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DATA EVALUATION RECORD

STUDY 1

CHEM 112602 CIMECTACARB §161-2

STUDY ID 42081403
Merritt, Andrew PHOTODEGRADATION OF ^{14}C-CGA-163935 IN pH 7 BUFFERED SOLUTION UNDER ARTIFICIAL SUNLIGHT. Sponsored and Performed by Ciba-Geigy Corporation, Greensboro, NC under Lab. Project ID ABR-91026; Study completed on 10 October 1991; Received by EPA 5 November 1991.

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CONCLUSIONS:

This photodegradation in water study is scientifically sound and is acceptable to fulfill the data requirement. No further photodegradation in water data for cimectacarb (CGA-163935) is needed at this time.

When exposed to a continuous light source (xenon arc lamp) for 372 hours, cimectacarb in pH 7 buffer solution degraded with a reported pseudo-first order kinetic half-life of 63.5 hours (equivalent to 5.3 days using intermittent light). The dark control samples did not degrade significantly (<2% of applied). Peak 1 (ethyl ester of tricarballylic acid) was the major degradate (maximum concentration of 55.69% at 372 hours) for the light exposed samples. Five other minor (<10% of applied) degradates (Peaks 1A (comprised of several components), 1B (not present in sufficient quantity to be identified), 2A & 2B (cis and trans isomers of CGA-163935, and 2 (CGA-179500) See Figure 1) were discernible in the light exposed samples reaching maximum concentrations of 2.67% of applied at 372 hours, 2.14% of applied at 372 hours, 6.26% of applied at 276 hours, 5.58% of applied at 276 hours and 5.41% of applied at 240 hours, respectively.

The data indicate that photodegradation plays an important role in the degradation of cimectacarb. The opening of the cyclohexane ring forming the ethyl ester of tricarballylic acid appears to be the major photolytic pathway.

MATERIALS AND METHODS:

Test Material: Radiolabelled CGA-163935 in the C1, C2, and C6 position of the cyclohexane ring was used which had a reported 97% radiochemical purity, specific activity of 39.0 μCi/mg, and solubility in pH 7 and 20°C of 2.7 g/100 mL.

Reference Standards: See Table IA.

Test Solution: The test solution was prepared in acetonitrile such that ≤1% by volume of test solution was added to test samples.

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Buffered Solution: The buffer solution (pH 7.0) was prepared by mixing 30 mL of 0.067M NaH_2PO_4 and 61 mL of 0.067M K_2HPO_4 and diluted with water. The buffer solution was then sterilized.

Sampling: Initial light exposed intervals: 10, 14, 16, 20, 28, 48, 66, 74, 96, and 145 hours.

Initial control intervals: 0, 5, 16, 66, 74, and 96 hours.

Supplemental test intervals: 0, 16, 74, 145, 240, 276, and 372 hours.

Test System: See Figures 2, 3, 4, and 5.

METHODOLOGY:

The glassware used in the test testing procedures was silylated. The silylating solution contained 5% dichlorodimethyl silone in methylene chloride (CH_2Cl_2). The glassware was soaked in the solution for ≥15 minutes. The glassware was then rinsed with methanol and methylene chloride, respectively, and baked at 110°C for two hours and sterilized by autoclaving.

Concentrated solutions of ^{14}C-cimectacarb, test solution, were prepared in acetonitrile. Aliquots of 10 μL to 29 μL (≤1% by volume) of the test solution were aseptically applied to each test vials. Each test vial contained ≈4.9 mL of pH7 buffer solution. Therefore, each test vials contained 9.425 to 11.300 ppm of test material. Replicate test vials were prepared for each sampling interval and condition.

The light exposed samples were exposed to a continuous artificial light source (xenon arc lamp) at 545 to 551 W/m^2 light intensity (See Figures 3 and 4). The natural sunlight intensity was reported to be 660 W/m^2 on 17 June 1991 at ≈12:00 (noon). Other readings on 11 July 1991 averaged 410 W/m^2 for the light hours (9:00 to 17:00) of the day. Ultraviolet waves below 290 nm were filtered out with a special glass filter. In addition, the waterbath in which the test vials were placed was monitored for temperature during the testing period (See Tables I and II).

The control samples were placed in a constant temperature chamber. They were incubated under dark conditions with the temperature monitored (See Figures 6 & 7).

Supplemental test (light exposed) and control samples were sampled at 0, 16, 74, 145, 240, 276, and 372 hours posttreatment. The initial test samples were sampled at 10, 14, 16, 20, 28, 48, 66, 74, 96, and 145 hours. However, the initial control samples were sampled at 0, 5, 16, 66, 74, and 96 hours. Samples were analyzed as soon as possible after collection. Extraction of samples was not necessary.

All radioactivity was measured by a liquid scintillation counter (LSC). Samples and HPLC eluates were assayed by direct aliquoting using TLC radioassay for reference, as well. HPLC was used for characterization, quantitation, and isolation of degradates. Each standard solution and sample used for HPLC was chromatographed and cochromatographed with the standards. In addition, all samples and selected standards were also characterized by normal phase 2D-TLC. TLC chromatography was used to confirm HPLC quantitation and characterization. The TLC solvent systems used were as follows:

SS1: Toluene:acetone:formic acid 75:25:1 (v:v:v)
SS2: Chloroform:methanol:formic acid:water 75:20:4:2 (v:v:v)

In addition, GC/MS or FAB/MS or MS/DIP analyses were used to confirm characterization of the cimectacarb residues. Peaks 1A, 1, 2A, 2B, and 3 were collected separately for the MS analyses.

Sterility of the test system was monitored throughout the testing period.

In addition, the pH of the test system was monitored during the testing period.

DATA SUMMARY:

When exposed to a continuous light source (xenon arc lamp) for 372, cimectacarb in pH buffer solution degraded with a reported pseudo-first order kinetic half-life of 63.5 hours (equivalent to 5.3 days using intermittent light). The rate constant was reported as 9.29×10^{-3} hours-1. The TLC analysis confirmed the HPLC analysis with a reported half-life of 62.1 hours and a rate constant of 1.118×10^{-2} (See Table XV). The dark control samples appeared to not degrade significantly (<2% of applied). Peak 1 (ethyl ester of tricarballylic acid) was the major degradate (maximum concentration of 55.69% at 372 hours) for the light exposed samples. Five other minor (<10% of applied) degradates products (Peaks 1A (comprised of several components), 1B (not present in sufficient quantity to identified), 2A & 2B (cis and trans isomers of CGA-163935, and 2 (CGA-179500) See Figure 1) were discernible in the light exposed samples reaching maximum concentrations of 2.67% of applied at 372 hours, 2.14% of applied at 372 hours, 6.26% of applied at 276 hours, 5.58% of applied at 276 hours and 5.41% of applied at 240 hours, respectively.

The data indicate that photodegradation is an important degradation pathway. The opening of the cyclohexane ring forming the ethyl ester of tricarballylic acid is the major photolytic pathway.

The material balance for the light exposed samples ranged from 93.48% to 102.73% of applied radioactivity. However, there was one replicate, 16-hour replicate B, which was not in this range. It had a reported balance of 89.25% of applied radioactivity (See Table VI). The material balance of the control samples ranged from 98.34% to 101.79% of applied radioactivity (See Table VII).

The temperature for the light exposed samples during the testing period ranged from 24.1 to 25.7°C. The control samples were incubated in a constant temperatrue chamber at 25 ± 1°C.

The sterility test of samples throughout the testing period indicated that the test system was sterile (See Tables III and IV).

The results of the pH monitoring of the test system is reported in Tables III and IV, as well.

COMMENTS:

1. A cimectacarb degradate (Peak 1B), which was discernible at a maximum concentration of ≈0.242 ppm (<10% of applied), was not identified in the study. In addition, Peak 1A, which was comprised of several components, was not characterized as to components and quantities.

2. Only percent recovery of applied radioactivity was reported. Actual levels of cimectacarb in samples was not reported.

3. An absorption spectrum of the test material in the test solution was not furnished. Therefore, the absorption wavelengths of cimectacarb are not known.

4. Peak 1, the major degradate and identified as the ethyl ester of the tricarballylic acid, was not confirmed by MS. However, HPLC and 2D-TLC showed Peak 1 to cochromatograph with the ethyl ester of the tricarballylic acid.

5. A supplemental experiment was carried out to determine the formation and decline of degradates. The initial experiment supplied data on samples exposed to light for periods up to 145 hours. The supplemental experiment supplied data for up to 372 days. Samples were assayed at sufficient time intervals to determine the half-life and formation and decline degradates. In addition, comparable results were reported for the initial and supplemental experiments.

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