DATA EVALUATION RECORD

STUDY 8

CHEM 129016 XRD-498 §165-1

STUDY ID 41931739

Hamburg, A.W., Byrne, S.L., and Harding, R.M. [5-^{14}C]DE-498 CONFINED ACCUMULATION STUDY IN ROTATIONAL CROPS: CONFIRMATION OF THE VALIDITY OF THE RESULTS FROM THE ORIGINAL STUDY STARTED ON MAY 5, 1987, AND REPORTED ON GH-C 2170 (30- AND 120-DAY PHASES) AND GH-C 2244 (365-DAY PHASE. Performed and Submitted by DowElanco; Midland, MI under Project ID 90069; Study completed on 14 June 1991; Received by EPA 19 June 1991; MRID 41931739.

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REVIEWED BY: G. Maske
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-8245

SIGNATURE:

APPROVED BY: Paul Mastradone, Chief
Supervisory Chemist
Review section #1
OPP/EFED/EFGWB

Signature: Paul J Mastradone

Date:

CONCLUSIONS:

The confined rotational crops study is not acceptable to meet Subdivision N Data Requirement for the following reasons:

The degradates were not identified.

The registrant must satisfactorily address the deficiency above for the study to fulfill the data requirement. If the registrant does not address the above deficiency, a new confined rotational crops study is required to fulfill the data requirement.

In this study, the results, where the application rate was confirmed by analysis of soil samples taken immediately after application rate, reported were similar to the results from MRID 41263232. There was limited accumulation of XRD-498 (<0.005 ppm) when rotational crops were planted in 30 day aged treated soil. Three degradates, which were labelled as Components A, B, and C were quantified. However, these degradates were only tentatively identified or not identified. Components A and B were present at ≥10% of applied and/or ≥0.01 ppm (See Table IX).

MATERIALS AND METHODS:

Test Material: The test substance was a mixture of [5-^{14}C]XRD-498 and non-radiolabelled XRD-498. The radiolabelled material had a specific activity of 10.9 mCi/mmole (74,400 dpm/μg. The non-radiolabelled test material was reported to have a 99.6% purity.

Standards: The non-radiolabelled test material used for the test standard was used in the test material, as well.

Two metabolites, N-(2,6-difluorophenyl)-5-hydroxymethyl-1,2,4-triazolo(1,5a)pyrimidine-2-sulfonamide and N-(2,6-difluorophenyl)-7-hydroxy-5-methyl-1,2,4-triazolo(1,5a)pyrimidine-2-sulfonamide, were used as standards and had reported purities of 87% and 97%, respectively.

Application Solution: [^{14}C]DE-498 was dissolved in acetone. Twenty-three point five mL of the solution and 25.8 mg of nonradiolabelled XRD-498 were combined.

The concentration of the radiolabelled and nonradiolabelled XRD-498 was found to be 0.291 mg/mL. The specific activity of the formulated test substance as applied was determined to be 39,000 dpm/μg (5.71 mCi/mmol)

Soil: Soil Characterization

62%	sand
22%	silt
16%	clay
2.5%	organic matter content
9.4 meq/100g	cation exchange capacity
12.81%	field moisture capacity at ⅓bar
7.5	pH

The pesticide use and crop history for the test system site for the previous three years is reported in Table I.

Sampling Intervals: 0 day, 29 days, 33 days posttreatment, 72 day (soybean forage), 86 days (wheat forage), 128 days (lettuce harvest), 142 days (wheat harvest) and soil samples, 159-160 days (carrot harvest), and 163 days (soybean harvest).

METHODOLOGY:

XRD-498 was applied to sandy loam soil at Midland Research Fields Station, Midland, MI. The dimensions of the plots were 0.61 m x 5.5 m (2 ft x 18 ft). Each plot was divided into one 0.2 m^2 sections. A 10 mL aliquot of the test solution was dribbled over each subsection with a 10 mL graduated pipet. For treatment of the last subsection only 4 mL of the test solution were applied to the soil surface using a graduated pipet. During application, the pipet was held 2-4 cm above the soil surface to prevent drift of the test solution. The surface of the soil was mixed with a hand tiller to a depth of 2-4 cm to distribute the applied test material. Soil samples were taken immediately after application.

At thirty days posttreatment, the test plot was tilled to a depth of 15-20 cm and soil samples were taken. The plot was fertilized with 21-0-0 ammonium sulfate at a rate of 67.2 kg n/ha. The test plot was subdivided into one 2.4 m, one 1-2 m, and two 0.92 m sections each 0.61 m wide. Wheat (4 rows) was planted in the 2-4 m section, soybeans (2 rows) were planted in the 1.2 m section; and carrots (3 rows) and lettuce (3 rows) were planted in each of the 0.92 m sections.

At fifty-four days posttreatment, three rows of carrots were planted. At fifty-five days posttreatment, three rows of lettuce were planted. These plantings were made because the previously planted carrots and lettuce appeared not to be growing. The sensitivity of carrots and lettuce to XRD-498 were observed in previous studies. The carrots and lettuce planted at 54-55 days posttreatment did grow until harvest.

The crops were irrigated with a sprinkler system as needed. Weed which were pulled up during the test period wee left on the soil surface.

The soil was sampled at prior to treatment, and 0 day, 29 days, 33 days, and harvest posttreatment. Both immature and mature tissues were collected for soybeans and wheat. Forage stage soybeans and wheat were collected at approximately pre-bloom stage forage and beginning boot, respectively. Only a single harvest at maturity was made for lettuce and carrots. Plants were harvested by pulling root crops or cutting above ground crops 2-3 cm from the soil line. Mature wheat was harvested at approximately Zadols[10] growth stage #83-85 due to the rainy weather. All crop harvests were stored frozen at -10 to -20°C unless further processing and combustion and/or extraction analysis were necessary.

To verify the accuracy of the results of the 1987 study, the harvests of mature wheat, mature soybeans, lettuce, and carrots were analyzed by the 1987 and 1990 method.

The soybean, lettuce, and wheat forage samples were chopped. Carrots were divided into tops and roots by slicing the tops from the root ≈0.5-1 cm toward the root side of the top/root interface. The top and root fraction were then chopped into 1-3 cm pieces and weighed.

The ^{14}C residue levels were determined by combustion. Aliquot size depended on crop fraction and moisture content which varied from 0.1-1 g for crop analysis. Two gram samples were used for soil samples. Moisture content was determined for crop fractions and soil samples.

Extraction analysis for soil samples was begun within 15 days of collection. To a 5 g aliquot of soil was added 15 mL of either 50:49:1 acetone:water:acetic acid or 50:50 acetone:water with 0.1M HCl. The mixture was shaken and centrifuged for an appropriate length of time. The extraction was repeated two more times, and the extracts were then combined and concentrated. Radioactivity was determined using LSC. The concentrated extracts were chromatographed using HPLC, and the extracted pellets were combusted.

Extraction analysis for soybean forage was begun 7 days after collection. Two-gram aliquots of soybean forage were extracted using three different procedures. (1) Six milliliters of 50:49:1 acetone:water:acetic acid were added. The mixture was homogenized, shaken, and centrifuged. The extraction procedure was repeated two more times. (2) The same extraction procedure as (1) was followed except 6mL of 75/25 water/acetonitrile was used. (3) Fifteen mL of 100% water was added to the sample, homogenized, and centrifuged. Extracts from all three procedures were concentrated. Radioactivity was determined by LSC (Extraction procedures were validated using spiked-control tissue. The concentrated extracts were chromatographed using HPLC, and the extracted pellets were combusted.

Extraction analysis of the wheat forage, wheat straw/chaff, and the soybean trash were begun 11 days after collection. To 2 g aliquot of wheat forage tissues was added 15 mL of either 80/20 acetonitrile, 50/50 water/acetonitrile or 100% water. To 2 g soybean trash sample was added 15 mL of 100%

water or 75/25 water/acetonitrile. The extract mixture was then homogenized, mixed, and centrifuged. The extraction procedure was repeated two more times. The extracts were then combined and concentrated. Radioactivity was determined by LSC. The concentrated extracts were chromatographed using HPLC, and the extracted pellets were combusted.

To a 1.5 to 2.5 g aliquot of the extracted wheat straw/chaff and soybean trash was added 30 mL of HCl. The mixture was heated to 80°C for ≈two hours, shaken well, and centrifuged. The supernatant was removed, and the pellets were then extracted twice in the same manner with 30 mL of a 50% aqueous acetonitrile solution. Radioactivity was determined by LSC. The extracted pellets were combined.

DATA SUMMARY:

Total XRD-498 residues were reported to be ≤0.01 ppm in lettuce, carrots, roots and tops, wheat grain, and soybeans. Wheat forage and soybean forage had reported total ^{14}C-residues of 0.039 and 0.056 ppm XRD-498, respectively. The highest total XRD-498 residues were reportedly found in soybean trash and wheat straw/chaff at 0.082 and 0.060 ppm, respectively. Three degradates were shown to be present which had concentrations ranging from 0.002 to 0.031 ppm. These were reported as Components A, B, and C. Component C was eluted in the region of the 5-hydroxymethyl and 7-hydroxy metabolites.

COMMENTS:

1. Components A and B were reported to be present at concentrations ≥10% of applied and/or ≥0.01 ppm. These Components could consist of two or more degradates. However, these Components were not identified in this study or further analyzed to determined if they consist of two or more degradates.

2. Storage stability data indicated that ≈3% average loss of XRD-498 occurred over a two year period.

3. Based on a meeting of 1 May 1990 in which it was agreed that the data from the original 1987 study (MRID 41263232 could be validated by repeating only the 30 day phase of a confined rotational crops study with [5-^{14}C]DE-498 with the above deficiencies addressed (minutes of meet ing), the registrant submitted this study to support MRID 41263232.

RIN # 4644-93 EFGW Review & Residue Chemistry Review for Flumetsulam (129016)

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