12-3-90

DATA EVALUATION RECORD

STUDY 17

CHEM 128974 Quinclorac §164-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41063565
Winkler, V. 1987. Confined field aquatic dissipation study of ^{14}C-BAS 514 H in rice paddy water. Registration Document No. 87/5064. Unpublished study performed by BASF Corporation Chemical Division, Research Triangle Park, NC/Parsippany, NJ.

DIRECT REVIEW TIME = 8

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CONCLUSIONS:

Dissipation - Aquatic Field

1. This study cannot be used to fulfill data requirements because the application rate of quinclorac was not confirmed. This was further confounded by the periodic removal of 10-20% of the water for use on other plots, and replenishing the plots with a like quantity of quinclorac-free water.

2. This study was not conducted using a typical end-use product; instead, the radiolabelled active ingredient that was used was dissolved in acetone.

3. Since the application rate of quinclorac was not confirmed, the problems with this study probably cannot be resolved

with the submission of additional data. A new study must be conducted.

4. Other problems that were noted by the reviewer are listed under the section below identified as "REVIEWERS COMMENTS".

METHODOLOGY:

An enclosed, small field plot (4 x 8 feet, 1 foot depth) of silty clay soil (9.6% sand, 40.4% silt, 50.0% clay, 2.2% organic matter, pH 6.5, CEC 33.18 meq/100 g) located in Greenville, Mississippi, was surrounded by aluminum and plastic walls which extended 1 foot above the surface of the plot. The plot was fertilized, and the soil was turned over to a depth of 8 inches and raked smooth to simulate disking. The plots were subsequently flooded. On May 1, 1984, the plots were planted to rice (6 rows with an average of 26 seeds/foot of row). On June 5, 1984, during a period of nonflooded conditions, [^{14}C]quinclorac (radiochemical purity 95.7%, specific activity 9.44 mCi/mMole, BASF) plus unlabeled quinclorac (purity unspecified), dissolved in acetone, was applied to the soil in the plot at 0.75 lb ai/A. At 7 days posttreatment, the plot was flooded. The water level was checked daily, and water was added as necessary to maintain flooded conditions. Water (10-20% of the total water in the plot) was removed for use on other experimental plots at 8, 36, 48, and 77 days posttreatment. Water samples were collected at intervals from 7 to 92 days posttreatment. Water samples were frozen for an unspecified period of time at an unspecified temperature prior to analysis.

All water samples were analyzed for total radioactivity by LSC; the detection limit was 0.001 ppm. Aliquots of water samples taken at 7, 35, 70, and 98 days posttreatment were selected for residue characterization. Aliquots (200 mL) were adjusted to an unspecified pH with 0.1 N hydrochloric acid and extracted twice with ethyl acetate; recovery of total radioactivity in the ethyl acetate extracts ranged from 70 to 90%. The ethyl acetate extracts were concentrated to dryness by rotary evaporation. The residues were redissolved in methanol and analyzed by TLC on silica gel plates developed in ethyl acetate:methanol:acetic acid (80:15:5). Radioactive areas on the TLC plates were located and quantified using a TLC linear analyzer, and identified by comparison to unlabeled reference standards of quinclorac and its degradate, 3-chloro-8-quinolinecarboxylic acid.

DATA SUMMARY:

An enclosed, small field plot of silty clay soil located in Greenville, MS, was planted to rice and treated with

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[^{14}C]quinclorac (radiochemical purity 95.7%) plus unlabeled quinclorac (purity unspecified) at 0.75 lb ai/A under nonflooded conditions. A permanent flood was established at 7 days posttreatment. Quinclorac residues dissipated from the floodwater with a half-life of <21 days. In floodwater, quinclorac residues were 0.005-0.013 ppm at 7-12 days posttreatment (0-5 days after flooding), 0.004 ppm at 28 days posttreatment (21 days after flooding), and were not detected (<0.001 ppm) after 70 days posttreatment (63 days after flooding) (Table I). In extracts of the floodwater, parent quinclorac decreased from 96.15% of the recovered radioactivity at 7 days posttreatment (immediately after flooding) to 67.92% of the recovered at 98 days posttreatment (91 days after flooding) (Figures 5-8).

REVIEWERS COMMENTS:

1. The application rate was not confirmed by collecting soil samples as suggested by Subdivision N Guidelines §164-2 (d)(4). Although quinclorac was applied to soil in the plot under nonflooded conditions, an immediate posttreatment soil sample was not taken. Therefore, the dissipation of quinclorac from the test site could not be adequately evaluated. This was further confounded by the periodic removal of 10-20% of the water for use on other plots, and replenishing the plots with a like quantity of water.

2. The experiment was not replicated in regards to treatments. Absence of replicates does not allow EFGWB to assess the experimental variation that may occur in soil and analytical procedures. Replication means that each treatment is replicated 2 or 3-fold. In the future, EFGWB suggests that the registrant establish at least two replicated (EFGWB prefers a minimum of 3-fold replication) experimental units for each treatment. The results should be given for each individual sample within a replicate and not as a composite. This is good laboratory practice and good science and gives an idea of the range and variability of possible results.

3. No pretreatment samples were taken to confirm that the site had not been contaminated with the test substance prior to the initiation of the study.

4. The study author stated that quinclorac residues in the water were determined to be exclusively parent quinclorac, based on TLC analysis of the water samples. However, the TLC radioscans do not support this conclusion (Figures 5-8); the TLC radioscans show that quinclorac decreased with time from 96.15% of the recovered radioactivity at 7 days posttreatment (immediately after flooding) to 67.92% of the recovered at 98 days posttreatment (91 days after flooding).

The question still remains as to what was the disposition of the other 32.08%.

5. It was stated that the water samples were frozen prior to analysis; however, the length of storage and the storage temperature were not reported. In a freezer storage stability study (STUDY ID 41063571 and 41063570, Study 28), it was determined that quinclorac residues were stable for up to 6 months in pond water that was stored frozen at <-5 °C.

6. Characteristics of the test water, including pH and dissolved oxygen content, were not reported.

7. Meteorological data were incomplete. The only temperature and precipitation data provided for the study period were monthly averages (Figures 3 and 4); minimum and maximum daily temperatures and total daily precipitation data for the duration of the study period were not provided. However, the study author stated that daily temperature and precipitation records were available.

8. Sampling procedures used to collect the water samples were not described; the sampling technique, the number of samples collected at each sampling interval, and the depth at which the water samples were taken were not reported.

9. EFGWB prefers that [^{14}C]residues in samples be separated by chromatographic methods (such as TLC, HPLC, or GC) 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 sample extracts were analyzed using one-dimensional TLC with one solvent system. Radioactive areas on the TLC plate were identified only by comparison to the location of known reference standards. It is unclear if reference standards were cochromatographed on the same plate or were chromatographed separately.

10. In a description of the analytical methodology, it was stated that aliquots of the water samples were "adjusted to 0.1 N hydrochloric acid" prior to extraction. Apparently, this is a typographical error, and the pH that the water was adjusted to using 0.1 N hydrochloric acid was inadvertently omitted.

11. Recovery efficiencies for quinclorac were not provided.

12. It was stated that the recoveries of total radioactivity in ethyl acetate extracts of water samples taken at 7, 35, 70,

and 98 days posttreatment averaged 80%, and ranged from 70 to 90%. Since recoveries at each sampling interval were not provided, data for the amount of parent quinclorac recovered from the extracts could not be converted to percent of the applied radioactivity.

13. This study was not conducted using a typical end-use product; instead, the radiolabelled active ingredient that was used was dissolved in acetone.

14. The conditions in this study were not typical of actual use conditions. The plot was (4 x 8 feet, 1 foot depth) was enclosed on the bottom and sides, with the walls extending 1 foot above the plot, so that water could not flow either vertically below the 1-foot soil depth or horizontally outside of the plot. However, it is not clear what impeded the movement of water past the 1-foot soil depth.

15. The registrant stated that the application rate of 0.75 lb ai/A is 1.5x the proposed maximum registered use rate.

16. The study author stated that the probable causes for dissipation of the test substance were dilution of residues by replacement of water removed from the plot, and a reduction of residues by plant uptake and absorption and/or diffusion into the soil. This reviewer notes that these causes of dissipation may be correct. However, until positive proof is presented in the form of acceptable data, the conclusions as to route of dissipation remain highly speculative. The question that still remains is how quinclorac dissipates from the aquatic system.

FACET 128974

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