

**Leaching of  
fluridone, hexazinone and simazine  
in sandy soils**

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## SUMMARY

Fluridone or simazine was sprayed in field experiments on coarse sand and on humic sand in spring or in late autumn, and hexazinone on coarse sand in spring only. Soil samples for residue analysis by bio-assay were taken to a depth of 60 cm at various times over an eight-month period.

After a spring application, residues of fluridone and hexazinone occurred in the profile of coarse sand from 0 to 60 cm, while simazine leached to a depth of at most 15 cm. Both fluridone and simazine were found in the profile to a depth of 60 cm after an application in late autumn.

In a laboratory experiment with simazine the loss of phytotoxicity in coarse sand was approximately the same as in humic sand. Lowering temperature and/or lowering water content delayed the disappearance. From this it can be concluded, that at winter conditions loss of phytotoxicity is not fast enough to prevent leaching of simazine to deeper soil layers of a coarse sand completely after autumn application.

Keywords: leaching, herbicide, fluridone, hexazinone, simazine.

## SAMENVATTING

In veldproeven gelegen op een grofzandige grond en op een humeuze zandgrond werden fluridon en simazin in het voorjaar of laat in het najaar gespoten. Hexazinon werd alleen op een grofzandige grond gespoten in het voorjaar. Op enkele tijdstippen gedurende een periode van acht maanden werden tot op een diepte van 60 cm grondmonsters voor een residubepaling met behulp van biotoetsen gestoken.

Residuen van fluridon en hexazinon werden na een toepassing in het voorjaar op een grofzandige grond tot op een diepte van 60 cm aangetroffen, terwijl simazin tot maximaal 15 cm diepte inspoelde. Na een toepassing laat in het najaar werden zowel fluridon als simazin tot op een diepte van 60 cm gevonden.

In laboratoriumproeven was in een grofzandige grond en in een humeuze zandgrond het verlies aan fytotoxiciteit van simazin ongeveer even snel. Door een verlaging van de temperatuur en/of het vochtgehalte van de grond werd dit proces vertraagd. Hieruit kan worden afgeleid, dat na een toepassing in het najaar de verdwijning van simazin niet snel genoeg verloopt om een uitspoeling naar diepere lagen in de bodem van een grofzandige grond volledig te kunnen voorkomen.

## INTRODUCTION

Herbicides applied to soils can be adsorbed to some extent by organic matter and clay minerals, which prevents leaching. On sandy soils with a very low content of organic matter and a high porosity, it may be expected that adsorption is too low to prevent leaching and contamination of soil water.

For bromacil Rhodes et al. (1970) found a high mobility in soils with a relatively low content of organic matter, but mobility was low in a muck soil. Bromacil, annually applied at a dose of 2.4 kg a.i. ha<sup>-1</sup> in apple orchards on silty clay loam and sandy loam (contents of organic matter in both soils below 3%), could be detected down to a depth of 100 cm at the end of a seven-year period (Leistra et al., 1975). On a sand bed of a railway track with a lower content of organic matter bromacil penetrated to depths around 100 cm within two years (Zandvoort et al., 1980).

In columns of quartz sand aminotriazole moved readily with the leaching water, but mobility was delayed at increasing adsorptive capacity (Day et al., 1961). After a single spring application of aminotriazole (7.2 kg a.i. ha<sup>-1</sup>) Zandvoort et al. (1981) detected this herbicide only in the top 20 cm of humic sand (organic matter 2.3%) and of sand beds of railway tracks (organic matter below 1%) over a period of eight months. After an autumn application the herbicide occurred below 40 cm in the sand beds of railway tracks within three months.

Fluridone is adsorbed strongly by organic matter as well as by montmorillonite clay surfaces (Weber et al., 1986). The desorption after that is not complete. For a sand from Texas (content of organic matter 0.9%) Loh et al. (1979) found an adsorption coefficient of 2.6 dm<sup>3</sup> kg<sup>-1</sup>.

In laboratory experiments Rhodes (1980) and Bouchard and Lavy (1985) found a weak adsorption of hexazinone in silt loam soils. Because of the data obtained by soil thin-layer chromatography (Rf = 0.8) Rhodes (1980) placed hexazinone in Class 4 (mobile) of the mobility classification scheme from 1 to 5 of Helling and Turner (Helling, 1971). Bouchard and Lavy (1985) found a rather strong adsorption in leaf litter and after repeated washings still 10% of hexazinone was not released.

Adsorption of simazine correlated highly positively with organic matter content and clay content (Talbert and Fletcher, 1965). For a loamy sand they found an adsorption coefficient of 1.0 dm<sup>3</sup> kg<sup>-1</sup>. After repeated washings of a silty clay still 10% of the simazine was not released.

Leaching will be favoured by conditions which retard degradation. The degradation of simazine is influenced by temperature and soil moisture content (Walker, 1976). In laboratory experiments microbial breakdown was important (Banks et al., 1979). After 210 days there was 5% to 62% remaining in the different soils. In field experiments the half-life of hexazinone varied from less than 28 days to 180 days (Rhodes, 1980). In laboratory experiments the half-life increased from 77 days at 30 °C to 372 - 564 days at 10 °C (Bouchard et al., 1985).

Information about leaching of herbicides in sandy soils with a high porosity and a low content of organic matter under field conditions is limited. Therefore, downward movement of fluridone and hexazinone (solubility in water at 25 °C 12 mg and 33 g per dm<sup>-3</sup>, respectively) was studied. These herbicides were chosen because of their very different solubility in water and of their different spectrum of weed control. Simazine (solubility in water 3.5 mg dm<sup>-3</sup> at 20 °C) was taken as a reference. The distribution of the residues in a sandy soil with a low content of organic matter was compared with that in a humic sandy soil.

## MATERIALS AND METHODS

### Field experiments

Field experiments on coarse sand beds of a railway track were laid out near Maarn and near Elst. Another experiment was situated on an agricultural field on a humic sand (experimental farm "Droevendaal") near Wageningen. Soil properties in different layers - determined by the Laboratory of Soil and Crop Testing at Oosterbeek - are given in Table 1. At Maarn the layer of 10 - 20 cm was not sampled.

Table 1. Properties of soil layers in the experimental fields.

Soil property	Experimental field and sampled layer (cm)					
	Maarn			Elst		Wageningen
	(coarse sand)			(coarse sand)		(humic sand)
	0-10	20-40	40-60	0-10	10-20	0-10
Organic matter (%)	2.4	0.6	0.2	1.0	0.4	2.0
Mineral fraction						
clay (<2 $\mu\text{m}$ ) (%)	3	2	2	2	2	6
silt (2-50 $\mu\text{m}$ ) (%)	4	-*	8	3	1	10
sand (50-105 $\mu\text{m}$ ) (%)	5	-	8	2	2	17
sand (>105 $\mu\text{m}$ ) (%)	89	-	82	91	94	65
pH-KCl	5.6	6.5	5.4	6.6	7.7	5.1
Bulk density (kg dm <sup>-3</sup> )	1.44	1.53	-	1.70	-	1.31
Field capacity (% w/w)	11.0	-	-	5.5	-	15.5

\*- = not detected

The relatively high content of organic matter in the top 10 cm at Maarn was partly due to the few degraded remainders of temporary storage of trunks in 1974. At Elst there was a dense layer of heavy clay at a depth of about 60 cm.

The sites were divided into four blocks with randomized treatments. The plots at Maarn, Elst and Wageningen were 200 x 500 cm, 200 x 700 cm and 200 x 600 cm, respectively. They were kept free from vegetation. Commercial wettable powder formulations of fluridone, hexazinone and simazine were applied in a volume of water equivalent to 600 dm<sup>3</sup> ha<sup>-1</sup> with a propane knapsack sprayer in coarse droplets at a pressure of 140 kPa. At Maarn, Elst and Wageningen fluridone or simazine was sprayed in spring. At Elst hexazinone was sprayed in spring, and fluridone or simazine in autumn as well. Soil cores were collected with a brass tube drilled into the soil to a depth of 30 cm. The core was pressed out with a plunger and divided into layers for residue analysis by bio-assays. Samples from deeper layers were collected with an open-faced sampling tube. If possible 10 subsamples from each plot were taken and combined to one sample per layer. On the sandy soils at Maarn and Elst occasionally the soil was so dry that the auger could not hold the core. For the same reason the quantity of soil collected from deeper layers was small.

The samples were dried at about 35 °C, sieved through a 0.3 cm sieve, intensively mixed and stored at -20 °C until analysis.

## Laboratory experiments

The loss of phytotoxicity of simazine was examined in fresh untreated soil from the top 10 cm. The soil from the three experimental fields was sieved through a 0.3 cm sieve, and put into 6 Mitscherlich pots (each pot filled with 8 kg of soil on a dry weight basis). Simazine (32 mg a.i. per pot) was added and carefully mixed through the soil. In three pots moisture content was brought to 90% of field capacity. Then the pots were covered with foil caps and incubated at temperatures of 10 °C, 20 °C and 30 °C. The moisture content of the three remaining pots was brought to levels of 60%, 40% and 20% of field capacity. After covering with foil caps these pots were incubated at 20 °C. The moisture content of the pots was kept more or less stationary by periodic addition of water. Samples were taken from each pot from the day of treatment over a period of 140 days and dried, sieved and stored until analysis as mentioned before.

## Bio-assay

For the detection of simazine in the soil samples from Maarn and Wageningen a jar of 1 dm<sup>3</sup> was filled with 200 g of dry soil of the sample and 850 cm<sup>3</sup> of a nutrient solution. Fodder radish (*Raphanus sativus* L. spp. *oleiferus* (DC.) Metzg.) cv. Siletina was used as a test plant (Zandvoort and Braber, 1981). Duplicates of samples and standard series were placed on turn-tables in the glasshouse.

For the detection of fluridone, hexazinone and simazine in the soil from Elst plastic cups (150 cm<sup>3</sup>) with a wad of cotton wool in the perforated bottoms were filled with the soil samples. A parallel standard series was prepared by adding various quantities of the formulated herbicide to the untreated soil of the same fields. Eight pre-germinated seeds of oat (*Avena sativa* L.) cv. Condor were planted about 1.5 cm deep in each cup. For simazine duplicates of the samples and standard series were placed on turn-tables in trays containing a layer of plastic foam of about 1 cm thick which was kept wet. Only when the surface of the soil was dry - especially at the beginning of the assay - the cups were sprinkled additionally, but leaching of water had to be avoided. Phytotoxic symptoms of fluridone and hexazinone, due to a diffusion of these herbicides through the foam from one cup to the other, could be expected with the soil from Elst. To exclude this each cup was placed in a separate tray and watered overhead in the first period. When the oat plants had developed to a height of about 2 cm (in an earlier stage the seedlings of oat are too weak to survive in a very wet soil), pipes (diam. 7 cm) closed at one side, were filled with water and placed upside down in each tray for watering.

On cloudy days and in winter natural light was supplemented by high pressure Hg-lamps to a daylength of at least 14 hours.

After 3 or 4 weeks fresh and dry weight of whole plants (grown on nutrient solution) or of shoots (plants grown on cups) were determined for each pot. To estimate the concentration of the herbicide in the sample the fresh and the dry weights were interpolated in the standard curve.

## RESULTS

### Field experiments

In the experimental field at Maarn the residues of fluridone (Table 2) and simazine (Table 3) were found mainly in the top 10 cm of the soil profile. Only small quantities of these herbicides were present in the next 10 cm. The residue contents over the whole profile were converted to percentages of the applied dosage. These percentages decreased during the experimental period.

Table 2. Content of fluridone in different soil layers (mg kg<sup>-1</sup> dry soil) at Maarn and the total amount recovered at different times after application of 2.5 kg a.i. ha<sup>-1</sup> on 12 May 1980.

Layer (cm)	Sampling time (days after application)			
	38	64	141	231
0-5	0.37	0.45	0.10	0.09
5-10		0.02	n.d.(s)***	0.02
10-15	*-	0.02	n.d.(s)	n.d.
15-20	-	n.d.**	n.d.	-
Total residue in % of applied dose	22	15	6	3

\*- = not sampled

\*\*n.d. = not detectable

\*\*\*n.d.(s) = not detectable, but typical symptoms of fluridone visible in some test plants

Table 3. Content of simazine in different soil layers (mg kg<sup>-1</sup> dry soil) at Maarn and the total amount recovered at different times after application of 2.5 kg a.i. ha<sup>-1</sup> on 12 May 1980.

Layer (cm)	Sampling time (days after application)				
	0	4	7	38	64
0-5	1.7	1.7	1.7	0.80	0.60
5-10				0.15	0.55
10-15	*-	-	-	0.07	n.d.**
15-20	-	-	-	0.10	n.d.
Total residue in % of applied dose	100	100	100	40	40

\*- = not sampled

\*\*n.d. = not detectable

The contents of fluridone in the soil samples from the field at Elst are shown in Table 4a and 4b, those of hexazinone in Table 5 and those of simazine in Table 6a and 6b. Immediately after the application the total amount of these herbicides were recovered in the top 10 cm. Soon thereafter leaching out from the top layer started, but the highest concentrations at all sampling dates were still present in the top 10 cm. After an application in autumn fluridone and simazine were detected to a depth of 60 cm next summer, but after a spring application fluridone could hardly be found at that depth in autumn, while simazine was not detected below 15 cm.

Table 4a. Content of fluridone in different soil layers (mg kg<sup>-1</sup> dry soil) at Elst and the total amount recovered at different times after application of 2.0 kg a.i. ha<sup>-1</sup> on 25 November 1981.

Layer (cm)	Sampling time (days after application)						
	7	42	134	167	223	330	
0-5	1.0	{	0.46	0.51	0.34	0.27	0.25
5-10			0.38	0.16	0.15	0.20	0.18
10-15	-*		0.05	0.08	0.15	0.10	n.d.(s)
15-20	-		0.06	0.05	0.05	0.03	n.d.(s)
20-30	-		0.06	n.d.(s)***	0.05	0.03	0.03
30-40	-		0.08	0.02	0.05	0.04	0.03
40-50	-		-	0.03	0.05	0.08	0.04
50-60	-		-	n.d.**	0.05	0.02	0.02
Total residue in % of applied dose	85	52	39	42	40	29	

Table 4b. Content of fluridone in different soil layers (mg kg<sup>-1</sup> dry soil) at Elst and the total amount recovered at different times after application of 2.0 kg a.i. ha<sup>-1</sup> on 13 April 1982.

Layer (cm)	Sampling time (days after application)					
	6	13	27	83	190	
0-5	1.1	1.0	{	0.64	0.66	0.70
5-10				0.15	0.10	0.33
10-15	-	-		0.08	0.07	0.04
15-20	-	-		-	0.02	0.03
20-30	-	-		-	0.02	0.03
30-40	-	-		-	0.02	0.03
40-50	-	-		-	-	0.04
50-60	-	-		-	-	n.d.(s)
Total residue in % of applied dose	94	85	41	39	54	

\*- = not sampled

\*\*n.d. = not detectable

\*\*\*n.d.(s) = not detectable, but typical symptoms of fluridone visible in some test plants.

Table 5. Content of hexazinone in different soil layers (mg kg<sup>-1</sup> dry soil) at Elst and the total amount recovered at different times after application of 3.0 kg a.i. ha<sup>-1</sup> on 13 April 1982.

Layer (cm)	Sampling time (days after application)					
	6	27	55	111	190	
0-5	} 1.6	{ 2.4	0.39	0.16	0.12	
5-10			0.35	0.09	0.05	n.d.**
10-15	-*	0.17	0.06	n.d.	n.d.	
15-20	-	0.17	0.06	n.d.	n.d.	
20-30	-	-	0.04	0.02	n.d.	
30-40	-	-	0.03	0.02	n.d.	
40-50	-	-	-	0.03	n.d.	
50-60	-	-	-	0.03	n.d.	
Total residue in % of applied dose	91	87	19	11	3	

\*- = not sampled

\*\*n.d. = not detectable

Table 6a. Content of simazine in different soil layers (mg kg<sup>-1</sup> dry soil) at Elst and the total amount recovered at different times after application of 2.0 kg a.i. ha<sup>-1</sup> on 25 November 1981.

Layer (cm)	Sampling time (days after application) content in dry soil					
	7	42	84	167	195	223
0-5	} 1.1	{ 0.90	0.85	0.12	0.08	n.d.**
5-10			0.37	0.42	0.11	0.08
10-15	-*	-	n.d.	0.07	n.d.	n.d.
15-20	-	-	n.d.	0.24	0.15	0.07
20-30	-	-	-	0.07	n.d.	0.10
30-40	-	-	-	n.d.	0.23	n.d.
40-50	-	-	-	-	-	n.d.
50-60	-	-	-	-	-	0.25
Total residue in % of applied dose	94	54	54	29	33	33



Table 6b. Content of simazine in different soil layers (mg kg<sup>-1</sup> dry soil) at Elst and the total amount recovered at different times after application of 4.0 kg a.i. ha<sup>-1</sup> on 13 April 1982.

Layer (cm)	Sampling time (days after application)					
	6	13	27	55	83	190
0-5	2.0	1.8	0.9	0.84	0.45	0.08
5-10			0.13	0.11	0.25	0.05
10-15	-	-	0.07	n.d.	n.d.	0.05
15-20	-	-	-	n.d.	n.d.	n.d.
Total residue in % of applied dose	85	77	23	20	15	4

\*- = not sampled

\*\*n.d. = not detectable

Similar to the experimental field at Maarn also in the field at Elst the highest content of the herbicides was found in the top 10 cm of the soil profile. During the period of sampling the total residue of the herbicides decreased. The disappearance of fluridone and simazine was faster in spring than in winter.

On the humic sand at Wageningen the residues of fluridone and simazine were mainly found in the top 2 cm of the soil profile (Table 7 and 8). Downwards to a depth of 10 cm the concentration of these herbicides decreased below the detection limit.

Table 7. Content of fluridone in different soil layers (mg kg<sup>-1</sup> dry soil) at Wageningen and the total amount recovered at different times after application of 2.0 kg a.i. ha<sup>-1</sup> on 4 May 1981.

Layer (cm)	Sampling time (days after application)			
	3	14	28	120
0-1	0.88	0.45	1.3	1.3
1-2			0.40	0.75
2-3			0.15	0.10
3-4			0.07	0.08
4-5			n.d.*	0.04
5-10			n.d.	n.d.(s)**
Total residue in % of applied dose	59	29	13	15

\*n.d. = not detectable

\*\*n.d. = not detectable, but typical symptoms of fluridone visible in some test plants

Table 8. Content of simazine in different soil layers (mg kg<sup>-1</sup> dry soil) at Wageningen and the total amount recovered at different times after application of 2.0 kg a.i. ha<sup>-1</sup> on 4 May 1981.

Layer (cm)	Sampling time (days after application)					
	3	14	28	56	120	156
0- 1 } 1- 2 } 2- 3 } 3- 4 } 4- 5 } 5- 7.5 } 7.5-10 }	1.5	1.3	{ 9.1 { 0.86 { 0.18 { n.d. ** { n.d. { -* { -	5.0 2.6 1.3 0.75 0.22 0.08 n.d.	2.0 0.87 0.24 0.14 0.09 n.d. n.d.	1.6 0.95 0.36 0.20 0.14 n.d. n.d.
Total residue in % of applied dose	98	85	65	66	22	21

\*- = not sampled

\*\*n.d. = not detectable

Duplicate results in a soil sample from the same plot were fairly close, but there could be a rather high variation between the replicate plots. The detection limit of fluridone and hexazinone was 0.02 mg kg<sup>-1</sup>, that for simazine 0.07 mg kg<sup>-1</sup>. In a number of samples the quantity of fluridone was below the detection limit, but visually the typical symptoms of this herbicide - retardation of growth and chlorosis in combination with a transverse constriction in the middle of the leaves - were present in the test plants.

#### Loss of phytotoxicity

Figure 1 and 2 summarize the results of laboratory experiments on loss of phytotoxicity of simazine in different sandy soils. Assuming the loss of phytotoxicity of simazine in the different soils to follow first-order kinetics the lines of best fit can be calculated by a linear regression analysis with the formula:  $\ln C_t = \ln C_0 - kt$  (1). In this formula  $C_0$  is the concentration at time zero,  $C_t$  the concentration remaining at time  $t$  of incubation and  $k$  the rate constant. With formula (1) the lines for the different temperatures and for the different contents of water are drawn. The calculated correlation coefficients varied from - 0.91 to - 0.99. In all experiments loss of phytotoxicity was accelerated by increasing the temperature from 10 °C to 30 °C (Fig.1). In the experiment with the soil from Maarn (10 - 20 cm) the rate of loss of phytotoxicity of simazine was delayed with a decrease of the water content of the soil (Fig.2). This effect was not clear for the other soilsamples. In these experiments soil type did not influence the loss of phytotoxicity of simazine.

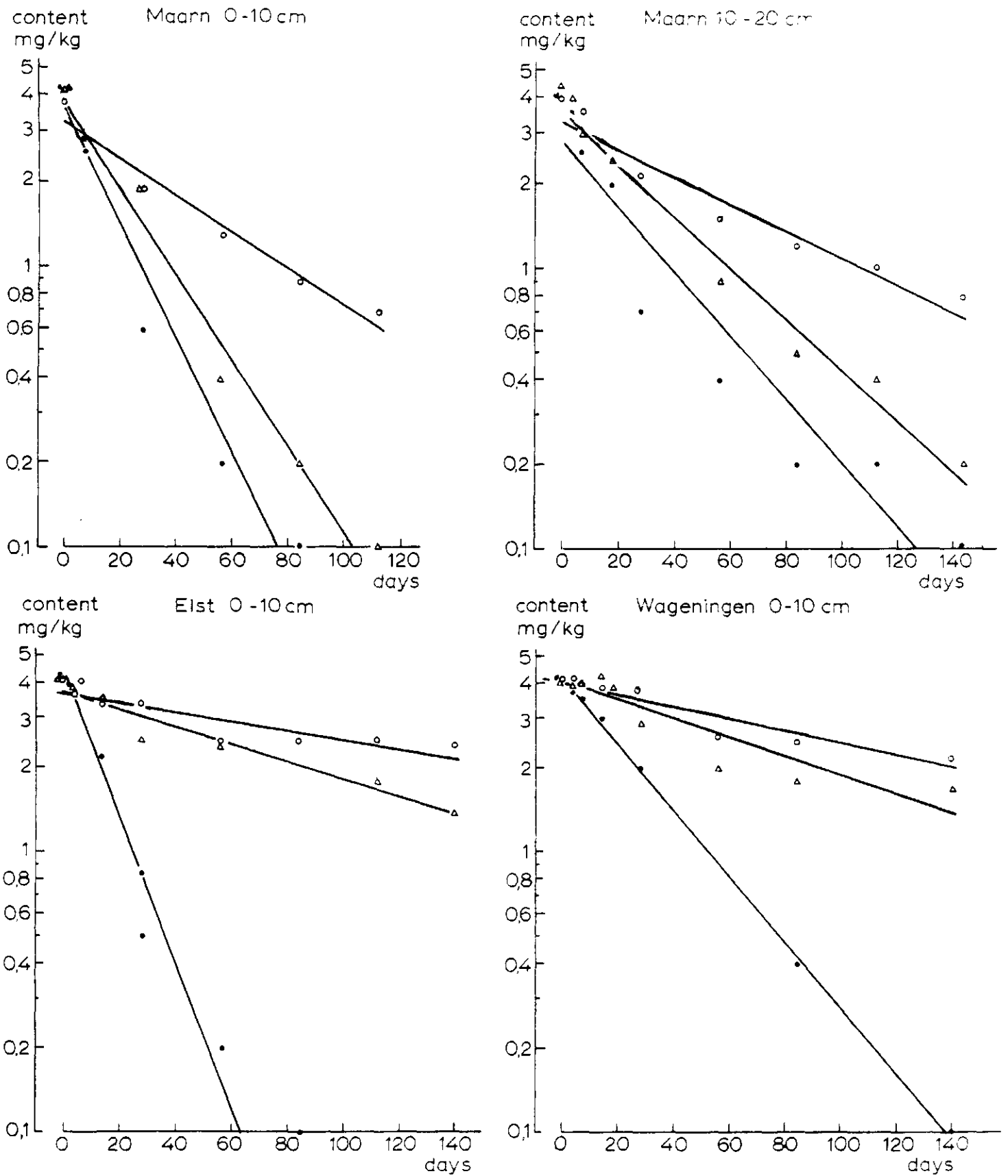


Fig.1 Loss of phytotoxicity of simazine in four soil samples (Maarn 0 - 10 cm and 10 - 20 cm, Elst 0 -10 cm and Wageningen 0 - 10 cm) with a moisture content of 90% of field capacity at three temperatures: 10 °C (o), 20 °C (Δ) and 30 °C (·). Regression lines calculated with the formula:  $\ln C_t = \ln C_0 - kt$ .

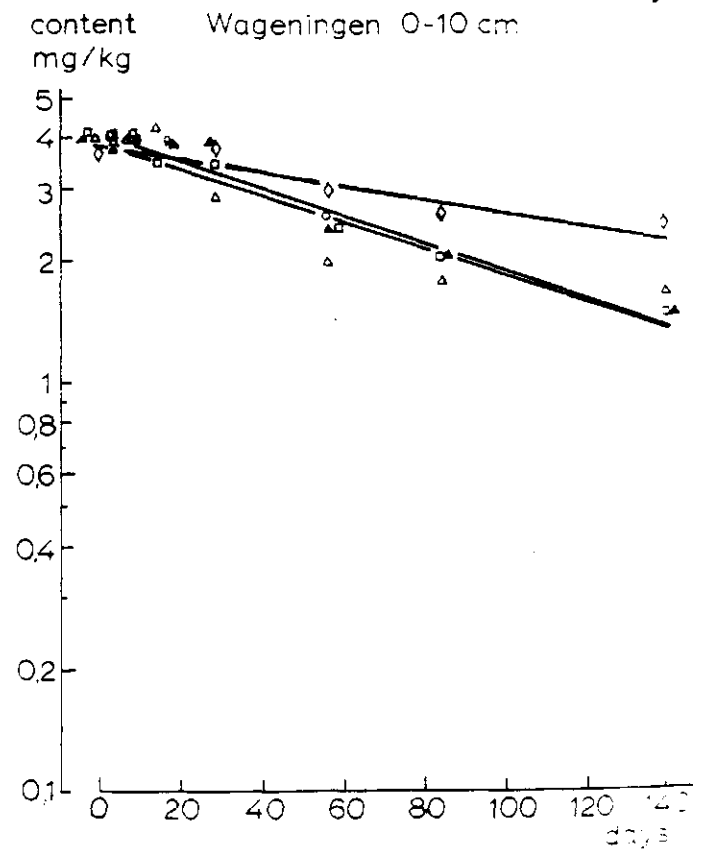
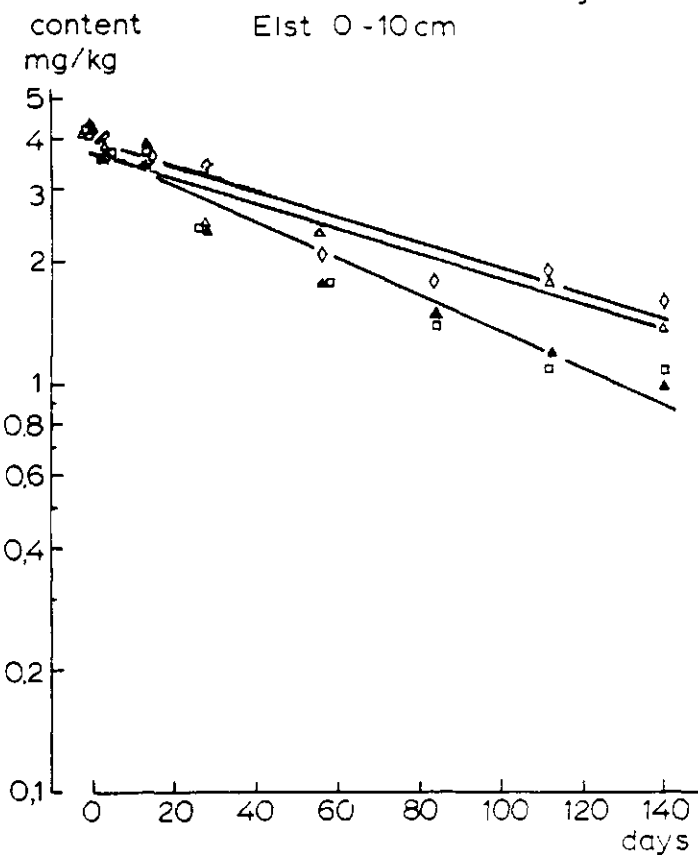
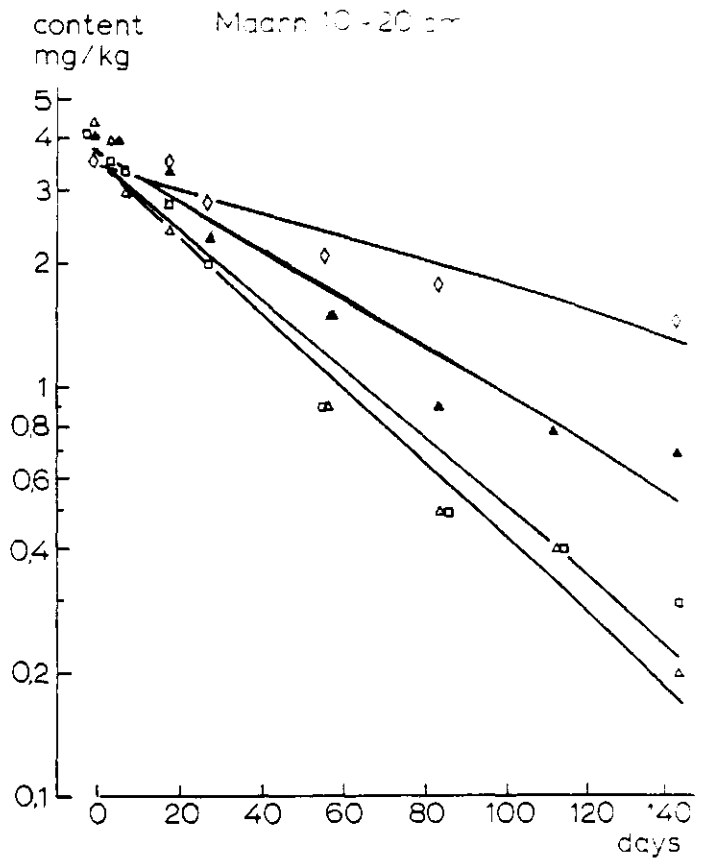
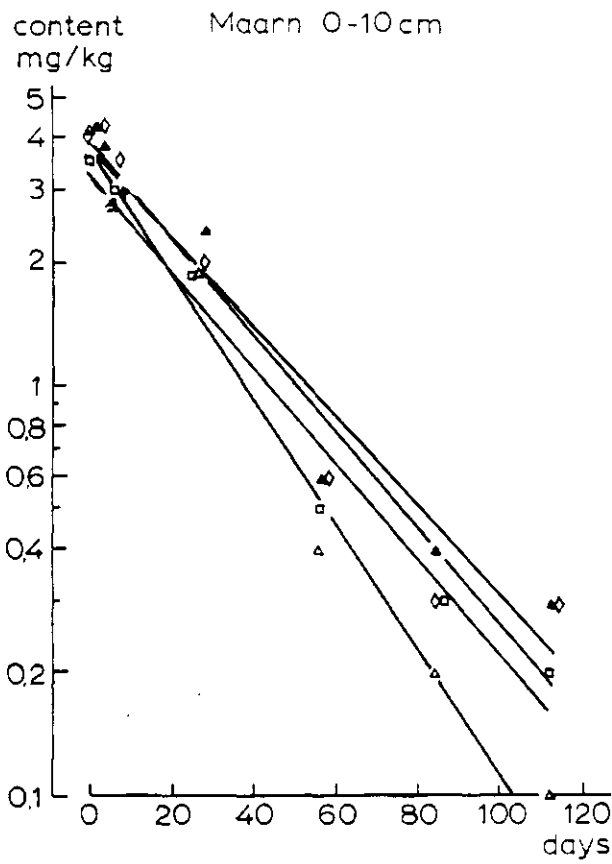


Fig.2 Loss of phytotoxicity of simazine in four soil samples (Maarn 0 - 10 cm and 10 - 20 cm, Elst 0 - 10 cm and Wageningen 0 - 10 cm) at 20 °C and a moisture content of 90% (Δ), 60% (◻), 40% (▲) and 20% (◇) of the field capacity. Regression lines calculated with the formula:  

$$\ln C_t = \ln C_o - kt.$$

## DISCUSSION

In agreement with the experiments of Weber et al. (1986) and Talbert and Fletchall (1965) the highest concentrations of fluridone (Table 2, 4a and 4b) and simazine (Table 3, 6a and 6b) were found in the top 10 cm of the soil profile at Maarn and Elst and in the upper 2 cm at Wageningen (Table 7 and 8). In these layers of the profile the content of organic matter was the highest. The experiments of Bouchard and Lavy (1985) suggested an adsorption of hexazinone by organic matter. Obviously the organic matter content in the top layer was sufficient to retard leaching from the upper 5 cm at Elst.

At Elst leaching was more important after autumn than after spring application of fluridone (Table 4a and 4b) and in particular of simazine (Table 6a and 6b). The low temperature in winter delayed degradation of fluridone and simazine (Banks et al., 1979; Walker, 1976) and with that the possibilities of leaching increased. The rainfall in the first period after spraying in autumn was higher than after spring application. Besides, in contrast with spring, evaporation of a bare soil was low in winter and consequently the precipitation excess was relatively high.

In the first month after the spring application at Maarn and Elst, the total rainfall was nearly the same (36 mm and 38 mm, respectively). In spite of this, due to the higher content of organic matter in the top layer, at Maarn leaching of both herbicides was less than at Elst.

In field experiments the half-life of hexazinone varied from less than 28 days to 180 days (Rhodes, 1980). In laboratory experiments the half-life increased from 77 days at 30 °C to 372 - 564 days at 10 °C (Bouchard et al., 1985). From this and from the recoveries in the experiment at Elst (Table 5) it can be concluded that degradation can be so slow, that contamination of deeper layers of a coarse sand with hexazinone is possible even after a spring application.

The data presented here and in former publications for bromacil (Zandvoort et al., 1980) and for aminotriazole (Zandvoort et al., 1981) show that contamination of deeper soil layers may occur on sandy soils with a low content of organic matter. To prevent this, it is recommended to apply only herbicides with a short or moderate persistence on sandy soils with a low content of organic matter, in spring.

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