DATA EVALUATION RECORD

I. Study Type: Photodegradation in Water

II. Citation:

Kuet, S.F. and S.T. Hadfield. 1994. ICIA5504: Aqueous Photolysis at pH 7. Performed by Zeneca Agrochemicals (Zeneca Limited), Berkshire, U.K. Submitted by Zeneca Agricultural Products (Zeneca Inc.), Wilmington, Delaware. MRID 43678173.

III. Reviewer:

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Organization: EFGWB/EFED/OPP 30 JUL 1996

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V. Conclusions:

The study provides supplemental data on the photodegradation of methyl(E)-2-{2-[6-(6-2-cyanophenoxy)pyrimidin-4-yloxy]pheny}-3-methoxyacrylate (ICIA5504) in pH 7 buffer solution. The data are deemed supplemental because unidentified radioactivity (7.6% per compound of applied radioactivity) was detected in irradiated buffer solutions at 30 days posttreatment. The data may be upgraded with the submission of additional information to substantiate the presence of multiple compounds at low concentrations (< 10% of applied radioactivity).

Radiolabeled ICIA5504 in pH 7 buffer solution had photodegradation half-lives of 11.1 to 17.1 Florida equivalent summer days ($k=-0.0418$ to -0.0624 days^{-1}) under a Xenon lamp. Photo-transformation products of ICIA5504 were methyl(Z)-2-{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]-3-methoxyacrylate as Compound 9 (15 % of applied), 2-hydroxybenzonitrile as Compound 13 (1.7% of applied), methyl{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}acetate as Compound 21 (<5.6% of applied), methyl 2-{2-[6-(2-cyanophenixy)pyrimidin-4-yloxy]phenyl}glycolate as Compound 24 (<2.6% of applied), 4-(2-cyanophenoxy)-6-hydroxypyrimidine as Compound 28 (<8.9% of applied), and 2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}acetate as Compound 30(<2.4% of applied).

The reported results indicate ICIA5504 should photodegrade in aquatic environments.

VI. Materials and Methods:

Aliqouts (25ml) of sterile, buffer solution (pH=7, 3,3 dimethyl-glutaric acid) were dispensed into sterile, photolysis vessels and glass scintillation vials. Each aliquot of test solution was amended with radiolabeled ICIA5504 (cycanophenyl labeled, SA=2479 Bq μg^{-1}, radiopurity=99% or pyrimidimyl labeled, S.A.=2458 Bq μg^{-1}, radiopurity=98.8% or phenylacrylate labeled, SA=2722 Bq μg^{-1}, radiopurity=98.5%) to yield concentrations of 3.04 to 3.29 μg/ml. The water solubility of ICIA5504 is 6 μg/ml. Each photolysis vessel was capped with a quartz lid prior to irradiation. The dark controls were capped, wrapped with aluminum foil, and then incubated at 25°C. Irradiated samples were exposed to Xenon light source at an equivalent light energy of 30 days of sunlight in Florida. The average light intensity of Xenon ranged from 27.46 to 35.93 W m^{-2} per Florida summer day. (Reviewer Note: The registrant selected Florida day equivalence because it is the southern most state in the United States and hence where greatest intensity of sunlight would be expected.) Duplicate samples were taken immediately posttreatment, 1, 5, 10, 20, and 30 days posttreatment.

A single photolysis vessel was equipped with a flow-through volatile trapping system. The photolysis vessel was continuously purged with sterile, humidified air to displace headspace gases into sequential gas traps of NaOH and ethanolamine.

Analytical

Residues in buffer solution were liquid-liquid extracted using water/ether or water/ethyl acetate.

Soluble residues were separated using HPLC equipped with a Sperisorb 150DS2 column and solvent systems of acetonitrile:water (70:30 v/v) or methanol: 0.2% aqueous formic acid (60:40 v/v). Soluble residues were also separated by the normal and reverse phase 1-D TLC and 2-D normal phase TLC. Separated residues were identified using co-chromatography with known standards. Identification of ICIA5504, Compound 9, and Compound 28 were confirmed using LC-MS and LC-MS-MS. The $_{14}C$ content in buffer solution and gas traps was determined by LSC. The $^{14}CO_2$ was identified using $BaCO_3$ precipitation.

VII. Study Author's Conclusions

A. The material balance of radioactivity ranged from 83 to 105.9% of applied ICIA5504 in irradiated and dark control treatments (Tables 7 and 8). (Reviewer Note: The registrant did not provide an explanation for the low material at 5 days posttreatment.)

B. Radiolabeled ICIA5504 in pH 7 buffer solution had photodegradation half-lives of 11.1 to 17.1 Florida equivalent summer days (k=-0.0418 to -0.0624 days^{-1}) under a Xenon lamp (Appendix 14B). The DT_{50} of ICIA5504 ranged from 8.7 to 13.9 Florida equivalent days (Appendix 14A). No degradation of ICIA5504 was observed in the dark control treatments.

C. Six phototransformation products of ICIA5504 were identified in TLC separations (Tables 9,10,11). The transformation products and their formation and decline patterns in irradiated treatments are shown below.

Compound 9 was detected in all radiolabeled ICIA5504 treatments. Compound 9 had a maximum concentration of 15% of applied at 5 to 10 days after irradiation and then declined to less than 7 % of applied at 30 days posttreatment.

Compound 13 was detected in only the cyanophenyl labeled treatment. Compound 13 had a maximum concentration of 1.7% at 30 days posttreatment.

Compound 21 was detected in all radiolabeled ICIA5504 treatments. Compound 21 had a maximum concentration of 5.6% at 30 days posttreatment.

Compound 24 was detected in all radiolabeled ICIA5504 treatments. Compound 24 had a maximum concentration of 2.6% at 30 days posttreatment.

Compound 28 was detected in the radiolabeled pyrimidinyl and cyanophenyl treatments. Compound 28 had a maximum concentration of 8.9% at 30 days posttreatment.

Compound 30 was detected in all radiolabeled ICIA5504 treatments. Compound 30 had a maximum concentration of 2.1% at 30 days posttreatment.

Unidentified radiolabeled residues were separated by the 2-D TLC with a choroform:methanol:water:formic acid and dichloromethane:acetonitrile:formic acid solvent systems. The maximum average corrected concentration would be 7.6% of the applied radioactivity per compound (Figure 14).

Unidentified radiolabeled residues, designated as Remainders or diffuse radioactivity between distinct bands, were also detected. The cumulative concentration of Remainders ranges from 23 to 25% of applied radioactivity (Figures 8, 10, and 11).

D. Volatile residues from photodegradation of radiolabeled phenylacrylate ICIA5504 were detected (6.2% of applied) in NaOH and ethanolamine volatility traps (Appendix 15). Radiolabeled CO_2 was identified (5.9% of applied) as the major volatile transformation product.

E. ICIA5504 had a maximum UV adsorption at 220 nm and then declined to minimal adsorption at 295 nm (Figure 2). The distribution of spectral irradiance of Xenon light correlated with autumn natural light from the United Kingdom. (Reviewer Note: The registrant did not provide a comparison of the spectral irradiance of Florida sunlight and the Xenon light.)

VIII. Reviewer's Comments

A. EFGWB notes the majority of the phototransformation products of ICIA5504 were separated as Unknowns (15 to 30% of applied at 30 days posttreatment) or as diffuse radioactivity between distinct bands (25% of applied at 30 days posttreatment). EFGWB cannot discern the number of compounds on TLC autoradiograms. (Please see Figures 8, 10, 11 and 14.) EFGWB believes the study provides supplemental data because unidentified radioactivity (cumulative concentration > 10% of applied radioactivity) was detected in irradiated buffer solutions. The data may be upgraded with the submission of additional information to substantiate the presence of multiple compounds at low concentrations (< 10% of applied radioactivity).

B. EFGWB notes the irradiation period was adjusted to mimic a typical Florida day. It is reasonable to assume the photodegradation rate of ICIA5504 should be longer at more Northern latitudes.

AZOXYSTROBIN

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