DATA EVALUATION RECORD

STUDY 3

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CHEM 061601 Paraquat dichloride §162-2

FORMULATION--00--ACTIVE INGREDIENT

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DP Barcode D164623
STUDY ID 41319302
Vickers, J.A., A.D. Hurt, and D.W. Bewick. 1989. Paraquat: degradation in anaerobic soil. Laboratory Project No. 88JH386/Report No. RJ0810B. *Unpublished study performed and submitted by ICI Americas Inc., Wilmington, DE.*

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CONCLUSIONS:

Metabolism -- Anaerobic Soil

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

2. Paraquat at 4.32 ppm did not degrade in sandy loam soil incubated under anaerobic conditions for 60 days following a 30-day aerobic incubation. Paraquat comprised 88.8% of the applied radioactivity at 90 days posttreatment [60 days of anaerobic incubation]. Most of the radioactivity was extracted with technical grade paraquat by isotopic exchange. Some radioactivity (0.29% of applied) was recovered in the water phase at 61 days posttreatment [30 days postflooding]. There was no volatile radioactivity. No degradates were reported from TLC or HPLC analyses.

3. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the anaerobic soil degradation of [$^{14}$C]paraquat in flooded sandy loam soil. No additional information on the anaerobic soil metabolism of paraquat is required at this time.

METHODOLOGY:

Sandy loam soil (64% sand, 22% silt, 14% clay, pH 6.5, organic matter 2.7%, CEC 10.8 meq/100 g) was collected, air-dried, sieved to 2 mm,



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and moistened to 40% of the moisture holding capacity. These samples were stored at 20 ± 2 C for 20 days. Samples (25 g dry weight) were weighed into glass sample dishes (3 cm deep; 3.7 cm diameter) and a mixture of [2,6-pyridyl-$^{14}$C]paraquat [1,1-dimethyl-4,4-bipyridinium dichloride; radiochemical purity >98%, specific activity 734 GBq/mMol, ICI) and unlabeled paraquat in water was added dropwise to the soil surface. The glass dishes were placed on coated wire racks which fit inside a glass column (Figure 3). Humidified, $CO_2$-free air was pumped into the glass column and then vented through a series of tubes containing 0.1 M HCl, 2-methoxyethanol, and ethanolamine as traps for volatile radioactivity. The glass columns were incubated in the dark at 20 ± 2 C for 30 days. After the 30 days of incubation, to create anaerobic conditions, the soil in the sample dishes was flooded to 2 cm above the soil surface and the gas pumped into the system was changed to $O_2$-free nitrogen. Duplicate glass dishes of soil were removed for analysis after 0 and 30 days of aerobic incubation and 31 and 60 days of anaerobic incubation [61 and 90 posttreatment].

At sampling, the floodwater was removed from the dishes with a Pasteur pipette and was analyzed by LSC. The soil samples were extracted three times; after the extraction the phases were separated by filtration and the filtrates were analyzed by LSC. Soil samples were first shaken with 100-150 mL of methanol for 1 hour. The second extract was for 2-4 hours with 100 or 150 mL of aqueous technical grade paraquat solution (7440 ppm paraquat cation) in order to desorb radiolabeled [$^{14}$C]paraquat by isotopic exchange. The soil was further extracted by a 5-6 hour reflux with 6 M HCl and the extracted soil was freeze-dried, ground, and analyzed by LSC following combustion.

The methanol extract did not contain detectable radioactivity and was not further analyzed. The extracts from the technical grade paraquat step and the acid reflux step were analyzed by one-dimensional TLC on silica gel plates developed with toluene:butanol:methanol:1 M HCl (10:20:50:20, v:v:v:v) or methanol:6 M HCL (65:35, v:v); samples were cochromatographed with reference standard paraquat. To detect paraquat, the developed plates were sprayed with a solution of potassium iodoplatinate; paraquat appeared as black-brown spots on a red-brown background. Radioactive areas were located and quantified by linear scan; the location was checked by autoradiography. For a confirmatory method, aliquots of the extracts were also analyzed by HPLC on a Beckman Ultrasphere-IP column eluted with a mobile phase of water:methanol (75:25, v:v). The HPLC mobile phase also contained: 11.35 g of octane sulphonic acid (sodium salt), 10.3 mL o-phosphoric acid, and 12.7 mL diethylamine per liter of mobile phase. Detection was by UV at 310 nm; fractions of the HPLC flow were collected and analyzed by LSC.

Derivatization of paraquat in the extracts : Aliquots from the 0 and 90 day samples were treated with a mixture of 9 M NaOH and 1% potassium ferric cyanide to derivatize the paraquat in the samples to

[$^{14}$C]1,1-dimethyl-4,4-bipyridyl-2,2-dione. These samples were analyzed by TLC on silica gel plates developed with chloroform:acetone:methanol (80:10:10, v:v:v); paraquat was located by fluorescence quenching. Radioactive areas were located and quantified by linear scan; the location was checked by autoradiography. HPLC was again used as a confirmatory technique using the same system described above except that the mobile phase contained only 2.27 g of octane sulphonic acid instead of 11.35 g.

Aliquots of the volatile trapping solutions were removed for LSC analysis at each sampling interval or at two weeks intervals.

DATA SUMMARY:

[$^{14}$C]Paraquat at 4.32 ppm (0.90 lb ai/A) did not degrade when incubated in anaerobic (flooded) sandy loam soil for 60 days after a 30-day aerobic incubation. The temperature was kept at 20 $\pm$ 2 C. [$^{14}$C]Paraquat comprised 99.5% of the radioactivity immediately posttreatment, 92.3% after 30 days of aerobic incubation, 90.7% at the sampling interval 30 days after flooding [61 days posttreatment] and was 88.8% after 60 days of anaerobic incubation [90 days posttreatment] (Table IV). At 30 days postflooding [61 days posttreatment], 0.29% (0.13 ppm) of the radioactivity was recovered from the floodwater; this radioactivity was not analyzed for specific compounds. No radioactivity was recovered from the volatile traps; radioactivity which was not removed by the three extraction methods ranged from 0.75-4.1% of the applied dose. Material balances were 93.0-106.8% throughout the study.

COMMENTS AND DISCUSSION:

1. At the 61 day sampling interval, 0.29% of the applied radioactivity was recovered from the water phase; by 90 days posttreatment there was no radioactivity in the water phase. The water samples were only analyzed by LSC; therefore, this radioactivity was not characterized. Since paraquat is extremely tightly bound to soil clay particles and the water samples were removed with a Pasteur pipette, it is probable that there was some soil in the water sample to account for this radioactivity.

2. The amount of radioactivity that was recovered as "dione" from the derivatized soil was approximately equivalent to the amount of paraquat identified in the HPLC extracts indicating that the radioactivity in the soil extracts was [$^{14}$C]paraquat.

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