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DATA EVALUATION RECORD

STUDY 4

CHEM 109101

Mepiquat Chloride

§164-1

STUDY ID 42353301

Eubanks, M.W. PIX PLANT REGULATOR (BAS 0823 05 W) SOIL DISSIPATION IN A COTTON USE PATTERN STUDY. Performed by Pan-Agricultural Laboratories, Inc., Medera, CA; Biosperics, Inc., Beltsville, MD; Harris Environmental Technologies, Lincoln, NE under BASF Study No. 9044 and Registration Document No. BASF 92/5083; Sponsored and Submitted by BASF Corporation, Research Triangle Park, NC; Completed June 1992; Received by EPA 12 June 1992.

STUDY ID 42369401

Burkey, J.D. IC METHOD FOR DETERMINATION OF MEPIQUAT CHLORIDE (BAS 083 W) RESIDUES IN SOIL. Submitted and Performed by BASF Corporation, Research Triangle Park, NC under Registration Document No. BASF 92/5067; Completed on 18 May 1992; Received by EPA 24 June 1992.

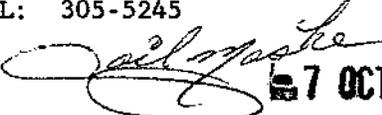
STUDY ID 42369402

Burkey, J.D. FREEZER STORAGE STABILITY OF BAS 083 W IN SOIL (5-MONTH INTERVAL) - INTERIM REPORT. Submitted and Performed by BASF Corporation, Research Triangle Park, NC under Registration Document BASF No. 92/5065; Completed on May 1992; Received by EPA 24 June 1992.

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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:

Terrestrial Field Dissipation

This terrestrial field dissipation study is of uncertain value because the following items make interpretation of the data impossible.

1. Time 0 posttreatment recoveries for the first applications (ranging from 23% to 53% of applied) were low.
2. Time 0 posttreatment recoveries for applications 2 and 3 (<0 to 37%, 53 to 74% of applied, respectively) were variable and low. Pretreatment samples were taken prior to application 1, 2, and 3. Mepiquat chloride residues present prior to applications 2 and 3 were taken into account in these calculations.
3. Recoveries for analytical methodology (MS soil = $74.9 \pm 10.9\%$, TX soil = $71.1 \pm 11.5\%$, CA soil $78.0 \pm 18.1\%$) were variable and low.
4. Recoveries for fortified storage stability samples were low ($\approx 77\%$ of fortified level) at 5 months and were not reported for up to the 10 months test samples in this study were stored.

In addition, the study cannot be used to fulfill the data requirement (164-1). Metabolites were not addressed during the study. All data submitted was cumulative for parent and metabolites. Metabolites should be analyzed for in field data to fully understand the degradation pathway and dissipation of mepiquat chloride in the environment.

These studies are of uncertain value but indicate that mepiquat chloride appears to degrade when applied to fields in the southwest and west. Half-lives of 3 days, 21 days, and 17 days were reported for mepiquat chloride residues when applied to bare ground sites in Mississippi, Texas, and California. Mepiquat chloride residue was not detected below the 0"-6" soil depth at the Mississippi and Texas sites. However, at the California site, mepiquat chloride was reported discernible at the 0"-12" soil depth immediately after the third application at a maximum level of 0.03 ppm.

MATERIALS AND METHODS:

Test Material: PIX Plant Growth Regulator manufactured by BASF Corporation Chemicals Division, Parsippany, NJ. The percent active ingredient, n,n-dimethylpiperidinium chloride, is 4.24%.

Reference Standards: N,N-dimethylpiperidinium chloride which had a chemical purity of 99.5% was used for analytical standard.

Soil: Greenville, MS - See Table I
Idalou, TX - See Table II
Medera, CA - See Table III

Sampling: Greenville, MS-Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 30, 35, 40, 45, 50, 60, 90, 181, 270, and 366 after third

Idalou, TX-Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 30, 35, 40, 45, 49, 60, 91, 180, 270, and 360 after third application.

Medera, CA-Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 31, 36, 41, 45, 50, 60, 90, 192, 270, and 360 after third application.

Test System: Bare soil was treated with three applications of PIX Plant Regulator at an average of 0.220, 0.110, and 0.328 lb/ai/A for Greenville, MS, at an average of 0.224, 0.108, and 0.350 lb/ai/A for Idalou, TX, at an average of 0.233, 0.113, and 0.353 lb/ai/A for Medera, CA.

METHODOLOGY:

Greenville, MS

PIX Plant Regulator was applied to a bare soil plot, 100' by 66.5', with a slope of <1% and a water table depth of ≈6' in Greenville, MS. The test plot sampled measured 90' by 50' and was divided into 135 subplots each measuring ≈6' by 6'. A control or untreated plot was located 130.5' up-slope from the test plot and measured 129.5' by 127'. The test plot had no history of pesticide use in the previous five years. However, Roundup (glyphosate) was used as a maintenance pesticide during the testing period. No other pesticides were applied to the test plot.

PIX was applied in three separate applications to the test plot using ground broadcaster-CO₂ pressurized tractor mounted boom sprayer. The first application, 0.220 lb ai/A, was made at the time a near by cotton field was in bloom. The second application, 0.110 lb ai/A, was made ≈14 days later. The third application, 0.328 lb ai/A, was made ≈30 days prior to crop harvest of near by cotton field. Therefore, a total application of 0.658 lbs ai/A was applied to the bare soil plot. This was typical application practice.

Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 30, 35, 40, 45, 50, 60, 90, 181, 270, and 366 after third application. In addition, 20 soil cores were collected from the control plot. Control core samples were collected as 0-48" depth soil cores and were sectioned into 0-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. Whereas, the soil core samples were sectioned into 0-3", 3-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. The treated soil samples consisted of separated 0-3" and 3-6" cores and which was followed by a

continuous core from 6-48". All samples were frozen and shipped to the analytical laboratory for analysis, BASF Corporation at Research Triangle Park, NC.

Total irrigation and precipitation during the testing period was 140.7% (6.59 inches) of the previous decade average, 1980-1989. Other weather data was furnished, as well.

Soil samples were refluxed in 0.5N NaOH after which contaminants were precipitate with an acidic medium. To form a mepiquat chloride-dipicrylamine complex, the acidic soil extracts were extracted with dipicrylamine and partitioned into dichloromethane. The dichloromethane soil samples were then extracted with hydrochloride acid and purified by column chromatography and analyzed by ion chromatography with conductivity detection.

Idalou, TX

PIX Plant Regulator was applied to a bare soil plot, 60' by 106', with a slope of <1% and a water table depth of \approx 250' in Idalou, TX. The test plot sampled measured 60' by 96' and was divided into 144 subplots each measuring \approx 6' by 6.67'. A control or untreated plot was located 40' upslope from the test plot and measured 50 by 40'. The test plot had no history of pesticide use in the previous five years. However, Roundup (glyphosate) and Prowl (pendimethalin) were used as maintenance pesticides during the testing period. No other pesticides were applied to the test plot.

PIX was applied in three separate applications to the test plot using ground broadcaster-CO₂ pressurized tractor mounted boom sprayer. The first application, 0.224 lb ai/A, was made at the time a near by cotton field was in bloom. The second application, 0.108 lb ai/A, was made \approx 14 days later. The third application, 0.350 lb ai/A, was made \approx 30 days prior to crop harvest of near by cotton field. Therefore, a total application of 0.682 lbs ai/A was applied to the bare soil plot. This was typical application practice.

Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 30, 35, 40, 45, 49, 60, 91, 180, 270, and 360 after third application. In addition, 20 soil cores were collected from the control plot. Control core samples were collected as 0-48" depth soil cores and were sectioned into 0-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. Whereas, the soil core samples were sectioned into 0-3", 3-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. The treated soil samples consisted of separated 0-3" and 3-6" cores and which was followed by a continuous core from 6-48". All samples were frozen and shipped to the analytical laboratory for analysis, BASF Corporation at Research Triangle Park, NC.

Total irrigation and precipitation during the testing period was 147.0% (36.3 inches) of the previous thirty year average, 1960-1989. Other weather data was furnished, as well.

Soil samples were refluxed in 0.5N NaOH after which contaminants were precipitate with an acidic medium. To form a mepiquat chloride-dipicrylamine complex, the

acidic soil extracts were extracted with dipicrylamine and partitioned into dichloromethane. The dichloromethane soil samples were then extracted with hydrochloride acid and purified by column chromatography and analyzed by ion chromatography with conductivity detection.

Medera, CA

PIX Plant Regulator was applied to a bare soil plot, 57' by 100', with a slope of <1% and a water table depth ranging from 88' to 107' in Medera, CA. The test plot sampled measured 57' by 92' and was divided into 154 subplots each measuring ≈4' by 7'. A control or untreated plot was sampled and assumed to be upslope from the test plot. The test plot had no history of pesticide use in the previous five years. However, Roundup (glyphosate) was used as maintenance pesticide during the testing period. No other pesticides were applied to the test plot.

PIX was applied in three separate applications to the test plot using ground broadcaster-CO₂ pressurized tractor mounted boom sprayer. The first application, 0.233 lb ai/A, was made at the time a near by cotton field was in bloom. The second application, 0.113 lb ai/A, was made ≈14 days later. The third application, 0.353 lb ai/A, was made ≈30 days prior to crop harvest of near by cotton field. Therefore, a total application of 0.699 lbs ai/A was applied to the bare soil plot. This was typical application practice.

Soil samples were collected prior to (T1) and immediately after (T1 same date) after 1st, 2nd (T2, T2 same date), and 3rd (T3, T3 same date) application, and days 1, 3, 5, 7, 9, 12, 15, 18, 21, 25, 31, 36, 41, 45, 50, 60, 90, 192, 270, and 360 after third application. In addition, 20 soil cores were collected from the control plot. Control core samples were collected as 0-48" depth soil cores and were sectioned into 0-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. Whereas, the soil core samples were sectioned into 0-3", 3-6", 6-12", 12-18", 18-24", 24-30", 30-36", 36-42", and 42-48" segments. The treated soil samples consisted of separated 0-3" and 3-6" cores and which was followed by a continuous core from 6-48". All samples were frozen and shipped to the analytical laboratory for analysis, BASF Corporation at Research Triangle Park, NC.

Total irrigation and precipitation during the testing period was 291.5% (29.27 inches) of the previous thirty year average, 1960-1989. Other weather data was furnished, as well.

Soil samples were refluxed in 0.5N NaOH after which contaminants were precipitate with an acidic medium. To form a mepiquat chloride-dipicrylamine complex, the acidic soil extracts were extracted with dipicrylamine and partitioned into dichloromethane. The dichloromethane soil samples were then extracted with hydrochloride acid and purified by column chromatography and analyzed by ion chromatography with conductivity detection.

DATA SUMMARY:

Analytical data prior to and immediately following treatment indicated that mepiquat chloride residues in the 0-3" soil depth degraded to ≤0.04 ppm by day

seven posttreatment at the Mississippi and Texas sites 0.07 ppm by day nine posttreatment at the California site and to undiscernible levels by days 7, 60, and 360 posttreatment, respectively. Mepiquat chloride residues were not discernible below the 0-6" soil depth at the Mississippi, and Texas sites. However, mepiquat chloride residues were detected at a maximum level of 0.03 ppm at the 0-12" soil depth immediately after the third application at the California site. Therefore, it appears that the 3-6" and 6-12" soil depths contained

This data is based on average recoveries of applied test material which ranged from 23% to <100% for the three applications at each site. Average recoveries (0-3" soil depth) for the three applications at Mississippi, Texas, and California were 23% (1st application), 27% (2nd application), and 73% (3rd application); 45% (1st application), 83% (2nd application), and 74% (3rd application); and 51% (1st application), <100% (2nd application), and 53% (3rd application), respectively.

The detection limit was reported as 0.01 ppm for IC analysis. However, soil samples from the three test sites were fortified at levels of 0.01, 0.05, 0.50, and 1.0 ppm mepiquat chloride. For soil samples fortified using soil from the Mississippi, Texas, and California sites, the average recovery was $74.9 \pm 10.9\%$ (Mississippi site), $71.1 \pm 11.5\%$ (Texas site), and $92.7 \pm 9.9\%$ (California site, Biosperics analysis) and $78.0 \pm 18.1\%$ (California site, Harris ENVIRO Tech. analysis). In addition, storage stability data furnished indicated that fortified soil samples were stable for at least 5 months which is 5 months short of the 10 month sample storage prior to analysis. However, the storage stability study is to be continued for up to 3 years (36 months) with the results being reported in a later document.

COMMENTS:

1. EFGWB prefers that samples for analytical analysis 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 IC. No confirmation analysis such as MS was furnished.

2. The detection limit was reported as 0.01 ppm. Two control samples were fortified with mepiquat chloride in each analytical set using fortification levels of 0.01, 0.05, 0.50, and 1.0 ppm. For soil samples fortified using soil from the Mississippi, Texas, and California sites, the average recovery was $74.9 \pm 10.9\%$ (Mississippi site), $71.1 \pm 11.5\%$ (Texas site), and $92.7 \pm 9.9\%$ (California site, Biosperics analysis) and $78.0 \pm 18.1\%$ (California site, Harris ENVIRO Tech. analysis).
3. Average recoveries for the three applications at Mississippi, Texas, and California were 23% (1st application), 27% (2nd application), and 73% (3rd application); 45% (1st application), 83% (2nd application), and 74% (3rd application); and 51% (1st application), <100% (2nd application), and 53% (3rd application), respectively.

4. Recoveries of fortified storage stability samples were $\approx 77\%$ of fortification level.
5. The author states that on-going storage stability data indicates that mepiquat chloride in soil is stable for at least 5 months. However, samples were stored up to 10 months prior to analysis. The additional data is needed prior to acceptance of this study to validate the analytical analysis.