



## ABSTRACT

### Mobility - Adsorption/Desorption

1. This study is scientifically valid and provides useful information on the mobility of 6-amino-7-fluoro-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one (APF) in four soils and one sediment.
2. This study is classified as supplemental. It does not satisfy the Subdivision N guideline requirement for mobility for the following reason:

mobility was studied in only sandy loam and loam soils.

In addition, information is needed on the mobility of the parent flumioxazin in four soils and one sediment.

3. The mobility of phenyl ring-labeled [U-<sup>14</sup>C]6-amino-7-fluoro-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one (APF), a degradate of flumioxazin, at nominal concentrations of 0.047-0.048, 0.13, 0.24-0.25, and 0.48-0.51 μg/mL, was investigated in four soils (three sandy loam and one loam) and one sediment that were equilibrated for 4, 16 or 17 hours (soils) or 24 hours (sediment) at 25 ± 1°C. Freundlich K<sub>ads</sub> values were 1.6110-5.9704 for the sandy loam soils, 4.2179 for the loam soil, and 4.9068 for the sediment; corresponding 1/n values ranged from 0.7800 to 0.9883. K<sub>oc</sub> values were 336-620 for the sandy loam soils, 391 for the loam soil, and 201 for the sediment. Freundlich K<sub>des</sub> values were 5.3150-18.6724 for the sandy loam soils, 6.3343-7.6085 for the loam soil, and 10.2141 for the sediment; corresponding 1/n values ranged from 0.6089 to 0.9334. Reviewer-calculated coefficients of determination (r<sup>2</sup>) values for K<sub>ads</sub> vs. percent organic matter, K<sub>ads</sub> vs. pH, and K<sub>ads</sub> vs. percent clay content were 0.2208, 0.4082, and 0.0278, respectively. Freundlich K<sub>des</sub> values were 5.3150-18.6724 for the sandy loam soils, 6.3343-7.6085 for the loam soil, and 10.2141 for the sediment; corresponding 1/n values ranged from 0.6089 to 0.9660. Reviewer-calculated coefficients of determination (r<sup>2</sup>) values for K<sub>ads</sub> vs. percent organic matter, K<sub>ads</sub> vs. pH, and K<sub>ads</sub> vs. percent clay content were 0.2208, 0.4082, and 0.0278, respectively. [<sup>14</sup>C]APF was stable in the soil/sediment slurries during the adsorption equilibration phase, but degraded in two sandy loam soils and the loam soil during the desorption equilibration phase, comprising 54.5-89.2% of the radioactivity recovered.

## MATERIALS AND METHODS

Five soils (Tulare sandy loam, Painesville loam, Madison sandy loam, Madera sandy loam and New Philadelphia clay), and one aquatic sediment (Painesville loam) were air-dried (soils only) and screened (2-mm) prior to use in the study (p. 22; Table I, p.38). However, based on the results of a preliminary stability study, the test substance was not stable in the New Philadelphia clay soil or in four additional silty clay loam and clay loam soils that were equilibrated for 24 hours (p. 33). Additional work with clay soils was discontinued when the results of another preliminary study indicated that the test substance was not stable in New Philadelphia clay soil:solution slurries equilibrated for 24 hours in the presence of bactericides (both 0.1% mercury (II) chloride and 0.1%

sodium arsenate in 0.01 M Ca (NO<sub>3</sub>)<sub>2</sub>.) Based on the results of a preliminary study on the adsorption of the test substance, equilibration (adsorption and desorption) periods of 4 and 24 hours were chosen for the Madera sandy loam soil and the four remaining soils, respectively. However, the definitive study conducted using the Madison and Tulare sandy loam soils and the Painesville loam soil was repeated using equilibration (adsorption and desorption) periods of 16-17 hours due to degradation of the test substance. Based on the results of a separate preliminary study, adsorption of the test compound to the glass tubes was not observed (p. 32).

For the adsorption phase of the definitive study, phenyl ring-labeled [<sup>14</sup>C]6-amino-7-fluoro-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one (APF; radiochemical purity 98.5%, specific activity 115 mCi/mmol, Sumitomo Chemical Company; pp. 21-22) in sterile 0.01 M Ca(NO<sub>3</sub>)<sub>2</sub> solution was added to subsamples (1.00 g; dry weight) of Tulare sandy loam soil, Painesville loam soil, Madison sandy loam soil, Madera sandy loam soil, and Painesville aquatic sediment at concentrations of 0.047-0.048, 0.13, 0.24-0.25, and 0.48-0.51 μg/mL (p. 27; Figures 17-29; ). Triplicate samples were prepared for each soil type/treatment combination. The soil:solution slurries were equilibrated by shaking for 4 hours (Madera sandy loam soil), 16-17 hours (Tulare sandy loam, Painesville loam, and Madison sandy loam soils) or 24 hours (Painesville aquatic sediment) at 25 ± 1°C. Following the equilibration period, the tubes were centrifuged (at approximately 3000 g) to pellet the soil and duplicate aliquots of the supernatant were analyzed by LSC. Selected aliquots were further analyzed by HPLC with radiochemical flow and UV detection (pp. 24-25).

#### HPLC Conditions

Column	Zorbax ODS column (250 mm x 4.6 mm i.d.)
Mobile Phase	A = Water containing 0.05% phosphoric acid B = Acetonitrile containing 0.05% phosphoric acid
Gradient (A:B)	85:15 to 0:100 (v:v)
Flow Rate	1.0 mL/min
Ultraviolet Detection	254 nm

For the desorption phase of the definitive study, an aliquot of pesticide-free 0.01 M Ca(NO<sub>3</sub>)<sub>2</sub> solution equal to the volume of supernatant that was removed following the adsorption phase was added to the soil pellets from the adsorption phase of the study (p. 28). The samples were equilibrated by shaking at 25 ± 1°C for 4 hours (Madera sandy loam soil), 16-17 hours (Tulare sandy loam, Painesville loam, and Madison sandy loam soils) or 24 hours (Painesville aquatic sediment). Following equilibration, the tubes were centrifuged (at approximately 3000 g) to pellet the soil and duplicate aliquots of the supernatant were analyzed by LSC. Selected aliquots were further analyzed by HPLC as previously described.

Following desorption, the soil pellets were combusted and analyzed by LSC to determine the total <sup>14</sup>C bound to the soil and in the remaining aqueous phase in the soil pellet (p. 28).

## RESULTS/DISCUSSION

The mobility of phenyl ring-labeled [U-<sup>14</sup>C]6-amino-7-fluoro-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one (APF; radiochemical purity 98.5%), at nominal concentrations of 0.047-0.048, 0.13, 0.24-0.25, and 0.48-0.51  $\mu\text{g/mL}$ , was determined in five soil/sediment:solution slurries that were equilibrated for 4 hours (Madera sandy loam soil), 16-17 hours (Tulare sandy loam, Painesville loam, and Madison sandy loam soils) or 24 hours (Painesville aquatic sediment) at  $25 \pm 1^\circ\text{C}$ .

Results were as follows:

Texture	Sandy loam	Loam	Sandy loam	Sandy loam	Sandy loam sediment
Source	Tulare, CA	Painesville, OH	Madison, OH	Madera, CA	Painesville, OH
% sand	61.2	50.4	70.4	69.2	38.4
% silt	30.0	32.0	20.0	24.0	44.8
% clay	8.8	17.6	9.6	6.8	16.8
% org. matter	1.44	1.87	2.05	0.44	4.21
CEC [meq/100 g]	6.41	5.44	6.16	2.16	9.32
soil pH	7.9	6.9	6.8	5.6	6.9
Equilibrium conc. range ( $\mu\text{g/mL}$ )	0.048-0.48 (16 hrs)	0.048-0.48 (17 hrs)	0.048-0.48 (16 hrs)	0.047-0.51 (4 hrs)	0.047-0.51 (24 hrs)
Adsorption Phase					
1/n	0.8645	0.7800	0.8367	0.9883	0.9278
$K_{\text{ads}}$	2.8022	4.2179	5.9704	1.6110	4.9068
$K_{\text{oc}}$	336	391	502	620	201
Desorption Phase					
1/n	0.7961- 0.8005	0.6089-0.7004	0.8632-0.8719	0.9660	0.9334
$K_{\text{des}}$	5.3150- 5.5360	6.3343-7.6085	18.2894- 18.6724	7.9086	10.2141
$K_{\text{oc}}$	637-663	587-704	1537-1569	3042	419

Data were obtained from Tables I, IV-V, VII, pp. 38, 41-42, 44.

Freundlich equation coefficients of determination ( $r^2$ ) values for all soil/sediment types were 0.9498-0.9984 following adsorption and desorption (Table VII, p. 44). Reviewer-calculated coefficients of determination ( $r^2$ ) values for  $K_{\text{ads}}$  vs. percent organic matter,  $K_{\text{ads}}$  vs. pH, and  $K_{\text{ads}}$  vs. percent clay content were 0.2208, 0.4082, and 0.0278, respectively (Attachment 2).

Phenyl ring-labeled [<sup>14</sup>C]APF was stable in the soil/sediment slurries during the adsorption equilibration phase, but degraded in the Tulare sandy loam, Painesville loam and Madison sandy loam soils during the desorption equilibration phase (Tables IV-V, pp. 41-42). [<sup>14</sup>C] APF accounted for 54.5-89.2% of the radioactivity recovered from these three soils following the desorption period.

Mean material balances ranged from 94.4 to 102.9% (Table VI, p. 43).

#### DEFICIENCIES/DEVIATIONS

1. Mobility was studied in three sandy loam and one loam soil. These four soils were too similar in particle size; a range of soil textures should have been studied. The failure of the test substance to remain stable in the soils with a higher clay content does not negate the need for information on its mobility in other types of soils.
2. It was not stated that the definitive study was conducted in the dark. Subdivision N guidelines specify that equilibration be conducted in the dark to minimize photodegradation. During the desorption phase of the definitive study, [<sup>14</sup>C]APF degraded in two sandy loam soils and the loam soil, comprising 54.5-89.2% of the radioactivity recovered (Tables IV-V, pp. 41-42). Clarification as to whether the definitive study was conducted in the dark is required.
3. Based on the calculated  $K_{oc}$  values reported in the study, APF would be classified as having medium mobility in the Tulare sandy loam and Painesville loam soils and in the aquatic sediment, and low mobility in the Madison and Madera sandy loam soils, according to the McCall Mobility Classifications (Table VIII, p. 45).
4. The soil series names of the soils and sediment were not reported. Instead, the soils and sediment were referred to by their geographical locations or descriptions of their locations.
5. The soil:solution ratios were not reported and could not be determined since it was only stated that "a measured volume of dosing solution" was added to the test tubes (p. 27).
6. Detection limit for LSC and HPLC analyses were not reported in the study.
7. The solubility of APF in water was reported to be >3 µg/mL (p. 31; Figure 3, p. 48).
8. The study authors stated that the Tulare sandy loam soil was obtained from the same location as the soil used in an aerobic soil metabolism study of [TMP-<sup>14</sup>C]-S-53482 (p. 19).
9. Good Laboratory Practice and Quality Assurance statements were submitted with the study (pp. 3,6).

ATTACHMENT 1  
Data Critical to the Study Interpretation

THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY  
SEE THE FILE COPY

Ricerca, Inc.  
 5855-93-0254-EF-001  
 Report/[Phenyl-<sup>14</sup>C]-APF

TABLE VII

SOIL SORPTION CONSTANTS ( $K_{oc}$ ) CALCULATED FROM  
 THE FREUNDLICH ADSORPTION COEFFICIENTS (K)

	Log K	1/n	R <sup>2</sup>	K	K <sub>oc</sub>	% Organic Carbon	Mobility Class
<b>Tulare Sandy Loam</b>							
<b>EFS 015</b>							
Adsorption	0.4475	0.8645	0.9952	2.8022	336	0.84	Medium
Desorption(1)	0.7432	0.8005	0.9959	5.5360	663	0.84	Low
Desorption(2)	0.7255	0.7961	0.9960	5.3150	637	0.84	Low
<b>Painesville Loam</b>							
<b>EFS 021</b>							
Adsorption	0.6251	0.7800	0.9984	4.2179	391	1.08	Medium
Desorption(1)	0.8017	0.6089	0.9498	6.3343	587	1.08	Low
Desorption(2)	0.8813	0.7004	0.9714	7.6085	704	1.08	Low
<b>Madison Sandy Loam</b>							
<b>EFS 022</b>							
Adsorption	0.7760	0.8367	0.9974	5.9704	502	1.19	Low
Desorption(1)	1.2712	0.8632	0.9965	18.6724	1569	1.19	Low
Desorption(2)	1.2622	0.8719	0.9969	18.2894	1537	1.19	Low
<b>Madera Sandy Loam</b>							
<b>EFS 026</b>							
Adsorption	0.2071	0.9883	0.9801	1.6110	620	0.26	Low
Desorption	0.8981	0.9660	0.9960	7.9086	3042	0.26	Slight
<b>Painesville Aquatic Sediment</b>							
<b>EFS 045</b>							
Adsorption	0.6908	0.9278	0.9937	4.9068	201	2.44	Medium
Desorption	1.0092	0.9334	0.9925	10.2141	419	2.44	Medium

The Freundlich adsorption coefficient (K) was derived from the analysis of the Freundlich equation.

(1) Data calculated assuming <sup>14</sup>C in the aqueous phase was 100% APF.

(2) Data calculated from HPLC analyses of % APF in the aqueous phase.

Ricerca, Inc.  
5855-93-0254-EF-001  
Report/[Phenyl-<sup>14</sup>C]-APF

TABLE VIII

CLASSIFICATION OF CHEMICAL MOBILITY IN SOIL

APPROXIMATE $K_{oc}$	MOBILITY CLASS
0-50	Very High
50-150	High
150-500	Medium
500-2000	Low
2000-5000	Slight
>5000	Immobile

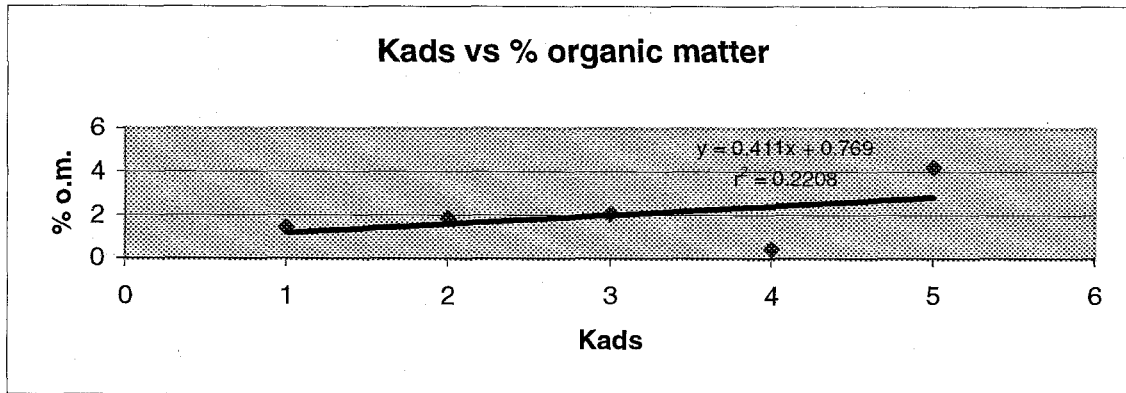
R. L. Swan, D.A. Laskowski, P.J. McCall, K. Vander Kuy and  
H.J. Dishburger, Residue Reviews, Volume 85, pg.23, 1983.



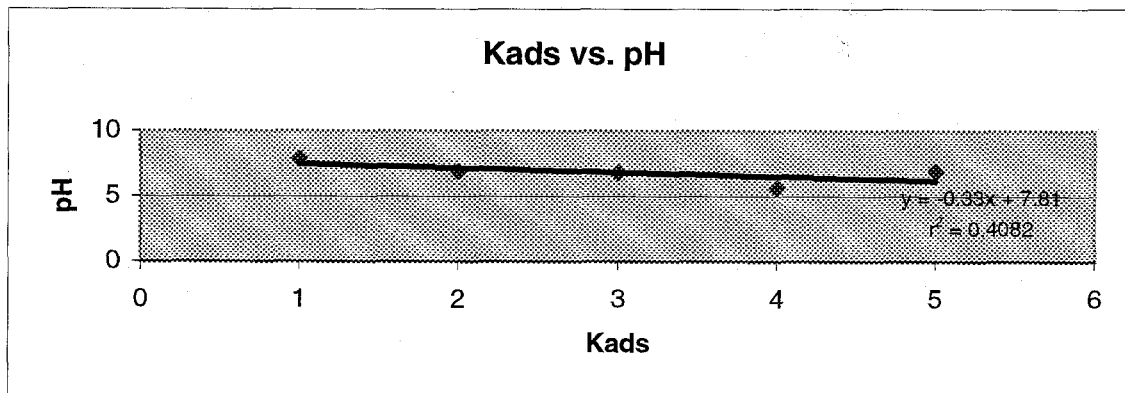
ATTACHMENT 2  
Excel Workbook

Chemical Name Flumioxazin (APF)  
 PC Code 129034  
 MRID 45309201  
 Guideline No. 163-1

Soil/Sediment	Kads	% Organic Matter
Tulare	2.8022	1.44
Painesville	4.2179	1.87
Madison	5.9704	2.05
Madera	1.6110	0.44
Painesville Aquatic	4.9068	4.21



Soil/Sediment	Kads	pH
Tulare	2.8022	7.9
Painesville	4.2179	6.9
Madison	5.9704	6.8
Madera	1.6110	5.6
Painesville Aquatic	4.9068	6.9



Soil/Sediment	Kads	% clay content
Tulare	2.8022	8.8
Painesville	4.2179	17.6
Madison	5.9704	9.6
Madera	1.6110	6.8
Painesville Aquatic	4.9068	16.8

