formed are polyhydroxy molecules. In addition, humic substances, complex polymers with essentially phenolic structures, have been known to establish binding with various xenobiotic chemicals (Dec and Bollag, 1997). Despite the fact that the formation of "bound residues" with sulfonylurea has not yet been mentioned in the literature, the presence of these hydroxy compounds (polyols, saccharides, humic substances) may also give rise to various alcoholysis reactions involving the formation of unexpected degradation products.

Additional investigation would therefore seem necessary to specify the potentiality of these alcoholysis reactions during chemical degradation of the sulfonylurea herbicides in soil. Analytical considerations prevent us from satisfactorily investigating these alcoholysis reactions directly in the soil. Consequently, the first experiments have been carried out using solutions as simplified soil models. The four herbicides previously studied, metsulfuron methyl, chlorsulfuron, chlorimuron ethyl, and bensulfuron methyl (Sabadie, 1990, 1991, 1995, 1996), were selected for this work. Their behavior in the presence of certain simple or complex alcohols (polyols, saccharides) was studied. The competitive effect of water was examined. Finally, their reactivity in the presence of phenol was investigated.

## EXPERIMENTAL PROCEDURES

**Chemicals.** Technical grade metsulfuron methyl, chlorsulfuron, chlorimuron ethyl, and bensulfuron methyl were



**Figure 1.** Alcoholysis and hydrolysis of sulfonylurea herbicides.

obtained from DuPont France and used without further purification. The preparation of their degradation products (hydrolysis, methanolysis, and ethanolysis) has already been described (Badon et al., 1990; Sabadie, 1991; Sabadie, 1995; Sabadie, 1996).

Isopropyl alcohol, 2-methyl-2-propanol (*tert*-butyl alcohol), glycerol (anhydrous), poly(ethylene glycol) (average  $M_w$  200), poly(vinyl alcohol) 99% hydrolyzed (average  $M_w$  89 000–98 000),  $\alpha$ -D-glucose (anhydrous), and phenol were obtained from Aldrich.

**General Operating Method for Isopropyl and *tert*-Butyl Carbamates.** Sulfonylurea herbicide (0.5 g) dissolved in 100 mL of alcohol (isopropyl or *tert*-butyl alcohol) was heated at 60 °C for 24 h. The alcohol was evaporated to dryness under vacuum, and the residue was dissolved in 50 mL of ethyl ether. The carbamate formed was extracted with 0.1 M aqueous sodium carbonate solution (2 × 25 mL). The aqueous solution was treated with ethyl ether (20 mL), acidified (HCl, pH 2), and then extracted with ethyl ether (4 × 25 mL). The organic solution was water-washed twice and evaporated to dryness. The viscous liquid obtained crystallized more or less rapidly. Recrystallization was carried out in ethanol–isooctane mixtures. The chemical structures of the four isopropyl- and *tert*-butylcarbamates obtained were characterized by infrared spectroscopy and mass spectrometry.

**Experimental Conditions.** Herbicide alcoholysis experiments were carried out in a thermoregulated water bath under appropriate conditions. Each experiment was run in duplicate.

Simple alcohol: herbicide concentration, 0.12 mmol L<sup>-1</sup>, 30 °C; poly(ethylene glycol) and glycerol, 5 mmol L<sup>-1</sup>, 30 °C; phenol, 3 mmol L<sup>-1</sup>, 50 °C.

Glucose effects were measured at 30 °C, using chlorsulfuron or chlorimuron (0.12 mmol L<sup>-1</sup>) in buffered solution (phosphate 0.07 M, pH 7.0) with 20% and 50% (by weight) of added anhydrous glucose.

With a stock solution of chlorsulfuron in absolute ethyl alcohol (0.28 mmol L<sup>-1</sup>) as starting material, various aqueous ethanol solutions (100, 90, 80, and 50% of ethanol by volume) with the same herbicide concentration (0.14 mmol L<sup>-1</sup>) were obtained by the addition of the appropriate volume of water and/or ethanol. Moderately acidic conditions (pH ~3) were obtained by adding concentrated HCl to the stock solution (1 drop to 100 mL), and a phosphate-buffered solution (0.07 M, pH 7.0) was used for neutral conditions.

Herbicide deposition on poly(vinyl alcohol) (5 mg g<sup>-1</sup>) was achieved either by a dry method (thorough crushing of the powder mixture) or by a liquid method (herbicide dissolution in acetone, addition of the powder, and then evaporation to dryness under vacuum at room temperature for 60 min). The herbicide-permeated powder (typically 2 g) was enclosed in 5 mL glass tubes, which were sealed and kept at 30 °C under continuous rotation-stirring.

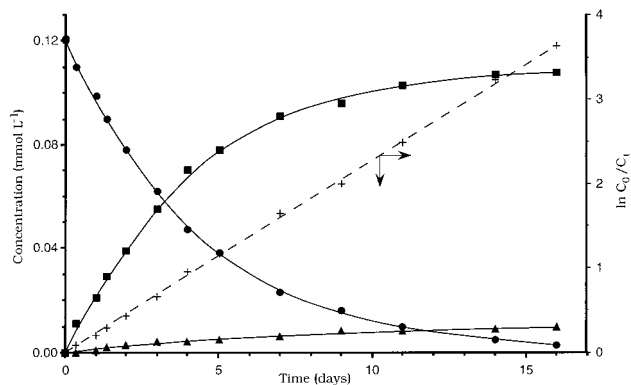
**Analytical Technique.** All the alcoholysis analytical samples (2 mL) were evaporated to dryness under vacuum and then stirred for 30 min in a mixture of chloroform, water, and acetic acid (3 + 2 + 0.1 mL). The phenolic analytical samples (50  $\mu$ L) were introduced directly into this extraction mixture.

**Table 1.** Alcoholysis of Sulfonylurea Herbicides at 30 °C

herbicide	rate constants <sup>a</sup> (days <sup>-1</sup> ) (rel selectivity <sup>b</sup> (%))			
	MeOH	EtOH	<i>i</i> -PrOH	<i>t</i> -BuOH
metsulfuron methyl	0.09 (99)	0.05 (99)	0.04 (86)	0.02 (83)
chlorsulfuron	0.20 (100)	0.11 (100)	0.09 (93)	0.05 (91)
chlorimuron ethyl	0.43 (100)	0.22 (100)	0.23 (92)	0.13 (92)
bensulfuron methyl	0.15 (100)	0.11 (100)	0.07 (95)	0.04 (90)

<sup>a</sup> Rate constants were determined assuming first-order kinetics.

<sup>b</sup> Relative selectivity measured the percentage of observed alcoholysis.



**Figure 2.** Alcoholysis of chlorimuron ethyl in isopropyl alcohol at 30 °C: concentration of chlorimuron ethyl (●), sulfonylamide (▲), and isopropyl carbamate (■) as a function of time. The first-order kinetics line was established from the loss of chlorimuron ethyl (- -).

Analytical samples from poly(ethylene glycol) or glycerol (50  $\mu$ L) and poly(vinyl alcohol) (30 mg) were stirred for 30 min in a mixture of chloroform and an aqueous potassium dihydrogen phosphate solution 0.07 M (3 + 2 mL).

Chloroform solution analysis was performed with a high-performance liquid chromatograph using a normal-phase column (Lichrosorb 5  $\mu$ m L5-25 F, 25 cm), a UV detector system (235 nm), and a mobile phase of isooctane + absolute ethanol + acetic acid (80 + 20 + 0.04 mL for metsulfuron methyl and chlorsulfuron; 90 + 10 + 0.04 mL for chlorimuron ethyl and bensulfuron methyl); the flow rate was 1.5 mL min<sup>-1</sup>.

## RESULTS AND DISCUSSION

**Simple Alcohols.** Selected sulfonylurea herbicides also underwent alcoholysis when dissolved in pure 2-propanol and *tert*-butyl alcohol (secondary and tertiary alcohols, respectively). The reaction rates could be described satisfactorily with first-order kinetics (Figure 2). Comparative rate constants, established at 30 °C, are listed in Table 1. The data show clearly that the alcoholysis reaction rate is fairly high using simple

**Table 2. Behavior of Chlorsulfuron Herbicide in Various Ethanol–Water Solutions (0.14 mmol L<sup>-1</sup>, 30 °C)**

operating conditions		ethanol concn			
		100%	90%	80%	50%
unbuffered water addition	rate constant <sup>a</sup> (days <sup>-1</sup> )	0.11	0.135	0.16	0.235
	rel selectivity <sup>b</sup> (%)	100	86	79	58
	obsd pH	6.5 <sup>c</sup>	6.2	5.9	5.5
buffered water addition (pH 7.0)	rate constant <sup>a</sup> (days <sup>-1</sup> )	0.11	0.0010	0.0008	0.0007
	rel selectivity <sup>b</sup> (%)	100	~15	~10	~10
	obsd pH	6.5 <sup>c</sup>	7.0	7.0	7.0
HCl addition	rate constant <sup>a</sup> (days <sup>-1</sup> )	0.345	0.23	0.25	0.40
	rel selectivity <sup>b</sup> (%)	100	84	78	60
	obsd pH	0.87 <sup>c</sup>	3.0	3.2	3.2

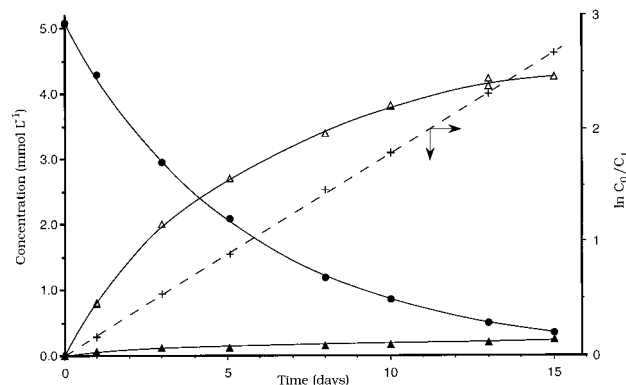
<sup>a</sup> Rate constants were determined assuming first-order kinetics. <sup>b</sup> Relative selectivity measured the percentage of observed alcoholysis. <sup>c</sup> Observed pH in pure ethanol solutions.

alcohol. The degradation rate appears to be governed by both the chemical structure of the sulfonylurea herbicide (chlorimuron ethyl > chlorsulfuron ≈ bensulfuron methyl > metsulfuron methyl in descending order) and the class of the alcohol (primarily > secondary > tertiary). The highest values were always observed in methanol. The relative selectivity for alcoholysis (Table 1) was established by comparing the amounts of sulfonyl carbamate and sulfonylamide (hydrolysis) obtained with regard to Figure 1. Although very high yields for the alcoholysis reaction were observed with the two primary alcohols, in accordance with previous results (Sabadie, 1991, 1995, 1996), a weak competitive hydrolysis (<20%) was also observed for isopropyl alcohol and *tert*-butyl alcohol. The presence of a trace of water may explain these results.

In the same way, chlorsulfuron and metsulfuron methyl underwent degradation in anhydrous glycerol (1,2,3-propanetriol). Elevated alcoholysis selectivity was observed (90 and 85%, respectively). However, the low solubility of the herbicide prevented the measurement of degradation rates.

**Behavior in Water–Ethanol Solutions.** For these investigations chlorsulfuron and ethanol were selected because of favorable analytical conditions and the formation of a stable sulfonylamide (absence of further cyclization). To inspect the water and pH effects, three different experiments were conducted: addition of distilled water (self-regulated pH), addition of buffered water pH 7.0 (phosphate 0.07 M), and the use of acidified ethanol (one drop of concentrated HCl dissolved in 100 mL). Table 2 shows the results obtained at 30 °C.

Under unbuffered conditions, an increase in the water content induced a proportional increase in the degradation rate, but the concomitant decrease in pH alone could explain this result (Beyer et al., 1988). However, the presence of water has an unfavorable effect on the alcoholysis path (Figure 1). Using buffered water, the pH of the three hydro-alcoholic solutions was fixed at 7.0. Under these conditions, the alcoholysis reaction appeared almost totally inhibited, and slow degradation by hydrolysis path prevailed. The addition of a small amount of acid allowed hydro-alcoholic solutions with pH values around 3 to be obtained. Although the presence of water favored hydrolysis, degradation rates remained lower than predicted (Sabadie, 1991), and it is difficult to explain the results obtained. One possible explanation is the presence of secondary transformation reactions, such as the breakdown of the heterocycle part of the herbicide molecule previously described for metsulfuron methyl (Badon et al., 1990).



**Figure 3.** Alcoholysis of chlorimuron ethyl in poly(ethylene glycol) (200) at 30 °C: concentration of chlorimuron ethyl (●), sulfonylamide (▲), and pyrimidamine (△) as a function of time. The first-order kinetics line was established from the loss of chlorimuron ethyl (---).

**Table 3. Degradation at 30 °C of Sulfonylurea Herbicides Dissolved in Poly(ethylene glycol) (5 mmol L<sup>-1</sup>) and Deposited on Poly(vinyl alcohol) (5 mg g<sup>-1</sup>)**

herbicide	poly(ethylene glycol)		<i>t</i> <sub>1/2</sub> <sup>c</sup> (days)	
	<i>k</i> <sup>a</sup> (days <sup>-1</sup> )	<i>S</i> <sup>b</sup> (%)	dry	liquid
metsulfuron methyl	0.03	78	~250	100
chlorsulfuron	0.07	90	~200	90
chlorimuron ethyl	0.18	95	60	25
bensulfuron methyl	0.10	94	>250	250

<sup>a</sup> Rate constants *k* were determined assuming first-order kinetics. <sup>b</sup> Relative selectivity *S* measured the percentage of alcoholysis by comparing the quantities of sulfonylamide and amino-heterocycle formed. <sup>c</sup> Observed half-life (days) as affected by the deposition method (dry or liquid).

The most significant result of these experiments is the low selectivity observed for alcoholysis reactions in buffered hydro-alcoholic solutions.

**Polymeric Alcohols.** Sulfonylurea herbicides underwent degradation when dissolved in liquid poly(ethylene glycol) (average *M<sub>w</sub>* 200) at 30 °C. The reaction rates are described satisfactorily with first-order kinetics (Figure 3). Because the formed carbamates remain unidentified, the selectivity of the reaction was established by comparing the quantities of sulfonylamide and amino-heterocycle obtained. Alcoholysis was preponderant, and the results presented (Table 3) are comparable to those already described for simple alcohols.

After deposition on powder poly(vinyl alcohol), very slow degradation of the four herbicides was observed at 30 °C. Nevertheless, the most homogeneous dispersion of the herbicide obtained using the liquid deposition

**Table 4. Degradation at 30 °C of Sulfonylurea Herbicides (0.12 mmol L<sup>-1</sup>) in Buffered Solution (pH 7) after Glucose Additions**

glucose addition (%, w/w)	obsd pH	rate constant <sup>a</sup> (10 <sup>-3</sup> days <sup>-1</sup> ) (30 °C)	
		chlorsulfuron	chlorimuron ethyl
0	7.0	2.7	4.5
20	6.8	3.7	6.5
50	6.6	4.6	7.4

<sup>a</sup> Rate constants (determined assuming first-order kinetics) as affected by anhydrous glucose addition (+20 and +50% weight to weight).

**Table 5. Degradation of Sulfonylurea Herbicides Dissolved in Phenol at 50 °C (3 mmol L<sup>-1</sup>)**

herbicide	rate constant <sup>a</sup> (days <sup>-1</sup> )	intermediate compd <sup>b</sup> (max %)
metsulfuron methyl	0.036	17
chlorsulfuron	0.035	14
chlorimuron ethyl	0.019	10
bensulfuron methyl	0.031	5

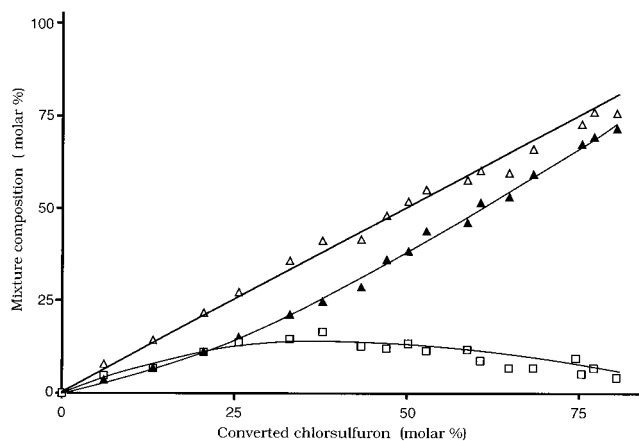
<sup>a</sup> Rate constants were determined assuming first-order kinetics.

<sup>b</sup> Maximum concentration observed for intermediate compound (probably phenyl carbamate).

method favored degradation (Table 3). The recovery of sulfonylamide and amino-heterocycle in almost equivalent yields suggested a predominant hydrolysis conversion, but quantitative interpretation was not possible. However, when an aliquot of correctly washed powder was subjected to an efficient hydrolytic treatment (70 °C, 24 h, pH 2.5), traces of sulfonylamide were recovered, which may attest to the presence of some carbamate links between the herbicide and its polymeric support. However, the presence of carbamate linkages remained impossible to detect using infrared spectroscopy.

**Saccharides.** Carbohydrates are present in soil in a variety of forms such as polysaccharides and monosaccharides (Lowe, 1978). These components, which are polyhydroxy molecules, may also react with sulfonylurea herbicides. With this in view, significant amounts of anhydrous glucose, the monosaccharide selected as model, were added to buffered sulfonylurea aqueous solutions. The degradation rates (Table 4) measured for chlorsulfuron and chlorimuron ethyl remained very low, in accordance with previous results about hydrolysis (Sabadie, 1991, 1995). The moderate rate increase that occurred after adding glucose seemed only to result from the accompanying decrease in pH. In addition, the ratio between the two products of hydrolysis (sulfonylamide and amino-heterocycle) remained unchanged. Consequently, alcoholysis reactions between these sulfonylurea herbicides and glucose were not observed.

**Phenols.** Humic substances, complex polymers with essentially phenolic structures, have been known to establish binding with various xenobiotic chemicals (Dec and Bollag, 1997). Because phenols are hydroxy molecules, it seemed necessary to examine the behavior of sulfonylurea herbicides when dissolved in phenol, which was chosen as model. The results (Table 5) show very low reaction rates despite a higher temperature (50 °C). Although only the two hydrolysis compounds remained, with similar yields, at the end of conversion, an intermediate product was also observed. This compound (probably phenyl carbamate) could not be isolated because it was too unstable. However, its concentration curve could be established from the difference between amino-heterocycle and sulfonylamide concentrations



**Figure 4.** Degradation of chlorsulfuron dissolved in phenol (3 mmol L<sup>-1</sup>, 50 °C): composition (molar %) of pyrimidamine ( $\Delta$ ), sulfonylamide ( $\blacktriangle$ ), and intermediate compound (likely phenyl carbamate) ( $\square$ ) as a function of converted chlorsulfuron (molar %).

(Figure 4). The low maximum concentration observed corroborates the instability of this intermediate product (Table 5).

## CONCLUSION

Extensive conversion was observed when the four selected sulfonylurea herbicides (metsulfuron methyl, chlorsulfuron, chlorimuron ethyl, and bensulfuron methyl) were dissolved in pure alcohols. Experiments were conducted in simple primary, secondary, and tertiary alcohols (methanol, ethanol, isopropyl alcohol, and *tert*-butyl alcohol), as well as in glycerol and in a polymeric alcohol (poly(ethylene glycol)). The reaction rates could always be described satisfactorily with first-order kinetics, and measured half-lives varied from 1.5 to 35 days at 30 °C. Degradation rates seemed to be governed by both the chemical structure of the sulfonylurea herbicide (chlorimuron ethyl > chlorsulfuron  $\approx$  bensulfuron methyl > metsulfuron methyl in descending order) and the class of alcohol used (primarily > secondary > tertiary).

Observed alcoholysis involves the breakdown of the urea part of the molecule and the formation of the corresponding sulfonyl carbamate (Figure 1). A minor hydrolysis reaction, which was observed concurrently, leads to the formation of sulfonylamide. However, it is the hydrolysis conversion that predominates after herbicide deposition on poly(vinyl alcohol) powder.

The addition of pure water (10–50%) induced an important hydrolysis conversion when chlorsulfuron was dissolved in ethanol. However, similar adducts of aqueous buffered solution (pH 7) completely inhibited alcoholysis. In the same way, hydrolysis was only observed after dissolution of chlorsulfuron and chlorimuron ethyl in buffered solution (pH 7), despite the addition of significant quantities of glucose.

In phenol solutions, the studied sulfonylurea herbicides essentially underwent hydrolysis at very low rates. However, an unknown and unstable compound (probably phenyl carbamate) was detected at a trace level.

A large proportion of soil organic residues consists of complex macromolecules such as polysaccharides, polyphenols, and humic substances. The results of our earlier alcoholysis studies suggested that these hydroxy compounds could also provoke various alcoholysis reactions involving the formation of unexpected degradation products or "bound residues".

However, the present observations lead to the conclusion that alcoholysis reactions only occurred under very particular conditions when sulfonylurea herbicides are dissolved in pure alcohols or in unbuffered hydroalcoholic solutions. Such situations unlikely appear in soil.

Although complementary investigations on soils seem necessary, these results suggested the improbability of alcoholysis reactions between sulfonylurea herbicides and soil hydroxy compounds.

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