DATA EVALUATION RECORD

I. Study Type: Batch Equilibrium

II. Citation:

Ferguson,R.E., K. Muller,and M.C.G. Lane. 1995. ICIA5504: Adsorption and Desorption Properties of R234886, a Major Soil Metabolite. Performed by Zeneca Agrochemicals (Zeneca Limited), Berkshire, U.K. Submitted by Zeneca Agricultural Products (Zeneca Inc.), Wilmington, Delaware. MRID 43678179.

III. Reviewer:

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Title: Soil Chemist
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30 JUN 1996

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30 JUL 1996

V. Conclusions:

The study provides acceptable data on (E)-2-{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]-3-methoxyacrylic acid (R234886) partitioning in mineral soils. These data in conjunction with the batch equilibrium data (MRIDs 43678180 and 43678177) fulfill the aged portion of the 163-1 data requirement. No additional data are needed at this time.

Radiolabeled R234886 had Freundlich adsorption coefficients of 0.82 ml/g (K_{oc}=28; 1/n=0.90) in the Kenny Hill sandy loam soil, 0.35 ml/g (K_{oc}=21; 1/n=0.76) in the East Anglia loamy sand soil, 1.4 ml/g (K_{oc}=490;1/n=0.79) in the Lilly Field sand, 6.8 ml/g (K_{oc}=420;1/n=.90) in a Nebo silty clay loam soil,0.85 ml/g (K_{oc}=49;1/n=0.85) in the Hyde Farm sandy clay loam soil, and 10 ml/g (K_{oc}=360;1/n=0.89) in the Pickett Piece clay loam soil.

The reported data indicate R234886 should be mobile to very mobile in terrestrial and aquatic environments.

VI. Materials and Methods:

The test soils from United Kingdom are classified as a Hyde Farm sandy loam, East Anglia loamy sand, Kenny Hill loamy sand, Lilly Field sand, Nebo silty clay loam, and a Pickett Place clay loam. Physicochemical properties of the test soils are shown in Table 2. The test soils were passed through a 2 mm sieve and then sterilized with gamma irradiation.

Preliminary Study

Subsamples (6g) of Hyde Farm or Nebo test soil were placed into each of 12 Teflon centrifuge tubes, suspended in 19 ml of sterile, pesticide-free 0.1M $CaCl_2$, and then mechanically shaken for 16 hours. After pre-equilibration, ten samples of each soil type were amended with radiolabeled R234886 (cyanophenyl; SA=2.33 Gbq $mmol^{-1}$;radiopurity > 98%) to yield a nominal concentration of 0.2 µg/ml. The concentration of acetonitrile co-solvent was < 0.25%. Two samples of each soil type were not amended with to serve as a treatment controls. In addition, two soilless samples of 0.01M $CaCl_2$ containing radiolabeled R234886 at a nominal concentration of 0.2 µg/ml were used to estimate adsorption onto the Teflon centrifuge tube.. Samples were mechanically shaken for 2, 4, 6, 16, 24, and 48 hours.

Definitive Study

Subsamples (6g) of each soil type were placed into each of 22 Teflon centrifuge tubes, suspended in 19 ml of sterile, 0.01 $CaCl_2$, and then mechanically shaken for 16 hours. After pre-equilibration, four samples of each soil type were amended with radiolabeled R234886 (cyanophenyl; SA=2.33 Gbq $mmol^{-1}$;radiopurity > 98%) to yield nominal concentrations of 0.05, 0.1, 0.2, 0.4, and 0.8 µg/ml. (Reviewer Note: The water solubility of R234886 was not reported.) The remaining sample of each soil type was not amended with R234886 to serve as a treatment control. The samples were mechanically shaken for 24 hours at 20°C. After equilibration, the samples were centrifuged to separate soil and water phases. Duplicate samples of each soil type were retained for chemical analysis. The remaining samples were used in the desorption study. These samples were treated exactly as described in the adsorption study except the soil pellet of each sample was retained for the desorption study. The soil pellet was suspended in pesticide-free 0.01M $CaCl_2$ and then mechanically shaken for 16 hours at 20°C. Supernatant and soil were taken for chemical analysis.

Analytical

All treatments of the Hyde and Nebo soils and a single sample at 0.2 µg/ml for the remaining soil types were analyzed for R234886. Soil samples were sequentially extracted with acetonitrile. Supernatant and soil extracts were liquid-liquid partitioned with diethyl ether, concentrated, and then separated residues were redissolved in acetone.

Soluble radiolabeled residues were separated by 1-D TLC using a hexane:ethyl acetate:glacial acetic acid 30:70:1 (v/v/v) solvent system. Soluble residues were identified by co-chromatography with known standards. The ^{14}C content in supernatant and soil extracts was determined by LSC. The ^{14}C content in extracted soil was determined by combustion-LSC.

VII. Study Author's Conclusions

A. Preliminary studies indicate radiolabeled R234886 reached a pseudo-equilibrium or steady-state after 16 hours of mechanical shaking (Figures 5 and 6). The registrant stated R234886 was stable in 0.01M $CaCl_2$ and had a low adsorption potential on Teflon centrifuge tubes. (Reviewer Note: The registrant did not provide data to support the adsorption potential of R234886 on Teflon centrifuge.)

B. The material balance of radioactivity ranged from 89 to 120% of applied R234886 in all treatments of the Hyde and Nebo soils and at a single sample at 0.2 µg/ml of the remaining soil types (Tables 10,11, 12).

C. The transformation product R234886 was stable (>90% of applied) during the 48 hour batch equilibrium study (Figures 19, 20, 21, and 22). (Reviewer Note: The registrant provided chromatograms with a single peak as evidence of the stability of R234886. The reviewer notes the registrant did not provide a standard chromatogram to substantiate identification of R234886.)

D. Radiolabeled R234886 had Freundlich adsorption coefficients of 0.82 ml/g (K_{oc}=28; 1/n=0.90) in the Kenny Hill sandy loam soil, 0.35 ml/g (K_{oc}=21; 1/n=0.76) in the East Anglia loamy sand soil, 1.4 ml/g (K_{oc}=490;1/n=0.79) in the Lilly Field sand, 6.8 ml/g (K_{oc}=420;1/n=.90) in a Nebo silty clay loam soil,0.85 ml/g (K_{oc}=49;1/n=0.85) in the Hyde Farm sandy clay loam soil, and 10 ml/g (K_{oc}=360;1/n=0.89) in the Pickett Piece clay loam soil (Table 6).

E. The registrant classified the mobility of R234866 from medium to very high according to McCall's mobility classification scale. There was an inverse correlation between soil pH and Freundlich adsorption coefficients for R234886 (Figure 3). This inverse correlation was explained by the deprotonation (H^+ dissociation) of the carboxylic acid (pK_a=4.78) functional group of R234866 in acid soils (Figure 4). The registrant stated the adsorption coefficient of R234886 increased with O.M. content for soils of comparable pH.

F. Radiolabeled R234886 had desorption coefficients, expressed as Koc, of 36 ml/µg in the Kenny Hill sand loam soil, 34 ml/µg in the East Anglia loamy sand soil, 520 ml/µg in the Lilly Field sand, 520 ml/µg in the Nebo silty clay loam soil, 440 ml/µg in Pickett Piece clay loam soil, and 68 ml/µg in Hyde Farm sandy clay loam soil (Table 8).

VII. Reviewer's Comments

A. The USDA soil taxonomy classification of test soils was taken from MRID 4378182. EFGWB appreciates the registrant's effort to cross-reference the United Kingdom soils into USDA soil taxonomy.

B. The registrant did not provide the water solubility of R234886. EFGWB notes the water solubility of R234886 is needed to evaluate the theoretical upper bound concentration for adsorption. EFGWB believes the absence of water solubility data should not influence acceptance of the study because R234886 is expected to be mobile to very mobile (K_d< 5 or K_{oc} < 500) in terrestrial and aquatic environments.

C. The registrant provided chromatograms with a single peak as evidence of stability of R234886 during equilibration. EFGWB notes the registrant did not provide a reference chromatogram for R402173. In future studies, the registrant should provide a standard chromatogram as reference for retention time.

D. The transformation product R234886 is cross referenced as Compound 2 in registrant data submissions.

AZOXYSTROBIN

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