

Mobility and Degradation of the Herbicide Imazosulfuron in Lysimeters under Field Conditions

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The mobility and degradation of imazosulfuron, labeled with carbon-14 at the imidazole ([imi-¹⁴C]-imazo) or pyrimidine ring ([pyr-¹⁴C]imazo), in lysimeters with 1 m² surface and 110 cm depth were investigated for three years. One lysimeter was treated with [imi-¹⁴C]imazo in two successive years at the rate of 50 g of active ingredient (ai)/ha each. The other two lysimeters were treated once with [pyr-¹⁴C]imazo and a mixture (1:1, w/w) of the two labeled imazosulfurons, respectively (50 g of ai/ha). In the first and second years of monitoring, the yearly mean concentration of ¹⁴C in the leachate water was <0.10 μg/L in each lysimeter. Although in the third year the concentration of ¹⁴C in the leachate water was 0.17 μg/L for the lysimeter treated twice with [imi-¹⁴C]imazo, the concentration of imazosulfuron and its degradation products in the leachate water ranged from 0.01 to 0.06 μg/L. At the study termination, the main portion of ¹⁴C recovered was found in the upper 30 cm soil layer in each lysimeter, and no ¹⁴C was detected below a depth of 50 cm. These findings indicated that imazosulfuron and its degradation products in soils translocated into groundwater only slightly.

KEYWORDS: Imazosulfuron; mobility; degradation; soil

INTRODUCTION

Imazosulfuron, 2-chloro-*N*-[[[4,6-dimethoxy-2-pyrimidinyl)-amino]carbonyl]imidazo[1,2-*a*]pyridine-3-sulfonamide, is a sulfonylurea herbicide with an imidazo[1,2-*a*]pyridine moiety (1). It exhibits potent herbicidal activities against most relevant weeds, such as *Capsella bursa pastoris* and *Matricaria* varieties, at an application rate of 25 g of active ingredient (ai)/ha with accompanying good safety to wheat and barley. The mode of action of imazosulfuron is an inhibition of acetolactate synthase, a key enzyme in the biosynthetic pathways of branched-chain amino acids such as isoleucine and valine (2, 3), and the crop selectivity is considered to reside in rapid metabolic inactivation by the tolerant plant (4), similar to other sulfonylurea herbicides (5–7).

Freundlich *K* values obtained from the batch adsorption technique and half-lives of pesticides in soils are frequently used to predict the behavior of pesticides in the environment (8, 9). In the prediction of the mobility of imazosulfuron in soils, imazosulfuron could be considered to indicate significant

movement in soils judging from relatively small Freundlich *K* values of 0.96–5.27 (10) and a half-life of 40 days in soils under laboratory conditions (11). On the other hand, soil desorption study has indicated that soil desorption constants of imazosulfuron changed as a function of time in soils (10) as reported for some pesticides (12–14). The soil desorption constants of imazosulfuron increased with time in soils by a factor of 2.4–36.4 during 60 days of aging. From these findings, it was assumed that imazosulfuron would become more strongly retained and that the mobility of imazosulfuron would decrease with time in soils. The present study was conducted to investigate the leaching behavior of imazosulfuron and its degradation products in soils under field conditions.

MATERIALS AND METHODS

Chemicals. [¹⁴C]Imazosulfuron labeled in the 3-position of the imidazole ring ([imi-¹⁴C]imazo) and labeled in the 5-position of the pyrimidine ring ([pyr-¹⁴C]imazo) (Figure 1) was synthesized by Nemoto & Co., Ltd.. The specific radioactivities were 5.30 MBq/mg for [imi-¹⁴C]imazo and 5.27 MBq/mg for [pyr-¹⁴C]imazo, and radiochemical purities were 99.0 and 98.1%, respectively, by HPLC analysis. The following authentic compounds were prepared in our laboratories: imazosulfuron, 2-chloro-*N*-[[[1,4-dihydro-6-methoxy-4-oxo-2-pyrimidinyl)amino]carbonyl]imidazo[1,2-*a*]pyridine-3-sulfonamide (HMS), 2-chloroimidazo[1,2-*a*]pyridine-3-sulfonic acid (IPSA), and 2-chloroimidazo[1,2-*a*]pyridine-3-sulfonamide (IPSN). 4,6-Dimethoxy-2-pyri-

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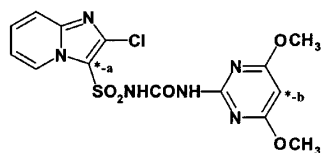


Figure 1. Chemical structure of imazosulfuron. Asterisks indicate positions of ^{14}C label: a, [imi- ^{14}C]imazo; b, [pyr- ^{14}C]imazo.

Table 1. Authentic Standards and Their R_f Values by TLC

compd	R_f value in solvent system ^a		
	A	B	C
imazosulfuron	0.71	0.89	0.43
ADPM	0.83	0.90	0.69
HMS	0.85	0.54	0.27
IPSA	0.54	0.40	0.28
IPSN	0.76	0.85	0.55

^a Solvent systems: A, butanol/acetic acid/water (6:1:1, v/v/v); B, ethyl acetate/methanol/acetic acid (8:1:1, v/v/v); C, hexane/ethyl acetate/methanol (2:2:1, v/v/v).

Table 2. Properties of Soil Monolith Used for Lysimeter

depth (cm)	particle size (%)			pH (CaCl ₂)	organic carbon (%)
	sand	silt	clay		
0–30	76	20	4	5.60	1.32
30–85	85	14	1	5.85	0.20
85–110	85	14	1	5.80	0.05

midinamine (ADPM) was purchased from Tateyama Kasei Co., Ltd. R_f values of authentic standards by TLC are shown in Table 1. All other chemicals were of reagent grade unless otherwise noted in this paper.

Lysimeter Installation. Undisturbed soil monoliths of 1 m² surface and 110 cm depth were collected on December 8, 1993, from an agricultural field in Münster-Handorf in Germany. The soil monoliths were transported to the lysimeter station of Covance Laboratories GmbH (Münster, Germany) and inserted in watertight outer containers. The soil characteristics of the lysimeters are shown in Table 2.

Application of Test Substance. Imazosulfuron was applied to lysimeters after formulation as an aqueous suspension. Specific radioactivities of [imi- ^{14}C]imazo and [pyr- ^{14}C]imazo were adjusted to 3.7 MBq/mg using nonlabeled imazosulfuron for the preparation of application suspension, and three lysimeters were treated with [imi- ^{14}C]imazo (lysimeter-Im), [pyr- ^{14}C]imazo (lysimeter-Pm), and a mixture (1:1, w/w) of [imi- ^{14}C]imazo and [pyr- ^{14}C]imazo (lysimeter-Im/Pm), respectively, at a rate of 18.5 MBq/m² corresponding to 50 g of ai/ha, which was equal to the highest application rate. The early postemergence applications to winter wheat were performed on November 21, 1994, for the first vegetation period. On November 15, 1995, only the lysimeter-Im was again treated with [imi- ^{14}C]imazo early postemergence to winter barley for the second vegetation period. No chemical was applied to oilseed rape for the third vegetation period. Fertilization and crop protection were conducted in accordance with agricultural practice during the 3-year study period.

Collection and Analysis of Leachate Water. The leachate water was collected in the lysimeter outer container. The amount of leachate water in the outer container was checked at weekly intervals at least, and leachate sample was collected if the amount of leachate water exceeded 1 L throughout the study. ^{14}C in the leachate sample was determined by LSC immediately after collection, and aliquots of the leachate sample were stored at -20 ± 2 °C. If the annual mean concentration of ^{14}C in the leachate water was >0.10 $\mu\text{g/L}$, further analysis of leachate samples was conducted as follows. An individual

leachate water sample or a combined sample considering their individual volumes was concentrated under reduced pressure to 1 mL. The supernatant and precipitate were separated by centrifugation, and the precipitate was extracted twice with CH_3CN (3 mL) by manual shaking for 3 min. The CH_3CN fraction was combined with the aqueous supernatant, and CH_3CN was removed from the solution under a gentle stream of nitrogen. An aliquot of the solution was subjected to TLC analysis to quantify imazosulfuron and its degradation products. The procedural recoveries of ^{14}C for leachate samples ranged from 94.2 to 98.3%.

Soil and Crop Analysis. At the termination of the 3-year study period, the upper three 10-cm layers of the soil monolith were removed using stainless steel shovels, and each layer was homogenized using a hand mixer. For the depth of 30–110 cm of the soil monolith, eight soil cores were taken by rotary driller equipped with poly(vinyl chloride) liners with 5-cm inner diameter. The liners containing the soil core were cut into 10-cm sections, and the soil sections of the respective depths were combined and mixed. The amount of ^{14}C in each soil layer was determined by combustion of aliquots of the mixed soils. Two moist mixed soil samples (~ 100 g each) from each of the upper four soil layers were extracted with $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ (1:1, v/v; 150 mL) in a centrifuge tube for 10 min and centrifuged for 10 min to separate supernatant and soil. The soil was subsequently extracted with CH_3CN ($\times 2$) and $(\text{CH}_3)_2\text{CO}$ by the shaking method described above. One selected sample from each lysimeter was extracted with an additional 100 mL of $(\text{CH}_3)_2\text{CO}$, and the extract was assayed separately to confirm that no significant amounts of ^{14}C were obtained by further extractions. The combined supernatants were concentrated under reduced pressures. A portion of the concentrate was subjected to TLC analysis to quantify imazosulfuron and its degradation products.

Harvested crops were air-dried in the dark. Dried crops were ground and subjected to combustion and LSC to quantify ^{14}C .

Radioassay. Carbon 14 radioactivity (^{14}C) in leachate water was quantified using a liquid scintillation counter (LSC; model 1409, Wallac). The limit of detection (LOD) of ^{14}C was 0.01 $\mu\text{g/L}$ as imazosulfuron equivalent for leachate water. ^{14}C in soils and crops was quantified by a combustion method using a sample oxidizer (model 307, Packard), and the LODs of ^{14}C for soils and crops were 0.14 and 0.14–0.34 $\mu\text{g/kg}$ of dry mass as equivalent, respectively.

Thin-Layer Chromatography (TLC). A Merck silica gel 60 F₂₅₄ chromatoplate (20 \times 20 cm) and the following solvent systems were used: A, butanol/acetic acid/water (6:1:1, v/v/v); B, ethyl acetate/methanol/acetic acid (8:1:1, v/v/v); C, hexane/ethyl acetate/methanol (2:2:1, v/v/v). The areas of ^{14}C on the TLC plate were detected and quantified using a Berthold LB 284 Multi-tracemaster 40 linear analyzer (Berthold). The non-radiolabeled standards on the TLC plate were visualized by irradiation with UV light at 254 nm.

Climatic Conditions and Irrigation. The climatic conditions at the lysimeter site correspond to northern European conditions. Irrigation was conducted to maintain good growing condition for crops and to reach the minimum of 800 mm of yearly precipitation. Comparison of the actual precipitation with long-term data of the Münster area on a monthly basis was used to decide if additional irrigation was necessary to achieve at least 800 mm per year.

RESULTS AND DISCUSSION

^{14}C in Leachate Water. The sum of precipitation and additional irrigation and the volumes of leachate water during the three years are shown in Figure 2. In all monitoring years, the required amount of water (at least 800 mm per year) was added to all lysimeters. The total amounts of leachate water for the first year were 397, 434, and 410 L in lysimeter-Im, lysimeter-Pm, and lysimeter-Im/Pm, respectively, and the mean amount for the three lysimeters was 414 L. The mean amounts of leachate water for the second and third years were 359 and 223 L, respectively.

The concentration of ^{14}C in leachate water was determined weekly. As shown in Figure 3, 11 weeks after the first

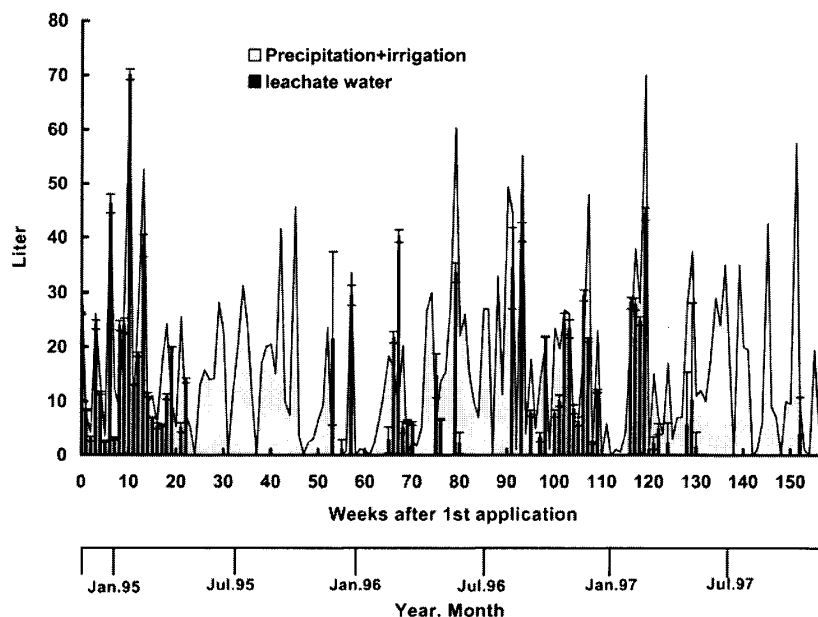


Figure 2. Amounts of precipitation, including irrigation, and leachate water during the study period. Data of leachate water are expressed as the mean of three lysimeters \pm SD.

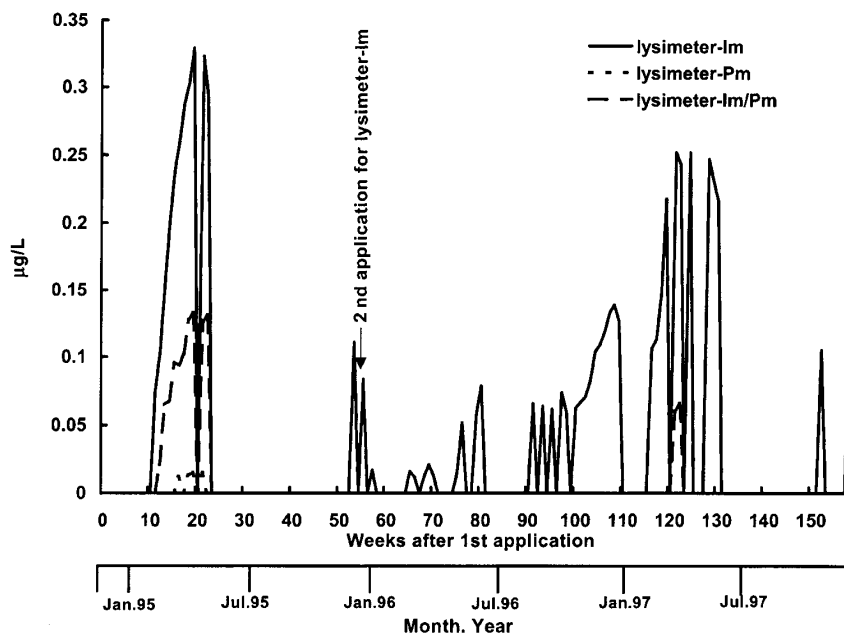


Figure 3. Time course changes of the radioactivity concentration in leachate water during the study period.

application (in February 1995), ^{14}C was detected in the leachate water for the first time in lysimeter-Im. Following a maximum in April 1995, the concentration of ^{14}C had significantly decreased, and no ^{14}C (less than the LOD) was detected from 23 weeks until 52 weeks. In lysimeter-Pm, a small amount of ^{14}C was detected in only March and April 1995. In lysimeter-

Im/Pm, ^{14}C was detected for the first time 12 weeks after application. After reaching a maximum in April 1995, the concentration decreased to less than the LOD, and no ^{14}C was detected from 23 weeks after application until the end of the first year. The mean concentrations of the first year were 0.08, 0.03, and <0.01 $\mu\text{g/L}$ for lysimeter-Im, -Pm and -Im/Pm,

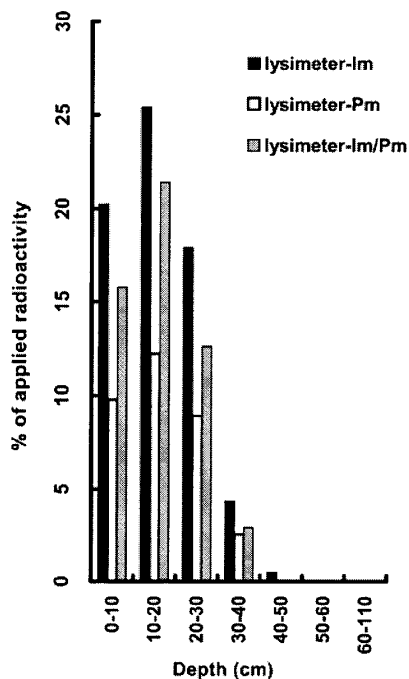


Figure 4. Percentages of applied ¹⁴C in the soil layers of the individual lysimeters at termination of the study.

Table 3. Yearly Mean Concentrations of Radioactivity in Leachate Water

year	$\mu\text{g/L}$		
	lysimeter-Im	lysimeter-Pm	lysimeter-Im/Pm
1	0.08	<0.01	0.03
2	0.05	<0.01	<0.01
3	0.17	<0.01	<0.01

respectively (**Table 3**). Determination of parent and degradation products in leachate water for each lysimeter was not performed due to the mean ¹⁴C concentration of <0.10 $\mu\text{g/L}$.

During the second year, leachate water occurred irregularly due to the distribution of the precipitation throughout the year and the impact of the water consumption of the crops. ¹⁴C was detected in only lysimeter-Im treated twice with [imi-¹⁴C]imazo, and the mean concentration was 0.05 $\mu\text{g/L}$ for the second year.

In the majority of leachate water samples of lysimeter-Im collected during the third year, the ¹⁴C concentration was >0.10 $\mu\text{g/L}$, and the mean concentration of the third year was

0.17 $\mu\text{g/L}$ in lysimeter-Im. In lysimeter-Pm and lysimeter-Im/Pm, the yearly mean concentrations of ¹⁴C in the leachate water were <0.01 $\mu\text{g/L}$, respectively. TLC analysis indicated that imazosulfuron and five ¹⁴C degradation products were present in the leachate water of lysimeter-Im. Imazosulfuron, HMS, and IPSA amounted to 0.06, 0.03, and 0.01 $\mu\text{g/L}$, respectively. Three unidentified degradation products ranged from 0.01 to 0.03 $\mu\text{g/L}$ as imazosulfuron equivalent.

¹⁴C in Soil. The lysimeters were disassembled after termination of three years of monitoring following the first application. The total amounts of ¹⁴C in the individual soil layers are presented in **Figure 4** as percent of the applied ¹⁴C. For lysimeter-Im, this percentage refers to the total amount of ¹⁴C applied with the two treatments performed. The total amounts of ¹⁴C present in the soil were 68.4, 33.5, and 52.7% of the applied ¹⁴C in lysimeter-Im, -Pm, and -Im/Pm, respectively. In all lysimeters, the majority of ¹⁴C was located in the upper two 10-cm layers, which were tilled after harvest of the crops, resulting in a relatively equal distribution of the ¹⁴C in these two layers. In the 40–50-cm layer of lysimeter-Im, 0.5% of the applied ¹⁴C was detected. No ¹⁴C was detected below the 40-cm layer of lysimeter-Pm and -Im/Pm.

TLC analysis of soil extracts was performed for the upper four layers of lysimeter-Im and -Im/Pm and for the upper two layers of lysimeter-Pm. Concentrations of imazosulfuron and its degradation products in soil layers at study termination are shown in **Table 4**. In lysimeter-Im treated with imazosulfuron in two successive years, the concentration of imazosulfuron was found to be 0.86–0.96 $\mu\text{g/kg}$ in the upper two 10-cm layers and decreased with depth. For lysimeter-Pm and lysimeter-Im/Pm, imazosulfuron amounted to 0.11–0.29 $\mu\text{g/kg}$ in the upper two 10-cm layers. As major degradation product, IPSN was determined in lysimeter-Im and in lysimeter-Im/Pm at concentrations of 1.29–1.77 and 0.25–0.35 $\mu\text{g/kg}$, respectively, in the upper three 10-cm layers. In the upper two layers of lysimeter-Pm, small amounts of ADPM were observed.

On the basis of the results of identified degradation products in leachate water and soils, the degradation pathways of imazosulfuron in soils were proposed as **Figure 5**.

Recovery of ¹⁴C Applied. Total amounts of ¹⁴C detected in leachate water, soil, and crops at the termination of the study period of three years were 70.2, 33.6, and 53.3% of applied ¹⁴C in lysimeter-Im, lysimeter-Pm, and lysimeter-Im/Pm, respectively (**Table 5**).

Although imazosulfuron could be considered to have significant mobility in soils on the basis of the results of laboratory adsorption and degradation studies, the concentrations of imazosulfuron in the leachate water never exceeded 0.10 $\mu\text{g/L}$ as a mean of a monitoring year in this study even after the applications of imazosulfuron labeled in the imidazopyridine ring ([imi-¹⁴C]imazo) in two subsequent years. Similar overestimations of mobility based on the results of

Table 4. Amounts of Imazosulfuron and Degradation Products in Lysimeters at Study Termination

soil layer (cm)	$\mu\text{g/kg}$ of dry mass											
	lysimeter-Im				lysimeter-Pm				lysimeter-Im/Pm			
	imazo ^a	IPSN	others ^b	nonextr ^c	imazo	ADPM	others	nonextr	imazo	IPSN	others	nonextr
0–10	0.96	1.29	1.50	14.47	0.11	0.05	0.13	4.12	0.29	0.35	0.50	5.61
10–20	0.86	1.77	1.91	14.87	0.17	0.06	0.14	4.75	0.29	0.42	0.40	6.95
20–30	0.56	1.49	0.25	7.80	– ^d	–	–	–	0.15	0.25	0.54	3.46
30–40	0.16	0.25	0.31	2.42	–	–	–	–	0.05	0.03	0.14	0.57

^a Imazosulfuron. ^b Total amount of unknown degradation products. ^c Nonextractable ¹⁴C. ^d Not analyzed.

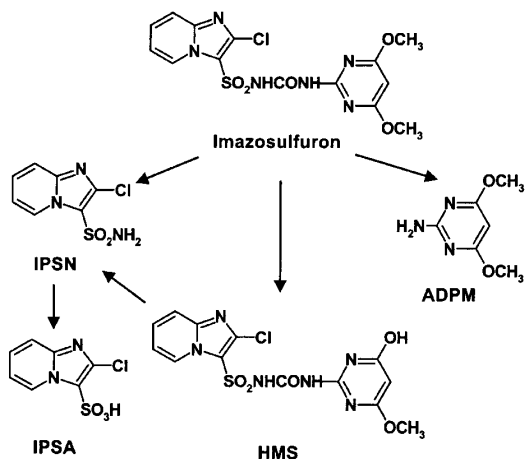


Figure 5. Proposed degradation pathways of imazosulfuron in soils.

Table 5. Distribution of Radioactivity Applied at the Termination of Study

fraction	% of applied ^{14}C		
	lysimeter-Im ^a	lysimeter-Pm	lysimeter-Im/Pm
leachate	1.3	<0.1	0.3
soil	68.4	33.5	52.7
crops ^b	0.4	0.1	0.2
total	70.1	33.6	53.2

^a Referring to the two applications performed in the first and second years.

^b Total amount of radioactivity detected in the crops harvested during the 3-year study period.

laboratory studies have been also reported for other sulfonylurea herbicides such as rimsulfuron (15) and sulfometuron-methyl (16). It has been reported for imazosulfuron that the soil desorption constants increased with increasing aging period in soils (10). Because soil desorption is one of the most important factors determining the mobility of pesticides, it was assumed that the increase of soil desorption constants in soil contributed to the low mobility of imazosulfuron in lysimeters observed in the present study. From these findings, it can be reasonably concluded that imazosulfuron will not reach environmentally significant concentrations in groundwater in practical field use.

Carry-over to succeeding crops has been reported for sulfonylurea herbicides (17). The difference between the amount of imazosulfuron in lysimeter-Im (~1 $\mu\text{g}/\text{kg}$) and those in lysimeter-Pm and -Im/Pm (0.1–0.3 $\mu\text{g}/\text{kg}$) at the study termination may be viewed as a rough estimate of imazosulfuron residue 2 years after a single application (0.7–0.9 $\mu\text{g}/\text{kg}$). This level of residue raises a potential carry-over concern to succeeding crops, and studies to address the concern are underway.

At the study termination, the majority of ^{14}C present in the soil was nonextractable residues. Imazosulfuron, IPSN, and unknown degradation products amounted to <2.0 $\mu\text{g}/\text{kg}$ in the upper 20-cm layer of the soil and decreased with depth. No ^{14}C was detected below a depth of 50 cm. The concentration of degradation products in the leachate water did not exceed 0.10 $\mu\text{g}/\text{L}$ as the mean of the three individual years of monitoring. It has been reported that the mobility of ^{14}C in aged soil columns treated with [imi- ^{14}C]imazo was less than that in nonaged soil columns under laboratory conditions, and Freundlich K values of ADPM and IPSN were larger than that of imazosulfuron (11).

On the basis of these findings, it was assumed that degradation products of imazosulfuron have little mobility in soils under field conditions.

The overall mass balances of the three treated lysimeters including soil, crops, and leachate water were different and amounted to 70.2% (lysimeter-Im), 33.6% (lysimeter-Pm), and 53.3% (lysimeter-Im/Pm) of the applied ^{14}C , respectively. These results implied that significant portions of the radiocarbon from the individual labeling positions had disappeared from the soil/plant/leachate system, particularly from the pyrimidine labeling position. It has been observed that a much higher amount of $^{14}\text{CO}_2$ had emerged from the soil treated with [pyr- ^{14}C]imazo compared to the soil treated with [imi- ^{14}C]imazo in a laboratory study (11). ADPM with a pyrimidine ring was not detected in the soil of lysimeter-Im/Pm, although IPSN with an imidazole ring was found to be from 0.03 to 0.42 $\mu\text{g}/\text{kg}$ in the upper 40-cm layer of the soil in the present study. These findings suggested that the labeling position of [pyr- ^{14}C]imazo degraded more rapidly to $^{14}\text{CO}_2$, which was emitted from the lysimeters to the atmosphere.

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