

DATA EVALUATION RECORD

STUDY 2

PC No. 129034 Flumioxazin §163-1

CAS No. 103361-09-7

DP Barcode D272504

FORMULATION- 00-ACTIVE INGREDIENT

STUDY ID 45309202

Shah, J.F. 1994. Adsorption and desorption of tetrahydrophthalic acid (THPA) to soil. Laboratory Project ID: 5854-93-0252-EF-001. Ricerca Study No: 93-0252. Unpublished study performed by Ricerca, Inc., Painesville, OH; and submitted by Valent U.S.A. Corporation, Walnut Creek, CA.

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ABSTRACT

Mobility - Adsorption/Desorption

1. This study is scientifically sound and provides useful information on the mobility of 3,4,5,6-tetrahydrophthalic acid (THPA) five soils and one sediment.
2. This study is classified as acceptable and provides information on the mobility of the flumioxazin degradate THPA in five soils and one sediment. Information is needed on the mobility of the parent flumioxazin in four soils and one sediment.
3. The mobility of [1,2-¹⁴C]THPA, at nominal concentrations of 0.026-0.027, 0.06-0.07, 0.13, and 0.26-0.27 $\mu\text{g/mL}$, was investigated in five soils (three sandy loam, one loam, and one clay) and one sediment that were equilibrated for 4-8 or 48 hours at $25 \pm 1^\circ\text{C}$. The soil:solution ratios were 2:7 for the sandy loam soils, 1:8 for the loam and clay soils, and 1:7.9 for the sediment. Freundlich K_{ads} values were 0.1078-0.8658 for the sandy loam soils, 2.6884 for the loam soil, 5.2614 for the clay soil, and 1.8357 for the sediment; corresponding $1/n$ values ranged from 0.8950 to 1.0147. K_{oc} values were 13-339 for the sandy loam soils, 248 for the loam soil, 191 for the clay soil, and 75 for the sediment. K_{oc} values ranged from 13 to 339 for all soil/sediment types and $1/n$ values ranged from 0.8950 to 1.0147. Freundlich K_{des} values were 0.1790-1.1350 for the sandy loam soils, 4.1860 for the loam soil, 6.9711 for the clay soil, and 2.8701 for the sediment; corresponding $1/n$ values ranged from 0.6413 to 0.9486. Reviewer-calculated coefficients of determinations (r^2) values for K_{ads} vs. percent organic matter, K_{ads} vs. pH, and K_{ads} vs. percent clay content were 0.4465, 0.365, and 0.2052, respectively.

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 (0.25-inch and #10 mesh sieves) prior to use in the study (p. 23, Table p. 39). Based on the results of a preliminary study of the adsorption of the test substance, equilibration (adsorption and degradation) periods of 4-8 hours were chosen for all of the test soils and the test sediment with the exception of the Madera sandy loam soil, which had equilibration periods of 48 hours (pp. 35-36). Based on the results of a separate preliminary study, adsorption of the test compound to the glass tubes was not observed.

For the adsorption phase of the definitive study, [1,2-¹⁴C]3,4,5,6-tetrahydrophthalic acid (THPA; radiochemical purity $\geq 90.0\%$, specific activity 110 mCi/mmol, Sumitomo Chemical Company; pp. 21-22) in sterile 0.01 M $\text{Ca}(\text{NO}_3)_2$ solution (7.0-8.0 mL) was added to subsamples (1.00-2.00 g; dry weight) of Tulare sandy loam, Painesville loam, Madison sandy loam, Madera sandy loam, and New Philadelphia clay soils, and in the Painesville aquatic sediment at concentrations of 0.026-0.027, 0.06-0.07, 0.13, and 0.26-0.27 $\mu\text{g/mL}$ (p. 30). The soil:solution ratios were 2:7 for the sandy loam soils, 1:8 for the loam and clay soils, and 1:7.9 for the sediment (p. 30). Triplicate samples were prepared for each soil type/treatment combination. The soil:solution slurries were equilibrated by shaking for 4 hours (Painesville loam soil and sediment), 5 hours (Tulare sandy loam

soil), 7 hours (Madison sandy loam soil), 8 hours (New Philadelphia clay soil), or 48 hours (Madera sandy loam soil) at $25 \pm 1^\circ\text{C}$. Following the equilibration period, the tubes were centrifuged 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. 25-26).

HPLC Conditions

| | |
|-----------------------|---|
| Column | Zorbax ODS column (250 mm x 4.6 mm i.d.) |
| Mobile Phase | A = Water containing 0.01% trifluoroacetic acid B = Acetonitrile containing 0.01% trifluoroacetic acid |
| Gradient (A:B) | 90:10 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 $\text{Ca}(\text{NO}_3)_2$ 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. 31). The samples were equilibrated by shaking for 4 hours (Painesville loam soil and sediment), 5 hours (Tulare sandy loam soil), 7 hours (Madison sandy loam soil), 8 hours (New Philadelphia clay soil), or 48 hours (Madera sandy loam soil) at $25 \pm 1^\circ\text{C}$. Following equilibration, the tubes were centrifuged 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 total ^{14}C bound to the soil and in the remaining aqueous phase in the soil pellet (p. 71).

RESULTS/DISCUSSION

The mobility of [1,2- ^{14}C]3,4,5,6-tetrahydrophthalic acid (THPA; radiochemical purity $\geq 90.0\%$) at nominal concentrations of 0.026-0.027, 0.06-0.07, 0.13, and 0.26-0.27 $\mu\text{g}/\text{mL}$ was determined in six soil/sediment:solution slurries that were equilibrated for 4 hours (Painesville loam soil and sediment), 5 hours (Tulare sandy loam soil), 7 hours (Madison sandy loam soil), 8 hours (New Philadelphia clay soil), or 48 hours (Madera sandy loam soil).

Results were as follows:

| | | | | | | |
|--|------------|-----------------|-------------|---------------|----------------------|---------------------|
| Texture | Sandy loam | Loam | Sandy loam | Sandy loam | Clay | Sandy loam sediment |
| Source | Tulare, CA | Painesville, OH | Madison, OH | Madera, CA | New Philadelphia, OH | Painesville, OH |
| % sand | 61.2 | 50.4 | 70.4 | 69.2 | 20.8 | 38.4 |
| % silt | 30.0 | 32.0 | 20.0 | 24.0 | 38.0 | 44.8 |
| % clay | 8.8 | 17.6 | 9.6 | 6.8 | 41.2 | 16.8 |
| % org. matter | 1.44 | 1.87 | 2.05 | 0.44 | 4.76 | 4.21 |
| CEC [meq/100 g] | 6.41 | 5.44 | 6.16 | 2.16 | 17.78 | 9.32 |
| soil pH | 7.9 | 6.9 | 6.8 | 5.6 | 4.7 | 6.9 |
| Equilibrium conc. range ($\mu\text{g/mL}$) | 0.026-0.26 | 0.026-0.26 | 0.027-0.27 | 0.027-0.27 | 0.027-0.27 | 0.026-0.26 |
| Adsorption Phase | | | | | | |
| 1/n | 0.9939 | 0.8950 | 1.0147 | 0.9592 | 0.9564 | 0.9952 |
| K_{ads} | 0.1078 | 2.6884 | 0.7851 | 0.8658 | 5.2614 | 1.8357 |
| K_{oc} | 13 | 248 | 66 | 339 | 191 | 75 |
| Desorption Phase | | | | | | |
| 1/n | 0.8548 | 0.8899 | 0.9486 | 0.6413-0.7136 | 0.9198 | 0.9288 |
| K_{des} | 0.1790 | 4.1860 | 1.0819 | 1.0323-1.1350 | 6.9711 | 2.8701 |
| K_{oc} | 21 | 386 | 91 | 405-445 | 253 | 118 |

Data were obtained from Tables I, IIIA-III B, V, pp. 39, 42-43, 46.

Freundlich equation coefficient of determination (r^2) values for all soil/sediment types were 0.9267-0.9989 following adsorption and desorption (Table V, p. 46). Reviewer-calculated coefficient of determination (r^2) values for K_{ads} vs. percent organic matter, K_{ads} vs. pH, and K_{ads} vs. percent clay content were 0.4465, 0.365, and 0.2052, respectively (Attachment 2).

$[^{14}\text{C}]\text{THPA}$ accounted for $\geq 90.1\%$ of the radioactivity present in the aqueous phase following adsorption (all soil/sediment:solution slurries), and $\geq 87.2\%$ of the radioactivity present in the aqueous phase following desorption (all soil/sediment:solution slurries with the exception of the Madera sandy loam which was 70.8%; Table III, pp. 42-43).

Material balances ranged from 93.5 to 100.8% (Tables IVA-IVB, pp 44-45).

DEFICIENCIES/DEVIATIONS

1. 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, [¹⁴C]THPA degraded in the Madera sandy loam soil during the desorption phase, comprising 70.8% of the radioactivity recovered (Table IIIB, p. 43). Clarification as to whether the definitive study was conducted in the dark is required.
2. Based on the calculated K_{oc} values reported in the study, THPA would be classified as having very high mobility in the Tulare sandy loam soil, high mobility in the Madison sandy loam soil and the Painesville loam aquatic sediment, and medium mobility in the Painesville loam, Madera sandy loam, and New Philadelphia clay soils according to the McCall Mobility Classifications (Table VI; p. 47).
3. 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.
4. Detection limits for the LSC and HPLC analyses were not reported in the study.
5. The solubility of THPA in water was reported to be >7 µg/mL (p. 34; Figure 4, p. 51).
6. The study author stated that the Madison sandy loam soil was obtained from the same location as the soil used in an aerobic soil metabolism study for [THP-¹⁴C]-S-23031, and that the Tulare sandy loam soil was obtained from the same location as the soil used in an aerobic soil metabolism study of [THP-¹⁴C]-S-53482 (p. 19).
7. Good Laboratory Practice and Quality Assurance statements were submitted with the study (pp. 3, 7).

ATTACHMENT 1
Data Critical to the Study Interpretation

THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY
SEE THE FILE COPY

TABLE V

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

| | Log K | 1/n | R ² | K | K _{oc} ¹ | % Organic Carbon | Mobility Class |
|--|---------|--------|----------------|--------|------------------------------|------------------|----------------|
| Tulare California Sandy Loam | | | | | | | |
| EFS 015 | | | | | | | |
| Adsorption | -0.9675 | 0.9939 | 0.9267 | 0.1078 | 13 | 0.84 | Very High |
| Desorption | -0.7471 | 0.8548 | 0.9888 | 0.1790 | 21 | 0.84 | Very High |
| Painesville Ohio Loam | | | | | | | |
| EFS 021 | | | | | | | |
| Adsorption | 0.4295 | 0.8950 | 0.9923 | 2.6884 | 248 | 1.08 | Medium |
| Desorption | 0.6218 | 0.8899 | 0.9884 | 4.1860 | 386 | 1.08 | Medium |
| Madison Sandy Loam | | | | | | | |
| efs 022 | | | | | | | |
| Adsorption | -0.1051 | 1.0147 | 0.9955 | 0.7851 | 66 | 1.19 | High |
| Desorption | 0.0342 | 0.9486 | 0.9922 | 1.0819 | 91 | 1.19 | High |
| Madera Sandy Loam | | | | | | | |
| EFS 026 | | | | | | | |
| Adsorption | -0.0626 | 0.9592 | 0.9972 | 0.8658 | 339 | 0.26 | Medium |
| Desorption ² | 0.0550 | 0.7136 | 0.9695 | 1.1350 | 445 | 0.26 | Medium |
| Desorption ³ | 0.0138 | 0.6413 | 0.9706 | 1.0323 | 405 | 0.26 | Medium |
| New Philadelphia Clay | | | | | | | |
| EFS 042 | | | | | | | |
| Adsorption | 0.7211 | 0.9564 | 0.9989 | 5.2614 | 191 | 2.76 | Medium |
| Desorption | 0.8433 | 0.9198 | 0.9829 | 6.9711 | 253 | 2.76 | Medium |
| Painesville Aquatic Sediment - Loam | | | | | | | |
| EFS 045 | | | | | | | |
| Adsorption | 0.2638 | 0.9952 | 0.9911 | 1.8357 | 75 | 2.44 | High |
| Desorption | 0.4579 | 0.9288 | 0.9964 | 2.8701 | 118 | 2.44 | High |

¹ Calculated using the K derived from the linear regression analysis of the Freundlich Equation

² Assuming no degradation of the test compound

³ Based on % THPA in desorption liquid phase as determined by HPLC

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TABLE VI

CLASSIFICATION OF CHEMICAL MOBILITY IN SOIL

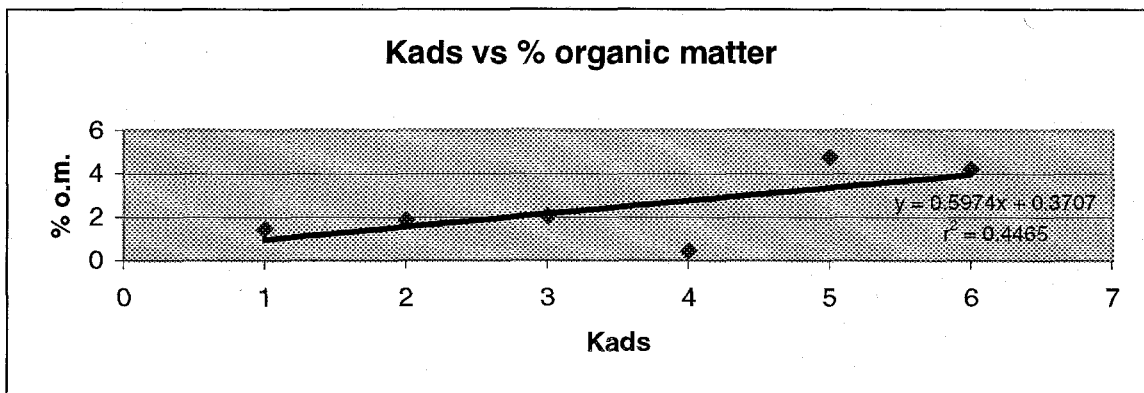
| APPROXIMATE K_{oc} | MOBILITY CLASS |
|-------------------------|-------------------|
| 0-50 | Very High |
| 50-150 | High |
| 150-500 | Medium |
| 500-2000 | Low |
| 2000-5000 | Slight |
| >5000 | Immobile |

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

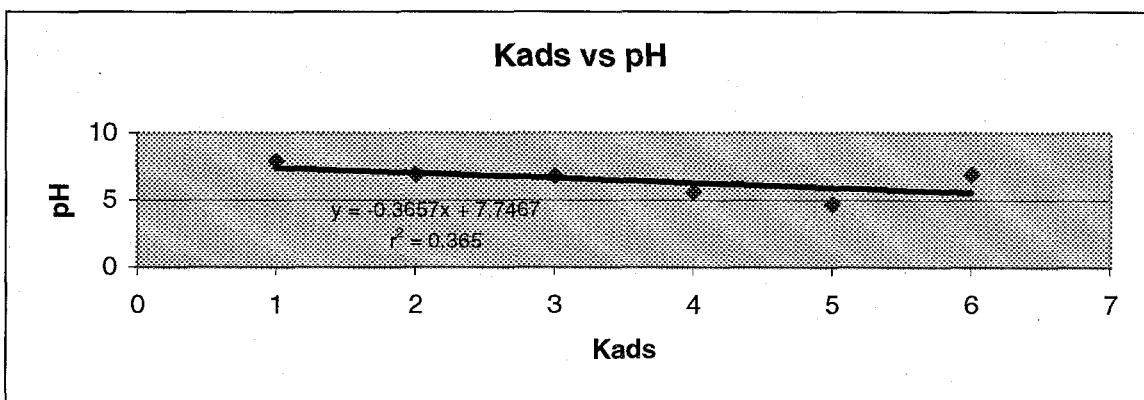
ATTACHMENT 2
Excel Workbook

Chemical Name Flumioxazin (THPA)
 PC Code 129034
 MRID 45309202
 Guideline No. 163-1

| Soil/Sediment | Kads | % Organic Matter |
|---------------------|--------|------------------|
| Tulare | 0.1078 | 1.44 |
| Painesville | 2.6884 | 1.87 |
| Madison | 0.7851 | 2.05 |
| Madera | 0.8658 | 0.44 |
| New Philadelphia | 5.2614 | 4.76 |
| Painesville Aquatic | 1.8357 | 4.21 |



| Soil/Sediment | Kads | pH |
|---------------------|--------|-----|
| Tulare | 0.1078 | 7.9 |
| Painesville | 2.6884 | 6.9 |
| Madison | 0.7851 | 6.8 |
| Madera | 0.8658 | 5.6 |
| New Philadelphia | 5.2614 | 4.7 |
| Painesville Aquatic | 1.8357 | 6.9 |



| Soil/Sediment | Kads | % clay content |
|---------------------|--------|----------------|
| Tulare | 0.1078 | 8.8 |
| Painesville | 2.6884 | 17.6 |
| Madison | 0.7851 | 9.6 |
| Madera | 0.8658 | 6.8 |
| New Philadelphia | 5.2614 | 41.2 |
| Painesville Aquatic | 1.8357 | 16.8 |

