[12-1-88]

DATA EVALUATION RECORD

STUDY 12

CHEM 059101 Chlorpyrifos §165-1

FORMULATION--12--EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00149415
Bauriedel, W. and J. Miller. 1985. Rotational crop using soil treated with ^{14}C-labeled chlorpyrifos. Report No. GH-C 1719. Performed and submitted by Dow Chemical USA, Midland, MI.

DIRECT REVIEW TIME = 7

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CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable because the application rate was not confirmed by immediate posttreatment sampling and there was insufficient sampling of the treated soil during the study; therefore, the extent of pesticide uptake by the rotational crops in relation to the concentration of pesticide in the soil could not be determined. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because the test substance was not of analytical grade or purer, [^{14}C]-residues were not characterized in all soil and crop samples, freezer storage stability data were not provided, and experimental and sampling procedures were inadequately described.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Chlorpyrifos residues accumulated in a leafy vegetable (lettuce and/or spinach), a root crop (turnips or beets), and a small grain (wheat

and/or soybeans) planted 30, 129, and 365 (wheat only) days after formulated (4 lb/gal, EC) ring-labeled [^{14}C]chlorpyrifos (radiochemical purity >98%) was applied at 5 lb ai/A to sandy loam soil. In the crops planted at 30 days posttreatment, [^{14}C]residues at final harvest were 0.16 ppm in lettuce, 0.22 ppm in spinach, 0.07 and 0.08 ppm in turnip tops and roots, and 1.78 and 0.42 ppm in wheat straw and grain. In the crops planted at 129 days posttreatment, total [^{14}C]residues at harvest were 0.06 ppm in lettuce, 0.23 and 0.04 ppm in beet tops and roots, 0.36 and 0.12 ppm in soybean foliage and beans, and 1.95 and 0.24 ppm in wheat straw and grain. In wheat planted at 1 year posttreatment, total [^{14}C]residues were 0.31 and 0.08 ppm in straw and grain. Total pyridinol compounds were detected at 0.17-0.35 ppm in wheat straw planted at 30, 129, and 365 days posttreatment and at 0.12 ppm in soybean foliage planted at 129 days posttreatment. HPLC analysis of lettuce and wheat straw (planted at 30 days posttreatment) extracts detected primarily polar compounds (≈70-83% of extractable radioactivity) and trace amounts of . . .

3,5,6-trichloro-2-pyridinol.

In wheat straw planted at 30 days posttreatment, ≈63% of the total radioactivity was contained in the lignin and cellulose fractions, and in the grain 27, 19, and 33% of the total radioactivity was detected in the starch, protein, and cellulose fractions.

In the soil (depth unspecified), total [^{14}C]residues were 2.9 ppm at 30 days posttreatment, declined to 0.68 ppm during the growth of the 30-day crops, declined from 0.58 to 0.44 ppm during the growth of the 120-day crops and declined from 0.65 to 0.48 ppm during the growth of the 365-day crop. Between 30 and 365 days posttreatment, chlorpyrifos decreased from 21.8 to 9.8% of the ether extractable radioactivity, . . .

2-methoxy-3,5,6-trichloropyridine

increased from 0.5 to 23.2%, and . . .

3,5,6-trichloro-2-pyridinol

plus polar compounds decreased from 76.7 to 56.1%.

DISCUSSION:

1. Residues in the soil were not analyzed at the time of treatment to confirm the application rate. The registrant reported that at 30 days posttreatment total residues were 90% of the theoretical application rate; ≈22% of these residues were parent chlorpyrifos. It should be noted that aerobic soil metabolism studies (Study 5) have found the dissipation of [^{14}C]residues resulting from the application of [^{14}C]chlorpyrifos to be quite variable depending on the soil type; 40-95% of the applied radioactivity present in the soil system at 30 days posttreatment.

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2. There was insufficient sampling of the soil during the study; the soil data are presented in the original document in a manner that implies the soil was only sampled once following each planting interval. It was reported in the Experimental section that soil was sampled "after each final harvest." Since crops were harvested at different intervals, it is unclear which interval the soil data represents. In addition, residues in the soil must be analyzed at the time of harvest of each rotational crop.

2. The test substance was formulated and, therefore, was not of analytical grade or purer.

3. Storage conditions for soil samples between sampling and analysis were not reported. No storage stability data for soil and crop samples were provided to confirm that the [^{14}C]residues did not degrade between sampling and analysis.

4. Extraction of the soil samples was incompletely described; the procedure was referenced but not provided for review.

5. [^{14}C]Residues in the soil were characterized only at the time of planting.

6. Total [^{14}C]residues in crops comprising up to 0.60 ppm were not characterized. Some of the crop samples were analyzed for chlorpyrifos residues (total pyrinidol compounds) using a nonspecific analytical method. The registrant indicated that no attempt was made to characterize the residues in crops, except lettuce and wheat straw, because of low quantities of [^{14}C]residues.

7. It was not described how soil samples were taken or to what depth.

8. The CEC value for the soil was not reported.

9. For the portion of the study conducted in a growth chamber (120-day rotation), the control plants contained up to 0.405 ppm of [^{14}C]residues. The registrant reported that trapping solutions (sodium hydroxide) in the growth chamber detected 1850-9770 dpm/g of $^{14}CO_2$; radioactivity decreased during the study in the growth chamber.

10. Recoveries from fortified soil and plant samples were not provided.

11. Rainfall and air temperature data were presented graphically and were difficult to interpret. Soil temperature data were not provided.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ring-labeled [^{14}C]chlorpyrifos (radiochemical purity >98%, specific activity 2.5 Ci/mole, Dow Chemical) was formulated as an emulsifiable concentrate (Lorsban 4E M-4519, 4 lb/gal EC). The formulated test substance was applied by pipette at 5.0 lb ai/A to a field plot (2 x 20 feet) of sandy loam soil (70% sand, 16% silt, 14% clay, 1.9% organic matter, pH 6.5) and hand-cultivated into the soil to a depth of 1 inch.

At 30 days posttreatment, the soil was hand-tilled to a depth of 6 inches and planted to lettuce, spinach, turnips, and spring wheat. Crops were harvested when immature and at maturity; 90 days posttreatment (60 days postplanting) for lettuce, spinach, and turnips and 154 days posttreatment (124 days postplanting) for wheat. At 126 days posttreatment, the upper 6 inches of treated soil was removed from a portion of the field plot, placed in nine lined steel drums (15- x 17-inches deep), and planted on day 129 posttreatment to lettuce, sugar beets, soybeans, and spring wheat. The drums were maintained in a growth chamber at 18-24°C, 40-70% relative humidity, on a 15-hour daylight photoperiod. Crops were harvested when immature and at maturity; 196 days posttreatment (67 days postplanting) for lettuce, 202 days posttreatment (73 days postplanting) for wheat, 230 days posttreatment (101 days postplanting) for beets, and 248 days posttreatment (119 days postplanting) for soybeans. At 1 year posttreatment, spring wheat was planted in the field plot and harvested at maturity; 458 days posttreatment (93 days postplanting). Plant samples were stored frozen until analysis. Soil samples (depth unspecified) were taken at 30, 126, and 365 days posttreatment. Storage conditions between sampling and analysis of the soil samples were not reported.

Soil samples were air-dried, ground, and sieved (#14); subsamples were analyzed for total radioactivity by LSC following combustion. Additional soil subsamples were converted to an acidic, aqueous soil slurry and extracted repeatedly with diethyl ether. The extracts were concentrated and analyzed by reverse-phase HPLC. The method was referenced, but not further described.

Plant samples were air-dried, finely ground, and subsamples were analyzed for total radioactivity by LSC following combustion. Additional subsamples were analyzed for total pyridinol by Dow Chemical Method ACR 71.19R.S6 (not provided for review); samples were hydrolyzed and analyzed for pyridinol by GC with electron-capture detection, the detection limit was 0.05 ppm. Reported recoveries from soybean (grain, straw) and wheat (grain, straw) samples fortified with 3,5,6-trichloro-2-pyridinol ranged from 69 to 102% of the applied. In addition, mature lettuce planted at 30 days posttreatment was extracted with 50% methanol, and the extract was analyzed by reverse-phase HPLC. Mature wheat straw planted at 30 days posttreatment was extracted as described in Figure 4 to determine the distribution of radioactivity in normal plant constituents (starch, cellulose, lignin, and protein).

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