

DATA EVALUATION RECORD

I. Study Type: Photodegradation on Soil

II. Citation:

Winter, L. and R.S.I. Joseph. 1995. ICIA5504: Soil Surface Photolysis. Performed by Zeneca Agrochemicals (Zeneca Limited), Berkshire, U.K. Submitted by Zeneca Agricultural Products (Zeneca Inc.), Wilmington, Delaware. MRID 43678174.

III. Reviewer:

Name: James A. Hetrick, Ph.D.
Title: Soil Chemist
Organization: EFGWB/EFED/OPP

James A. Hetrick

30 JUL 1996

IV. Approved by:

Name: Paul J. Mastradone, Ph.D.
Title: Section Chief
Organization: EFGWB/EFED/OPP

Paul J. Mastradone

30 JUL 1996

V. Conclusions:

The study provides acceptable data on the photodegradation of methyl(E)-2-{2-[6-(6-2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}-3-methoxyacrylate (ICIA5504) on Hyde Farm sandy loam soil. No additional information is needed at this time.

Radiolabeled ICIA5504, at 1.0 lbs ai/A, had an average 50% degradation time (DT_{50}) of ICIA5504 was 11 days. The first order photodegradation half-life of ICIA5504 ranged from 17.6 to 28.4 days. There was minimal degradation of ICIA5504 in the dark control treatments. Phototransformation products of ICIA5504 were identified as methyl (E)-2-{2-(6-hydroxypyrimidin-4-yloxy)phenyl}-3-methoxyacrylate as Compound 3 (0.7% of applied), methyl(Z)-2-{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]-3-methoxyacrylate as Compound 9 (9.0% of applied), 2-hydroxybenzonitrile as Compound 13 (< 3.5% of applied), methyl 2-{2-[2-6-(2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}oxoacetate as Compound 19 (< 6.0% of applied), methyl 2-{2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}glycolate as Compound 24 (<3.2% of applied), 4-(2-cyanophenoxy)-6-hydroxypyrimidine as Compound 28 (<6.5% of applied), 2-[6-(2-cyanophenoxy)pyrimidin-4-yloxy]phenyl}acetate as Compound 30 (<5.0% of applied), and U13 (<5.2% of applied). Unidentified transformation products (individually < 3.0% of applied) were also detected in soil extracts.

The reported results indicate ICIA5504 should photodegrade in terrestrial environments.

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VI. Materials and Methods:

The United Kingdom test soil was described as Hyde Farm sandy loam soil. Physicochemical properties of the test soils are shown in Appendix 1. The soil was passed through a 2 mm sieve, stored at ambient room temperature for 12 days and then stored at 4°C prior to the experiment.

Subsamples (1 g) of test soil were suspended in aliquots (700 μ l) of distilled water. Each suspension was uniformly spread over the bottom of side-arm photolysis flasks at an approximate thickness of 1 mm. Each soil layer was amended with radiolabelled ICIA5504 (cyanophenyl labeled, SA=2479 Bq μ g⁻¹; radiopurity=99%; pyrimidinyl labeled, SA=2458 Bq μ g⁻¹; radiopurity=98.8%; phenylacrylate labeled, SA=2722 Bq μ g⁻¹, radiopurity=98.5%) to yield application rates of 463 to 498 g ha⁻¹ or 1.01 to 1.1 lbs ai/A.

Each photolysis vessel was capped with a quartz lid and then connected to a closed flow-through air system. Headspace gases were purged with CO₂-free, humidified air and then passed through sequential gas traps of 1M NaOH and ethanolamine. Photolysis vessels were wrapped with aluminum foil to serve as dark control treatments. The dark control samples were maintained at 20°C. The photolysis vessels were irradiated with a Suntest Xenon lamp. The cyanolabeled treatments were irradiated with a single Xenon lamp. The pyrimidinyl and phenylacrylate labeled treatments were irradiated with two Xenon lamps. The irradiated samples were maintained at 25°C during irradiation. The average light intensity of Xenon ranged from 33.54 to 41.88 W m⁻² per Florida summer day. (Reviewer Note: The registrant selected Florida day equivalence because its the southern most state in the United States and hence where greatest intensity of sunlight would be expected.)

Duplicate samples were taken immediately posttreatment, 3, 6, 10, 16, and 30 days posttreatment.

Analytical

Soil samples were extracted within 3 days of the sampling date. Each irradiated soil sample was sequentially extracted with sonicated acetone:water (80:20 v/v) and acetone/0.1 M HCl (80:20 v/v). Dark control samples were extracted with sonicated acetone:water (80:20 v/v). Soil extracts were stored frozen prior to chemical analysis.

Soluble residues were separated using normal and reverse phase 1-D TLC and 2-D TLC. Separated residues were identified using co-chromatography with known standards. Identification of residues was confirmed by HPLC-MS and GC-MS. The total ¹⁴C content in soil was determined by combustion-LSC. The total ¹⁴C content in soil extracts and gas traps was determined by LSC. Radiolabelled CO₂ in NaOH gas traps was identified by BaCO₃ precipitation.

VII. Study Author's Conclusions

A. The material balance of radioactivity ranged from 90 to 129.9% of applied ICIA5504 in irradiated and dark control treatments (Tables 6, 7, 8). (Reviewer Note: EFGWB notes an extremely high material balance (129.9% of applied ICIA5504) was detected at 9.8 days posttreatment. EFGWB believes a single outlier in material balance does not invalidate the study because it does not indicate a pattern of analytical or experimental error.)

B. Radiolabeled ICIA5504, at 1.0 lbs ai/A, had a DT_{50} of 11 days (Figure 27a and 27b). There was minimal degradation of ICIA5504 in the dark control treatments (Tables 9, 10, 11). [Reviewer Note: EFGWB calculated the first order photodegradation half-life of ICIA5504 as 17.6 to 28.4 days.]

C. Eight transformation products of ICIA5504 were identified in irradiated treatments (Tables 9, 10, and 11).

Compound 3, formed by oxidative cleavage of the diphenyl ether linkage, was detected in only the phenylacrylate labeled experiment. The maximum concentration of Compound 3 was 0.7% of applied at 16.4 days posttreatment and declined to less than 0.5% of applied at 30.2 days posttreatment.

Compound 9, Z-isomer of ICIA5504, was detected in all the labeled experiments. The maximum concentration of Compound 9 ranged from 2.8 to 9.0% of applied during the 31 day irradiation experiment. No observable pattern of formation and degradation were observed.

Compound 13 was detected in only the cyanophenyl labeled experiment. The concentration of Compound 13 was < 3.5% of applied during the 31 day irradiation experiment. No observable pattern of formation and degradation was observed.

Compound 19, formed by oxidation of compound 24, was detected in all labeled experiments. The concentration of Compound 13 was < 6.0% of applied during the 31 day irradiation experiment.

Compound 24, formed by oxidative cleavage of the diphenyl ether, was detected (<3.2% of applied) in all labeled experiments.

Compound 28, formed by oxidative cleavage of the diphenyl ether link, was detected (<6.5% of applied) in the cyanophenyl labeled experiments.

Compound 30, formed oxidation of Compound 19, was detected (<5.0%) in all labeled experiments.

Compound U13, formed by oxidative rearrangement of ICIA5504, was detected (<5.2% of applied) in all labeled experiments.

D. Volatile residues from photodegradation of radiolabeled phenylacrylate ICIA5504 were detected (6.2% of applied) in NaOH and ethanolamine volatility traps (Appendix 15). Radiolabeled CO₂ 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.

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