

DATA EVALUATION RECORD

STUDY 1

CHEM 129016 XRD-498 \$161-3

STUDY ID 41931728

Wolt, J.; Shepler, K.; Marc, M.; McGovern, P.; and Ruza, L.O.; PHOTODEGRADATION OF [¹⁴C-PYRIMIDINE]XRD-498 ON SOIL BY NATURAL SUNLIGHT.
Preformed by Pharmacology and Toxicology Research Laboratory, Richmond, CA under PTRL Report No. 208W; Submitted by DowElanco; Midland, MI under Dow Protocol No. 89088; Study completed on 29 January 1991; Received by EPA 19 June 1991.

DIRECT REVIEW TIME - 1.0 day

REVIEWED BY: G. Maske
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-8245

SIGNATURE:

APPROVED BY: Paul Mastradone, Chief
Supervisory Chemist
Review section #1
OPP/EFED/EFGWB

Signature: 

Date: 

CONCLUSIONS:

The photodegradation on soil study alone is not acceptable to meet Subdivision N Data Requirement. However, in combination with MRID's 41931729 and 41931730) the photodegradation on soil data requirement is fulfilled. No further photodegradation on soil data for [¹⁴C-pyrimidine]XRD-498 is required at this time.

This soil photodegradation study of XRD-498 (the ¹⁴C-pyrimidine]XRD-498 portion of the material) appears to exhibit limited degradation in both light exposed and dark control samples. The reported extrapolated half-lives were 87 days and 321 days for light exposed and dark control data, respectively. The photodegradates were CO₂ (not >1.0% of applied) and numerous unidentified degradates which were formed in insignificant amounts (none >6% of applied radioactivity)(See Table II). The material balance averaged 97.6% for the testing period. The photodegradation of the ¹⁴C-aniline portion of XRD-498 is discussed in DER-Study 2 and 3 of this review. Very similar results were reported.

MATERIALS AND METHODS:

Test Material: [¹⁴C-pyrimidine]XRD-498 was used which was reported to have a specific activity of 10.9 mCi/mmol and radiochemical purity of 99.5%.

An unlabeled standard was obtained from DowElanco for analytical purposes. The purity of this standard was given as 99.7% which was confirmed by TLC analysis.

Stock Solution: Stock solution was prepared by adding an acetone solution of [¹⁴C-pyrimidine]XRD-498 (1.23 mg in 1350 μL, 9 x 10⁷ dpm) to acetone (2650 μL). The solution was mixed prior to use.

Soil: A commercial silt loam soil from Wayside, MS was used for the test.

See Table I for soil characterization.

Sampling: 0, day 5, day 10, day 13, day 20, and day 30

Test System: See Figure 4.

The irradiation source was natural sunlight. The test was conducted in Richmond, CA at 37.45°N latitude and 122.26°W longitude.

METHODOLOGY:

Silt loam soil from Wayside, MS was passed through a 2 mm sieve, air dried, and sterilized. Aliquots of the sterile silt loam soil (3.1 gm) were weighed onto 50 mm Petri dishes. Distilled water (3 mL) was added to each dish and the slurries allowed to air dry, forming a thin soil layer (≈ 0.5 mm) on the bottom surface of each petri dish. The soil dishes were then autoclaved for an additional 20 minutes.

Aliquots (100 μ L) of the [14 C-pyrimidine]XRD-498 stock solution were transferred onto each dish as evenly as possible in a circular pattern. After treating the petri dishes with the proposed application rate of 9.8 ppm, the plates were then placed in a quartz covered stainless steel temperature controlled chamber in natural sunlight. The temperature was continuously monitored and recorded at 20 minute intervals throughout the study (See Table IV). The mean temperature for the light exposed and dark control samples was $23.3^{\circ}\text{C} \pm 0.7$ and $23.1^{\circ}\text{C} \pm 0.7$.

Ethylene glycol, 10% H_2SO_4 , and 10% NaOH were used to trap volatile organic compounds and CO_2 , respectively. Ambient air was drawn through sterilized bacterial filters into both light and dark sample chambers into separate sets (light and dark) of the three traps. Gas dispersion tubes were used to maximize the trapping efficiency. Trapping efficiency for $^{14}\text{CO}_2$ was determined, using the identical system, by introducing a measured amount of ^{14}C -sodium bicarbonate as an aqueous solution into a petri dish and adding an excess of glacial acetic acid while air was being drawn through the system.

Sunlight intensity and cumulative sunlight energy were measured (250-700 nm integration range) and recorded at 10 minute intervals throughout the study using an International Light Photodetector equipped with a Model SED 400 probe oriented at a 30 degree angle with respect to the vertical and located approximately four feet above and six feet behind the soil chambers. The photodetector was calibrated by the manufacturer. All soil chambers were oriented in the same manner such that the chambers were perpendicular to the sun's path. Cloud cover data was provided by the City of Richmond Public Works Department. Experiments were carried out in Richmond, CA at latitude 37.45N longitude 122.26W for a period of 30 days.

Duplicate light exposed and dark control samples were removed from their respective soil chambers at the following times - Day 0, 5, 10, 13, and 30. The samples were extracted immediately with acetone/water/acetic acid, 90:5:5 (v/v/v) and the extracts analyzed. Aliquots of the extracts were subjected to LSC (3 x 0.5 mL) and then analyzed by HPLC. Selected samples were analyzed by 2-dimensional TLC.

Total volumes in each gas dispersion trap were measured and aliquoted (3 x 0.5 mL) for radioassay (LSC) on each sampling day. Recovered radiocarbon from each trap was divided proportionately among the contributing samples.

DATA SUMMARY:

The half-life for [¹⁴C-pyrimidine]XRD-498 when applied to silt loam soil and exposed to natural light was extrapolated to be 87 days from a first-order plot having a correlation coefficient of 0.97. The half-life of [¹⁴C-pyrimidine]XRD-498 in the dark controls was 321 days. These values were considered by the author to overestimate the reactivity of XRD-498 since unextracted radiocarbon (Tables VI and VII) may consist largely of unreacted parent material based on soil properties. There was decreased extractability of XRD-498 with aging in soil which was well documented prior to initiation of the study. Soils for sampling times of day 20 and day 30 exhibited >10% non-extractable ¹⁴C. However, these were subjected to additional extraction in an attempt to desorb additional radiocarbon. Exhaustive extraction released levels of radiocarbon not extracted initially (Table VI). TLC analysis of selected additional extracts shows a single band with the same R_f as XRD-498 in both light and dark samples.

The material balance was determined by summing the radiocarbon remaining in soil, the soil extracts, and radiocarbon volatilized. Radiocarbon recoveries averaged 97.6% based on nominal applied radioactivity.

[¹⁴C-pyrimidine]XRD-498 does appear to slowly photodegrade when applied to sterile silt loam soil, producing carbon dioxide and numerous low yield (none >0.3% of applied) unidentified degradates. The data obtained in this study was consistent with other photodegradation data for XRD-498.

COMMENTS:

1. Dry soil was used for the experiment. The guidelines specify a soil moisture content of 75% of 1/3 bar should be maintain and monitored during the testing period.
2. The soil was sterilized twice. Once before putting in petri dishes and once after being placed in petri dishes. Sterization of soil may change the nature of the soil. The characteristics of the soil given may no longer be valid; and therefore, the data is not acceptable. However, photodegradation on soil data was furnished using non-sterile soil.
3. The experiments were carried out in Richmond, CA at latitude 37.45N longitude 122.26W for a period of 30 days (8 December to 7 January) using natural sunlight. The intensity of sunlight in late fall is less than the intensity of sunlight during the summer growing season. Therefore, this study did not reflect the greatest intensity of sunlight and perhaps the greatest amount of photodegradation that will occur in the environment. No comparison of sunlight intensity was given for the two seasons of year.
4. No ultraviolet spectrum of XRD-498 in water was provided from 290nm to 750nm.
5. Due to the widely varying radiocarbon recoveries obtained in the study, the half-life was calculated using the percent [¹⁴C-pyrimidine]XRD-498 detected in soil extracts by HPLC expressed as a percent of recovered radiocarbon for each sample.

0.57 for the light exposed samples and the dark samples, respectively. The authors stated these reflected the relative small amount of degradation that occurred over the 30 day testing period. However, in the [¹⁴C-aniline]XRD-498 study there was a correlation coefficient of 0.98 reported. The data provided was consistent with previous aerobic soil metabolism and photodegradation data submitted. Based on available data for [¹⁴C-pyrimidine and aniline]XRD-498, additional data is not needed to understand the environmental fate of XRD-498.

TABLE 1

% sand	36%
% silt	52%
% clay	12%
% organic material	0.5%
% pH	7.4%
CEC	8.8 meq/100g

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