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Pesticide Mobility in Soils: Assessment of the Movement of Isoxaben by Soil Thin–Layer Chromatography

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During the past 20 years, researchers concerned by the dynamics of pesticides in soil have developed experimental techniques to assess the soil mobility and the potential hazards of groundwater contamination. The two basic methods, soil columns and soil thin-layer chromatography, have a fundamental analogy in conventional chromatography ; they allow a direct leaching measurement.

Soil column chromatography is, by and large, more widely used than soil TLC. Nonetheless, soil TLC developed by Helling and Turner (1968) is a technique which allows a quick assessment and comparison of pesticide mobility in soil. This technique has, however, remained neglected because its actual interest only appears with the use of labelled molecules, which makes visualization of pesticide movement much easier. Various technical improvements were previously performed by Jamet and Thoisy (1984) especially for a better Rf-value determination. Then, a comparative study of the mobility of isoxaben and four other pesticides (aldicarb, atrazine, chloridazon and metamitron) on seven agricultural soils was achieved.

Isoxaben, known as EL-107 is a benzamide herbicide (N-[3-(1-ethyl -1-methylpropyl)-5-isoxazolyl]-2, 6-dimethoxylbenzamide)) developed by ELI-LILLY and especially used in Europe. Isoxaben, a pre-emergence herbicide, is applied for controlling broadleaved weeds in winter cereal crops. Results from field trials were first described (1982) and summarized (1984) by Huggenberger et al. Since little was known about isoxaben mobility in soil the objective of this study was to determine its movement using soil TLC and to compare it with to various widely used molecules.

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MATERIALS AND METHODS

Seven samples of agricului soils were collected from various French departments. Each sample was air-dried, passed through a 2 mm-meshed sieve and characterized by texture, organic matter content, cation exchange capacity and pH (Table 1). Then, soils were passed through a 100 µm-meshed screen ; a soil-water slurry was prepared with this fine fraction (approximately 40 g of soil for 100 ml of distilled water) ; glass plates (20 x 20 cm) were then coated at a thickness of 250 µm. After preparation the plates were allowed to dry at room temperature.

Approximately 1650 Bq (100 000 dpm) of each ^{14}C-labelled molecule (in an organic solvent solution) were applied to the soil thin layers as a spot with a microsyringe at 2 cm from the bottom edge. The solvent was then allowed to evaporate. A horizontal line was scribed 17 cm above the base to stop water movement during developement. Then a strip of soil, 1-cm wide, was removed from the two side edges ; two spacers, in plastic material, 0.3 cm thick, were placed on these edges to support a 20 x 20 cm glass plate to tail off water evaporation during development.

Two paper-clips were used to keep the glass plates in position. A third spacer was placed at the base of the soil thin-layer to hold a sheet of chromatography paper (Whatman 1 ; medium flow rate) used to control the water flow ; during developement with distilled water the whole device was hold horizontal. After development the soil plate was air-dried.

Visualization of movement was obtained by a linear analyzer (BERTHOLD LB 2832) with a multichannel analyzer (SILENA SYSTEM BS 27/N). The distances moved on the soil thin-layer relative to water, i.e. Rf. value were determined by visual inspection of the radiochromatograms. The digital spectra (1018 values each) were transmitted to a computer (DPS 8/70 MULTICS) via a microcomputer (KONTRON PSI-80). The method of smoothing data proposed by Proctor and Sherwood (1980) was used ; then the smoothed radiochromatograms were drawn on a BENSON plotter with GPGS-F graphic software.

RESULTS AND DISCUSSION

According to Helling (1971a) the three methods suitable for detecting movement of radioactive pesticides on soil TLC plates are (1) extraction and analysis of successive zones (2) autoradiography and (3) radiochromatogram scanning.

While the first method was too much time-consuming, autoradiography appeared especially satisfactory ; however, as several days or weeks were usually necessary before measuring Rf-values, this delay can be reduced to a few hours with a radiochromatogram scanner.

Table 1. Soil properties

Soil Type	Origin (*)	% Clay (<2 µm)	% organic matter	CEC (meq/100 g)	pH	Texture
Castel-Maurou	(31)	13.1	1.27	5.9	6.1	silt loam
Landerneau	(29)	19.6	4.90	12.9	6.0	loam
Loches	(37)	7.5	0.06	3.6	7.6	sandy loam
Maisnil-Dohem	(62)	14.7	1.87	8.6	7.4	silt loam
Plomodiern	(29)	16.0	6.03	12.5	6.3	silt loam
Sillery	(51)	21.5	2.37	6.2	8.0	sandy clay loam
Versailles	(78)	20.5	1.94	10.0	6.4	silt loam

(* : French department code)

Table 2. Mobility of 5 pesticides on 7 soils, using soil TLC plates

Soil Type	Rf				
	aldicarb	atrazine	chloridazon	isoxaben	metamitron
Castel-Maurou	0.96	0.52	0.10	0.00	0.51
Landerneau	0.68	0.04	0.10	0.00	0.19
Loches	0.64	0.05	0.07	0.00	0.39
Maisnil-Dohem	0.90	0.49	0.42	0.00	0.63
Plomodiern	0.71	0.09	0.15	0.00	0.27
Sillery	0.67	0.12	0.07	0.00	0.39
Versailles	0.87	0.35	0.32	0.00	0.48
Rf mean	0.78	0.24	0.18	0.00	0.41
(range)	(0.64-0.96)	(0.04-0.52)	(0.07-0.42)	(0.00-0.00)	(0,19-0.63)
mobility class (*)	4	2	2	1	3
(range)	(3-5)	(1-3)	(1-3)	(1-1)	(2-4)

* mobility is classified according to the system of Helling and Turner

	class	**Rf**
1	immobile	0.00 - 0.09
2	slightly mobile	0.10 - 0.34
3	moderately mobile	0.35 - 0.64
4	mobile	0.65 - 0.89
5	very mobile	0.90 - 1.00

With the linear analyzer described by Filthuth (1982) radiochromatograms were obtained after a data acquisition of 2000 s. By spotting 1650 Bq of each ^{14}C-pesticide the detection efficiency ranged from 2 to 4 % ; such a low yield was due to the absorption of the beta-particules of carbon-14 into the soil thin-layer.

Figures 1 to 5 represent soil TLC smoothed radiochromatograms of each pesticide studied ; movements on the plates are clearly distinct. The Rf values measured on the smoothed radiochromatograms are listed in Table 2. They ranged from 0.00 for isoxaben in all soils to 0.96 for aldicarb in Castel-Maurou soil. It appears from these results that aldicarb was very mobile in the agricultural soils studied as indicated previously by Jamet and Thoisy (1984).

Isoxaben, for which all Rf-values were 0.00, was classified as an immobile herbicide according to the mobility scale (Table 2) published by Helling and Turner (1968).

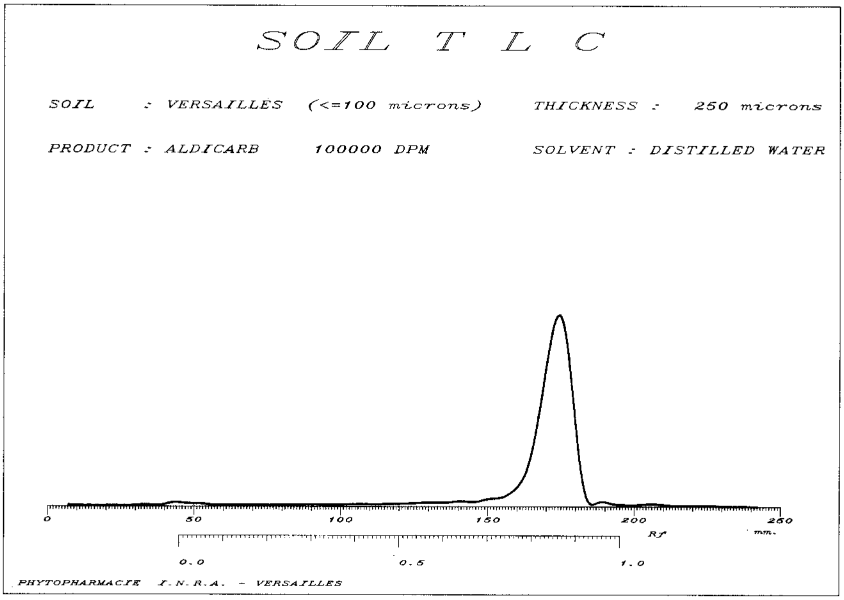

Figure 1 : Typical smoothed radiochromatogram of aldicarb in a thin layer of Versailles soil.

Among the three other herbicides, metamitron was the more mobile while atrazine and chloridazon showed approximtely the same mobility in all soils (Table 2). In our experiments atrazine appeared less mobile than in those of Helling (1971a,b). This author indicated (1971c) high simple correlation coefficients between atrazine Rf-values and soil organic matter content (-0.656 significant at 5 % level) as well as water flow (0.749 significant at 1 % level). Comparison of Rf-values (Table 2) with

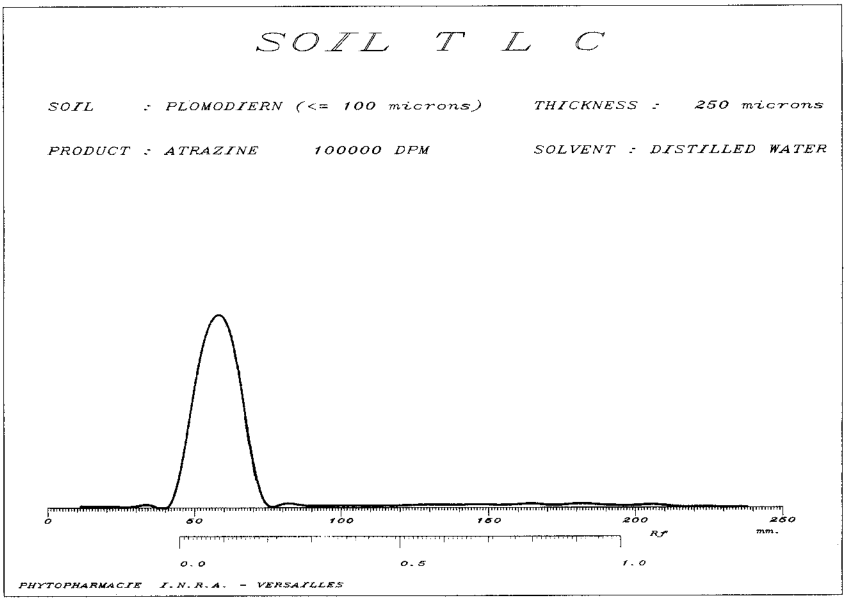

Figure 2 : Typical smoothed radiochromatogram of atrazine in a thin layer of Plomodiern soil.

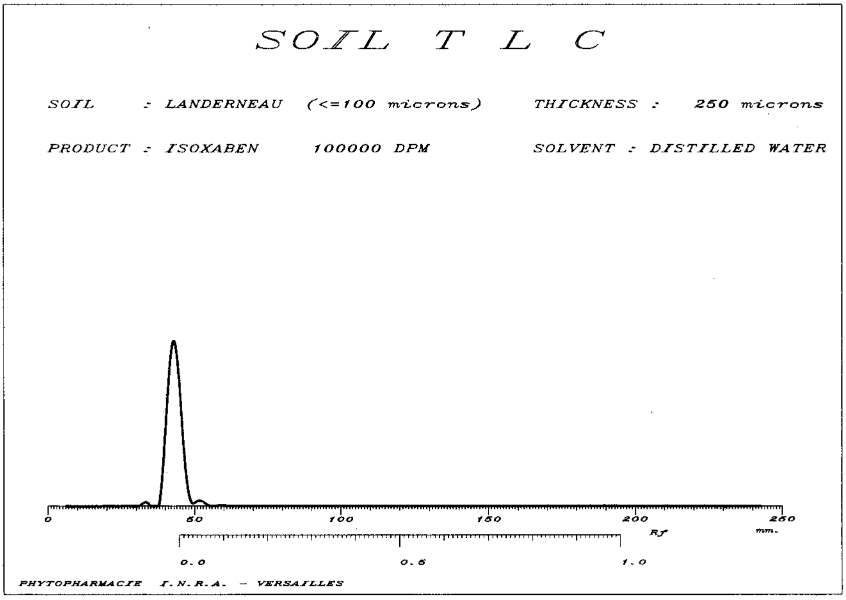

Figure 3 : Typical smoothed radiochromatogram of isoxaben in a thin layer of Landerneau soil.

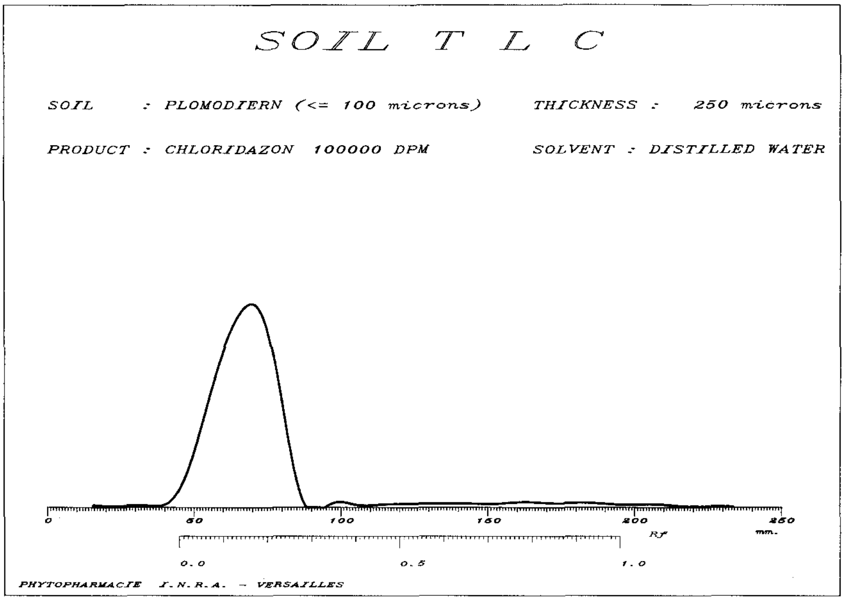

Figure 4 : Typical smoothed radiochromatogram of chloridazon in a thin layer of Plomodiern soil.

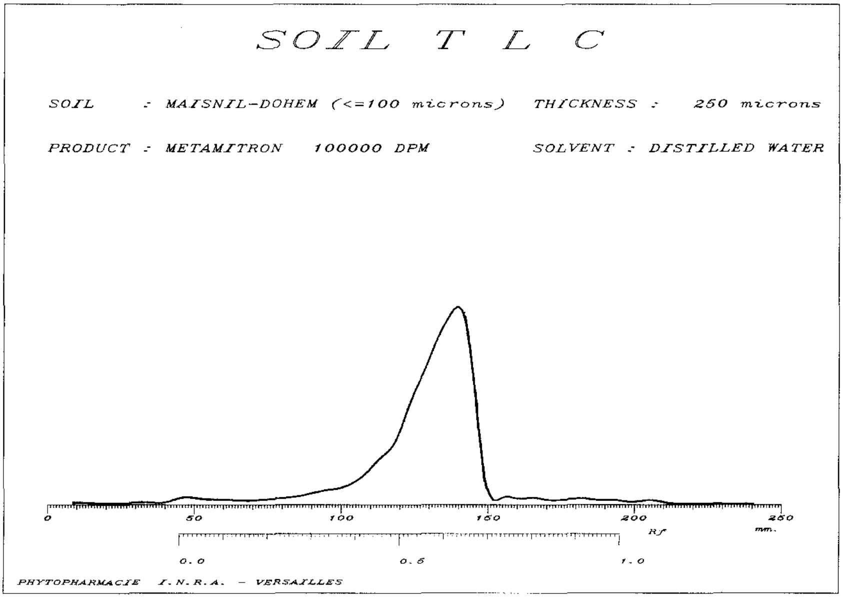

Figure 5 : Typical smoothed radiochromatogram of metamitron in a thin layer of Maisnil-Dohem soil.

soil properties (Table 1) indicates that the soil organic matter content was one of the major factor in controlling the movement of pesticide in soil : the lowest Rf-values were measured in soils with the highest organic matter contents, i.e. Plomodiern and Landerneau.

However, in reducing agricultural soils to a particule size range of 1 to 100 µm a considerable part of the soil was lost ; soil structure was then markedly altered and probably a part of the organic matter was removed with the rejected fractions. Nevertheless, soil TLC remains a quick inexpensive method to assess and compare pesticide mobility in soils ; the application of the linear analyzer and the use of computer for processing the measured chromatograms stimulates new interest in the study of pesticide mobility by soils thin layer chromatography.

On the basis of our results and of the mobility scale as suggested by Helling and Turner (1968) the following classification of the pesticides studied, expressed in a decreasing order of mobility was obtained whatever the soil :

aldicarb > metamitron > atrazine-chloridazon > isoxaben.

According to Huggenberger (personal communication 1984) isoxaben is slightly mobile in soil ; it leaches less than atrazine and chlortoluron, and remains at the top of the soil profile available in the zone where weeds germinate. Our experiments confirmed these first results and indicated that isoxaben would not be a potential environmental hazard due to leaching toward groundwater.

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