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DATA EVALUATION RECORD

STUDY 13

CHEM 059101 CHLORPYRIFOS §165-4

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40056401
Murphy, P. and N. Lutenske. 1986. Bioconcentration of chlorpyrifos in rainbow trout (Salmo gairdnerii Richardson). Laboratory Project Identification ES-928. Unpublished study prepared by Dow Chemical U.S.A. 49 p.

DIRECT REVIEW TIME = 10

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CONCLUSIONS:

Laboratory Accumulation - Fish

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the accumulation of chlorpyrifos in fish.

SUMMARY OF DATA BY REVIEWER:

Total [^{14}C]chlorpyrifos residues in rainbow trout ranged from 46 to 385 ppb in edible tissues (muscle), 158 to 1174 ppb in nonedible tissues (head, skin, viscera, skeleton), and 139.5 to 821.5 ppb in whole fish during 30 days of exposure to [2,6-^{14}C]chlorpyrifos at 0.30 ppb in a flow-through aquarium system. Maximum bioconcentration factors were 1280x in edible tissues, 3903x in nonedible tissue, and 2729x in whole fish. In general, the concentration of residues in the fish increased with exposure time. After 28 days of exposure, chlorpyrifos comprised up to 98.6% of the total radioac-

tivity in the edible tissues, 77.2% in the nonedible tissues, and 80.4% in whole fish. The degradate ...

3,5,6-trichloro-2-pyridinol (TCP)

...comprised up to 38.2% of the total radioactivity in the fish, and two glucuronide conjugates of 3,5,6-trichloro-2-pyridinol each comprised up to 28.8 and 36.5%. After 16 days of depuration, [^{14}C]residues in edible tissues, nonedible tissues, and whole fish were 5, 16, and 8 ppb, respectively.

Throughout the study, the temperature of the treated water was 11.3-12.8°C, the pH ranged from 7.9 to 8.1, and the dissolved oxygen content ranged from 8.5 to 9.4 mg/L; values were comparable to the control aquarium. Total [^{14}C]residues in the treated water were 0.25-0.34 ppb during the exposure period.

DISCUSSION:

1. In the original document, it was stated that the exposure period of the study was originally designed to last 28 days, but was extended to 30 days due to a brief power outage on day 3 which resulted in static conditions in the control and treated aquaria for approximately 9 hours. As a result, the concentration of chlorpyrifos in the treated aquarium on day 4 had decreased by ≈50%. However, the study authors reported that this temporary decrease in concentration had no apparent effect on the [^{14}C]residue levels in the fish.

2. The physical condition of the fish during the exposure period was not reported. It was stated that three fish died during the exposure period. Two of these deaths were attributed to "unknown causes"; the remaining fish was accidentally killed during the study.

3. The length of the acclimation period prior to exposure was not reported.

4. Based on previous studies, the 96-hour LC_{50} value of chlorpyrifos for rainbow trout was approximately 10 µg/L. In view of these results, the study authors chose an exposure level of 0.37 µg/L (<1/10 of the 96-hour LC_{50} value) for the bioaccumulation study.

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MATERIALS AND METHODS

MATERIALS AND METHODS:

Rainbow trout (Salmo gairdnerii; length and weight of 3.5-5 cm and 0.6-0.7 g, respectively) were held in stainless steel tanks on a 16-hour daylight photoperiod for an unspecified period of time prior to the initiation of the study. Flow-through aquatic exposure systems were prepared using two 40-L aquaria. Water from Saginaw Bay of Lake Huron and treated at the City of Midland Water Treatment Plant (pH 7.7-8.1, dissolved oxygen 8.4-10.1 mg/L, hardness 74-82 mg/L as $CaCO_3$, alkalinity 43-52 mg/L as $CaCO_3$) was provided to each aquarium at a rate of 5 turnovers per day. The aquaria were immersed in a water bath and maintained at 12°C.

Rainbow trout (85) were placed in each aquarium, and one aquarium was continuously treated with [2,6-^{14}C]chlorpyrifos (radiochemical purity ≥98%, specific activity 15.78 mCi/mmole, Dow Chemical) at an average concentration of 0.30 µg/L. The second aquarium served as an untreated control. Following a 30-day exposure period, fish remaining in the [2,6-$_{14}$C]chlorpyrifos-treated aquarium were transferred to a 40-L flow-through aquarium supplied with untreated water for a 16-day depuration period. The treated water was sampled prior to exposing the fish, and then daily during the exposure period (with the exception of days 9, 10, 14, 23, 24, 26, and 27). Fish (5) were taken from the treated and control aquaria after 0.5, 1, 2, 4, 8, 14, 21, and 30 days of exposure. During the depuration period, water samples were taken daily and [^{14}C]chlorpyrifos-treated and untreated fish were taken on days 1, 3, 6, 9, 12, and 16.

Radioactivity in the water samples was quantified using LSC. Periodic water samples were acidified with phosphoric acid and were then extracted with chloroform. The aqueous phase was analyzed for total radioactivity by LSC. Chloroform extracts were combined and evaporated to dryness. The remaining residue was dissolved in acetone:water (1:1) and analyzed for total radioactivity by LSC and for chlorpyrifos and its degradates by reverse-phase HPLC. Extraction recoveries of parent chlorpyrifos from water samples that were fortified with [^{14}C]chlorpyrifos (rate unspecified) averaged 97.5 ± 1.25%.

Samples of whole fish, edible tissues (muscle), and nonedible tissues (head, skin, viscera) were analyzed for total radioactivity by LSC following combustion. Reported recoveries of [^{14}C]residues from fish tissues fortified with [^{14}C]chlorpyrifos (rate unspecified) averaged 93.8 ± 4.2%.

Additional samples of edible tissue, nonedible tissues, and whole fish were extracted three times with 1% phosphoric acid in acetone, shaken for 1 hour between extractions, concentrated using nitrogen. The concentrated extracts were redissolved in acetone and then analyzed for total radioactivity by LSC, and for parent chlorpyrifos and its degradates using reverse-phase HPLC. The residue remaining from the extraction procedure was extracted three times with water.

The aqueous phase was analyzed for radioactivity by LSC, and unextractable [^{14}C]residues remaining in the fish tissues were quantified by LSC following combustion. Extraction recoveries of parent chlorpyrifos from fish tissues fortified with [^{14}C]chlorpyrifos (rate unspecified) averaged 116.7 ± 14.7%.

In order to characterize polar degradates in the acetone extracts, aliquots of the two extracts containing the highest amounts of these polar degradates were mixed with β-glucuronidase at pH 6.8 and 37°C. The solutions were acidified with concentrated phosphoric acid and extracted with diethyl ether. The ether extracts were concentrated to dryness, and the residues were dissolved in acetone:water (70:30) and analyzed by LSC and HPLC.

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