

## Isoxaflutol Herbicide Soil Persistence and Mobility in Summer Corn and Winter Wheat Crops

J. Rouchaud,<sup>1</sup> O. Neus,<sup>1</sup> D. Callens,<sup>2</sup> R. Bulcke<sup>2</sup>

<sup>1</sup>Laboratory of Phytopharmacy, Catholic University of Louvain, 3, Place Croix du Sud, SCl. 15D, 1348 Louvain-la-Neuve, Belgium

<sup>2</sup>Weed Research Center, University of Gent, Coupure Links 653, 9000 Gent, Belgium

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Isoxaflutol (5-cyclopropyl-4-(2-methanesulphonyl-4-trifluoromethylbenzoyl)isoxazole, RPA 201772) is a new experimental 5-cyclopropyl-4-benzoyl-isoxazole herbicide (Luscombe et al., 1995). It provides excellent selective control of both grass and broad-leaf weeds in corn, after pre-emergence application at the relatively low rates of 75 to 125 g a.i. ha<sup>-1</sup>. Isoxaflutol disrupts pigment biosynthesis via inhibition of phydroxyphenyl pyruvate dioxygenase and as such brings a new mode of action to combat the risks of resistance to current products. Isoxaflutol has a low solubility in water (6 mg litre<sup>-1</sup>). In the present work the soil persistence and mobility of isoxaflutol have been studied in summer corn and winter wheat crops made in several sites different as to their soil textures and compositions.

### MATERIALS AND METHODS

A spring corn crop was made in 1997 at Melle on sandy loam soil at different pH, with isoxaflutol pre-sowing application. A 24 x 30 m field at Melle, Belgium (clay 7%, silt 38%, sand 55%, organic matter 1.87% -%C x 2-, sandy loam) was divided in 6 x 10 m plots located in randomized block design. Since 9 years, the soil of the plots was stabilized at different pH (5.5, 6.1 or 7.2 ; relative to water) by addition of controlled amounts of calcium carbonate, i.e. the foams from the sugar beet industry. No organic fertilizer was applied on the field plots since 6 years. For each soil pH, there were 4 replicate plots. The field was tilled on 11-12-1996 (day-month-year). On 25-3-1997, the field was rotary-tilled to 15 cm depth, and made ready as for sowing and 75 g isoxaflutol ha<sup>-1</sup> was applied by spraying the emulsion of isoxaflutol (water dispersible granules containing 75% isoxaflutol, Rhone-Poulenc Agrochimie, Belgium) in water (300 L ha<sup>-1</sup>). Corn (cv. Aviso) was sown on 21-4-1997. At intervals after isoxaflutol application, samples were taken separately (and analyzed separately) in the 0-8 cm surface soil layer of each of the four replicate plots. Sampling dates (day-month in 1997), the days after isoxaflutol treatment, and the cumulative rainfall (mm) respectively were : 26-3, 1, 0; 14-4, 20, 4; 30-4, 36, 19; 13-5, 49, 62; 21-5, 57, 97; 17-6, 84, 168. For each soil sample, 15 cores (2.5 cm diameter) were taken from each replicate plot at random points, the cores from each replicate plot were bulked together and then stored at -25°C until analyzed.

A summer corn crop was made in 1996 on sandy loam soil at Melle, with isoxaflutol pre-emergence application. A 120 x 140 m field was tilled on 19-4-1996 (clay 7%, silt 38%, sand 55% ; organic matter 2.28% ; pH 6.6 ; sandy loam). At the end of August 1995 (8 months before the isoxaflutol application), 40 tons cow slurry ha<sup>-1</sup> was applied. In the past, 40 tons cow manure ha<sup>-1</sup> (or alternately 40 tons cow slurry ha<sup>-1</sup>) was applied once every 3 years. On 20-4-1996, corn (cv. Aviso) was sown. On 25-4-1996, 75 g isoxaflutol ha<sup>-1</sup> was applied. Four 10 x 12 m replicate plots were localized at random in the field. At intervals after isoxaflutol application, soil samples were taken in the 0-8 cm surface soil layer separately (and analyzed separately) in each of the 4 replicate plots, in the same way as in the pH trial. Sampling dates (day-month in 1996), the days after isoxaflutol treatment, and the cumulative rainfall (mm) respectively were : 25-4, 0, 0 ;

9-5, 14, 20 ; 30-5, 35, 66 ; 14-6, 50, 93 ; 28-6, 64, 117; 15-7, 81, 131 ; 31-7, 97, 147; 19-8, 116, 192.

Summer corn crops were made in 1997 in four sites having different soil types. Isoxaflutol was applied pre-emergence at the same date (except at Zingem where the isoxaflutol treatment was made one week later than at the 3 other sites) on four corn crops (about 130 x 145 m each) located in 4 sites different as to their soil textures and compositions, and located about 40 km apart in Belgium : 1. Melle: clay 7%, silt 38%, sand 55%, organic matter 1.71%, pH 5.6, sandy loam; no organic fertilizer was applied in spring 1997 nor in September 1996; in the past, 40 tons cow manure ha<sup>-1</sup> (or alternately 40 tons cow slurry ha<sup>-1</sup>) was applied once every 3 years. 2. Zingem: clay 10%, loam 11%, sand 79%, organic matter 1.72%, pH 7.0, loamy sand; in April 1997 (one month before isoxaflutol treatment) 30 tons cow manure ha<sup>-1</sup> was applied; in the past, this treatment was applied once every 4 years. 3. Koksijde: clay 34%, loam 47%, sand 19%, organic matter 2.06%, pH 7.6, clay loam; in September 1996 (8 months before isoxaflutol treatment), 30 tons cow slurry ha<sup>-1</sup> was applied; winter wheat was the preceding crop; in the past, 30 tons cow slurry ha<sup>-1</sup> was applied once every 3 years. 4. Zarlardinghe: clay 14%, loam 51%, sand 35%, organic matter 1.72%, pH 6.3, loam; in April 1997 (one month before isoxaflutol treatment) 30 tons cow manure ha<sup>-1</sup> was applied, sugar beet was the preceding crop; in the past, corn and sugar beet crops alternated and 30 tons cow manure ha<sup>-1</sup> was applied every year in the spring. The fields were tilled at the beginning of May 1997 ; some days later, corn (cv. Aviso) was sown. On 12-5-1997, 125 g isoxaflutol ha<sup>-1</sup> was applied pre-emergence on the fields at Melle, Koksijde and Zarlardinghe. On 20-5-1997, the same isoxaflutol treatment was applied pre-emergence on the field at Zingem. Four 10 x 12 m replicate plots were localized at random in each field. At intervals after isoxaflutol application, in each field and at the same date soil samples were taken in the 0-10 cm surface soil layer separately (and analyzed separately) in each of the 4 replicate plots, as in the pH trial (Table 1). In the 0-2, 2-4, 4-6, 6-8, 8-10, 10-15 and 15-20 cm surface soil layers, soil samples were taken in each replicate field plots, but the samples from two replicate field plots were mixed giving a total of 2 separate replicates -for each surface soil layer- which were analyzed separately. At Melle, the sampling dates (day-month in 1997), and the days after isoxaflutol treatment respectively were : 12-5, 0; 21-5, 9; 17-6, 36; 8-7, 57; 28-7, 77; 20-8, 99. At Zingem, these values respectively were : 21-5, 1; 31-5, 10; 17-6, 28; 8-7, 49; 28-7, 69; 20-8, 92. At Koksijde, these values respectively were : 12-5, 0; 21-5, 9; 17-6, 36; 8-7, 57; 28-7, 77; 20-8, 99. At Zarlardinghe, these values respectively were : 12-5, 0; 21-5, 9; 17-6, 36; 8-7, 57; 28-7, 77; 20-8, 99.

Winter wheat crops were made in 1996-1997 in four sites of different soil types, with isoxaflutol pre-emergence treatment (except at Melle where isoxaflutol was applied post-emergence) at the same date. The fields (130 x 145 m each) were located 40 km apart in Belgium : 1. Melle: clay 7%, silt 38%, sand 55%, organic matter 1.92%, pH 7.0, sandy loam; in September 1996, 40 tons cow manure ha<sup>-1</sup> was applied; in the past, 40 tons cow manure ha<sup>-1</sup> (or alternately 40 tons cow slurry ha<sup>-1</sup>) was applied once every 3 years. 2. Zingem: clay 10%, loam 11%, sand 79%, organic matter 1.98%, pH 7.0, loamy sand; no organic fertilizer was applied before winter wheat sowing; sugar beet was the preceding crop ; at harvest, their leaves and tops were soil incorporated in September 1996; in the past, 30 tons cow manure ha<sup>-1</sup> was applied once every 4 years. 3. Sint Denijs: clay 16%, loam 49%, sand 35%, organic matter 1.17%, pH 8.1, loam; no organic fertilizer has been applied since more than 5 years in the past; sugar beet was the preceding crop : at harvest, their leaves and tops were soil incorporated in September 1996. 4. Zevekote: clay 36% loam 45%, sand 19%, organic matter 2.94%, pH 8.0, clay loam; in September 1996, 40 tons pig slurry ha<sup>-1</sup> was applied; the preceding crop was flax; in the past, every year 40 tons pig slurry ha<sup>-1</sup> or 40 tons cow manure ha<sup>-1</sup> were alternately applied. At Melle, the field was tilled and sown with winter wheat (cv. Fidel) on 15-10-1996. At Zingem, Sint Denijs and Zevekote, the fields were tilled and sown with winter wheat at the end of October 1996. On the four fields, 125 g isoxaflutol ha<sup>-1</sup> was applied on 8-11-1996 preemergence at Zingem, Sint Denijs and Zevekote, and post-emergence at Melle. In each field there were four 10 x 12 m replicate plots located at random in the fields. At intervals after the isoxaflutol application, in each field and at the same dates, samples were taken in the 0-10 cm surface soil layer separately (and analyzed separately)

**Table 1.** Isoxaflutol soil concentrations, half-lives (in the 0-10 cm surface soil layer) and mobility in the summer corn crops made in 1997 in 4 sites different as to their soil types.

in the summer corn crops made in 1997 in 7 sites different as to their soil types.

Days after isoxaflutol treatment	Cumulative rainfall, mm	Surface soil layers depths, cm						
		0-10	0-2	2-4	4-6	6-8	8-10	10-15
		Isoxaflutol concentrations ( $\mu\text{g kg}^{-1}$ dry soil) in the surface soil layers <sup>a</sup>						
1. Sandy loam at Melle:								
0	0	93±5	465±33					
9	40	51±3	217±15	38±3	nd	nd	nd	nd
36	110	25±2	54±4	58±4	9±2	4±2	nd	nd
57	178	16±2	15±2	20±2	36±3	6±2	2±2	nd
77	218	12±2	6±2	18±2	27±2	7±2	2±2	nd
99	225	9±2	2±2	10±2	23±2	7±2	4±2	nd
Corr. coeff.: 0.9840; $k=1.01698 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 10.6±0.6 days.								
2. Loamy sand at Zingem:								
1	0	91±5	455±36					
10	2	55±3	275±16	nd	nd	nd	nd	nd
28	72	35±2	126±7	39±3	11±2	nd	nd	nd
49	140	20±2	26±2	50±3	10±2	7±2	5±2	2±2
69	180	15±2	9±2	17±2	34±2	11±2	3±2	2±2
92	187	13±2	7±2	12±2	28±2	14±2	4±2	nd
Corr. coeff.: 0.9897; $k=0.7620 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 14.1±0.7 days								
3. Clay loam at Koksijde:								
0	0	98±6	490±39					
9	33	58±3	232±16	58±4	nd	nd	nd	nd
36	92	26±2	39±3	72±5	20±2	nd	nd	nd
57	226	17±2	16±2	18±2	38±3	9±2	3±2	2±2
77	244	12±2	6±2	11±2	32±2	8±2	2±2	2±2
99	253	11±2	8±2	10±2	30±2	7±2	nd	nd
Corr. coeff.: 0.9873; $k=0.8654 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 12.4±0.6 days								
4. Loam at Zarlarding:								
0	0	88±6	440±35					
9	47	71±4	302±21	53±4	nd	nd	nd	nd
36	100	34±2	58±5	112±8	nd	nd	nd	nd
57	171	22±2	29±2	68±6	13±2	nd	nd	nd
77	228	18±2	18±2	50±4	18±2	5±2	nd	nd
99	240	14±2	16±2	32±4	17±2	4±2	nd	nd
Corr. coeff.: 0.9836; $k=0.5294 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 20.3±1.0 days								

a. In the 0-10 cm surface soil layer, means of 4 replicates ± SD. In the 0-2, 2-4, 4-6, 6-8, 8-10, 10-15 and 15-20 cm surface soil layers, means of 2 replicates ± SD. nd=Not detected. Isoxaflutol was not detected in the 15-20 cm surface soil layer.

**Table 2.** Isoxaflutol soil concentrations, half-lives (in the 0-10 cm surface soil layer) and mobility in the winter wheat crops made in 1996-1997 in 4 sites different as to their soil types

in the winter wheat crops made in 1976-1977 in 4 sites different as to their soil types

Days after isoxaflutol treatment	Cumulative rainfall, mm	Surface soil layers depths, cm						
		0-10	0-2	2-4	4-6	6-8	8-10	10-15
		Isoxaflutol concentrations ( $\mu\text{g kg}^{-1}$ dry soil) in the surface soil layers <sup>a</sup>						
1. Sandy loam at Melle:								
0	0	93±6	465±33					
15	55	66±4	264±19	66±6	nd	nd	nd	nd
40	110	42±3	126±10	63±5	17±2	4±2	nd	nd
76	128	25±2	50±5	50±4	15±2	5±2	5±2	nd
112	201	14±2	18±2	35±3	11±2	4±2	4±2	nd
140	219	8±2	10±2	20±2	6±2	2±2	2±2	nd
173	236	5±2	6±2	13±2	4±2	nd	nd	nd
220	385	nd	nd	3±2	nd	nd	nd	nd
Corr. coeff.: 0.9734; $k=0.5193 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 20.7±1.0 days								
2. Loamy sand at Zingem:								
0	0	98±5	490±39					
15	55	63±4	252±18	47±4	16±2	nd	nd	nd
40	110	34±2	43±3	119±8	9±2	nd	nd	nd
76	128	17±2	7±2	33±2	35±3	7±2	3±2	nd
112	201	9±2	5±2	11±2	23±2	5±2	2±2	nd
140	219	6±2	3±2	8±2	15±2	3±2	nd	nd
173	236	6±2	nd	8±2	14±2	6±2	nd	nd
220	385	nd	nd	nd	4±2	nd	nd	nd
Corr. coeff.: 0.9687; $k=0.8903 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 12.1±0.7 days								
3. Loam at Sint Denijs:								
0	0	88±6	440±35					
15	57	41±2	21±2	185±15	nd	nd	nd	nd
40	117	19±2	10±2	14±2	67±5	5±2	nd	nd
76	127	10±2	3±2	5±2	10±2	28±2	3±2	3±2
112	195	8±2	2±2	4±2	6±2	18±2	10±2	2±2
140	210	4±2	nd	2±2	3±2	9±2	5±2	nd
173	225	nd	nd	nd	nd	2±2	nd	nd
220	379	nd	nd	nd	nd	nd	nd	nd
Corr. coeff.: 0.9854; $k=1.06895 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 10.1±0.6 days								
4. Clay loam at Zevekote:								
0	0	95±7	475±33					
15	70	72±4	324±25	29±3	7±2	nd	nd	nd
40	120	53±3	212±17	48±4	5±2	nd	nd	nd
76	128	35±2	75±6	88±7	9±2	4±2	nd	nd
112	228	24±2	36±3	66±6	12±2	6±2	nd	nd
140	250	18±2	27±2	50±5	9±2	5±2	nd	nd
173	270	8±2	12±2	22±2	4±2	2±2	nd	nd
220	427	5±2	8±2	14±1	3±2	nd	nd	nd
Corr. coeff.: 0.9874; $k=0.2724 \cdot 10^{-3} (\mu\text{g kg}^{-1})^{-1} \text{ days}^{-1}$ ; isoxaflutol soil half-life: 39.5±2.0 days								

a. As in Table 1.

in each of the 4 replicate plots, as in the pH trial (Table 2). In the 0-2, 2-4, 4-6, 6-8, 8-10, 10-15 and 15-20 cm surface soil layers, soil samples were taken in each replicate field plots, but the samples from 2 replicate field plots were mixed, giving a total of 2 separate replicates -for each surface soil layer- which were analyzed separately. Sampling dates (day-month-year) and days after isoxaflutol treatment were, respectively : 8-11-1996, 0 ; 23-11-1996, 15 ; 18-12-1996, 40 ; 23-1-1997, 76 ; 28-2-1997, 112 ; 28-3-1997, 140 ; 30-4-1997, 173 ; 16-6-1997, 220.

For the isoxaflutol soil analysis, soil (100 g) was stirred in acetone/water (8/2 vol./vol., 200 ml, 20°C, 1 hr). The mixture was filtered, and the extraction was repeated during 30 min. The filtrates were gathered, water (100 ml) was added and the acetone removed in a vacuum rotary evaporator (30°C). NaCl (15 g) was added, the aqueous solution was extracted two times with ethyl acetate (2 x 200 ml), the ethyl acetate solution was dried (stirring during 1 hr at 20°C with  $\text{Na}_2\text{SO}_4$ ), concentrated in a one litre flask to 40 ml in a vacuum rotary evaporator at 30°C, further concentrated to 15 ml in a 50 ml flask in a vacuum rotary evaporator at room temperature, and then further concentrated to 0.5 ml under a slow stream of nitrogen (20°C). The concentrate was applied as a band to a first silica gel 60 F 254 20 x 20 cm, 0.2 mm thick, thin-layer chromatography (TLC) plate. The isoxaflutol standard was applied on another part of the TLC plate, next to the band of the sample solution. Elution with acetone/hexane (1/4 vol./vol.) gave the band containing isoxaflutol at  $R_f = 0.65$ . This band was scraped off, the silica gel extracted with ethyl acetate (40 ml) in a small glass column, the extract was concentrated successively to 15 ml in a vacuum rotary evaporator at room temperature, and to 0.5 ml under a slow stream of nitrogen (20°C), and was applied onto a second TLC plate. After a first elution with dichloromethane/hexane (2/1, vol./vol.), the TLC plate was taken off the bath, quickly air dried and placed again in the same TLC bath for a second elution. After this second elution, isoxaflutol was in the band at  $R_f = 0.40$ . This band was scraped off, the silica gel extracted with ethyl acetate (40 ml), the extract was concentrated successively to 15 ml in a vacuum rotary evaporator (20°C) and to 1 ml under a slow stream of nitrogen (20°C). The extract was analyzed for isoxaflutol by gas-liquid chromatography (GLC) and, for several samples, by combined GC-mass spectrometry (GC-MS).

GLC conditions were the following. Electron capture detection. Injection and detection at 280 and 255°C, respectively. Glass column 1.80 m x 2 mm i.d containing 5% SE 30 on Chromosorb W-HP 80-100 mesh. Nitrogen gas at 50 ml min<sup>-1</sup>. With column oven at 215°C, the isoxaflutol retention time was 2.8 min. Mass spectra were recorded at 70 eV in the electron impact mode. At the 20 and 5 µg isoxaflutol kg<sup>-1</sup> level in soil, recoveries respectively were 87-98 and 79-91%. The analytical limit of sensitivity was 2 µg isoxaflutol kg<sup>-1</sup> dry soil.

The linear regression  $1/y - 1/y_0 = kt$  was made between the reverse of the isoxaflutol soil concentration ( $y = \mu\text{g kg}^{-1}$  dry soil) and the time  $t$  (days) following isoxaflutol treatment ( $y_0$  is the isoxaflutol soil concentration at zero time). For the spring corn crop made in 1997 at Melle on sandy loam soils at different pH, and for the summer corn crop made at Melle in 1996, the periods for which the linear regression was applied respectively were 84 and 97 days after isoxaflutol treatment, and the correlated isoxaflutol soil concentrations were in the 0-8 cm surface soil layer in both trials. For the summer corn crops made in 4 different sites in 1997, the period for which the linear regression was applied was 99 days at Melle, Koksijde and Zarlardingne and 92 days at Zingem after the isoxaflutol treatment, and the correlated isoxaflutol concentrations were in the 0-10 cm superficial soil layer at the 4 sites. For the winter wheat crops made in 4 different sites in 1996-1997, they were 112 days and 0-10 cm, respectively. The isoxaflutol soil half-lives, and the times for 80% isoxaflutol soil dissipation with their 95% confidence intervals were obtained using the SAS logical CMS SAS 5.18 (1984, 1986, SAS Institute Inc., Cary, NC 27512).

For preparation of the isoxaflutol analysis standard, the formulation of isoxaflutol (20 g; water dispersible granules containing 75% isoxaflutol; Rhone-Poulenc Agrochimie, Belgium) was stirred in dichloromethane (300 ml) with heating to reflux (10 mm). The hot mixture was filtered, and the filter cake was extracted a second time in the same way. The filtrates were

gathered, and the dichloromethane was evaporated in a vacuum rotary evaporator. The solid was recrystallized in dichloromethane/hexane, giving isoxaflutol (13.8 g, 92%) of a purity greater than 99.9% as shown by TLC and GLC. Spectra of isoxaflutol:  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz): 1.20-1.33 (m, 2 H, cyclopropyl  $\text{CH}_2$ ); 1.33-1.48 (m, 2 H, cyclopropyl  $\text{CH}_2$ ); 2.59 (m, H, cyclopropyl CH); 3.34 (s, 3 H,  $\text{SO}_2\text{CH}_3$ ); 7.63 (d, 1 H, 6-H benzoyl); 8.04 (d, 1 H, 5-H benzoyl); 8.17 (s, 1 H, 3-H isoxazol); 8.44 (s, 1 H, 3-H benzoyl). IR (KBr disc,  $\text{cm}^{-1}$ ): 3099, 3028, 1671 (CO), 1572, 1493, 1393, 1319, 1248, 1159, 1121, 1082, 957, 939, 907, 862, 779, 762. MS (70 eV, electron impact;  $m/e$ , relative abundance, %): 359 ( $\text{M}^+$ , 7); 344 ( $\text{M-CH}_3$ , 9); 331 ( $\text{M-CO}$ ,  $\text{M-C}_3\text{H}_5$ , 4); 302 ( $344-(\text{CH}_2)_2\text{CH-H}$ , 5); 280 ( $\text{M-SO}_2\text{CH}_3$ , 8); 251 ( $\text{M-(C}_3\text{HNO)(C}_3\text{H}_5$ ), 100). MS (CI,  $\text{NH}_3$ ): 360 ( $\text{M}^+ + 1$ , 100); 377 ( $\text{M}^+ + 1 + \text{NH}_3$ , 30); 251 ( $\text{M-(C}_3\text{HNO)(C}_3\text{H}_5$ ), 8).

## RESULTS AND DISCUSSION

During the 3 to 4 months period following isoxaflutol application, in all the corn and winter wheat crops, there was a positive relationship between the reverse of the isoxaflutol soil concentrations and the time following isoxaflutol treatment (second order kinetics). To this unusual kinetic corresponds a fast decrease of the isoxaflutol soil concentrations during the first month following isoxaflutol application; thereafter the isoxaflutol soil dissipation becomes much slower, and concentrations corresponding to 15-25% of the dose remain during a long time (about 3 months) in soil. On account of the isoxaflutol high herbicide activity, these concentrations are sufficient for the protection against weeds.

In the spring corn crop with isoxaflutol treatment made pre-sowing in 1997 at Melle on plots at different soil pH, isoxaflutol soil half-lives were  $14.4 \pm 0.7$ ,  $15.0 \pm 0.8$  and  $12.1 \pm 0.7$  days at pH 5.5, 6.1 and 7.2, respectively. The rate of isoxaflutol soil dissipation was slightly lower at lower soil pH. The low basicity of the isoxazole ring should enable proton addition to isoxaflutol at low soil pH. This should increase isoxaflutol soil adsorption by cation exchange, and reduce the rate of isoxaflutol soil dissipation at lower soil pH (Bailey et al., 1968; Ladlie et al., 1976; Goetz et al., 1986). In the summer corn crop made in 1996 on sandy loam soil at Melle, the isoxaflutol soil half-life was  $12.2 \pm 0.6$  days. The time for 20% isoxaflutol remaining in soil was  $49 \pm 2$  days.

Soils of the 4 sites on which were made the summer corn crops in 1997 were different as to their textures, pH, organic matter content, and the recent organic fertilizer treatments. This was also the case with the winter wheat crops made in four different sites. The influences of these soil parameters -on the rates of isoxaflutol soil dissipation- thus combined. Interpretations of the results thus are tentatively made. The results of the pH trial (spring corn crop at Melle in 1997) indicate that the isoxaflutol soil dissipation is slightly faster at pH 7.2 than 5.5. In soils having organic matter contents lower than 2%, herbicide adsorption is related with low correlation coefficients to the soil structure: adsorption is lower in sandy soils than in loamy and clay soils (Savage, 1976; Sanchez-Martin and Sanchez-Camazano, 1991). Herbicide adsorption, persistence and mobility in soils containing 2% or more of organic matter are correlated by the soil organic matter content, and not by the soil texture: herbicide adsorption and persistence are greater -and mobilities lower- in soils containing more organic matter. On the other hand the recent soil organic matter due to the recent organic fertilizer treatments- is more able to slow down the pesticide soil dissipation, relative to the old soil organic matter (Rouchaud et al., 1994). Soil organic matter ageing (humification) leads to its aromatization (coalification) and loss of the chemical functions (carboxylic...) able to bond with the pesticide in soil. Yearly applications of recent organic fertilizers are more efficient to slow down the pesticide soil dissipation, than applications made once every 3 or 4 years. This effect of the recent organic fertilizers disappears faster with pig or cow slurries than with cow manure (Rouchaud et al., 1996).

In the summer crops made in 1997 in four sites different as to their soil types, the isoxaflutol soil half-lives were  $10.6 \pm 0.6$  days in sandy loam (Melle),  $14.1 \pm 0.7$  days in loamy sand (Zingem),  $12.4 \pm 0.6$  days in clay loam soil (Koksijde), and  $20.3 \pm 3$  in loam soil (Zarlardingne) (Table 1). The times for 20% isoxaflutol remaining in soil (80% dissipated) were  $42 \pm 2$  days at Melle,  $58 \pm 3$  days

at Zingem,  $47 \pm 2$  days at Koksijde, and  $86 \pm 4$  days at Zarlarding. There is no relationship between soil textures (the percentages of sand loam and clay) and pH, and the isoxaflutol soil persistences. According to the soil pH, the longest isoxaflutol soil persistence indeed was expected at Melle. According to the soil texture, the longest isoxaflutol soil persistence was expected at Koksijde. The soil organic matter concentrations were similar (1.7%) at Melle, Zingem and Zarlarding, and slightly higher at Koksijde (2.1%). According to the soil organic matter content, the longest isoxaflutol soil persistence was expected at Koksijde. The soil organic matter contents thus do not explain the isoxaflutol soil persistences. The greatest isoxaflutol soil half-life in the loam soil at Zarlarding should be related to the cow manure treatment made one month before the isoxaflutol application; moreover, this organic fertilizer treatment was repeated every year in the past; the loam texture and acid pH also combined to increase the isoxaflutol soil persistence. The low isoxaflutol soil persistence in the sandy loam soil at Melle should be related to the absence of organic fertilizer treatment during the 12 months period preceding the isoxaflutol application; this low isoxaflutol soil persistence occurred in spite of the low soil pH and the sandy loam texture.

In the summer corn crops made in 1997 in different sites, isoxaflutol generally did not reach the 10-15 and 15-20 cm surface soil layers. There was a surface soil layer containing the greatest isoxaflutol soil concentration. It contained 50 to 100 g% of the total isoxaflutol soil residue. This maximum isoxaflutol concentration soil layer after isoxaflutol application was the 0-2 cm surface soil layer; it then progressively moved down in the 2-4 and 4-6 cm surface soil layers in the crops at Melle, Zingem and Koksijde, the moving down being fastest at Melle (Table 1). At Zarlarding, the maximum isoxaflutol concentration soil layer did not move at a depth lower than the 2-4 cm surface soil layer. This should be explained by the reasons suggested to explain the isoxaflutol soil persistence; especially the cow manure application made one month before the herbicide treatment, and the repetition every year of this cow manure treatment. This maximum isoxaflutol concentration soil layer insures high local isoxaflutol concentrations and herbicide efficiencies. It is at the opposite of a potential uniform diffusion of isoxaflutol in all the soil surface layers; this should dilute the isoxaflutol soil residue and reduce its herbicide efficiency.

In the winter wheat crops made in 1996-1997 in four sites different as to their soil types, the isoxaflutol soil half-lives were  $20.7 \pm 1.0$  days in sandy loam (Melle),  $12.1 \pm 0.7$  days in loamy sand (Zingem),  $10.1 \pm 0.6$  days in loam (Sint Denijs), and  $39.5 \pm 2.0$  days in clay loam soil (Zevokote) (Table 2). The times for 20% isoxaflutol remaining in soil (80% isoxaflutol dissipated) were  $83 \pm 4$  days at Melle,  $46 \pm 2$  days at Zingem  $43 \pm 2$  days at Sint Denijs, and  $155 \pm 8$  days at Zevokote. The greater isoxaflutol soil persistence at Zevokote should correspond to the greater soil organic matter content, and to the pig slurry treatment made 2 months before the isoxaflutol application. The clay loam soil texture at Zevokote added its positive effect on the isoxaflutol soil persistence, and the high soil pH combined its negative effect. The low isoxaflutol soil persistence at Sint Denijs should be related to the low soil organic matter content and the absence of organic fertilizer treatment during several years in the past. The high soil pH added its negative influence onto the isoxaflutol soil persistence, whereas the loam structure combined its positive effect.

In the winter wheat crops made in different sites, isoxaflutol generally did not reach the 10-15 and 15-20 cm surface soil layers. There was a maximum isoxaflutol concentration soil layer containing 50 to 100 g% of the total isoxaflutol soil residue. At Sint Denijs, it moved down as far as the 6-8 cm surface soil layer (Table 2). At Zingem, as far as the 4-6 cm surface soil layer. At Melle and Zevokote, it did not move deeper than in the 2-4 cm surface soil layer; this should be explained by the same reasons as for the longer isoxaflutol soil persistence at Zevokote, especially the greater soil organic matter concentration and the pig slurry application made 2 months before isoxaflutol application. As in the summer corn crops, in the winter wheat crops isoxaflutol did not move at soil depths greater than 10 cm. In the winter, the maximum isoxaflutol concentration soil layer however could move at a depth (6-8 cm) lower than in the summer corn crop (4-6 cm). This occurred in spite of the lower rainfall during the winter crops than during the summer ones. Rainfalls thus do not explain the somewhat greater soil mobility of isoxaflutol in winter than in summer. This however should be related to the generally greater

water concentrations in soil during winter than summer. In summer, the continuous evaporation of water from soil is greater than in winter, the soil is dryer, and there is an upward movement of water in soils (Weerts, 1996). In winter, the water movement is mainly downward.

At the end of September 1996 and 1997, some days before harvest of each of the corn crops (the spring corn crop made in 1997 with soil pH trial, the summer corn crop made in 1996 at Melle, and the summer corn crops made in 1997 in 4 different sites), no isoxaflutol was detected in each of the analyzed surface soil layers (0-8 cm for both first crops; 0-10, 0-2, 2-4, 4-6, 6-8, 8-10, 10-15 and 15-20 cm in the summer corn crops made in 4 different sites). The same occurred at the middle of July 1997 before harvest of the winter wheat crops made in 1996-1997 in 4 different sites.

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## REFERENCES

- Bailey GW, White JL, Rothberg T (1968) Adsorption of organic herbicides by montmorillonite: role of pH and chemical character of adsorbate. *Soil Sci Soc Am Proc* 32: 222-234
- Goetz AJ, Wehtje G, Walker RH, Ben Hajek (1986) Soil solution and mobility characterization of imazaquin. *Weed Sci* 34: 788-793
- Luscombe BM, Pallett KE, Loubiere P, Millet JC, Melgarejo J, Vrabel TE (1995) RPA 201772: A novel herbicide for broad leaf and grass weed control in maize and sugar cane. *Brighton Crop Prot Conf -Weeds* 1: 35-42
- Ladlie JS, Meggitt WF, Penner D (1976) Effect of soil pH on microbial degradation, adsorption, and mobility of metribuzin. *Weed Sci* 24: 477-481
- Rouchaud J, Gustin F, Wauters A (1994) Soil biodegradation and leaf transfer of insecticide imidacloprid applied in seed dressing in sugar beet crops. *Bull Environ Contam Toxicol* 53: 344-350
- Rouchaud J, Thirion A, Wauters A, Van de Steene F, Benoit F, Ceustermans N, Gillet J, Marchand S, Vanparys L (1996) Effects of fertilizer on insecticides adsorption and biodegradation in crop soils. *Arch Environ Contam Toxicol* 31: 98-106
- Sanchez-Martin MJ, Sanchez-Camazano M (1991) Adsorption of chloridazon by soils and their components. *Weed Sci* 39: 417-422
- Savage KE (1976) Adsorption and mobility of metribuzin in soil. *Weed Sci* 24: 525-528
- Weerts J (1996) Director. Research and Development Department. Ministry of Middle Classes and Agriculture, Brussels, Belgium.