DATA EVALUATION RECORD

STUDY 1

CHEM 109101 MEPIQUAT CHLORIDE §161-3

STUDY ID 41889009

Wood, N.R. PHOTOLYSIS OF MEPIQUAT CHLORIDE ON SOIL. Performed by BASF Corporation Agricultural Chemicals, Research Triangle Park, NC under Registration Document No. BASF 91/5079 and BASF Report No. M9117; Sponsored and Submitted by BASF Corporation Agricultural Chemicals, Research Triangle Park, NC; Completed 17 May 1991; Received by EPA 27 May 1991.

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REVIEWED BY: G. Maske
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 305-5245
SIGNATURE: 7 OCT 1993

Approved by: P. Mastradone
TITLE: Chief, Section 1
ORG: EFGWB/EFED/OPP
TEL: 305-5335
7 OCT 1993

CONCLUSIONS:

Photodegradation on soil

The photodegradation on soil study is of uncertain value and cannot be used to fulfill the data requirement (161-3). In order to validate the TLC analysis, EFGWB prefers a confirmatory method such as MS in addition to comparison to the Rf of reference standards. In addition, the vitality and moisture content of soil were not monitored during the testing period. It appears there was low microbial activity in the soil since there was a discrepancy between the reported half-lives (stable vs. 1-21 days) for mepiquat chloride in the photodegradation on soil control results and the aerobic soil metabolism (MRID 00127749-$t_{1/2}$=1-17 days and 42412103-$t_{1/2}$=3-21 days) and the aerobic portion of the anaerobic soil metabolism (MRID 41889010) results.

Mepiquat chloride was reported by the registrant to be photolytically stable when applied to sandy loam soil and exposed to an intermittent light source. At termination of the study, mepiquat chloride recovered in light exposed samples and dark control samples was 85.5% and 88.6%, respectively.

MATERIALS AND METHODS:

Test Material: 1,1-dimethylpiperidinium chloride was used which was reported to have a specific activity of 6.11 Ci/mole, radiochemical purity of 97.2% and chemical purity of >95%.

Soil: A sandy loam soil collected from the BASF Research Station in Dinuba, CA was used which had the following characteristics:

pH: 6.4
CEC(meq/100g): 18.79

% Organic matter:	4.3
% Field moisture:	14.22
Bulk density (g/cm^3):	1.26
% Sand:	54
% Silt:	32.4
% Clay:	10.4

Sampling: Light exposed soil samples and volatile traps were sampled at 0, 7, 14, 22, and 30 days posttreatment. The dark control samples were sampled at 0, 14, and 30 days posttreatment.

Test System: See Figures 1 and 2.

METHODOLOGY:

Five gram soil samples were weighed into each half of the glass trays and 5.0 mL of methanol containing 32.17 µg of mepiquat chloride was added to each. The methanol was allowed to evaporate to give a 2-mm thick soil layer with a surface area of 10.62 sq. cm in each half of the tray. Fortification of soil was equivalent to 0.28 kg/ha (0.31 lb/A).

The trays were sealed in the reaction vessels and exposed to an intermittent light source at 1800 microeinsteins m^{-2} sec^{-1} using a xenon arc lamp equipped with filters to mimic natural sunlight (wavelengths <290 nm were filtered out). A light intensity of 1800 microeinsteins m^{-2} sec^{-1} is equivalent to that at noon on a cloudless day in the middle of November at the Research Triangle Park, NC. The samples were exposed to the light source for 12 hours followed by a 12 hour dark period which was repeated for a total of 30 cycles.

To maintain the soil at a constant temperature of 25±1°C, water at 25±1°C from a cooling bath was circulated through the water jacket at each reaction vessel.

To collect any volatiles produced during the study, air was drawn through the reaction vessels and a series of scrubbing bottles containing 150 mL of liquid. On the inlet side was water to moisten the air, and on the outlet side was carbitol, which is a general solvent for organic volatiles. Sulfuric acid solution (0.5N) was used to trap basic volatiles, and 0.5N NaOH was used to trap CO_2.

At intermediated intervals of 7 cycles, a soil sample and the volatile traps were removed for analysis. Liquid samples were radioassayed by liquid scintillation. Soil samples were radioassayed by combustion. The aliquots of carbitol and sulfuric acid traps (1 mL) and NaoH traps (0.1 mL) were analyzed directly.

Soil samples were extracted immediately after the sampling interval. Each sample was transferred to an Erlenmeyer flask, and then 25 mL of water and 100 mL of DCM containing 7.5 mg of dipicrylamine were added. The flask was shaken for one hour and the slurry filtered. After separation of the DCM extract, it was made up to exactly 100.0 mL and radioassayed.

When a second extraction was necessary, the soil and the aqueous solution from the first extraction was combined with an additional 100 mL of DCM containing 7.5 mg of dipicrylamine in a blender. After 22 days a third extraction was performed. The extraction sequence is depicted in Figure 3.

TLC was performed using Solvent System A: acetonitrile:methanol:conc. hydrochloric acid (60:39:1); Solvent system B: methanol:acetic acid:water

(25:4:8) and Solvent System C: butanol:acetic acid:water (40:10:50) to develop.

Control experiments were also performed in which samples were kept in the dark at 25±1°C.

Material balances are shown in Table 1 for the dark control soils and in Table 2 for the light exposed soils.

DATA SUMMARY:

Mepiquat chloride, appears to be photolytically stable under the test conditions. Therefore, the registrant stated that a photolytic half-life for mepiquat chloride on soil could not be determined.

All combined DCM extract were examined by TLC using System A. The samples appeared to contain only mepiquat chloride as the radioactive residue. However, this finding was confirmed with System C. The registrant noted that the complex of mepiquat chloride with dipicrylamine in DCM decomposed to mepiquat chloride during the conditions employed in TLC.

Data in Tables 1 and 2 show that with time the mepiquat chloride was more strongly bound to the soil. Initially a single extraction with a mixture of water and dichloromethane containing the ion-pairing reagent dipicrylamine was sufficient to remove most of the applied mepiquat chloride into the organic layer. Later two such extractions were required. After 22 days, a third extraction was required in which the soil was boiled with 0.5N NaOH before addition of the extractant.

The extractability averaged 95.0% for the light exposed soils and 95.9% for the dark control soils (See Tables 3 and 4).

Volatile residue produced during light exposure appeared to be negligible. Volatile results are shown in Table 5.

The material balance averaged 95.7% for the light exposed soils and 94.3% for the dark control soils. See Table 2 and Table 1

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 Rf of reference standards.

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

2. The soil moisture content was not monitored during the testing period. A soil moisture content of 75% of ⅓ bar field capacity should be maintained during the testing period.

3. Methanol was used as the pesticide solvent for treatment of the test soil. In other studies, water has been the pesticide solvent.

4. Vitality of the soil was not monitored.

5. There is a discrepancy between the half-lives of mepiquat chloride reported for the photodegradation on soil study and the aerobic soil metabolism study. The half-life reported for mepiquat chloride when applied to sandy

loam soil and exposed to a light source and dark was stable (no degradation). The half-lives reported for mepiquat chloride when applied to sandy loam soil and clay loam soil and incubated in the dark under aerobic conditions were reported to be 1 to 17 days.

6. Control samples were taken at ever other sampling interval. EFGWB prefers that control samples be taken at each sampling interval.

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Mepiquat Chloride MRID 41889009

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