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DATA EVALUATION RECORD 1

CHEM 123000 Isoxaflutole §163-1
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STUDY ID 43940301
Burr, C.M. 1995. RPA 202248: Adsorption/desorption to and from four soils. Laboratory Project ID: P 95/062. Unpublished study performed by Rhône-Poulenc Agriculture Limited, Essex, ENGLAND, and submitted by Rhône-Poulenc Ag. Co., Essex, ENGLAND.

STUDY ID 44065801
Burr, C.M. 1996. [^{14}C]-RPA 202248: Adsorption/desorption to and from four soils and an aquatic sediment. Laboratory Project ID 11486. Unpublished study performed by Rhône-Poulenc Agriculture Limited, Essex, ENGLAND, and submitted by Rhône-Poulenc Ag. Co., Essex, ENGLAND.

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CONCLUSIONS

Mobility - Leaching and Adsorption/Desorption

1. These studies are acceptable, and partially satisfy the aged portion of the 163-1 data requirement for isoxaflutole. The 163-1 data requirement is partially satisfied with the combination of this study and MRID's 43588008 and 43588009, reviewed on 6/29/95. These studies provide information on the adsorption and desorption of the isoxaflutole for the metabolite RPA 202248 in five U.S. and three U.K. soils and in one U.S. aquatic sediment. To fully satisfy the 163-1 data requirement, the registrant should conduct a batch equilibrium study for RPA 203328.

2. In the first study (MRID 43940301), the isoxaflutole metabolite [^{14}C]RPA 202248, at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L, was very mobile in loam (U.K.), loamy sand (U.K.) and two sandy loam (one U.S., one U.K.) soil:solution slurries (1:5, w:v) that were equilibrated in darkness at 20 ± 2°C for 72 hours. Freundlich K_{ads} values were 2.54 for the loam soil, 0.63 for the loamy sand soil, and 0.71 and 2.18 ml/g, respectively, for the U.S. and U.K. sandy loam soils. K_{oc} values for the four soils were 134, 57, 54 and 99 ml/g, respectively. Respective K_{des} values for the loam, loamy sand and two sandy loam soils were 3.99, 0.77, 1.27 and 3.50, with associated K_{oc} values of 210, 70, 97 and 159 ml/g, respectively. The 1/n values ranged from 0.89-0.97.

In the second study (MRID 44065801), [^{14}C]RPA 202248, at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L, was very mobile in clay, sand, loamy sand and silt loam soil:solution slurries and loam sediment:solution slurries (1:5, w:v) that were equilibrated in darkness at 20 ± 2°C for 72 hours. Freundlich K_{ads} values for the sand, loamy sand and silt loam soils were 0.44, 0.54 and 0.70 ml/g, with associated K_{oc} values of 117, 159 and 149 ml/g, respectively. Freundlich K_{ads} values for the clay soil and the high organic carbon (5.08%) loam sediment were 1.15 and 6.71, with associated K_{oc} values of 94 and 135 ml/g, respectively. Freundlich K_{des} values were 1.10, 1.62 and 1.56 ml/g for the sand, loamy sand and silt loam soils, and were 1.30 and 8.96 ml/g for the clay soil and loam sediment, respectively. Values of K_{oc} associated with desorption were 293, 474 and 333 ml/g, respectively, for the sand, loamy sand and silt loam soils; values for the clay soil and loam sediment were 106 and 180 ml/g, respectively. The 1/n values ranged from 0.81-0.96.

METHODOLOGY

This study (inclusive of two MRID's referred to here as "Study One" and "Study Two" for purposes of clarification) was conducted to determine the adsorption/desorption properties of the isoxaflutole metabolite RPA 202248 (2-cyano-3-cyclopropyl-1-(2-methylsulphonyl-4-trifluoromethylphenyl)propan-1,3-dione). Study One evaluated the mobility of RPA 202248 in three U.K. soils and one U.S. soil; Study Two evaluated the mobility of RPA 202248 in four additional U.S. soils and one aquatic sediment (U.S.). A prior study (MRID 43588008) was used to partially fulfill the data requirement on the mobility of isoxaflutole by providing data on the leaching of the parent compound and degradates in soil columns.

Study One

Duplicate subsamples (15-g oven-dried equivalent wt.) of loam soil (U.K.), loamy sand soil (U.K.) and two sandy loam soils (one U.S. and one U.K. soil) (MRID 43940301; Table 7.1, p. 25) were partially air dried, sieved to 2 mm, weighed into acid-washed borosilicate glass tubes and dosed with 75 mL of 0.01 M $CaCl_2$ solutions containing uniformly ring-labeled [^{14}C]RPA 202248 (radiochemical purity 97.3%, specific activity 909.1 MBq/mmol) at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L. Tubes containing the 1:5 (w:v) soil:solution slurries were shaken by hand then placed on an end-over-end shaker and maintained in the dark at 20 ± 2°C. Following 72 hours (determined in a preliminary study) of equilibration, all tubes were removed from the shaker and centrifuged for 15 minutes at 1500 rpm. The supernatant (adsorbate) was decanted and triplicate aliquots of the adsorbate were mixed with 10 mL Aqualuma-plus scintillation cocktail and analyzed by liquid scintillation counting (LSC) for total radioactivity. No representative soil samples were analyzed by combustion following adsorption.

For the desorption phase of the experiment, 75 mL of pesticide-free 0.01 M $CaCl_2$ solution was added to the tubes with the soil from the adsorption phase of the experiment. The tubes were shaken for 24 hours (determined in a preliminary study using only two of the four soils, p. 14) on an end-over-end shaker and maintained in the dark at 20°C. Tubes were centrifuged and the supernatant (desorbate) was analyzed for total radioactivity as described for the adsorption phase. This procedure was repeated four times in order to obtain five desorbates for each soil.

Following desorption, single replicates of soil representing the high-dose level for each of the four soils was extracted by shaking for 20 minutes in 50 mL of acetonitrile:water (1:1, v:v). Tubes were centrifuged for 10 minutes and the supernatant was decanted and analyzed in triplicate by LSC. All soil samples (the extracted samples plus all unextracted samples) were then air dried, ground to a fine powder. Triplicate subsamples of soil were combusted and radioactivity was determined using LSC.

To confirm the stability of RPA 202248 during the experiment, aliquots of selected adsorbates and desorbates were analyzed by HPLC using an Ultrabase C_8 column with a mobile phase of 45:55 acetonitrile:water (v:v) plus 0.5% trifluoroacetic acid, and both UV (275 nm) and radio (β-ram) detection.

Study Two

Duplicate subsamples (15 g oven-dried equivalent wt.) of air dried, sieved (2 mm) clay, sand, loamy sand and silt loam soils and loam sediment (MRID 44065801; Table 7.1, p. 27) were weighed into acid-washed borosilicate glass tubes and dosed with 75 mL of 0.01 M $CaCl_2$ solutions containing uniformly ring-labeled [^{14}C]RPA 202248 (radiochemical purity 97.3%, specific activity 909.1 MBq/mmol) at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L. Tubes containing the 1:5 (w:v) soil:solution and sediment:solution slurries were shaken during a 72-hour equilibration period using an end-over-end shaker maintained in the dark at 20 ± 2°C. Following equilibration, all tubes were removed from the shaker and centrifuged for 10 minutes at 1500 rpm. The supernatant (adsorbate) was decanted and triplicate aliquots of the adsorbate were mixed with 10 mL Starscint scintillation cocktail and analyzed by LSC for total radioactivity. No representative soil samples were analyzed by combustion following adsorption.

For the desorption phase of the experiment, 75 mL of pesticide-free 0.01 M $CaCl_2$ solution was added to the tubes containing the soil from the adsorption phase of the experiment. The tubes were shaken for 24 hours on an end-over-end shaker and maintained in the dark at 20°C. Tubes were centrifuged and the supernatant (desorbate) was analyzed for total radioactivity as described for the adsorption phase. This procedure was repeated four times in order to obtain five desorbates for each soil. Some soil:solution slurries were not shaken for the entire equilibration period of the third cycle due to equipment failure (See Comment 4).

Following desorption, single replicates of soil representing the high-dose level for each of the four soils was extracted by shaking for 20 minutes in 75 mL of acetonitrile:water (1:1, v:v). Tubes were centrifuged for 10 minutes and the supernatant was decanted and analyzed in triplicate by LSC. All soil samples (the extracted samples plus all unextracted samples) were then air dried, ground to a "fine powder" and analyzed by combustion; radioactivity was determined using LSC.

To confirm the stability of RPA 202248 during the experiment, aliquots of selected adsorbates and desorbates were analyzed by HPLC using a Kromasil KR100 5C8 column with a mobile phase of 50:50 acetonitrile:water (v:v) plus 0.5% trifluoroacetic acid, and both UV (275 nm) and radio (β-ram) detection.

DATA SUMMARY

Study One

Uniformly ring-labeled isoxaflutole metabolite [^{14}C]RPA 202248 (radiochemical purity 97.3%), at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L, was very mobile in loam, loamy sand and two sandy loam soil:solution slurries (1:5, w:v) that were equilibrated in the dark at 20 ± 2°C for 72 hours. Freundlich K_{ads} values were 2.54 for the loam soil containing 1.9% OC; 0.63 for the loamy sand soil containing 1.1% OC; and 0.71 and 2.18 ml/g, respectively, for the U.S. and U.K. sandy loam soils containing 1.3 and 2.2% OC, respectively (Table 7.5, p. 27). Respective K_{oc} values associated with the adsorption coefficients were 134, 57, 54, and 99 ml/g. Freundlich K_{des} values determined following one equilibration period (24 hours) were 3.99 for the loam soil, 0.77 for the loamy sand soil, and 1.27 and 3.50 ml/g for the U.S. and U.K. sandy loam soils, respectively (MRID 43940301; Table 7.6, p. 27). Respective K_{oc} values associated with the desorption coefficients obtained following 24 hours of equilibration were 210, 70, 97, and 159 ml/g. The registrant also reported desorption coefficients associated with the four 24-hour equilibration periods following the initial desorption period. The 1/n values were 0.93, 0.97, 0.92 and 0.93 for adsorption and 0.93, 0.93, 0.89 and 0.92 for desorption, respectively.

A slight degradation of [^{14}C]RPA 202248 in solution occurred during the experiment (Table 7.15, p. 37).

Material balances for all soil:solution systems ranged from approximately 98 to 102% of the applied (p. 20).

Study Two

Uniformly ring-labeled isoxaflutole metabolite [^{14}C]RPA 202248 (radiochemical purity 97.3%), at nominal concentrations of 5, 1, 0.2 or 0.04 mg/L, was very mobile in clay, sand, loamy sand and silt loam soil:solution slurries and loam sediment:solution slurries (1:5, w:v) that were equilibrated in the dark at 20 ± 2°C for 72 hours. Freundlich K_{ads} values for the sand, loamy sand and silt loam soils containing less than 1% OC were 0.44, 0.54 and 0.70, respectively; associated K_{oc} values were 117, 159 and 149 ml/g, respectively (MRID 44065801; Table 7.5, p. 29). Freundlich K_{ads} values for the clay soil (1.25% OC) and the high organic carbon (5.08%) loam sediment were 1.15 and 6.71, respectively; associated K_{oc} values were 94 and 135 ml/g, respectively (MRID 44065801; Table 7.5, p. 29). Freundlich K_{des} values determined following one equilibration period (24 hours) were 1.10, 1.62 and 1.56 for the sand, loamy sand and silt loam soils, respectively, and were 1.30 and 8.96 ml/g for the clay soil and loam sediment, respectively (Table 7.6, p. 29). Values of K_{oc} associated with the desorption phase were 293, 474 and 333, respectively, for the sand, loamy sand and silt loam soils; values for the clay soil and loam sediment were 106 and 180 ml/g, respectively. The registrant also reported desorption coefficients associated with the four 24-hour equilibration periods following the initial desorption period. The respective 1/n values associated with the adsorption of RPA 202248 to sand, loamy sand, silt loam and clay soils were 0.95, 0.96, 0.95 and 0.92; the 1/n value for the loam sediment was 0.89. The respective 1/n values associated with the desorption of RPA 202248 from sand, loamy sand, silt loam and clay soils were 0.91, 0.94, 0.91 and 0.81; the 1/n value for the loam sediment was 0.89.

A slight degradation (<5%) of [^{14}C]RPA 202248 in solution occurred during the experiment (MRID 44065801; Table 7.17, p. 41).

Material balances for all soil:solution and sediment:solution systems ranged from approximately 94 to 100% of the applied with the exception of one soil:solution slurry representing the low dose with loamy sand soil system (MRID 44065801, p. 22). Due to a recovery of 148.23% which indicated possible misdosing, data from that sample were not used to calculate coefficients (MRID 44065801, pp. 22, 42).

COMMENTS

1. In the first study, initial results indicated that the radiolabeled metabolite was being adsorbed to the glass tubes, as only 91.6% of the applied dose was recovered following 24 hours of shaking (MRID 43940301, p. 17). To alleviate this problem, all tubes were acid washed prior to use in the adsorption phase. This amended procedure allowed for an approximately 102% recovery of applied radioactivity.

2. Soils from the adsorption phase were not analyzed by combustion in either study to confirm that the adsorption to soil occurred and to provide a complete material balance for the adsorption phase of the studies. It is probable that the amended procedure (i.e., using acid-washed glass tubes) precluded the adsorption of the test chemical to the sides of the glass tubes. Mean recoveries indicated that significant material losses did not occur overall.

3. In Study One, the determination of the desorption equilibration period was performed using only two of the four test soils, the loam and U.K. sandy loam which had higher organic carbon contents than the two soils not included in the preliminary desorption experiment.

4. In Study Two, desorption data on the loamy sand soil (95/06) for the 3rd desorption step was not determined due to equipment failure. However, this had no adverse impact on the data.

5. In each study, HPLC data indicated that RPA 202248 degraded slightly during the adsorption and desorption phases of the experiment. At the equilibrium times (72 or 24 hours, respectively), however, less than 5% degradation was observed (Table 7.3: MRID 43940301, p. 26; MRID 44065801, p. 28).

6. No microbial data were included for the three foreign soils, as necessary to indicate their similarity to U.S. soils.

7. The U.K. soil No. 95/02 is referred to in the study as a clay-loam soil based on the ADAS textural classification system. The USDA classification of "loam" is used in this review.

8. The qualitative classifications of soil mobility reported in the data summary were determined by the reviewer using "Table III: The general relationship between the soil/solution partition coefficient K, R_f, and soil mobility" (*Federal Register*, Vol. 44, No. 53) and are based on the K_{ads} values reported by the registrant. The qualitative determinations reported by the registrant in the form of a McCall mobility class were based on the K_{oc} values associated with the adsorption phase of the experiment; these class determinations were not reported by the reviewer. While the K_{oc} is useful in determining which soil components (organic vs. mineral) have the greatest effects on pesticide mobility, the level of organic matter in a system varies with time of sampling and, thus, may lead to different results with regard to the mobility class of the pesticide. It is generally more acceptable to report the mobility class based on K_{ads} values.