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DATA EVALUATION RECORD

STUDY 5

CHEM 128974 Quinclorac §162-1

FORMULATION--00--ACTIVE INGREDIENT

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Clark, J. 1988. BAS 514-^{14}C laboratory aerobic soil metabolism study using a terrestrial system. BASF Registration Document # 88/5046. Unpublished study performed and submitted by BASF Corporation Chemicals Division, Research Triangle Park, NC/Parsippany, NJ.

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This study was originally reviewed by EFGWB (2/13/90). The review has been reformatted for inclusion in the Registration Standard by Dynamac Corporation; the conclusions are those of the EFGWB reviewer and were not altered by Dynamac.

CONCLUSIONS:

Metabolism - Aerobic Soil

1. This study can be used to fulfill data requirements.

2. Quinclorac was persistent in aerobic silt loam soils; after 365 days of incubation at 23 °C, "practically all" of the extractable residues in the soils (84-98% of the applied) were unchanged quinclorac.

3. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the aerobic soil metabolism of [2,3,4-^{14}C]quinclorac in silt loam soil.

4. No additional information on the aerobic soil metabolism of quinclorac is required at this time.

METHODOLOGY:

Silt loam soils from Illinois and Mississippi (Table I) were treated at 0.5 ppm with [2,3,4-^{14}C]quinclorac (radiochemical purity 96.6%, specific activity 9.74 Ci/Mol, Nippon Soda) dissolved in acetone. The solvent was evaporated from the treated soils, and the soil moisture content was adjusted to 15% by weight (equivalent to 50 or 70% of 0.33 bar in the Mississippi and Illinois silt loam soils, respectively). Subsamples of the moistened treated soils were weighed (50 g) into glass dishes (3 x 5.5-cm diameter), and the dishes were placed on a wire rack that fitted inside a tall glass cylinder (the "soil reactor").

Humidified CO_2-free air was pumped continuously into the bottom of the cylinder; the air flowed out of the top of the cylinder through a tube of Oxifluor-CO_2 or Carbon 14 Cocktail trapping solutions. The treated soils were incubated in the dark at 23 $\pm$ 0.86 °C for up to 12 months. Duplicate dishes were removed from the glass cylinder at 0, 7, and 14 days and 1, 2, 4, 6, and 12 months posttreatment. The trapping solutions were changed at 1-month intervals. Soil samples were stored at -20 °C prior to analysis (length of storage not specified).

Soil samples that were collected between 0 and 4 months posttreatment were analyzed for total extractable quinclorac residues (Figure 2). The soils were refluxed with 0.1 N NaOH for 1 hour, and the extracts were adjusted to pH 2 and partitioned three times with methylene chloride. Radioactivity in the two fractions was analyzed using LSC, and radioactivity remaining in the extracted soil was analyzed using LSC following combustion. The ethyl acetate fraction was analyzed for quinclorac using one-dimensional TLC on silica gel plates developed in ethyl acetate:-methanol:acetic acid (80:15:5); the samples were cochromatographed with [^{14}C]quinclorac. Radioactive areas were located and quantified using a TLC linear analyzer and autoradiography. The radioactive zone identified as quinclorac was scraped from the plates, desorbed from the silica gel with methanol, and reacted with diazomethane. The solution was analyzed using two-dimensional TLC with

ethyl acetate:methanol:acetic acid (80:15:5) and toluene:-methanol (70:30) solvent systems.

Soil samples that were collected at 14 days and 6 and 12 months posttreatment were analyzed so that free, ionically-bound, and covalently-bound residues could be differentiated (Figure 1). The soils were extracted three times with water (free residues) by shaking, and the extracts were combined and analyzed by LSC. The water-extracted soils were further extracted with room temperature 0.1 N NaOH (ionically-bound residues) by shaking, and the extracts were combined and analyzed by LSC. The water and room temperature NaOH extracts were separately adjusted to pH 2 and partitioned three times with ethyl acetate; the ethyl acetate fractions were analyzed by LSC and one-dimensional TLC on silica gel plates developed in ethyl acetate:methanol:acetic acid. Radioactive areas were located and quantified using a TLC linear analyzer and autoradiography. The extracted soils were then refluxed with 0.1 N NaOH for 1 hour to extract the covalently-bound residues. Radioactivity in the NaOH extracts was determined by direct LSC; radioactivity in the extracted soils was determined by LSC following combustion. The NaOH extracts were adjusted to pH 2 and partitioned three times with ethyl acetate; the ethyl acetate fractions were combined and analyzed by LSC and one-dimensional TLC as described above.

Radioactivity in the gas trap solutions was quantified by LSC.

DATA SUMMARY:

[2,3,4-^{14}C]Quinclorac (radiochemical purity 96.6%), at 0.5 ppm, degraded slowly with a half-life of >1 year in two silt loam soils that were incubated in the dark at 23 C and a moisture content of 50-70% of 0.33 bar for 365 days (Table VII, Figure 5). At 12 months posttreatment, 83-100.6% of the [^{14}C]residues applied to the soil were extractable: 47.1-58.8% of the applied as "free" residues; 17.2-21.3% as ionically-bound residues; and 15.7-22.1% as covalently-bound residues. Quinclorac was the only [^{14}C]compound detected (using two-dimensional TLC) in the extracts, with the exception of

3-chloro-8-quinolinecarboxylic acid (BH 514-1)

which was recovered from the ionically-bound residue fraction in trace amounts in the sodium hydroxide extract (Figure 5).

At 12 months posttreatment, unextractable [^{14}C]residues comprised 2.5-11.3% of the applied and evolved [^{14}C]volatiles accounted for <0.1% (Table VII). The materials balance during the study ranged from 95.5-109.7% of the applied (Tables V and VII).

REVIEWERS COMMENTS:

1. The concentrations of quinclorac and 3-chloro-8-quinoline-carboxylic acid (BH 514-1) were not quantified; it was simply stated that "practically all" of the extractable residues were quinclorac and there was "a trace amount" of the degradate.

2. The method detection limits and recovery efficiencies from fortified soil samples were not reported.

3. EFGWB prefers [^{14}C]residues in samples be separated by chromatographic methods (such as TLC or HPLC) with at least three solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the R_f of reference standards.

   In this study, the majority of samples were analyzed by one-dimensional TLC with a single solvent system. Several samples were analyzed by two-dimensional TLC. Radioactive areas on the TLC plates were identified only by comparison to the location of known reference standards chromatographed on the same plates.