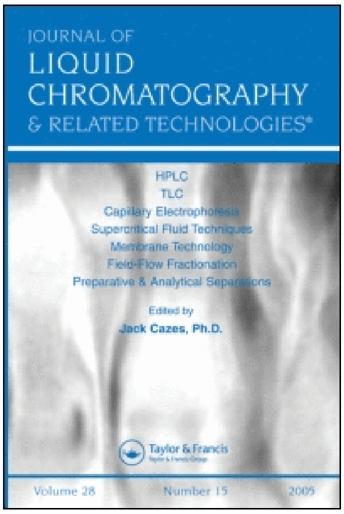

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Investigation of Selected Sulfonylurea Herbicides by TLC and HPLC

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To cite this Article Miszczyk, M. and Pyka, A.(2006) 'Investigation of Selected Sulfonylurea Herbicides by TLC and HPLC', Journal of Liquid Chromatography & Related Technologies, 29: 16, 2437 — 2449
To link to this Article: DOI: 10.1080/10826070600864916
URL: http://dx.doi.org/10.1080/10826070600864916

Journal of Liquid Chromatography & Related Technologies®, 29: 2437–2449, 2006

ISSN 1082-6076 print/1520-572X online
DOI: 10.1080/10826070600864916

Investigation of Selected Sulfonylurea Herbicides by TLC and HPLC

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Abstract: The following selected sulfonylurea herbicides have been investigated: thifensulfuron methyl (10), triasulfuron (11), chlorsulfuron (12), rimsulfuron (13), amidosulfuron (14), and tribenuron methyl (15) by NP-TLC, RP-TLC, and RP-HPLC. The best separations of sulfonylurea herbicides were obtained by NP-TLC (on silica gel and benzene-methanol mobile phase in volume composition 90:10), but the technique did not enable complete separation of all compounds, chlorsulfuron was not separated from thifensulfuron methyl. This pair can be separated by RP-TLC and use of a mobile phase with an organic mixture (acetonitrile-methanol, 1:1, v/v) - 0.1% H_3PO_4 in the volume composition 70:30. The separation of all investigated herbicides is not possible in applied chromatographic RP-TLC conditions. The retention times (t_R), the peak resolutions (R_S), and the separation factors (α) obtained by RP-HPLC indicate that the mobile phases acetonitrile – methanol – 0.1% H_3PO_4 in volume compositions 25:25:50, as well as 30:20:50 are the best for separations of this group of herbicides. Degradation of sulfonylurea herbicides happened in the range of 75–100% after 168 h. This fact permits accepting investigated pesticides as an unstable class of compounds (degradation after 1–12 weeks) in a water environment. From among investigated herbicides, the weakest stability in a water environment involved rimsulfuron and tribenuron methyl. Their concentration after 24 h was below 1% of initial concentration.

Keywords: TLC, HPLC, Sulfonylurea herbicides, Durability in water

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INTRODUCTION

The sulfonylurea herbicides are a new generation of plant protection products. High herbicidal activity of these compounds allows use of doses containing about 10 g/ha of active ingredient for sufficient protection of crops. This group of herbicides is generally used for protection of grains. The sulfonylurea compounds works by interfering with branched chain amino acids production.[1–3]

Stability in the environment is one of the most important criterion taken into account when permission to use the particular compound is given. In the case of organic compounds, their durability in the environment is expressed by half-life ($t_{1/2}$) or by time of their degradation in the environment. Taking into account the time of degradation, pesticides are divided into following classes:

- very stable, indefinite stability, about 20–30 years;
- stable, degradation in environment after 2–5 years;
- moderate stable, degradation in range 1–18 months; and
- unstable, degradation proceeds in time of 1–12 weeks.[4]

Taking into account that sulfonylurea derivatives are active ingredients of plant protection products, which were commercialized, in the early 90's (ten years after the introduction of the first sulfonylurea herbicide – chlorsulfuron), they have characterized these substances by high efficiency and quick degradation.[5]

Two groups of the selected pesticides: 1st group – monolinuron (1), chlorotoluron (2), diuron (3), isoproturon (4), linuron (5); and 2nd group – dimefuron (6), diflubenzuron (7), teflubenzuron (8), and lufenuron (9) have been investigated by RP-HPLC, PR-HPTLC, and NP-TLC in earlier works.[6–9] Earlier, we also investigated retention parameters of selected sulfonylurea herbicides separated by the LC technique.[9,10] The presented work is a continuation of these investigations.

The aim of our study was to work out the optimum conditions of the separation of selected sulfonylurea herbicides: thifensulfuron methyl (10), triasulfuron (11), chlorsulfuron (12), rimsulfuron (13), amidosulfuron (14), and tribenuron methyl (15) by normal and reversed-phase thin layer chromatography (NP-TLC and RP-TLC), as well as reversed-phase high performance liquid chromatography (RP-HPLC), and evaluation of their durability in water, of the mentioned-above herbicides.

EXPERIMENTAL

Chemicals and Sample Preparation

The components of mobile phases: benzene, methanol, ethanol (POCh, Poland), acetonitrile (Merck, Germany), and water (Millipore, France) were for HPLC analysis. Potassium dichromate and sulfuric acid (POCh, Poland) were used to prepare the visualizing reagent. The commercial samples of thifensulfuron methyl, rimsulfuron, tribenuron methyl (DuPont, France), amidosulfuron, chlorsulfuron (Labor Dr. Ehrenstorfier, Germany), and triasulfuron (Ciba Geigy, Switzerland) were used as test solutes. The above mentioned sulfonylurea herbicides (about concentration 0.1 mg mL^{-1} of each pesticide) were dissolved in mixtures of methanol, water, and phosphate buffer ($pH = 6.8$), in the volume proportion 2:2:1. The purity of the studied standard samples was at least 98.6%.

Thin Layer Chromatography

Adsorption Thin-Layer Chromatography

Adsorption TLC was performed on 20 × 20 cm glass plates, precoated with 0.25 mm layer of a silica gel $60F_{254}$ (E. Merck, #1.05715). The plates were activated at 120°C for 30 min. Solutions of the standards (10 μL) were spotted manually on chromatographic plates. The particular compounds were separately spotted on plates. The pesticides were separated using benzene-methanol and benzene-ethanol in volume compositions 85:15, 90:10, 95:5, 98:2, and 100:0 as mobile phases. The mobile phase (50 mL) was placed in a classical chamber (Camag, Switzerland) for development of 20 cm × 20 cm plates, and the chamber was saturated with the mobile phase for 30 min. The chromatograms were developed at room temperature. The development distance was 14 cm. The plates were dried at room temperature in a fume cupboard. The pesticides were then detected with iodine vapor, or by treatment with a solution of potassium dichromate (5 g) in sulfuric acid (40%; 100 g) then heated 150°C.

Reversed Phase Thin-Layer Chromatography

Reversed phase thin-layer chromatography was performed on 10 × 10 cm glass HPTLC plates, coated with RP-$18WF_{254}$ (Merck, #1.13124). Solutions of the standards (10 μL) were spotted manually on the chromatographic plates. The mixture organic (acetonitrile-methanol, 1:1, v/v)-0.1% aqueous orthophosphoric acid (H_3PO_4), in volume compositions 90:10, 80:20, 70:30, 60:40, and 50:50 were used as mobile phases. Plates were developed to a distance of 8 cm at room temperature (18 ± 1°C) in a classical flat bottom chamber (Camag, Switzerland) for development of 20 cm × 20 cm plates previously saturated with the mobile phase for 30 min. After development and drying the spots were visualized with the UV light ($\lambda = 254$ nm).

Chromatographic Parameters

Chromatography was performed in triplicate and mean R_F values were calculated.

The separation factor (α)[11] and the constant of the pair separation (R_F^{α})[12] were calculated for all the chromatograms. The separation factor (α) was calculated by use of the formula:

$$\alpha = \frac{1/R_{F1} - 1}{1/R_{F2} - 1} \tag{1}$$

where R_{F1} and R_{F2} are the R_F values of two adjacent spots, and $R_{F1} < R_{F2}$.

The constant of the pair separation (R_F^{α}) was calculated by use of the formula:

$$R^{\alpha}_{F(1,2)} = \frac{R_{F1}}{R_{F2}} \tag{2}$$

where R_{F1} and R_{F2} are the R_F values of two adjacent spots, and $R_{F1} > R_{F2}$.

ΔR_F values were calculated by use of the formula:

$$\Delta R_{F(1,2)} = R_{F1} - R_{F2} \tag{3}$$

where R_{F1} and R_{F2} are the R_F values of two adjacent spots, and $R_{F1} > R_{F2}$.

Reversed-Phase High Performance Liquid Chromatography

The chromatographic investigations were conducted using the liquid chromatograph obtained from Gynkotek firm (Germany). Dionex Softron GmbH certified the chromatograph. The conditions of applied HPLC were the following: pump – P 580 LPG, detector UVD 340 S, column – Alltima C18 5 u (250 mm × 4.6 mm), injection volume – 20 mL, eluent flow – 1 mL/min. The detection of the sulfonylurea herbicides investigated was conducted at 230 nm. Acetone was selected for the determination of the retention time of an unretained compound.

Isocratic elution was applied using, as mobile phase, a mixture of methanol, acetonitrile, and 0.1% water solution of orthophosphoric acid (H_3PO_4). Fifty percent of the 0.1% H_3PO_4 was always in the mobile phase. However, the content of acetonitrile was comprised from 20% to 40% in mobile phase.

The logarithm of the retention factor ($\log k$) was calculated using the formula:[13]

$$\log k = \log \frac{t'_R}{t_M} = \log \frac{t_R - t_M}{t_M} \tag{4}$$

where t_R is the retention time of the investigated compound, t'_R is adjusted retention time of the investigated compound; whereas t_M is retention time of an unretained compound (acetone).

The separation factor (α) was calculated using the equation:[13]

$$\alpha = \frac{\mathrm{k}_2}{\mathrm{k}_1} \tag{5}$$

where k_2 and k_1 are retention factors of two adjacent peaks ($k_2 > k_1$).

The peak resolution (R_S) was calculated using the formula:[13]

$$R_S = \frac{2(t_{R2} - t_{R1})}{w_{b1} + w_{b2}} \tag{6}$$

where t_{R2} and t_{R1} are retention times of two adjacent peaks in the chromatogram ($t_{R2} > t_{R1}$), whereas w_{b1} and w_{b2} are the peaks width at the base.

Evaluation of Durability of Selected Urea Derivatives in the Water Environment by the RP-HPLC Method

For determination of half-life of investigated compounds in the water environment by the RP-HPLC method, methanolic solutions of these compounds were prepared. Weights of analytical standards of particular substances were dissolved with methanol in volumetric flasks (10 mL), then 1 mL was taken, and transferred to a volumetric flask (10 mL), and diluted to mark with water. Each pesticide was investigated separately. Weights of compounds were established according to literature data about solubility in methanol and water.[14] Usually weights were about 0.01 g (accuracy 0.0001 g). Chromatographic investigation of durability selected pesticides in water environment was carried out using as mobile phase: acetonitrile – methanol -0.1% H_3PO_4 in 25:25:50 (v/v).

During investigations pH of water solutions selected pesticides were measured using pH-meter Knick 912 with gel electrode Double Por (Hamilton). Flasks with water solutions were kept in room temperature (18° ± 2°C).

Determination of Half-Life ($t_{1/2}$)

For the investigated urea derivatives, we established a first-order degradation model. According to this model of half-life,[5] the following equation is calculated:

$$t_{1/2} = \frac{0.693}{\mu} \tag{7}$$

where μ is the reaction rate constant (T^{-1}).

The first order degradation constant can be expressed by the following equation:

$$\mu = \frac{\ln \frac{C_o}{C}}{t} \tag{8}$$

where C is the substrate concentration at time (t), C_o is the initial substrate concentration.

RESULTS AND DISCUSSION

R_F values, obtained by NP-TLC using benzene-methanol and benzene-ethanol mobile phases for examined sulfonylurea herbicides, are presented in Table 1. Adsorption chromatography (NP-TLC) on silica gel with benzene-methanol and benzene-ethanol, 85:15, 90:10, and 95:5, as mobile phases separated the sulfonylurea herbicides into four subgroups: 1. tribenuron methyl (15), 2. rimsulfuron (13), 3. triasulfuron (11), and amidosulfuron (14), 4. chlorsulfuron (12) and tifensulfuron methyl (10).

Chlorsulfuron cannot be separated from tifensulfuron methyl by NP-TLC under the conditions used. The best separation of tribenuron methyl, rimsulfuron, triasulfuron, amidosulfuron, and chlorsulfuron was obtained by use of benzene-methanol mobile phase in volume composition 90:10 ($\Delta R_{F(15/13)} = 0.14$, $R^{\alpha}_{F(15/13)} = 1.26$, $\alpha_{(15/13)} = 1.81$; $\Delta R_{F(13/11)} = 0.25$, $R^{\alpha}_{F(13/11)} = 1.86$, $\alpha_{(13/11)} = 2.87$; $\Delta R_{F(11/14)} = 0.06$, $R^{\alpha}_{F(11/14)} = 1.26$, $\alpha_{(11/14)} = 1.37$; $\Delta R_{F(14/12)} = 0.05$, $R^{\alpha}_{F\ (14/12)} = 1.28$, $\alpha_{(14/12)} = 1.36$).

The separation of all investigated herbicides is not possible in applied chromatographic RP-TLC conditions (Table 2). Rimsulfuron can be separated from triasufluron ($\Delta R_{F(13/11)} = 0.07$, $R^{\alpha}_{F(13/11)} = 1.32$, $\alpha_{(13/11)} = 1.45$) and rimsulfuron from tribenuron methyl ($\Delta R_{F(13/15)} = 0.10$, $R^{\alpha}_{F(13/15)} = 1.83$, $\alpha_{(13/15)} = 2.07$) on $RP18WF_{254}$ plates by use of the organic modifier – 0.1% H_3PO_4 mobile phase in volume composition 50:50. Amidosulfuron can be separated from rimsulfuron ($\Delta R_{F\ (14/13)} = 0.09$, $R^{\alpha}_{F\ (14/13)} = 1.25$, $\alpha_{(14/13)} = 1.45$) and rimsulfuron from tribenuron methyl ($\Delta R_{F\ (13/15)} = 0.06$, $R^{\alpha}_{F(13/15)} = 1.50$, $\alpha_{(13/15)} = 1.31$) by using the organic modifier – 0.1% 60:40 (v/v), as mobile phase. Tifensulfuron methyl was separated from chlorsulfuron ($\Delta R_{F(10/12)} = 0.04$, $R^{\alpha}_{F(10/12)} = 1.07$, $\alpha_{(10/12)} = 1.18$), chlorsulfuron from rimsulfuron ($\Delta R_{F\ (12/13)} = 0.04$, $R^{\alpha}_{F\ (12/13)} = 1.08$, $\alpha_{(12/13)} = 1.17$) and amidosulfuron from tribenuron methyl ($\Delta R_{F(14/15)} = 0.04$, $R^{\alpha}_{F(14/15)} = 1.08$, $\alpha_{(14/15)} = 1.17$) by using the organic modifier-0.1% H_3PO_4, 70:30 (v/v) as mobile phase.

The best separations of sulfonylurea herbicides were obtained by NP-TLC, but the technique did not enable complete separation of all compounds – chlorsulfuron was not separated from tifensulfuron methyl.

Table 1. R_F values[a] of investigated sulfonylurea herbicides using NP-TLC and benzene-methanol as well as benzene-ethanol in different volume composition as mobile phases

	Benzene-methanol, *v*/*v*					Benzene-ethanol, *v*/*v*				
Pesticide	85:15	90:10	95:5	98:2	100:0	85:15	90:10	95:5	98:2	100:0
Thiensulfuron methyl (10)	0.21	0.18	0.14	0.01	0.00	0.38	0.19	0.08	0.03	0.00
Triasulfuron (11)	0.33	0.29	0.19	0.03	0.00	0.45	0.27	0.11	0.04	0.00
Chlorsulfuron (12)	0.24	0.18	0.14	0.03	0.00	0.35	0.19	0.06	0.03	0.00
Rimsulfuron (13)	0.62	0.54	0.51	0.19	0.00	0.56	0.46	0.29	0.18	0.00
Amidosulfuron (14)	0.34	0.23	0.22	0.16	0.00	0.42	0.29	0.15	0.08	0.00
Tribenuron methyl (15)	0.73	0.68	0.59	0.14	0.00	0.69	0.58	0.34	0.16	0.00

[a] Average of 3 measurements

Table 2. R_F values[a] of investigated sulfonylurea herbicides using RP-TLC and organic mixture (acetonitrile – methanol, 1:1, v/v) – 0.1% H_3PO_4 in different volume composition as mobile phases

	Organic mixture (Acetonitrile – Methanol, 1:1, v/v)\rm 0.1% H_3PO_4, v/v				
Pesticide	50:50	60:40	70:30	80:20	90:10
Thiensulfuron methyl (10)	0.32	0.46	0.60	0.65	0.72
Triasulfuron (11)	0.29	0.45	0.61	0.65	0.72
Chlorsulfuron (12)	0.33	0.45	0.56	0.63	0.71
Rimsulfuron (13)	0.22	0.36	0.52	0.60	0.69
Amidosulfuron (14)	0.35	0.45	0.51	0.62	0.69
Tribenuron methyl (15)	0.12	0.30	0.47	0.56	0.66

[a] Average of 3 measurements

This pair can be separated by RP-TLC and use of a mobile phase with an organic modifier volume fraction of 0.70.

The retention time values (t_R) from RP-HPLC were introduced for the investigated sulfonylurea herbicides, as well as acetone as an unretained compound in acetonitrile – methanol – 0.1% H_3PO_4 as mobile phase in various volume fractions (Table 3). The peak resolutions (R_S), as well

Table 3. Retention time values (t_R) [min] of investigated sulfonylurea herbicides and acetone using acetonitrile – methanol – 0.1% H_3PO_4 in different volume fraction as mobile phases

	Acetonitrile – Methanol – 0.1% H_3PO_4, v/v				
	20:30:50	25:25:50	30:20:50	35:15:50	40:10:50
Pesticide	t_R [min][a]				
Thiensulfuron methyl (10)	12.615	11.515	10.369	9.226	8.185
Triasulfuron (11)	13.911	13.109	12.077	10.910	9.749
Chlorsulfuron (12)	18.432	16.487	14.508	12.619	10.910
Rimsulfuron (13)	21.077	18.431	15.808	13.392	11.265
Amidosulfuron (14)	21.825	19.382	16.969	14.698	12.646
Tribenuron methyl (15)	29.792	27.013	24.035	21.105	18.248
Acetone	3.192	3.181	3.181	3.208	3.201

[a] Average of 3 measurements

as the separation factors (α), of investigated herbicides with acetonitrile – methanol – 0.1% H_3PO_4 in different volume fractions as mobile phase were presented in Table 4. The highest values of peak resolutions (R_S), the separation factors (α) as well as the largest differences in retention times, indicate that acetonitrile – methanol – 0.1% H_3PO_4 mobile phases in the volume compositions 20:30:50, 25:25:50, 30:20:50, and 35:15:50 separated completely of all six studied herbicides. However, from a practical point of view the retention time in liquid chromatography is the most important. The retention times should be comparatively short, giving, simultaneously, the total separation of individual components of the studied mixture. The retention times (t_R) of studied substances, the peak resolutions (R_S), and the separation factors (α) obtained for the studied herbicides, indicate that the optimum mobile phase for separation of all investigated sulfonylurea herbicides were the mobile phases containing about 25% and 30% of acetonitrile in acetonitrile – methanol – 0.1% H_3PO_4 mobile phase. The chromatogram obtained using acetonitrile – methanol – 0.1% H_3PO_4 mobile phase in the volume composition 25:25:50 is presented in Fig. 1.

Next investigations were conducted by using RP-HPLC, because this technique enables complete separation of the all examined herbicides. Percentage changes of concentration of water solutions of investigated herbicides and pH values from time of their storing are presented in Tables 5–7. Obtained data shows that between selected herbicides the weakest stability in the water environment have rimsulfuron and tribenuron methyl. Their concentration in the solution after 24 h is below 1% of initial concentration. The most stable among the investigated group of

Table 4. Peak resolutions (R_S) and separation factors (α) of investigated sulfonylurea herbicides using acetonitrile – methanol – 0.1% H_3PO_4 in different volume fraction as mobile phases

	R_S	Acetonitrile – Methanol – 0.1% H_3PO_4, v/v				
	α	20:30:50	25:25:50	30:20:50	35:15:50	40:10:50
	R_{S10-11}	2.656	3.594	4.408	4.997	5.162
	R_{S11-12}	7.946	6.496	5.519	3.418	3.261
R_S	R_{S12-13}	3.918	3.289	2.661	1.849	0.907
	R_{S13-14}	1.006	1.534	2.239	2.941	3.523
	R_{S14-15}	9.184	10.333	11.101	11.360	11.844
α	α_{10-11}	1.138	1.191	1.238	1.280	1.314
	α_{11-12}	1.422	1.340	1.273	1.222	1.177
	α_{12-13}	1.174	1.146	1.115	1.082	1.046
	α_{13-14}	1.042	1.062	1.092	1.128	1.171
	α_{14-15}	1.428	1.471	1.513	1.558	1.593

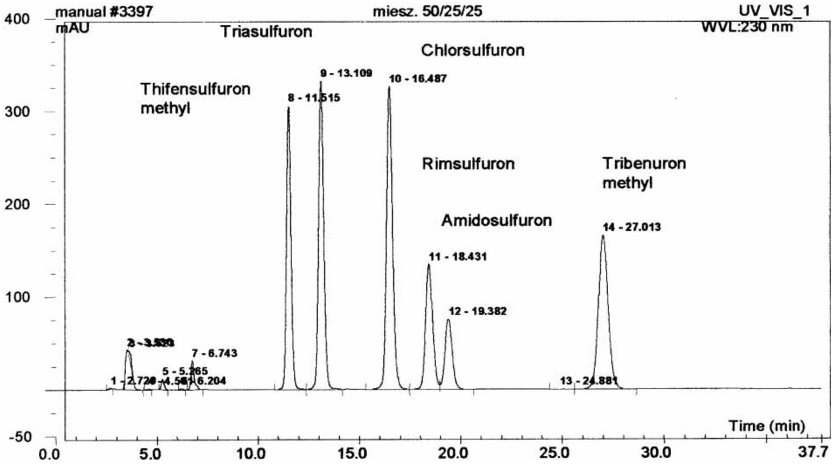

Figure 1. Chromatogram with acetonitrile – methanol – 0.1% H_3PO_4 (25:25:50, *v*/*v*) as mobile phase for the investigated sulfonylurea herbicides.

compounds are: thifensulfuron methyl, triasulfuron, and chlorsulfuron. Their concentrations in water solutions after 24 h are in the range 85.8–93.5% of initial concentrations. In this time, the contents of amidosulfuron drops to about 50%. Similar concentrations (about 50%) thifensulfuron methyl, triasulfuron, and chlorsulfuron are reached after 72 h. A degradation of sulfonylurea herbicides in the range 75–100% was observed after 168 h, which allows us to accept the substances of this group as an unstable

Table 5. Percentage changes of concentration of aqueous solutions of rimsulfuron and tribenuron methyl and pH values from time of their storing

	Rimsulfuron		Tribenuron methyl	
Time of storage (h)	Change of concentration (%)	pH	Change of concentration (%)	pH
1	100	4.09	100	4.35
2	80.5	4.27	78.1	4.68
3	72.7	4.14	61.2	4.86
4	60.6	4.12	47.7	5.20
5	51.3	4.16	36.6	5.46
6	42.3	4.03	27.5	5.99
7	35.2	4.01	24.5	6.04
8	28.0	4.04	20.1	6.15
9	23.5	3.96	18.2	6.25
24	0.9	3.97	0.5	6.55

Table 6. Percentage changes of concentration of aqueous solutions of thiensulfuron methyl, triasulfuron, and chlorsulfuron and pH values from time of their storing

	Thiensulfuron methyl		Triasulfuron		Chlorsulfuron	
Time of storage (h)	Change of concentration (%)	pH	Change of concentration (%)	pH	Change of concentration (%)	pH
1	100	3.92	100	4.36	100	3.87
24	93.5	3.97	85.8	4.75	87.8	3.92
48	87.2	4.09	66.2	4.73	76.2	4.01
72	55.4	4.24	48.3	4.77	45.1	4.05
96	43.4	4.67	39.5	4.78	34.8	4.22
120	33.7	4.74	31.6	4.82	26.1	4.28
144	26.7	4.80	28.7	4.88	20.4	4.35
168	21.9	4.90	25.0	4.93	16.4	4.43

class of compounds (degradation after 1–12 weeks) in the water environment. Table 8 shows obtained half-life values ($t_{1/2}$) of sulfonylurea herbicides according to the Equation (7). For all compounds, except rimsulfuron and tribenuron methyl, the last measure data of time (t) versus concentration (C) were taken into account. In the case of rimsulfuron, $t = 9$ (h) and this corresponded with its measured concentration;

Table 7. Percentage changes of concentration of aqueous solution of amidosulfuron and pH values from time of their storing

	Amidosulfuron	
Time of storage (h)	Change of concentration (%)	pH
1	100	4.08
4	94.6	3.91
9	90.5	3.98
24	53.8	4.06
25	50.6	4.05
30	46.4	4.09
48	40.6	4.09
54	36.1	4.16
72	33.8	4.07
78	31.4	4.16
120	28.6	4.17
168	26.1	4.20

Table 8. Half-life values of investigated sulfonylurea herbicides

Sulfonylurea herbicide	$T_{1/2}$ (h)
Thiensulfuron methyl (10)	77.0
Triasulfuron (11)	86.6
Chlorsulfuron (12)	64.2
Rimsulfuron (13)	4.3
Amidosulfuron (14)	86.6
Tribenuron methyl (15)	3.2

however, for tribenuron methyl, $t = 6$ (h) and its corresponding concentration was taken (in accordance with values from Table 5). In comparing data from Table 8 with data presented in Tables 5–7, high accordance was identified for thifensulfuron methyl, chlorsulfuron, rimsulfuron, and tribenuron methyl; it was lower for triasulfuron and the lowest was for amidosulfuron

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Received March 28, 2006
Accepted April 27, 2006
Manuscript 6863