DATA EVALUATION RECORD

I. Study Type: Anaerobic and Aerobic Soil Metabolism

II. Citation:

Warinton, J.S., I. Chalofti, and B.R. Harvey. 1995. ICIA5504: Degradation of 14C-Labelled Compound in Soil Under Laboratory Conditions. Performed by Zeneca Agrochemicals (Zeneca Limited), Berkshire, U.K. Submitted by Zeneca Agricultural Products (Zeneca Inc.), Wilmington, Delaware. MRID 43678175.

III. Reviewer:

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Organization: EFGWB/EFED/OPP 30 JUL 1996

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V. Conclusions:

Aerobic Soil Metabolism

The study provides supplemental data on the metabolism of methyl(E)-2-{2-[6-(6-2-cyanophenoxy)pyrimidin-4-yloxy]pheny}-3-methoxyacrylate (ICIA5504) in aerobic mineral soils. The data are deemed as supplemental because it was not conducted for 365 days. (Please see Section VIII for more details.) The data may be upgraded with submission of ICIA5504 residue concentrations in soil from 120 to 365 days posttreatment.

Radiolabeled ICIA5504, at 0.57 µg/g, had a 50% dissipation time (DT_{50}) of 7.7 weeks (53.9 days) in the Hyde Farm soil, 12.2 weeks (85.4 days) in the 18 Acre soil, and 23.4 weeks (163.8 days) in the Visalia soil. The first-order degradation half-lives of ICIA5504 was 72 days in the Hyde Farm soil, 85 days in the 18 Acre soil, and 163.8 days in the Visalia soil. Transformation products of ICIA5504 were identified as (E)-2-{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]-3-methoxyacrylic acid as Compound 2 (12 to 20% applied), methyl (E)-2-{2-(6-hydroxypyrimidin-4-yloxy)phenyl}-3-methoxyacrylate as Compound 3 (< 3% of applied), 4-(2-cyanophenoxy)-6-hydroxypyrimidine as Compound 28 (3.1% of applied), and (E)-2-{2-[6-(2-carbamoylphenoxy)pyrimidin-4-yloxy]phenyl}-methyoxyacrylic acid as Compound 36 (< 2.8% of applied). Unidentified bands/spots were also detected (cumulative 0.8 to 11.6% of applied) on the TLC plates. Unextractable radiolabelled soil residues ranged from 16 to 22% at 120 days posttreatment. The cumulative concentration of $^{14}CO_2$ ranged from 2% to 27% of applied radioactivity.

The reported data indicate ICIA5504 should be moderately persistent in terrestrial environments.

Anaerobic Soil Metabolism

The study provides acceptable data on the soil metabolism of ICIA5504 in anaerobic soil-water systems. No additional data are needed at this time.

Radiolabeled ICIA5504, at 0.57 µg/g, had DT_{50}s of 7 to 8 weeks in flooded Hyde Farm and 18 Acre soils. The DT_{50} of radiolabeled ICIA5504 was < 2.1 days in the test water and 8.6 to 9.2 weeks in anaerobic Hyde Farm and 18 Acre soils. Transformation products of ICIA5504 were identified as Compound 2 (58 % of applied), Compound 3 (0.2 % applied), Compound 28 (< 4.2% of applied), and Compound 36 (< 2.8% of applied). Unidentified bands/spots were also detected (cumulative < 8.0 of applied) on the TLC plates. Unextractable radiolabelled soil residues ranged from 5 to 10% of applied at 120 days posttreatment. The cumulative concentration of $^{14}CO_2$ was < 5% of applied radioactivity.

The reported data indicate ICIA5504 should be moderately persistent in flooded terrestrial environments.

VI. Materials and Methods:

Test soils were taken from Jealott's Hill Farm in Berks, U.K. and a site in California, USA. The United Kingdom test soils were classified as a Hyde Farm sandy loam and 18 Acres sandy clay loam. These soils were cross-referenced to the USDA great group Eutic Hapludoll. The test soils were not treated with any pesticide for at least five years prior to the study.

The United States soil was classified as a Visalia soil (Aquic Haploxeroll). The registrant indicated the soil was taken from a site in California. The soil was taken from a site spot treated with paraquat in 1993.

Soil samples were taken at depth of 10-to-15 cm. The test soils were passed through a 2mm sieve, stored at 4°C for the U.K. soils or ambient temperature for the U.S. soil, and then incubated at 20°C. Physicochemical properties of the test soils are shown in Table 2.

Soil samples were pre-incubated under aerobic or anaerobic conditions prior to definitive experiments. Subsamples (30g) of each soil type were placed into glass pots. The United Kingdom soils were flooded with 2 cm of ultra-pure water to induce anaerobic conditions. The anaerobic soil samples were incubated in flow-through N_2 atmosphere for 3 weeks. The water content of the remaining soil samples were adjusted to 75% of 1/3 bar to induce aerobic conditions. The aerobic samples were incubated in a flow through CO_2-free atmosphere for < 21 days.

Each soil sample was amended with radiolabelled ICIA5504 (cycanophenyl labeled, SA=2479 Bq μg^{-1};radiopurity=99%; pyrimidimyl labeled, SA=2458 Bq μg^{-1}; radiopurity=98.8%; phenylacrylate labeled, SA=2722 Bq μg^{-1}, radiopurity=98.5%) at a nominal application rate of 0.5667 $\mu g/g$. A treatment set, representing a soil type and a radiolabelled position ICIA5504 and an incubation condition, were incubated in a common flow-through volatile trapping system connected to sequential gas traps of activated carbon and ethanolamine. (Reviewer Note: There were 21 common flow-through systems for the aerobic and anaerobic incubations.) The aerobic soil samples were incubated under a CO_2-free atmosphere at 1/3 bar soil moisture content. The anaerobic soil samples were incubated under N_2-free atmosphere at a flooded or saturated soil moisture content. Triplicate soil samples were taken immediately posttreatment. Duplicate soil samples were taken at 7, 14, 30, 62, and 120 days posttreatment. (Reviewer Note: The registrant stated they will submit additional data for 180 and 365 days sampling periods.)

Analytical

Redox potentials in anaerobic soil samples were determined using a Pt electrode. Anaerobic soil samples were centrifuged to separate the soil and water phases. Each soil sample was sequentially extracted with acetone:water (9:1 v/v) to remove 95% of applied radioactivity. The test water from anaerobic soil samples (or supernatant) was diluted in acetone and then stored for chemical analysis.

Soluble radiolabelled residues in soil extracts and test water were separated using normal and reverse phase TLC. Separated residues were identified by co-chromatography with known standards. (Reviewer Note: The registrant did not provide additional confirmation of soil transformation products.) The ^{14}C content in soil extracts, test water, and gas traps was determined by LSC. The ^{14}C content in extracted soil was determined by combustion-LSC.

VII. Study Author's Conclusions

A. The material balance of radioactivity ranged from 94.7 to 100.7% of applied ICIA5504 in aerobic and anaerobic experiments (Tables 3, 4, 5, 6, and 7). (Reviewer Note: A low material balance (68.2 to 86.5% of applied ICIA5504) was detected in the 18 Acre soil amended with labeled phenylacrylate ICIA5504 at 62 and 120 days posttreatment. The registrant believes the low material balance is the result of a leak in the air-line of the flow-through system.)

Aerobic Soil Metabolism

B. Radiolabeled ICIA5504, at 0.57 µg/g, had DT_{50}s of 7.7 weeks (53.9 days) in the Hyde Farm soil, 12.2 weeks (85.4 days) in the 18 Acre soil, and 23.4 weeks (163.8 days) in the Visalia soil (pages 120, 121 and 122; Figure 6). The first-order degradation half-lives of ICIA5504 was 72 days in the Hyde Farm soil, 85 days in the 18 Acre soil, and 163.8 days in the Visalia soil. The slow degradation rate of ICIA5504 in the Visalia soil was attributed to a low biomass of the soil. (Reviewer Comment: The registrant stated a 2nd order degradation model provided a best fit for ICIA5504 degradation in the Hyde Farm and Visalia soils. Therefore, the registrant described the degradation as a DT_{50}.).

C. Four transformation products of ICIA5504 were identified in aerobic soils (Tables 8, 9, 10).

Compound 2 in the United Kingdom soil had a maximum concentration of 12 to 20% of applied at 62 days and then declined to 9.5 to 15.2% of applied. The maximum concentration of Compound 2 in the Visalia soil was 12.1% of applied at 120 days posttreatment.

Compound 3 had a maximum concentration 1.5% of applied in the at 30 days posttreatment in the Hyde soil, 1.1% of applied at 62 days posttreatment in the 18 Acre soil, and 3.1% of applied at 120 days posttreatment in the Visalia soil. The concentration of compound 3 in the Hyde and 18 Acre soils declined to 0.5% of applied at 120 days posttreatment.

Compound 28 in the Visalia soil had a maximum concentration of 2.1% of applied at 120 days posttreatment.

Compound 36 in all test soils had a maximum concentration of 0.4 to 2.8% of applied at 120 days posttreatment.

Unidentified bands/spots were also detected (cumulative 0.8 to 11.6% of applied) on the TLC plates. The registrant stated the unidentified radioactivity within a designated TLC band did not exceed 5% of the applied or extracted radioactivity.

D. Volatile residues in aerobic soils were tentatively identified as CO_2. The cumulative concentration of $^{14}CO_2$ ranged from 19 to 27% of applied radioactivity in aerobic Hyde Farm soil, 2% of applied in aerobic 18 Acre soil, and 2% of applied in aerobic Visalia soil. No radiolabeled residues were detected in the activated carbon traps.

Anaerobic Soil Metabolism

E. The DT_{50} of radiolabeled ICIA5504, at 0.57 µg/g, ranged from 7 to 8 weeks in flooded Hyde Farm and 18 Acre soil (Tables 11 and 14; Figure 7). The DT_{50} of radiolabeled ICIA5504 was < 2.1 days in the test water of flooded Hyde Farm and 18 Acre soils (Tables 12 and 15). The DT_{50} of radiolabeled ICIA5504 ranged from 8.6 to 9.2 weeks in anaerobic Hyde Farm and 18 Acre soils.

F. Four transformation products of ICIA5504 were identified in anaerobic United Kingdom test soils (Tables 11, 12, 13, 14, 15; Figures 9).

Compound 2 was detected in the surface water and soil extracts. The maximum concentration of Compound 2 was 58 % of applied in the total anaerobic water-soil systems of applied. The concentration of Compound 2 ranged from 16 to 19% of applied in the test water and 39 to 42% of applied in soil extracts at 120 days posttreatment.

Compound 3 had a maximum concentration of 0.2 % applied in the test water of 18 Acre soil. This transformation product was not detected in the Hyde Farm soil.

Compound 28 in the total soil-water system had a maximum concentration of 1.8 to 4.2% of applied and then declined to 0.8 to 3.3% of applied at 120 days posttreatment. This compound was predominately detected in soil extracts.

Compound 36 had a maximum concentration of 0.4 to 2.8% of applied at 120 days posttreatment.

Unidentified bands/spots were also detected (cumulative < 8.0 of applied) on the TLC plates. (Reviewer Note: The registrant did not provide secondary confirmation of identification. The registrant also did not provide any information on the number of separate compound on the unidentified TLC spots or baseline.)

G. The redox potential in the anaerobic soil/waters system range from 0 to -50 mV. EFGWB notes the redox potential appeared to be poised at -50 mV. (Reviewer Note: The pe+pH of the neutral anaerobic soils would range from 6 to 7).

H. Volatile residues in anaerobic soils were tentatively identified as CO_2. The cumulative concentration of 14CO2 was < 5% of applied.

I. Unextractable radiolabeled soil residues in aerobic soils ranged from 16 to 22% at 120 days posttreatment. Unextractable radiolabeled soil residues in anaerobic soils ranged from 5 to 10% of applied at 120 days posttreatment.

VIII. Reviewer's Comments

A. The aerobic soil metabolism study was conducted for 120 days. As per Subdivision N guidelines, the aerobic soil metabolism study should be conducted for 365 days. EFGWB notes the registrant indicated in the data submission that additional metabolism data from 120 to 365 days will be submitted. The aerobic soil metabolism data is deemed as supplemental pending submission of additional metabolism data from 120 to 365 days posttreatment.

B. The registrant did not provide confirmation of the identity of transformation products Compounds 2, 3, and 36. Secondary analytical confirmation of Compound 28 was established in MRID 43678174. EFGWB notes that 1-D TLC chromatograms indicate clear separations of ICIA5504 and its transformation products in normal and reverse phase 1-D TLC systems. Confirmation of residue identification should be conducted using 2-D TLC or HPLC with solvent systems of different polarity or GC-MS. Since the registrant used a different 1-D TLC systems, EFGWB believes there is adequate identification of the transformation products. In future studies, the registrant should confirm residue identification using a secondary analytical technique.

C. The degradation rate of ICIA5504 was reported as a DT_{50}. EFGWB calculated the first-order degradation half-lives for ICIA5504.

D. The USDA soil taxonomy classification of test soils was taken from MRID 4378182. EFGWB appreciates the registrant's effort to cross-reference the United Kingdom soils into USDA soil taxonomy.

D. The registrant measured the redox potential in anaerobic soils. EFGWB appreciates the reporting of redox potential because it provides a measure of anaerobicity of the soil-water system.

AZOXYSTROBIN

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