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DATA EVALUATION RECORD

STUDY 6

CHEM 106701

Fosamine ammonium

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FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 40133704

Priester, T.M. and G.D. Sheftic. 1987. Batch equilibrium (adsorption/desorption) and soil thin-layer chromatography studies with [carbonyl-¹⁴C]-fosamine ammonium. Laboratory Project ID AMR-632-86. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 12

REVIEWED BY: J. Harlin

TITLE: Staff Scientist

EDITED BY: T. Colvin-Snyder

TITLE: Staff Scientist

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD
TEL: 468-2500

APPROVED BY: Elizabeth A. Resek

TITLE: Chemist

ORG: EFGWB/EFED/OPP

TEL: 557-7709

SIGNATURE: 

CONCLUSIONS:

EFGWB concludes that the study submitted does not satisfy environmental fate data requirements for leaching and adsorption/desorption. The study cannot be made acceptable upon the submission of additional information, therefore, a new study is required. Soil TLC experiments are not sufficient for the determination of mobility of compounds; they may be used as supplemental information. (It should also be noted that aquatic nonfood uses require batch equilibrium studies to satisfy the leaching and adsorption/desorption requirement). In addition, the batch equilibrium portion of the submitted study is not acceptable. The study authors failed to use compound specific methodology to analyze for fosamine ammonium (see DISCUSSION) and to provide a complete material balance. Also, no desorption values were reported (the desorption portion of the experiment was conducted using only one concentration; see DISCUSSION).

Since the method of analysis (LSC) was not specific to the compound, no conclusion can be drawn as to the mobility of fosamine ammonium. It appears as though in one case the compound degraded during the course of the study.

MATERIALS AND METHODS:

Batch Equilibrium

Two sandy loam and two silt loam soils (Table 1) were treated with 0.01 N calcium sulfate solutions (20 g soil:20 mL solution) of carbonyl-labeled [^{14}C]fosamine ammonium (radiochemical purity 80%, specific activity 6.83 uCi/mg, du Pont) at 0.2, 0.5, 1.0, 2.5, and 6.0 ug/mL. The solutions were shaken for 24 hours at 25 C, centrifuged, and the supernatants were analyzed for total radioactivity by LSC.

Duplicate aqueous solutions from the slurries treated at 6 ug/mL for each soil were combined and concentrated by evaporation under a stream of nitrogen. The concentrates were cochromatographed with radiolabeled fosamine ammonium and carbamoyl phosphonic acid (CPA) reference standards on cellulose TLC plates developed in 0.5 M ammonium carbonate:methanol:water (5:60:35). Fosamine ammonium and the degradate CPA were identified by comparison to the reference standards.

Desorption of fosamine ammonium residues was investigated in the soil:solution slurries treated at 6.0 ug/mL. The removed supernatants were replaced with untreated 0.01 N calcium sulfate solution to reestablish the 1:1 soil:solution ratio. The soil:solution slurries were shaken for 24 hours at 25 C and then centrifuged. The supernatant from each solution was filtered and analyzed by LSC. This procedure was repeated five additional times.

The aqueous solutions were extracted and analyzed as described in the adsorption portion of the study. The soil samples were extracted three times ultrasonically and then one time by shaking with methanol:0.5 M ammonium carbonate (1:1). The soils were again extracted by shaking with 0.5 M ammonium carbonate following storage of soil:ammonium carbonate slurries overnight at 4 C. Then, the soils were extracted twice by shaking with 0.5 M ammonium carbonate adjusted to pH 10 with 28% ammonium hydroxide. Aliquots (500 uL) from each of the resulting seven extracts were analyzed for radioactivity by LSC, and then extracts from duplicate soil samples were combined. The soils were further extracted two times with 0.5 M sodium hydroxide and one time with 0.5 M sodium hydroxide:methanol (1:1); the sodium hydroxide and sodium hydroxide:methanol extracts were combined. All extracts were concentrated by rotoevaporation. The concentrated sodium hydroxide:methanol extracts were adjusted to pH 8.5-10 using 85% phosphoric acid, followed by 1 M sodium hydroxide

and then 9 M ammonium hydroxide. All concentrated extracts were then analyzed by TLC on cellulose plates as described above. Extracted soil samples were analyzed for unextractable radioactivity by LSC following combustion.

Soil TLC

Soil TLC plates were prepared by coating glass plates (20 x 20 cm) with the two sandy loam and two silt loam soils used in the batch equilibrium experiment to a thickness of 400 μm . The plates were spotted with carbonyl-labeled [^{14}C]fosamine ammonium (described above). [2- ^{14}C]Terbacil and phenyl-labeled [^{14}C]diuron were used as reference pesticides. The plates were developed in water to a distance of 10 cm. Following development, the plates were air-dried for 24 hours. Radioactive areas were visualized using autoradiography.

REPORTED RESULTS:

Batch Equilibrium

The authors concluded that carbonyl-labeled [^{14}C]fosamine ammonium (radiochemical purity 80%), at 0.2-6.0 $\mu\text{g}/\text{mL}$, was very mobile in Woodstown and Cecil sandy loam soils, mobile in Flanagan silt loam soil, and immobile in Keyport silt loam soil that were equilibrated in soil:0.01 M calcium solution slurries (20 g:20 mL) at 25 C for 24 hours. Adsorption values were 0.22 for the Woodstown sandy loam, 0.54 for the Cecil sandy loam, 2.7 for the Flanagan silt loam soil, and 350 for the Keyport silt loam soil; respective K_{oc} values were 20, 26, 62, and 7400 (Table 6). After adsorption, fosamine ammonium in the solutions treated at 6 $\mu\text{g}/\text{mL}$ comprised 98% of the radioactivity recovered from the solution phase in the sandy loam slurries, 81% of the recovered in the Flanagan silt loam slurry, and 9% of the recovered in the Keyport silt loam slurry; the remaining radioactivity in the solutions was comprised of unidentified polar compounds (Table 12). The registrant attributed the higher Freundlich K_{ads} values in the silt loam soils to degradation of fosamine ammonium in these slurries. Adsorption increased with increasing soil organic matter content; however, correlation between adsorption values and organic matter contents was poor (correlation coefficient 0.64), indicating that other factors have a significant influence on the adsorption of fosamine ammonium.

Fosamine ammonium residues were desorbed from the four soils in slurries treated at 6 $\mu\text{g}/\text{mL}$ after five days of desorption (Tables 7-10). Fosamine ammonium degraded extensively by the end of the desorption period. After five days of desorption, fosamine ammonium comprised <3% of the radioactivity recovered from the soil and $\leq 8\%$ of the radioactivity recovered from the solutions. Remaining radioactivity was comprised of unidentified polar compounds, CPA, and radioactivity that was unextractable from the soil (Table 13).

Soil TLC

Based on soil TLC studies, carbonyl-labeled [¹⁴C]fosamine ammonium (radiochemical purity 80%) was very mobile (R_f 0.92-0.98) in two silt loam and two sandy loam soils (Table 14).

DISCUSSION:

The following points raised should be addressed by the registrant:

1. Since the method of analysis (LSC) was not specific to the compound, no conclusion can be drawn as to the mobility of fosamine ammonium. It appears as though in one case the compound degraded during the course of the study. (Fosamine ammonium was determined to be mobile in Flanagan silt loam soil and immobile in Keyport silt loam based on the batch equilibrium experiment, but was determined to be very mobile in these two soils based on the soil TLC experiment. The registrant attributed the decreased mobility of fosamine ammonium in the silt loam soils in the batch equilibrium experiment to degradation of fosamine ammonium in these slurries).
2. Soil TLC is not sufficient to determine the mobility of compounds; it may be used as supplemental information.
3. The purity of the fosamine ammonium test substance (radiochemical purity 80%) was inadequate. The presence of fosamine ammonium degradates may have affected the mobility of fosamine ammonium. Since, as demonstrated in the hydrolysis and photolysis studies (Studies 1-3), fosamine ammonium can be purified to >98% radiochemical purity, a purer test substance should have been used to minimize the effect of fosamine ammonium degradates on adsorption and desorption. Since fosamine ammonium was very mobile in all four soils in the soil TLC experiment, any decrease in mobility due to the presence of degradates is not considered significant and, therefore, repeating the soil TLC experiment is not necessary. However, the lack of purity of the test substance is considered a deficiency in the batch equilibrium portion of the study.
4. The study authors should have reported whether soils were sieved, and sieve size. Since fosamine ammonium was very mobile in all four soils in the soil TLC experiment, any decrease in mobility due to sieving is not considered significant and, therefore, repeating the soil TLC experiment is not necessary. However, the lack of information concerning soil sieving is considered a deficiency in the batch equilibrium portion of the study.
5. The water solubility for fosamine ammonium was reported as 1790 g/L (at 25 C).
6. The study authors failed to analyze the soil to confirm adsorption and to provide a complete material balance. Instead, the

concentration of radioactivity adsorbed onto the soil after equilibration was determined by subtracting the concentration of radioactivity in the aqueous solution at equilibrium from the initial concentration in the standard solution. The study authors stated that soils from slurries treated at 6 ug/mL were analyzed after extraction by LSC following combustion, but data for radioactivity in these soils were not provided.

7. The methods of identification and quantification of radioactive areas on the TLC plates were not reported.
8. The study authors adjusted values for total radioactivity in solution before and after equilibrium to correct for the concentration of parent fosamine ammonium in solution, and used these adjusted values to calculate adsorption values.
9. The study authors reported Freundlich K_{des} values (Table 11) and stated that desorption isotherms were generated; however, desorption was conducted only using 6 ug/mL solutions. Since several concentrations are needed to construct desorption isotherms and calculate Freundlich K_{des} values, these values cannot be true Freundlich K_{des} values and therefore were not reported in this review.
10. Although adsorption of fosamine ammonium residues increased with increasing soil organic matter content, correlation between adsorption values and organic matter contents was poor (correlation coefficient 0.64), indicating that other factors have a significant influence on the adsorption of fosamine ammonium residues. The study authors suggested that other factors that may affect adsorption of fosamine ammonium include cation/anion exchange capacity, pH, and degradation to carbamoyl phosphonic acid.

PAGES 6.6 THROUGH 6.9 HAVE BEEN REMOVED FROM THIS DOCUMENT. THOSE PAGES
CONSIST OF REGISTRANT-SUBMITTED DATA.