2-5-93

DATA EVALUATION RECORD

STUDY 5

CHEM 128101 RH-5287 §163-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41845005
Olson, G.L. and L.J. Lawrence. 1991. Leaching of $^{13/14}$CRH-5287 in four soil types following 30 days or one half-life of aerobic aging in sandy loam soil. Laboratory Project ID: PTRL Report No. 1296, PTRL Project No. 297, Rohm and Haas Technical Report No. 34-90-67. Unpublished study performed by PTRL East, Inc. and submitted by Rohm and Haas Company, Spring House, PA.

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CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

1. This soil column leaching is acceptable and partially satisfies 163-1 data requirements by providing information on the mobility of aged residues of RH-5287 in four different soils. However, data are not available on the mobility of aged RH-5287 in an aquatic sediment.

2. Aged (21 days) RH-5287 residues were immobile in columns of sand, sandy loam, silt loam, and silty clay loam soils; between 80.2 and 100.4% of the applied [^{14}C]residues remained in the upper 6 cm of the

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soil columns. RH-5287 was the only compound identified in the soil fractions.

3. An aquatic sediment should have been included in the study. This was recommended in EFGWB review #90498 (8/28/89).

4. The requirement for data on the mobility of unaged RH-5287 has also been satisfied (Study 4; 41845004).

5. The registrant must provide an explanation to the discrepancy in half-lives of aerobic incubation (soil) in this study (calculated as 21 days) and those observed in anaerobic and aerobic aquatic sediments (162-3/162-4 studies), which were reported as being less than one-day. The explanation should also extend to the nature of degradates, since the major residue (greater than 45%) was the parent compound in the soil column leading study.

METHODOLOGY:

Samples (50 g) of sandy loam soil (60% sand, 30% silt, 10% clay, 3.2% organic matter, pH 7.6, CEC 13 meq/100 g) were weighed into separate 500 mL flasks and treated at 0.5 ppm with [^{14}C]RH-5287 (radiochemical purity >99%, specific activity 55.39 mCi/g, Rohm and Haas), dissolved in acetonitrile. The solvent was evaporated and the soil moisture was adjusted to 75% field capacity with deionized water. The flasks were stoppered, and the soil was aged for 21 days in the dark at 24.8 $\pm$ 1 C. At intermediate periods (10 or 11 days), and 21 days posttreatment, the flasks were flushed with oxygen and the exhaust gases were vented through ethylene glycol or 10% potassium hydroxide trapping solutions. At the time of flushing, the flasks were weighed and soil moisture was maintained at 75% of field capacity by adding water. Two flasks of treated soil were used to characterize [^{14}C]residues in the soil after 0 and 21 days of aging.

Portions of untreated sand, sandy loam, silt loam, and silty clay loam soils were air-dried and sieved (2 mm). The soils were packed into duplicate polyvinylchloride columns (2-cm id) that had been covered at the bottom with six layers of gauze. The moisture content of the soil was adjusted to 75% of field capacity by placing the columns in a container of 0.01 M calcium chloride, then allowing the saturated columns to drain.

Following the 21-day aging period, the aged treated soil (50 g) was placed on the surface of the soil columns (two columns per soil type) and covered with a filter paper disk; the final length of the soil columns was 30 cm. The flasks that held the treated soil were rinsed with 0.01 M calcium chloride, and the rinsate was applied to the columns. The columns were then leached with 0.01 M calcium chloride to bring the total application to 1030 mL, equivalent to 20 inches (50.8 cm). The time required to leach the columns with 20 inches of water was 2.8 hours for the sand soil, 17 hours for the sandy loam

soil, 49-55 hours for the silt loam soil, and 7.8-8 hours for the silty clay loam soil. The leachates were collected in 250-mL fractions, which were stored at 4 C until analysis (length of storage not reported). Following leaching, each soil column was cut into five 6-cm segments, which were placed in plastic resealable bags and stored at -20 C prior to analysis (length of storage not reported).

Aliquots of the leachates were analyzed for total radioactivity by LSC. Each soil section was thawed at room temperature and mixed thoroughly. Three subsamples (500 mg) were analyzed for total radioactivity by LSC following combustion; the remaining soil was refrozen at -20 C. The 0-6 cm depth from each soil column (which were the only fractions containing $\geq$10% of the applied radioactivity) was thawed and extracted three times with methylene chloride:methanol (1:1, v:v) by shaking; the first and third extractions were shaken for 30 minutes each and the second extraction was shaken overnight. Following each extraction, the samples were centrifuged for 10 minutes, and the supernatant decanted. Following the third extraction, the supernatants were combined and analyzed for total radioactivity by LSC. The extracts were concentrated under nitrogen and analyzed with HPLC on a reverse-phase L-18 column eluted with a methanol:water gradient (75:25 to 100:0) and with UV/VIS (220 nm) and radioactivity detection. The extracted soils were further extracted with methanol by shaking for 20 minutes, followed by centrifuging for 10 minutes; the supernatants were analyzed for total radioactivity by LSC.

Further extractions were performed on several of the 0- to 6-cm soil segments in an attempt to release additional radioactivity. The 0- to 6-cm segment from one sandy loam soil column was Soxhlet-extracted with methanol for 24 hours; the methanol extract was analyzed by HPLC (described previously). The 0- to 6-cm segments from the second sandy loam soil column and one silty clay loam soil column were extracted with 0.1 N sodium hydroxide by shaking overnight. The samples were centrifuged for 10 minutes, and the supernatants decanted and analyzed by LSC. The extracted soils were further extracted with 1 N sodium hydroxide by shaking for 20 minutes, centrifuged for 10 minutes, and the supernatant decanted and analyzed by LSC. The soils were then extracted with 1 N sodium hydroxide by shaking for 5 hours, centrifuged for 10 minutes, and the supernatants analyzed by LSC. All remaining soil column segments were extracted with 1 N sodium hydroxide by shaking overnight; the samples were centrifuged for 10 minutes and the supernatants were analyzed by LSC. The extracted soils were air-dried and analyzed for unextracted radioactivity using LSC following combustion.

The 1 N sodium hydroxide extracts of several soil samples were further analyzed in an attempt to characterize the radioactivity. The 1 N sodium hydroxide extract from a replicate of each soil type was acidified with concentrated hydrochloric acid to pH 2.5-3.0. The supernatants (fulvic acid) were partitioned with methylene chloride and each fraction was analyzed by LSC. The aqueous fraction was

neutralized to approximately pH 7 with 4 N sodium hydroxide and the procedure was repeated. The neutralized aqueous fraction was made basic (pH 10-11) with 4 N sodium hydroxide and the procedure was repeated.

The ethylene glycol and 10% potassium hydroxide trapping solutions from the aging phase of the study were analyzed for total radioactivity by LSC.

DATA SUMMARY:

Based on column leaching studies, aged (21 days) [^{14}C]RH-5287 residues were immobile in columns (30-cm length) of sand, sandy loam, silt loam, and silty clay loam soils that were leached with 20 inches of water. Prior to leaching, the columns had been topped with 50 g of soil that had been treated with 0.5 ppm of [^{14}C]RH-5287 (radiochemical purity >99%) and aged aerobically for 21 days. [^{14}C]Residues in the soil columns remained in the upper 6 cm, and ranged from 80.2 to 100.4% of the applied (Tables X-XIII). Of the remaining radioactivity, $\leq$0.9% of the applied was in the leachates and 11.6-19.9% of the applied was [^{14}C]volatiles. Following leaching, material balances for the four soil columns plus leachates and volatiles were 93.0-115.5% of the applied (Tables X-XIII).

In the treated soils after 21 days of aging and prior to leaching, 30.5-47.3% of the applied was RH-5287 and 0.9-1.4% was unidentified ("Unknown") (Table IX). Following leaching, RH-5287 was the only compound identified in the upper 6 cm of the four soils, comprising 47.0-64.6% of the applied (Table XVI). An additional 14.5-32.7% of the applied radioactivity was extracted from the surface soil segments with sodium hydroxide and up to 6.1% was extracted with methanol (residues not characterized); 21.5-42.6% was unextracted (Table XIV). Based on further analyses of the sodium hydroxide solutions of each soil type, 7.0-11.6% was in the fulvic acid, 2.1-6.8% was in the humin, and 0.1-3.3% was in the humic acid (Table XV).

COMMENTS:

1. Based on the results of a preliminary study, the half-life of RH-5287 was calculated to be 21.2 days. The study authors stated that subsequent reinterpretation of the data indicated that the actual half-life was 34.9 days. The authors concluded that although the definitive study was conducted using the shorter aging interval of 21 days, the results should not be affected since there was approximately 50% parent in the soil after 21 days of aging. However, based on the data provided in Table IX, RH-5287 comprised only 30.5% of the applied in the sand and sandy loam soils, and 47.3% of the applied in the silt loam and silty clay loam soils, following 21 days of aging. However, since Subdivision N guidelines require that only one treated soil be aged under aerobic conditions for 30

days or one half-life, (whichever comes first), and approximately 50% parent remained in the silt loam and silty clay loam soils after 21 days of aging, these data are considered adequate.

2. The study authors stated that distribution coefficients for the four soil types could not be calculated since there was no appreciable movement of [^{14}C]residues through the columns into the leachates.

3. Recovery efficiencies and method detection limits for fortified soil samples were not reported.

4. An aquatic sediment should have also been included.

5. Discrepancies in half-lives of parent and nature of degradates in soils incubated under aerobic conditions and in aquatic sediments were noted, but no explained.

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