DATA EVALUATION RECORD

STUDY 5

CHEM 129016 XRD-498 §162-1

STUDY ID 41931732

Havens, P.L. and Miller, J.R. AEROBIC SOIL METABOLISM OF $_{14}$C-(ANILINE)-DE-498 IN TWO SOILS. Preformed and Submitted by DowElanco; Midland, MI under Laboratory Project ID ENV91006.00; Study completed on 7 June 1991; Received by EPA 19 June 1991.

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CONCLUSIONS:

The aerobic soil metabolism study is marginally acceptable to fulfill the data requirement for EUP only. However, the study is not acceptable to meet Subdivision N Data Requirement for the following reason:

In order to fully understand the environmental fate, EFGWB requires that the pattern of formation and decline of degradates and XRD-498 be explained.

A new aerobic soil metabolism study is required which fully addresses the pattern of formation and decline of degradates and XRD-498. A higher application rate or lower detection limit will probably be required in order to determine the degradation process in the new studies.

This study is of the [^{14}C-aniline]XRD-498 material which had a half-live of two months when applied to sandy clay loam soil and silt loam soil at 25 ± 1°C and 75% of 1/3 bar moisture. Two degradates were reported to be present in small quantities (not >0.08% of applied)(See Tables VII and VIII). The average material balances were 95.27% and 93.67% of applied for the sandy clay loam and silt loam, respectively. The [^{14}C-pyrimidine]XRD-498 material was discussed in a previous review (WGM;06/22/90) which has very similar reported results.

MATERIALS AND METHODS:

Test Material: [^{14}C-aniline]XRD-498 was used which was reported to have a specific activity of 28.0 mCi/mmol

An unlabeled standard was obtained from DowElanco for analytical purposes. The purity of this standard was 99.6%.

Primary Stock Solution: Stock solution was prepared by dissolving 52 µCi of the material in 1 mL of acetone.

Stock Solution: A 250μL aliquot of the primary stock solution was diluted to 10 mL with a 80/20 mixture of acetone/0.005M NH_4OH. This solution was found to be 96%+ radiochemically pure.

Soil: See Table 1 for soil characterization.

Sampling: 0, day 3, day 7, day 14, day 28, day 56, day 101 or day 108.

Test System: See Figure 4.

METHODOLOGY:

Clay loam and silt loam soils were sieved through a 2 mm sieve. Fifty-gram (oven-dry basis) quantities of soil were weighed into biometric flasks an 105μL of stock was added dropwise over the soil surface in each biometer flask yielding a soil concentration of 0.07 ppm on an oven-dry basis. The soils were then thoroughly mixed with a spatula and an appropriate volume of sterile glass-distilled water was added to bring the soils to 75% of 1/3 bar moisture tension. The soils were again mixed and 100 mL of 0.2N NaOH was added to the caustic trap compartment. The flasks were the sealed with stopcock grease, the expansion bulbs were installed, and then the flasks were attached to the oxygen manifold at 25 ± 1°C. Time zero samples were prepared as above and were immediately sacrificed. Homogeneity and repeatability of the spiking solution was confirmed by taking aliquots of solution after spiking every fifth sample.

Sampling intervals were zero, 3 days 1, 2, 4, 8, and 14 weeks. Additional samples aged for 32 and 52 weeks will be sacrificed after the completion of this report and may be reported in a revision or addendum.

At each sampling interval, the caustic traps were removed to a vial and triplicate 500 μL aliquots were counted by LSC to measure mineralization. The soil samples were placed in glass containers and a small sample was taken for moisture determination.

Five grams of soil were transferred to tared 50 mL centrifuge tubes for extraction. Initial organic solvent extractions were carried out using 15 mL of 90% acetone/10% 0.1N HCl. After addition of the solvent, samples were vortexed, sonicated, and vortexed again. This was followed by centrifugation and pipetting of an aliquot of extract to empty tubes. The weighing of the samples was recorded at each step.

Following extraction, triplicate aliquots of the acetone extracts were counted by LSC. A portion of each extract was then partially evaporated (by about 90%) and reconstituted, to its original volume with 0.005N HCl. One mL aliquots were injected into the HPLC for reuse-phase analysis. Mobile phase A was water acetonitrile + 0.5% acetic acid and phase B was water + 0.5% + 0.5% glacial acetic acid. Both phases also contained 0.05% n,n-dimethyloctylamine to improve chromatography. Minute fractions were collected using a fraction collector, and individual fractions were counted by LSC.

The extracted soils were allowed to air dry in their tubes before being extracted with 0.5N NaOH. The combined extractions were brought to 50 mL total volume and triplicate 0.5 mL aliquots were analyzed by LSC. Those caustic extracts containing >10% of applied radioactivity were further analyzed. The soil remaining after exhaustive extraction was oven dried and placed in a combustion unit to complete radiocarbon analysis.

DATA SUMMARY:

Based on initial rate data, XRD-498 degrades with an apparent first order half-life of 27 days when applied to sandy clay loam soil and 42 days when applied to silt loam soil. However, the degradation of XRD-498 exhibits multiphasic behavior with simple first order curve points possibly neglecting the importance of initial points along the degradation curve. The authors believe that initial rate data may be more indicative of the degradation rate of bioavailable XRD-498. The two most prominent metabolites of

XRD-498 had a maximum concentration of 6 to 8% of applied radioactivity at day 56. Therefore, based on their being <10% of applied and <0.01 ppm, they were not identified.

Material balances averaged 95.27% of applied radioactivity for sandy clay loam soil and 93.67% of applied radioactivity for silt loam soil. The material balances were reported to range from 89.23 to 98.60%.

COMMENTS:

1. In a previous storage stability experiment indicated that XRD-498 was stable for up to 8 months in frozen acetone:acetic acid:water extract (Table VII). Soil samples were extracted by "hand" by shaking with acetone:acetic acid:water for 1 hour. Then after 6-8 months of frozen storage, the same soil samples were extracted by vortexing for 5 minutes using a robot (the extraction procedure used in the actual study). This study a few samples were frozen prior to analysis. Their stability was shown by reextracting and analyzing the day zero samples after 24 days of frozen storage. The percentage of XRD-498 in these samples was found to average 94.4 ± 1.8% of applied.

2. The registrant attempted to characterize radioactivity in the caustic extracts (fulvic/humic acid fractions). The caustic extracts containing >10% of applied radioactivity were neutralized to ≈ pH 5.8N HCl to precipitate the humic soil organic matter fraction. The supernatant remaining was the fulvic soil organic matter fraction. The fulvic portion was counted by LSC to determine the partitioning between the two soils organic matter compartments. Some of the fulvic extract samples were analyzed by HPLC, as well.

3. Assuming first order degradation kinetics, the registrant calculated two half-lives for each soil. A half-life using data for all sampling intervals, and a first order half-life based on initial rate data. Data for all the sampling intervals did not readily conform to first order kinetics. The authors presented the half-life based on initial rate data in the study which they felt was most representative of bioavailable XRD-498.

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