Influence of Soil Properties on the Adsorption–Desorption of Sulcotrione and Its Hydrolysis Metabolites on Various Soils

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The retention process of sulcotrione and its two hydrolysis products, 1,3-cyclohexanedione (CHD) and 2-chloro-4-methylsulfonylbenzoic acid (CMBA), were studied in four different soils (Belgium, Landes, Perpignan, and Martinique) under laboratory conditions. Adsorption isotherms were well fitted by a Freundlich relationship. The values of K_{fa} ranged from 0.4 to 27.0, and the most adsorbed compound was CHD regardless the soil type. Sulcotrione and CMBA exhibited similar retention behaviors. According to adsorption and desorption results, sulcotrione presents a moderate affinity with soil components; however, its leaching capacity needs to be carefully assessed. Clay content seemed to be the most important factor influencing the adsorption capacity of each compound, whereas organic matter and pH had little influence. The soils were classified according to their adsorption capacity in the following decreasing order: Martinique, Belgium, Landes, Perpignan.

INTRODUCTION

A recent triketone herbicide, sulcotrione, 2-(2-chloro-4-methylsulfonylbenzoyl)-1,3-cyclohexanedione (weak acid, pK_a = 2.9), was discovered (*1*) by Zeneca Ag Products (now Syngenta) and marketed in Europe by Bayer CropScience SA under the tradename of Mikado (liquid concentrate, 300 g·L⁻¹ of active ingredient). In maize and sugar cane fields, sulcotrione is applied pre- and post-emergence at a relatively low rate (300–450 g·ha⁻¹) for the control of a broad range of annual and perennial broadleaf weeds. Accumulation of uncolored pigments and decrease of plastoquinone and α-tocopherol rate lead to external bleaching symptoms, occurring especially on leaves. Internal activity of sulcotrione affects the photosynthetic apparatus with *p*-HPPD enzyme as molecular target (*2–4*). The degradation pathway of sulcotrione (**1**) leads to 2-chloro-4-methylsulfonylbenzoic acid (CMBA, **3**) in plants and hydroxysulcotrione in mammals. In soil, the most well-known degradation pathway is hydrolysis, which gives 1,3-cyclohexanedione (CHD, **2**) and CMBA (**3**) (**Figure 1**).

Degradation and retention processes are among the most important factors influencing the fate of an agrochemical in a soil environment. Several authors reported that, in some soils, sulcotrione is slightly adsorbed and relatively easily desorbed (*5–11*). Our purpose was to undertake the retention study of sulcotrione and its two hydrolysis metabolites for which, to our
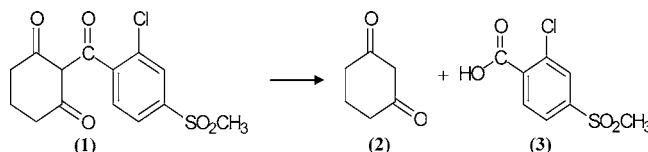

Figure 1. Hydrolysis pathway of sulcotrione with sulcotrione (**1**), CHD (**2**), and CMBA (**3**).

Table 1. Physicochemical Characteristics of the Three Molecules

compound	molar mass (g·mol⁻¹)	water solubility (mg·L⁻¹)	pK_a
sulcotrione ($C_{14}H_{13}ClO_5S$)	328	165	2.9 ± 0.2
CHD ($C_6H_8O_2$)	112	soluble	5.3 ± 0.2
CMBA ($C_{14}H_{15}ClO_6S$)	346	soluble	4.6 ± 0.2

knowledge, there is no available information. Some physicochemical properties of the soil (clay and organic matter contents, pH), acting in the retention process, were also examined.

MATERIALS AND METHODS

Chemicals. Standards of sulcotrione (purity = 99.2%), CHD (97%), and CMBA (95%) were purchased, respectively, from Riedel-deHaën Gmbh, Fluka Chemica, and Acros Organics. The physicochemical properties of these molecules are given in **Table 1**.

Sulcotrione was also extracted from Mikado (liquid formulation, 300 g·L⁻¹ of sulcotrione) according to the method published by Rouchaud et al. (*8*). Twenty grams of Mikado (density = 1.12) was lyophilized during 10 h, and the product was dissolved in acetone. The resulting solution was filtered, and the filtrate was evaporated under reduced

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Table 2. Classification of Selected Soils According to Taxonomy and Physicochemical Properties[a]

	Landes	Perpignan	Belgium	Martinique
FAO classification	sandy	clayey sandy loam	sandy loam	sandy clayey loam
% clay	6.3	13.9	23.0	29.7
% silt	22.7	60.5	41.4	32.9
% sand	71.0	25.6	35.6	37.4
pH	6.2	8.1	6.6	6.5
% organic carbon	2.1	0.9	2.0	1.9
CEC (mequiv 100 g^{-1})	4.0	15.5	9.4	17.6
Ca^{2+}/CEC (%)	64.0	214.0		55.0
% SMC	0.9	3.4	1.60	5.1
previous treatment with sulcotrione	none	none	each year, for 10 years, until May 2003	none
culture	corn crop	lucerne	corn crop	sugar beet

[a] CEC, cation exchange capacity; % SMC, soil moisture content.

pressure conditions. The residue was recrystallized twice in chloroform to afford 3.88 g (11.8 mmol) of sulcotrione. To verify its purity, it was analyzed by nuclear magnetic resonance (NMR), mass spectrometry (MS), and high-performance liquid chromatography (HPLC): ^{1}H NMR δ 2.07 (2H, q), 2.43 (2H, t), 2.79 (2H, t), 3.07 (3H, s), 3.61 (1H, s), 7.36 (1H, d), 7.86 (1H, dd), 8.01 (1H, d). Organic solvents (methyl alcohol and acetonitrile for HPLC) were purchased from Carlo Erba, trifluoroacetic acid (TFA, 99%) and calcium chloride (CaCl$_2$, 2 H$_2$O, 98%) were from Sigma Aldrich, water was of Milli-Q quality.

Stock solutions of each component (sulcotrione, CHD, and CMBA) were prepared at 1000 mg·L^{-1} in methanol. Working solutions (0.25, 0.5, 1, 2.5, 10, 20, and 30 mg·L^{-1}) were prepared in distilled water or in 0.01 M CaCl$_2$ aqueous solution. Sulcotrione solutions were denoted, respectively, as Sw$_1$–Sw$_7$ (water solution) and Sc$_1$–Sc$_7$ (CaCl$_2$ solution). For CHD and CMBA, dilutions were prepared only in water and denoted Dw$_1$–Dw$_7$ and Cw$_1$–Cw$_7$.

Soils. The soils were selected according to their physicochemical properties (**Table 2**). They were sieved to <2 mm and air-dried at ambient temperature. Pedological analyses were carried out by the Laboratoire d'Analyses Agricoles, Perpignan, France, except for the Belgian soil, which had been previously analyzed by the Laboratory of Herbology, Gent University, Belgium. Soil moisture content in samples was determined in our laboratory according to the method published by Mathieu and Pieltain (*12*).

Batch Sorption Study. The adsorption–desorption experiment required a kinetic study to evaluate the agitation time needed to reach equilibrium. For each compound, we used the ratio of soil (1 g) to solution (5 mL) (Sw$_7$ and Sc$_7$ for sulcotrione, Dw$_7$ for CHD, and Cw$_7$ for CMBA). Seven samples, using centrifugation glass tubes, were prepared in duplicate and shaken mechanically for 0.5, 1, 2, 3, 4, 8, and 24 h on a vertical shaker. The obtained soil suspension was centrifuged at 3500g for 10 min. An aliquot of the supernatant was analyzed by HPLC. Calculations were performed using the indirect method proposed by OECD guideline (*13*) based on the difference between initial and equilibrium concentrations of the compound in the supernatant.

The adsorption kinetics were obtained by plotting the solid-phase concentration of sulcotrione C_s (mg·kg^{-1}) against agitation time (t) expressed in minutes. Although it is minor in comparison to K_{fa}, the calculation of K_d [the distribution coefficient for adsorption at equilibrium (cm^3·g^{-1})] was established according to OECD guideline (*13*) as given in

$$K_d = (A_{eq}V_0)/(100 - A_{eq}) \times m_{soil} \qquad (1)$$

with A_{eq} = adsorption percentage at the equilibrium state, V_0 = initial volume of liquid phase, and m_{soil} = mass of the solid phase.

Agitation time (8 h for sulcotrione and 24 h for CHD and CMBA) was deduced from the kinetic results, using a security factor close to 2, to ensure equilibrium for all samples. Adsorption isotherms were obtained using a batch equilibrium method (*5*, *11*, *13*). Glass tubes containing 1 g of soil and 5 mL of each working solution (Sw$_1$–Sw$_7$, Sc$_1$–Sc$_7$, Dw$_1$–Dw$_7$, and Cw$_1$–Cw$_7$) were shaken during the agitation

period. Samples were then centrifuged at 3500g for 10 min. Supernatants were analyzed to determine the amount of each compound adsorbed on the soil at equilibrium status. Two blanks, soil with aqueous solution free of chemicals and aqueous solution without soil, were performed under the same conditions.

Adsorption isotherms were obtained by plotting C_s (mg·kg^{-1}) solid-phase concentration versus $C_{e,ads}$ (mg·L^{-1}) liquid-phase concentration at adsorption equilibrium (*13*). The Freundlich equation was used to describe the results of adsorption isotherms.

$$C_s = K_{fa}(C_{e,ads})^{1/n_{fa}} \qquad (2)$$

K_{fa} and $1/n_{fa}$ are empirical constants representing, respectively, the Freundlich coefficient for adsorption and the slope of the isotherm, which expresses its degree of nonlinearity.

Only highest concentration samples for adsorption procedure (i.e., Sw$_7$, Sc$_7$, Dw$_7$, and Cw$_7$) were used for desorption isotherms. Supernatant was removed, replaced by the same volume of water or 0.01 M CaCl$_2$ solution, and agitated for 8 h for sulcotrione and 24 h for CHD and CMBA. Supernatants were analyzed by HPLC. This desorption step was repeated three times consecutively. The amount of each chemical remaining adsorbed in the soil was calculated after each desorption step. Desorption isotherms were obtained by plotting C_s (solid-phase concentration of the compound remaining adsorbed on the soil after each desorption step) versus C_e (compound concentration in liquid phase after each desorption step). The Freundlich relationship (eq 3) was used to characterize the desorption process.

$$C_{s,0} - C_s = K_{fd}(C_{e,0} - C_e)^{1/n_{fd}} \qquad (3)$$

$C_{s,0}$ is the solid-phase concentration (mg·kg^{-1}) of each compound adsorbed on the soil at equilibrium status; $C_{e,0}$ is the compound concentration in liquid phase at equilibrium after adsorption, K_{fd} and $1/n_{fd}$ are, respectively, the empirical coefficient for desorption and the slope of the curve expressing desorption intensity of each compound from the soil.

Chromatographic Method. Supernatants were analyzed using a high-performance liquid chromatograph (Shimadzu) equipped with an SIL-10AD*vp* autosampler injector, two HPLC LC-10AD*vp* pumps, a Supelco ODS Hypersil C$_{18}$ column, 250 × 4.6 mm i.d, 5 μm, and an SPD-10 A*vp* UV–vis detector set at the wavelength of 254 nm. The mobile phase consisted of a mixture of water acidified by 0.3% TFA (AW) and acetonitrile (ACN) delivered at a flow rate of 1 mL·min^{-1}. The delivery mode was isocratic for isotherm studies, respectively, 80: 20, 70:30, and 50:50 (AW/ACN) for CHD, CMBA, and sulcotrione. A gradient system [8 min with 70:30 (AW/ACN) and then 10 min with 50:50 (AW/ACN)] was used to determine potential degradation products during sulcotrione kinetic study.

Statistical Analysis. To identify which physicochemical properties influence the retention process of all molecules, nonparametric correlation was carried out between soil properties (clay, silt, and sand contents, CEC, pH) and adsorption kinetic and isotherm coefficients. Analyses were run using SPSS 10.0 software.

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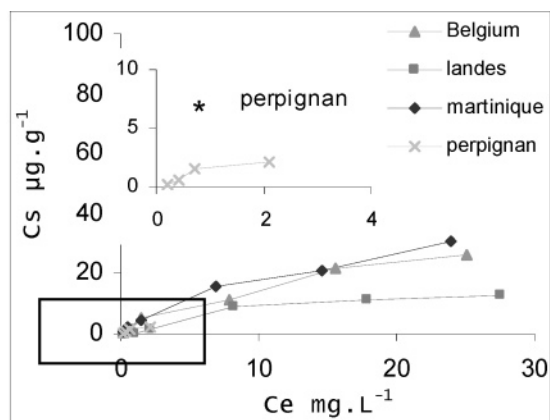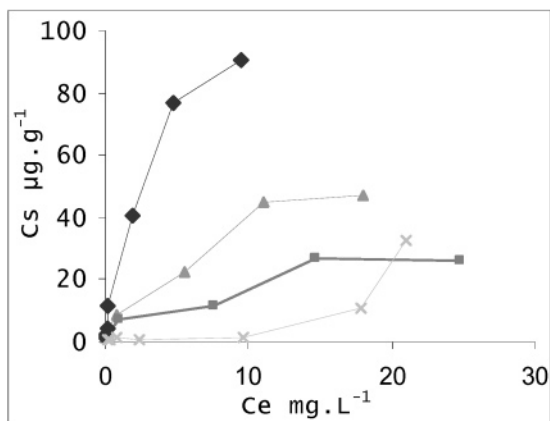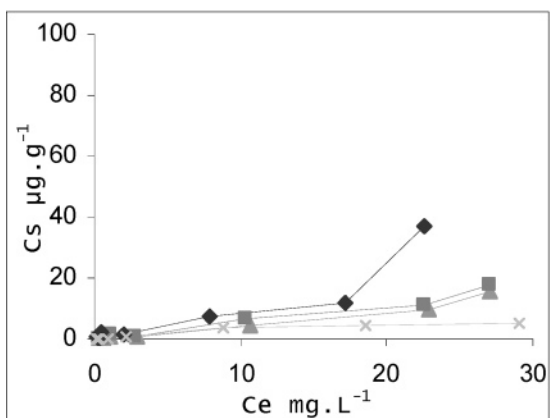
Figure 2. Adsorption isotherms of (**a**) sulcotrione, (**b**) CHD, and (**c**) CMBA in Belgium (▲), Landes (■), Martinique (◆), and Perpignan (*) soils. (Inset) Enhanced adsorption of sulcotrione at low concentrations in Perpignan soil.

RESULTS AND DISCUSSION

Adsorption Kinetics. Equilibrium status was reached after 4 h for sulcotrione, for most of the studied soils, and after 12 h for CHD and CMBA.

The K_d (0.25−2.5 $cm^3 \cdot g^{-1}$ for sulcotrione, 0.2−10 $cm^3 \cdot g^{-1}$ for CHD, and 0.2−1.7 $cm^3 \cdot g^{-1}$ for CMBA), which represents an expression of inherent mobility of chemicals in soils (*13*), led us to foresee a preliminary classification following this increasing order: Perpignan, Landes, Belgium, Martinique.

Martinique soil, with K_d values >1 $cm^3 \cdot g^{-1}$ for the three molecules, seemed to present moderate adsorption capacity (*13*) toward sulcotrione, CHD, and CMBA. These results will be confirmed further by corresponding K_{fa} values.

Adsorption Isotherms. Adsorption isotherms for the three molecules were established, primarily, with distilled water as liquid phase and are presented in **Figure 2**. The sorption behavior of the three molecules was evaluated by the Freundlich relationship (eq 2). Experimental data, for most of the soils, fitted well this model, and respective values of K_{fa}, $1/n_{fa}$, K_{oc}, and r^2 for selected soils for sulcotrione and its metabolites are given in **Table 3**.

Sulcotrione and its major metabolite, CMBA (*11*), exhibited similar patterns, as shown by K_{fa} and $1/n_{fa}$ (close to 1) values and by the shape of their respective isotherms (**Figure 2** and **Table 3**). The two molecules, according to the classification of Giles (*14*), exhibited L-type isotherms in all soils. This type is commonly seen for most of the herbicides (*15*). This shape expresses a moderate affinity between the soil and the herbicide and a decrease of the availability of adsorption sites with increasing liquid concentration (*16*). As shown by K_{fa} values, the metabolite CHD presented the higher adsorption capacity compared to sulcotrione and CMBA regardless of soil type, as previously predicted by K_d values. According to Cox et al. (*17*), the smaller molecular size and the absence of an active functional group in or close to the cyclic ring (which could experience some steric hindrance to reach the sorption sites) could explain the adsorption behavior of CHD. Two different shapes for CHD isotherms could be described (**Figure 2**). For Perpignan soil, the S-shape isotherm is explained by the increasing of the adsorption as well as the solute concentration, which means a low affinity of the adsorbate CHD toward soil components. For the other soils, isotherms presented L-type shapes.

For all molecules, Perpignan soil had the lowest K_{fa} values. Even if it is difficult to classify soils using K_{fa} values, when $1/n_{fa}$ is significantly different from 1 as shown in the case of CHD, it seemed possible to consider Perpignan soil to have the lowest adsorption capacity. This soil is characterized by low values for organic carbon content (0.9%) and slightly alkaline pH when compared to the other soils. Nevertheless, correlation coefficients (**Table 4**) calculated for organic carbon and pH indicated their limited influence on the molecule's adsorption. Martinique soil presented the highest adsorption capacity. This could mainly be related to its high percentage of clay content and, less so, its CEC, parameters that appeared to be larger than those of Belgium and Landes soils even though they have the same values of organic matter (OM) content. Several authors (*18−21*) observed the same correlation for some other acidic molecules (2,4-D, pentachloronitrobenzene, pentachloroaniline, bentazone, etc.). Exchangeable ions play some role in the adsorption on clay surfaces, according to Pusino et al. (*22*). The lower adsorption capacity of Landes soil toward sulcotrione and its two metabolites could be explained by the high sand content when the OM percentage is similar. The same observation was reported (*9*) and showed a decrease of sulcotrione persistence in the soil presenting the highest sand content.

According to K_{fa} results and those previously reported for K_d, soils followed the decreasing order Martinique, Belgium, Landes, and Perpignan for all studied molecules.

Several authors reported that the difference in K_{fa} values can be linked to operating conditions, that is, the soil/solution ratio (*15*). In the case of sulcotrione, we compared our results to those

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Table 3. Values of K_{fa}, n_{fa}, K_{oc}, and r^2 Obtained for the Adsorption of Sulcotrione, CMBA, and CHD on Selected Soils

soils/adsorption coefficient	sulcotrione				CHD				CMBA			
	K_{fa}	$1/n_{fa}$	r^2	K_{oc}[a]	K_{fa}	$1/n_{fa}$	r^2	K_{oc}[a]	K_{fa}	$1/n_{fa}$	r^2	K_{oc}[a]
Belgium	1.54	1.02	0.89	74.0	10.0	0.52	0.99	492.0	0.39	1.02	0.90	20.0
Landes	0.66	0.95	0.95	33.0	5.59	0.50	0.96	260.0	0.39	1.12	0.78	19.0
Martinique	2.82	0.80	0.98	142.0	26.63	0.60	0.96	1370.0	1.59	0.80	0.87	81.0
Perpignan	1.30[b]	0.87[b]	0.90	144.0[b]	1.4	0.64	0.70	155.0	0.35	0.87	0.89	39.0

[a] K_{oc}, partition coefficient normalized on the soil organic carbon content (% OC) and calculated according to OECD (*13*) as $K_{oc} = (K_{fa} \times 100)/\%$ OC. [b] For sulcotrione in Perpignan soil, adsorption isotherm was done only for low concentrations.

Table 4. Spearman Correlation Coefficients at the Significance Level of 0.05 (∗, Significant Values)

correlation coefficient	clay	OC	pH	CEC
K_{fa}, sulcotrione	+1.00∗	−0.40	+0.20	+0.80∗
K_{fa}, CHD	+0.80∗	+0.20	−0.40	+0.40
K_{fa}, CMBA	+0.63∗	+0.32	−0.63*	+0.32

previously published (*5*, *10*, *11*) where respective ratios are 0.1, 0.5, and 0.4. Our K_{fa} values, comparable to recent results (*11*) but quite different from the other ones, did not allow us to come to any conclusions on the hypothetical role of the soil/solution ratio.

Influence of Liquid-Phase Composition on Sulcotrione Adsorption. No adsorption was noted in Perpignan soil for sulcotrione using concentrated (30, 150 mg·L^{-1}) aqueous solution, whereas the percentage of adsorption reached 5% of sulcotrione dissolved in CaCl$_2$ solution. A slightly alkaline pH of this soil enhanced the predominance of the anionic form of the herbicide. This could lead to a repulsion phenomenon toward the soil components. The use of a CaCl$_2$ (Ca^{2+}) aqueous solution of sulcotrione allowed the setting up of a salt bridge between negatively charged sulcotrione and soil particles (clay and organic matter). Isotherms (**Figure 3**) and experimental K_{fa} values (**Table 5**) supported this hypothesis, already reported (*20*) on 2,4-D, a weak acidic herbicide.

Desorption Isotherms for Sulcotrione and Its Metabolites. The Freundlich relationship was tried to evaluate our desorption results. For sulcotrione, the K_{fd} values (lower than K_{fa} values) and mainly the percentage of desorption [>60% (**Table 6**) after three desorption steps] showed its relatively higher desorption capacity. Similar results were published for sulcotrione (*11*) and showed a desorption of >94% after four desorption steps.

For CHD and CMBA, the Freundlich equation did not fit well our results. Therefore, only desorption percentages are reported, showing their lower aptitude to the desorption under our working conditions.

Conclusion. This study, carried out in four different soil types, furnished information that allowed us to extend the existing knowledge about the behavior of sulcotrione and its metabolites.

According to our retention results, sulcotrione presents a moderate affinity with soil components, indicating that its leaching capacity must not be neglected but needs to be carefully assessed. Its hydrolysis products appeared to be also moderately adsorbed, but their desorption seemed to be more difficult. Therefore, complementary GLP studies (degradation rates, migration in microlysimeters, field dissipation testing) and calculations of predicted environmental concentrations under the scenarios developed by the European FOCUS group (*23*) must be undertaken to draw any conclusions on the potential risk of sulcotrione and its metabolites toward groundwater as
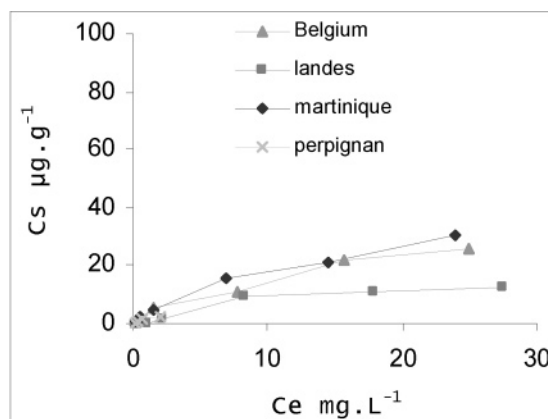
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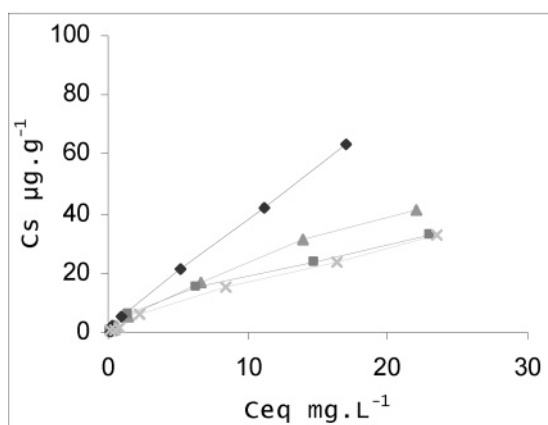
With CaCl$_2$ solution

Figure 3. Adsorption isotherms of sulcotrione with distilled water and CaCl$_2$ solution in Belgium (▲), Landes (■), Martinique (◆), and Perpignan (∗) soils.

Table 5. Values of K_d and K_{fa} Obtained for the Adsorption of Sulcotrione with Distilled Water (DW) and CaCl$_2$ Solution

soil	K_d DW	K_d CaCl$_2$	K_{fa} DW	K_{fa} CaCl$_2$
Belgium	0.66	2.43	1.54	2.89
Landes	0.32	0.85	0.66	2.34
Martinique	1.09	2.52	2.82	3.73
Perpignan	0.0	0.25	1.30[a]	1.76

[a] For Perpignan soil, adsorption isotherms were done only with low concentrations.

part of the standard regulatory data package required to register agrochemical active ingredients.

Moreover, a second degradation pathway of sulcotrione in the soil has been described (*8*), which led to other derivatives: 5,7-diketo-7-(2-chloro-4-methylsulfonylphenyl)heptanoic acid, presented by the authors as having herbicidal activity, and

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Table 6. K_{fd}, n_{fd}, and Total Desorption Percentage of Studied Compounds on the Four Soils[a]

soil/% desorption	sulcotrione				CHD	CMBA
	K_{fd}	$1/n_{fd}$	r^2	TD	TD	TD
Belgium	0.026	2.0	0.74	91.00	2.50	0.00
Landes	6×10^{-4}	2.5	0.92	80.00	10.00	0.00
Martinique	2.22	0.68	0.96	67.00	2.40	77.50
Perpignan					11.00	4.00

[a] TD, percentage of total desorption of each compound after three desorption steps; k_{fd} and n_{fd}, empirical coefficients for desorption.

1-acetyl-2-chloro-4-methylsulfonylbenzene. Our further purpose consists of the confirmation of this probable pathway in the soil as well as in water samples and the study of the retention behavior of these two other metabolites under the same conditions.

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