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DATA EVALUATION RECORD

STUDY 2

CHEM 109101 Mepiquat Chloride §162-1

STUDY ID 42412103

Huber, R. FURTHER INVESTIGATIONS INTO THE AEROBIC DEGRADATION OF MEPIQUAT CHLORIDE IN SOIL. Sponsored and Submitted by BASF Corporation, Research Triangle Park, NC; Performed by BASF, Limburgerhof, FRG under Registration Document No. BASF 91/10952 and Laboratory Study Code P87-M009; Completed October 1991; Received by EPA 23 July 1992.

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CONCLUSIONS:

Aerobic Soil Metabolism

The aerobic soil metabolism study is scientifically valid and can be used as supplemental data at this time. However, it cannot be used to fulfill the data requirement. Even though the data indicate that mepiquat chloride metabolizes to CO^2 ($t_{1/2}$=3-21 days), degradates present and the degradation pathway for metabolism of mepiquat chloride under aerobic condition cannot be determined from this study. In addition, confirmatory analysis of Radio-TLC data for parent material and degradates was not furnished. The initial analysis of TLC data should be supported by other analytical methodology (preferably MS). These data are needed to make a complete environmental assessment of mepiquat chloride.

Mepiquat chloride appears to degrade to CO_2 when applied to aerobic sandy loam and loam soils (USDA classifications could not be determined-see COMMENT 2) and exposed to aerobic conditions. Observed and calculated half-lives of 2.72 and 21.06 days for sandy loam and loam soils, respectively, were reported. Potential metabolites like N-methylpiperidine and piperidine were not discernible during the testing period. Therefore, it appears that the heterocycle ring was completely broken down. In addition, soil bound residues appeared to not increase significantly.

MATERIALS AND METHODS:

Test Material: 1,1-dimethylpiperidinium chloride was used which was reported to have a specific activity of 1.804 mCi/mMol radiochemical purity of >98%. Chemical purity was not furnished.

(1)

Test Solution: 3922.2 μg/10 mL

Reference Standards: No chemical purity was given.

1,1-dimethylpiperidinium chloride
1-methyl-piperidine
piperidine

Soil: A sandy loam soil collected in Limburgerhof, Germany and loam soil collected in the USA were purchased and were used for the study. See Appendix 3 for characterization of soils.

Sampling Intervals: Samples were taken at 0, 2, 7, 14, 29, 61, 90, 25, 152, 244, and 365 days posttreatment.

Test System: See Appendix 4.

METHODOLOGY:

Two different soil textures, sandy loam and loam, were used for the study. However, the soil texture for the soils used could not be verified due to soil size distribution reported in the data. Prior to application of mepiquat chloride, the test soils were sieved through a 2 mm sieve and the soil moisture to adjusted to 75% of ⅓ bar field capacity.

A total of 1805.87 g and 2021 g of moist sandy loam and loam soil, respectively, were fortified at a concentration of 1 mg/kg or 1 kg/ha. This is equivalent to 1 ppm. The fortified soil was distributed into petri dishes each containing 120 g dry soil (≈140 g moist soil). The test dishes were immediately placed in a soil reactor. The system was closed and connected to absorption traps and aeration. The test soil reactors were kept in a dark room at 20 ± 2°C during the testing period. During sampling intervals samples were exposed to some light.

Samples were collected at days 0 (2 hr), 2, 7, 14, 30, 60, 90, 125, and 152 posttreatment for sandy loam soil and at 0 (2 hr), 2, 7, 14, 30, 60, 90, 125, 152, 244, and 365 posttreatment for the loam soil. Samples were immediately analyzed in duplicate upon collection. Volatiles from traps were collected at each sampling interval and radioassayed by LSC immediately, as well.

The soil samples were extracted with dipicrylamine in dichloromethane. The dichloromethane orange phase was collected. The aqueous phase was again extracted with dipicrylamine-dichloromethane and the dichloromethane orange phase collected. The soil was then extracted with methanol and the methanol phase collected.

Bound residues were determined by drying the preextracted soil and adding 0.5 N HCl. The HCl-washing solution contained fulvic acid which was collected. The remaining soil was extracted with 0.5 N NaOH. The NaOH was collected and acidified with HCl conc. to precipitate humic acid.

Radio-TLC was used to determine the radioactive components in the dichloromethane extracts. The concentrated extracts were streaked onto the TLC plates and developed with methanol/acetone/HCl conc. (90/10/4;v/v/v). Non-radiolabelled standards were cochromatographed on the TLC plates for characterization/identifications.

LSC was used to determine quantity of radioactive material present.

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Due to the soil reactor set up and the continual introduction of fresh air, the authors concluded that the system was aerobic. Therefore, no measure of aerobicity was taken during the study.

The individual balance values amounted only to ≈70% with the average balance values being 87.6%. The authors believed this was due to the rapid and strong evolution of CO_2 which resulted in losses due opening of soil reactor for sample collection and/or absorption capacity of CO_2 traps was not adequate. Therefore, additional material balance testing was conducted.

Soil moisture capacity was monitored during the testing period.

Half-lives were calculated using first order kinetics.

DATA SUMMARY:

Based on first order kinetics, the half-life reported for mepiquat chloride applied to sandy loam soil was 2.72 days using data for days 0-14 posttreatment/incubation and 21.06 days using data for days 0-90 posttreatment/incubation when applied to loam soil. It was believed that after 14 and 90 days, respectively, any signification degradation had ceased due to microorganism exhaustion in the test system. Cochromatographies of reference standards and test samples indicated that neither N-methylpiperidine nor piperidine were not present in discernible concentrations. In addition, non-extractables increased from days 0-7 posttreatment/incubation. However, day 125 samples had similar results for non-extractables indicating that after day 7 there was no significate increase of non-extractables.

Due to what the authors reported as rapid and strong CO_2 production, the individual balances were 70% plus with an average balance of 87.6%. The low balance values could have been due to absorption capacity of the CO_2 traps, as well. This is indicated by the balance range (97 to 104%) for days 20 to 51 when CO_2 production had decreased. However, an additional study was conducted and submitted under MRID 41889010 to indicate that acceptable balance values could be obtained. In this study (Anaerobic soil Metabolism Study-MRID 41889020) the average balance value was 88.5% (range 77.5 to 99.9%) of the applied radioactivity. The study author felt the material balance was adequate considering the tremendous difficulties in achieving adequate balances when a lot of initial CO_2-evolution is involved.

COMMENTS:

1. EFGWB prefers that [^{14}C]residues in samples be separated by chromatographic methods (such as TLC, HPLC, and GC) with solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the R_f of reference standards.

   In this study, the samples were analyzed using TLC using one solvent systems and Radio-TLC. Radioactive areas on the TLC plates identified only by comparison to the location of known reference standards.

2. The soil fractions for the test soil was reported in a manner that the USDA soil texture of the soils used in the study could not be verified. However, it does appear that both soils are similar and that the soil texture reported as loam would not be a loam soil under USDA classification. For loam soils with clay content of ≤15% the sand content is <55% using USDA classification. See Appendix 3 for details.

Soil Size Reported for Sandy Loam and Loam			USDA Soil Sizes	
0.2-0.02	-sand=82%	=68%	2.0-0.05	-sand
0.02-0.002	-silt=10%	=17%	0.05-0.002	-silt
<0.002	-clay= 8%	=15%	<0.002	-clay

3. The half-life reported for this aerobic study was consistent with the half-life reported for the aerobic portion of the anaerobic study. However, sample intervals were insufficient to determine a degradation pathway.

4. The detection limit was not furnished. However, data was reported to the third decimal place for parts per million (0.002 ppm).

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