

DATA EVALUATION RECORD

STUDY 1

CHEM 109101

MEPIQUAT CHLORIDE

\$164-1

STUDY ID 43415401

Evans, J.R. and Anderson, S.E. 1991 PONNAX VINEYARD SOIL DISSIPATION STUDY SUMMARY REPORT. Sponsored and Submitted by BASF Corporation Chemical Division, Research Triangle Park, NC under Study No. 91007 and Report No. ER93024; Chemical Analysis Performed by Harris Laboratories, Inc., Lincoln, NE and Biospherics Incorporation, Beltsville, MD; Study completed on 14 October 1991; Received by EPA 19 October 1994.

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CONCLUSIONS:

Dissipation - Terrestrial field:

This terrestrial field dissipation study is scientifically valid. In addition, it can be used to partially fulfill the data requirement (\$164-1). These data submitted are only for the bare ground portion of the study. They do not include the vineyard portion of the study. It is EFGWB understanding that the vineyard portion of the study will be submitted at a later date. These data are needed to better understand the environmental fate of mepiquat chloride when applied to vineyards.

These data indicate that mepiquat chloride is relatively non-persistent under New York field conditions to moderately persistent under Washington and California field conditions. In addition, mepiquat chloride appears to be relatively non-mobile under all three field conditions. Half-lives for mepiquat chloride ranged from 6.5 to 87.2 days for the three test sites, and mepiquat residues were not detected below the 0-6 inch soil depth, except for two test samples during the test periods. Furthermore, except for the New York test site, these data indicate that higher mepiquat chloride application rates have longer half-lives.

For the New York Test site, a non-linear regression half-life of 6.5 days was calculated with a correlation coefficient of 0.87. The highest residue concentration, 0.78 ppm, was detected on day 9 posttreatment. However, the discernible residues concentration dropped to 0.05 and 0.01 ppm by day 30 posttreatment. All detectable residues were located in the top 0-6 inch soil depth. In addition, the data indicated that the majority of the residues remained in the 0-3 inch soil depth as opposed to the 3-6 inch soil depth.

For the Washington Test site, a half-life of 71.9 days was calculated with a correlation coefficient of 0.93. The highest residue concentration, 0.38 ppm, was detected on day 24 posttreatment. However, the discernible residues concentration dropped to 0.01 ppm by day 360 posttreatment. All detectable residues were located in the top 0-6 inch soil depth except for one replicate at 90 days posttreatment where 0.02 ppm was discernible in the 6-12 inch soil depth sample. In addition, the data indicated that the majority of the residues remained in the 0-3 inch soil depth as opposed to the 3-6 inch soil depth.

For the California Test site, a half-life of 87.2 days was calculated with a correlation coefficient of 0.94. The highest residue concentration, 0.68 ppm, was detected in the immediately after application sample (T1). However, the discernible residues concentration dropped to 0.39 ppm by day 150 posttreatment and 0.03 ppm by day 360 posttreatment. All detectable residues were located in the top 0-6 inch soil depth except for the sample taken immediately after application (T1). A mepiquat chloride concentration of 0.04 ppm was reported in the 6-12 inch soil depth sample. However, no mepiquat chloride residue was detected below the 0 to 6 inch soil depth the following day nor at any other sampling interval.

#### MATERIALS AND METHODS:

**Test Material:** Ponnax is the end product used for the study. It is a solution (active ingredient is mepiquat chloride) formulated (23.0% a.i. BAS 083 19W) for vineyard uses.

**Reference Standards:** Mepiquat chloride with a chemical purity of 99.3%

**Soil and Test sites:** See Figure 1

New York - 2.5 miles south of Phelps, NY in Ontario County. For characterization of the soil on the New York test plot see Table 1. The soil on the plot site was reported to be sandy loam for the 0 to 36" soil depth and loam for the 36 to 48" soil depth.

Washington - 0.25 mile north of Vancouver, WA in Clark County. For characterization of the soil on the Washington test plot see Table 3. The soil on the plot site was reported to be sandy loam for the 0 to 48 inch soil depth.

California - 6 miles southeast of Chualar, CA in Monterey County. For characterization of soil on the California test plot see Table 5. The soil on the plot site was reported to be sandy clay loam except for the 12 to 24 inch soil depth (characterized to 48 inches) which was reported to be silt loam.

**Sampling intervals:** Soil cores were collected immediately prior to application (-T1), immediately after application (T1), and at 1, 3, 5, 7, 9, 11, 13, 15, 18, 21, 24, 30, 35, 40, 45, 50, 60, 90, 120, 150 (New York site testing terminated), 330, and 360 days posttreatment.

The pre-application sampling interval (-T1) was

the only time samples were collected from the control plot.

Analytical Method: Ion pair Chromatography

Testing Period: Approximately 1 year (360 days) for the Washington and California sites and 150 days for New York site

METHODOLOGY:

In 1991 three field sites (New York, Washington, California) were chosen for testing the end-product Ponnax (active ingredient is meperquat chloride). Each field site was divided into three plots (a control or untreated plot, a treated vineyard plot, and a bare ground treated plot. The vineyard and bare ground plots further divided into subplots and treated with Ponnax at an application rate of 0.75 which is 3X the recommended label application rate.

The New York test site was located 2.5 miles south of Phelps, NY in Ontario County. The sandy loam bare ground plot measured 60' by 75', and was treated with a backpack sprayer (See Table I). The control plot appears to be 26' from the treated bare ground plot directly to the west. Precipitation (rainfall + irrigation) during the testing period for the New York site was similar to the 10 year average (36.4 vs 31.9, respectively) (See Table II).

The Washington test site was located 0.25 miles north of Vancouver, WA in Clark County. The sandy loam bare ground plot measured 30' by 150', and was treated with a ground application sprayer (mounted boom) (See Table III). The location of the control plot was not reported. Precipitation (rainfall + irrigation) during the testing period for the Washington site was approximately 2X the 10 year average (88.1 vs 40.0, respectively) (See Table IV).

The California test site was located 6 miles southeast of Chualar, CA in Monterey County. The sandy clay loam (12 to 24" soil depth was silt loam) bare ground plot measured 26.67' by 175', and was treated with a tractor mounted boom (See Table V). The location of the control plot was not reported. Precipitation (rainfall + irrigation) during the testing period for the California site was approximately 4.5X the 10 year average (56.1 vs 12.2, respectively).

Soil cores from each of the test sites were collected immediately prior to application (-T1), immediately after application (T1), and at 1, 3, 5, 7, 9, 11, 13, 15, 18, 21, 24, 30, 35, 40, 45, 50, 60, 90, 120, 150 (New York site testing terminated), 330, and 360 days posttreatment. The pre-application sampling interval (-T1) was the only time soil cores were collected from the control plot. Soil cores were collected to a depth of 48 inches at each sampling interval in triplicate from each of five randomly selected subplots. Soil cores were cut into 6 inch segments (0 to 6, 6 to 12, 12 to 18, 18 to 24, 24 to 30, 30 to 36, 36 to 42, and 42 to 48 inch). The 0 to 6 inch soil segment was further cut into a 0 to 3 inch and 3 to 6 inch soil segments. Test samples were placed in coolers for transfer to field laboratory. Prior to shipment to BASF Corporation the three replicate soil segments were composited and stored and shipped in freezers. BASF Corporation, upon receiving the frozen test samples, shipped them to Harris Laboratories, Inc. or to Biospherics Incorporated for analysis (See Table VIII). Depending on detections in the two previous soil depths and sampling times, some test samples were determined to unnecessary and not analyzed.

Soil samples were extracted by refluxing in 0.5N NaOH. After an acidic medium was used to precipitate contaminants, the test material was isolated

in the form of a dipicrylamine-complex and partitioned in dichloromethane. The dipicrylamine-complex was then extracted with an acidic solution to decomplex the test material. The acidic solution extract was then purified by alumina column chromatography and analyzed by ion chromatography (See Figure 2).

The half-lives were calculated using a first order non-linear model. The regression curve used to fit the residue values for soil describes the data empirically and should not be used to imply the only one process of dissipation is involved.

Fortified samples were analyzed by both the Harris and Biospherics Laboratories. In addition, weather data for the testing period and detailed information of each test site was furnished in the study.

#### DATA SUMMARY:

This terrestrial field study was initiated to support registration of mepiquat chloride on vineyards. Therefore, the application rate reflects a higher application rate of 0.75 lb a.i./A. Previous field dissipation data reviewed for mepiquat chloride reflected the lower application rate (0.022 to 0.044 lb a.i./A) for cotton. Except for the New York test site (half-life = 6.5 days) longer half-lives were reported for the higher application rate (3 to 21 days vs 71.9 to 87.2 days). (See Table VII).

For the New York Test site, a non-linear regression half-life of 6.5 days was calculated with a correlation coefficient of 0.87. The highest residue concentration, 0.78 ppm, was detected on day 9 posttreatment. However, the discernible residues concentration dropped to 0.05 and 0.01 ppm by day 30 posttreatment. All detectable residues were located in the top 0-6 inch soil depth. In addition, the data indicated that the majority of the residues remained in the 0-3 inch soil depth as opposed to the 3-6 inch soil depth (See Table IX).

For the Washington Test site, a half-life of 71.9 days was calculated with a correlation coefficient of 0.93. The highest residue concentration, 0.38 ppm, was detected on day 24 posttreatment. However, the discernible residues concentration dropped to 0.01 ppm by day 360 posttreatment. All detectable residues were located in the top 0-6 inch soil depth except for one replicate at 90 days posttreatment where 0.02 ppm was discernible in the 6-12 inch soil depth sample. In addition, the data indicated that the majority of the residues remained in the 0-3 inch soil depth as opposed to the 3-6 inch soil depth (See Table X).

For the California Test site, a half-life of 87.2 days was calculated with a correlation coefficient of 0.94. The highest residue concentration, 0.68 ppm, was detected in the immediately after application sample (T1). However, the discernible residues concentration dropped to 0.39 ppm by day 150 posttreatment and 0.03 ppm by day 360 posttreatment. All detectable residues were located in the top 0-6 inch soil depth except for the sample taken immediately after application (T1). A mepiquat chloride concentration of 0.04 ppm was reported in the 6-12 inch soil depth sample. However, no mepiquat chloride residue was detected below the 0 to 6 inch soil depth the following day nor at any other sampling interval (See Table XI).

Therefore, these data indicate that mepiquat chloride is relatively non-persistent under New York field conditions to moderately persistent under Washington and California field conditions. In addition, mepiquat chloride appears to be relatively non-mobile under all three field conditions.

The verification of the application rate for each site was based on the 0-6 inch soil depth test sample results. The initial recoveries (T1) for the

application rate was 115%, 93%, and 178% of applied test material for New York, Washington, and California test sites, respectively

Samples fortified at 0.01, 0.05, 0.50, and 1.00pp were analyzed with concurrently with test samples. Samples analyzed at Harris Laboratories, Inc had average recoveries of  $100.2\% \pm 6.2$  for the New York test soil,  $80.7\% \pm 7.6$  for the Washington test soil, and  $83.1\% \pm 7.8$  for the California test soil. Samples analyzed by Biospherics Incorporated had average recoveries of  $87.0\% \pm 9.7$  for the New York test soil,  $85.3\% \pm 14.7$  for the Washington test soil, and  $87.7\% \pm 10.2$  for the California test soil. The residues levels reported were not corrected for these recoveries.

COMMENTS:

1. Since these data did not include the treated vineyard portion of the study, methodology concerning the vineyard portion of the study was not addressed in this study. These data will be submitted and reviewed at a later date.
2. Soil cores were divided into six inch soil segments. However, the surface 0-6 inch soil segment was further divided into a 0-3 inch soil segment and a 3-6 inch soil segment. Even though most of the test material was detected in the 0-3 inch soil segment, the data was averaged and reported for the 0-6 inch soil segment as opposed to the 0-3 inch soil segment. Half-lives and dissipation rates may differ for the 0-3 inch soil segment and should be calculated, as well.
3. It appears that at all three test sites there was an increase in the concentration of the test material at approximately 7-15 days posttreatment. This was not addressed by the study authors. These increases do not appear to be hot spots since triplicate random samples were taken at each sampling interval and all test samples had similar test results. In addition, it appears that this increase in residues was a factor in the longer field half-lives reported especially for the Washington and California test sites. However, the study authors state that this trend was found in rotational crop studies (half-lives = 11 to 18 days for NC and 175 days for CA).
4. The initial recovery (T1) for the California site was 178% of applied test material. Based on the concentration mepiquat chloride in the 0-3 and 3-6 inch soil samples, there is a concern that the application rate of the liquid formulation was not 0.75 lb a.i./A at the test site. However, these data do indicate that higher mepiquat chloride application rates have longer half-lives, but the residues are still relatively non-mobile.
5. The test samples were stored for up to 39 months prior to completion of analysis. Storage stability data for mepiquat chloride at 40 months in a Texas soil (only one test site) were previously submitted in this action. It should be noted that EFGWB prefers that storage stability samples be prepared in the field (at each test site) at concentrations which reflect the application rate(s) and handled in the same manner as the test samples. This testing method for storage stability data is believed to better reflect stability of the test samples during transfer and storage. Future storage stability studies should be carried out in this manner.
6. In a previous terrestrial field study using the application rate for cotton, the analytical methodology used in this study was considered

not sensitive enough. The application rate used for cotton is 0.022 to 0.044 lb a.i./A (not to exceed 0.066 lb a.i./A/season). Therefore, since the limit of quantitation for the analytical methodology is 0.010 ppm, approximately 25 to 50% of the applied test material for cotton would not be accounted for using this analytical methodology, and the analytical method was not sensitive enough to determine the fate of mepiquat chloride for cotton uses. However, the sensitivity of the analytical method is sufficient to understand the fate of mepiquat chloride when applied to vineyards which has an application rate of 0.25 lb a.i./A.

7. This terrestrial field dissipation study appears to be generally in agreement with previous supplemental terrestrial field data. Both laboratory and previous supplemental field dissipation data indicated that mepiquat chloride was relatively non-mobile (Kds of 10-25 and detected only in the 0-6 inch soil depth). However, shorter half-lives are usually reported for field dissipation data than for laboratory data. The half-life for the NY test site was approximately the same (6.5 vs 3 to 21 days) as the half-lives reported for laboratory data. However, the Washington and California test sites had longer half-lives (71.9 and 87.2 days). No explanation was given for the longer half-lives, but irrigation/rainfall did not appear to be a factor. Vegetation prior to study initiation may have been a factor in soil microbial population.
8. Test samples were analyzed by IC. IC analysis was previous used in laboratory or supplemental field data. Method validation and confirmation of methodology by mass spectrometry (MS) was included with the data.

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Mepiquat Chloride MRID 41889006

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