10-26-94

DATA EVALUATION RECORD

STUDY IDENTIFICATION:

Yen, P. Y., and S. N. Wendt. 1994. Leaching of Aged Residues of ^{14}C-Phostebupirim. Study performed and submitted by Miles, Inc., Kansas City, Missouri. Report No. 105173. MRID No. 430927-01.

TYPE OF STUDY:

Aged Leaching/Adsorption/Desorption column study (163-1)

REVIEWED BY:

George Tompkins, Ph.D. Entomologist
Review Section 1, EFGWB, EFED

Signature: George Tompkins
Date: 26 OCT 1994

APPROVED BY:

Paul J. Mastradone, Ph.D. Section Chief
Review Section 1, EFGWB, EFED

Signature: Paul J Mastradone
Date: 26 OCT 1994

CONCLUSIONS:

1. This study is satisfactory and provides information on the leaching potential of aged MAT 7484 and fulfills the aged portion of the 163-1 data requirement. This study combined with the information provided in MRID No. 42005469 and 42005470 fulfills the leaching/adsorption/desorption data requirement.

2. The reported MAT 7484 residues found in the leachates following leaching were 0.5% from the silty clay loam soil, 1.4% from the silt loam soil, 3.7% from the sandy loam soil, and 5.8% from the sand soil. Parent MAT 7484 was not identified in the leachates. The metabolites TBHP (0.33-0.40% of applied radioactivity) and OMAT (0.62-0.66%) were identified. Two other polar metabolites were not identified by GC/MS because of insufficient quantities.

3. The main component in the soil columns was parent ^{14}C-phostebupirim. Four metabolites were detected (I-IV). Metabolites TBHP ($\leq$1.5% of applied radioactivity) and OMAT ($\leq$2.5%) were identified and insufficient quantities of the other two metabolites were in the soil extracts to analyze by GC/MS. The average recovered radioactivity from the columns after leaching ranged from 96.5% to 103.0% of the applied radioactivity. The majority of this radioactivity remained in the top 6 cm of the columns (81.6% in the sandy loam soil, 82.1% in the sand soil, 85.6% in the silt loam, and 90.0% in the silty clay loam soil). The above information indicates that phostebupirim and its metabolites were not very mobile in the soils tested.

MATERIALS AND METHODS:

Four soils (Indiana sandy loam, Florida sand, Illinois silt loam, and a Mississippi silty clay loam, see Table 3 for characteristics) were air dried and sieved (2 mm) prior to use. The sandy loam soil was selected for aging phostebupirim and microbial activity was determined prior to application (3.6×10^5 colony forming units per gram of soil). The phostebupirim used in this study had a specific activity of 68 uCi/mg and a radiochemical purity of 98.4%. Based on a maximum application rate of 0.084 ppm (0.15 lb ai/A), and a cross sectional area (19.6 cm^2) of the glass columns (60 cm long, inside diameter 5.0 cm), the application rate of phostebupirim for each column was 0.34 mg/column. Approximately 800 g of a sandy loam soil was treated with ^{14}C-phostebupirim. Prior to incubation five sub-samples of about 0.2 g of each were oxidized to determine the uniformity of application and two 25-g sub-samples of the treated soil were extracted and analyzed to verify the application rate (1.23 ug phostebupirim per g of treated soil). The treated soil was maintained at 75% of 1/3 bar of moisture (10.4% soil moisture content) and transferred into a reaction vessel covered with aluminum foil. The soil was incubated under aerobic conditions at approximately 22^{0}C in a flow through air system for 30 days (Fig. 2). All traps were changed and analyzed at 15 days and 30 days post-application. Sub-samples (30.6 g) of the aged soil contained an equivalent of 0.034 mg (2.31 uCi) of ^{14}C-phostebupirim residues were weighed out into individual glass storage jars and stored at -22^{0}C until the soil columns were prepared for leaching.

The bottom of the glass column was threaded to fit a teflon end piece equipped with a flow regulator valve. Each soil column was packed with 100 g of washed sea sand. On top of the sandy layer each column was packed with the dried, sieved soil to a height of 30 cm above the sand layer. Bulk density of each packed column was calculated (Table 4). Each column was initially saturated (from bottom to top) with 0.01 M aqueous $CaCl_2$ solution. A glass filter paper was placed on top of the saturated column, and 30.6 g of aged soil was added on top of it. Each column was then leached under saturated conditions with about 1000 ml of $CaCl_2$ solution (equal to approximately 20 inches of rainfall) for 48-72 hours. The flow rate through the column was manually adjusted to maintain an average positive constant pressure. At the end of the leaching process, the total volume of the leachates was divided into approximately equal (200 ml) sequential fractions. The amount of radioactivity collected in each fraction was determined by LSC. The leachate fractions were extracted with ethyl acetate (3 x 100 ml). The combined ethyl acetate and aqueous phases were concentrated and analyzed by HPLC.

After leaching, the soil columns were frozen at approximately -22^{0}C to facilitate removal of the intact soil cores from the glass columns. The bottom end of frozen soil was

pushed out of the glass column first. The glass columns were rinsed with methanol and the radioactivity in the column rinses was determined by LSC. The soil core was sectioned into five 6-cm segments plus the aged soil and sand layers. Each soil segment was air dried, homogenized, and weighed. The distribution of aged ^{14}C-phostebupirim residues within the soil column was determined by oxidizing triplicate aliquots of each segment. Soil segments containing greater than 10 ppb of aged ^{14}C-phostebupirim residues were extracted twice for 3 hr with methanol. A soil to solution ratio of about 1:4 was maintained. After solvent extraction, the soil samples were refluxed for approximately 3 h with acetonitrile. All filtrates were combined and evaporated to 1 ml and analyzed by HPLC and TLC. The TLC data was used to verify the HPLC results. Mass spectral (GC/MS) analysis was used for confirmation.

REPORTED RESULTS:

1. After 30 days aerobic incubation 5.7% of the applied radioactivity was detected as ^{14}C-volatiles, 84.5% as parent ^{14}C-phostebupirim, 6.5% as four ^{14}C-metabolites, and 5.1% as ^{14}C-bound residues (Table 6).

2. The reported radioactivity (^{14}C-phostebupirim residues) found in the leachate following leaching was 0.5% in the silty clay loam soil, 1.4% in the silt loam soil, 5.8% in the sand soil, and 3.7% in the sandy loam soil. Parent phostebupirim was not detected in the leachates. Four minor metabolites were detected in the leachates. Two of the four metabolites were identified as TBHP (0.33-0.40% of applied radioactivity) and OMAT (0.62-0.66%). Metabolite identification of the other two polar unidentified metabolites was not possible by GC/MS due to insufficient quantities.

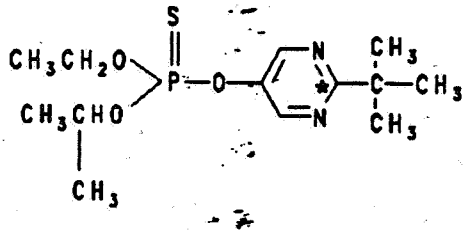
3. The average recovered radioactivity from the soil columns after leaching ranged from 96.5% to 103.0% of the applied radioactivity (Table 7, Appendix 6). The majority of this radioactivity remained in the 6 cm of the columns which were an average of 81.6% in the sandy loam soil columns, 82.1% in the sand soil columns, 85.6% in the silt loam soil columns, and 90.0% in the silty clay loam columns.

4. The main component of the aged ^{14}C-residues in the soil columns was parent ^{14}C-phostebupirim. Four metabolites were detected (I-IV). Metabolite II was identified as TBHP ($\leq$1.5%) and metabolite III was OMAT ($\leq$2.5%). Insufficient quantities of Metabolites I and IV were in the soil extracts to analyze by GC/MS.

DISCUSSION:

1. Although TLC data was used to verify the HPLC results, the solvent system(s) used for the TLC was(were) not specified.

Table 1. Identity, physical and chemical properties of active ingredient.

Proposed Common Name:	Phostebupirim
Chemical Name (CAS):	O-[2-(1,1-dimethylethyl)-5-pyrimidinyl] O-ethyl O-(methylethyl)phosphorothioate.
Structural Formula:	

* position of radiolabel carbon

CAS-No:	96182-53-5
Molecular Weight:	318.4
Appearance and Form:	Colorless liquid (MR 100610)[5]
Melting Point:	Does not apply (MR 100610)[5]
Boiling Point:	152°C at standard pressure (MR 100604)[6]
Vapor Pressure:	5×10^{-5} hPa @ 20°C; 1×10^{-4} hPa @ 25°C (MR 90477)[7]
Relative Density:	1.10 g/mL @ 20°C (MR 100606)[8]
pH:	5.83 in a 2% suspension of test substance in a solution of 0.1% of NaCl in water (MR 100608)[9]
Octanol/Water Partition Coefficient:	85,000 @ 22°C (MR 99607)[10]
Water solubility:	5.5 mg/L in neutral water @ 20°C (MR 99606)[11]
Solubility:	Completely miscible @ 20°C with n-hexane, toluene, dichloromethane, 2-propanol, polyethyleneglycol, octanol, acetone, dimethylformamide, acetonitrile, and dimethylsulfoxide. (MR 100609)[12]

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