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DATA EVALUATION RECORD

STUDY 6

CHEM METALAXYL 164-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY MRID 422598-03
Leech, G. and T. Wiepke. 1992. Aquatic dissipation of metalaxyl (Ridomil 2E) in a California rice paddy. Pan-Agricultural Laboratories, Inc. Study No. EF-90-326, Twin City Testing Corporation Study No. 31/90-CGA.1 and Ciba-Geigy Protocol No. 91/90. Performed by Pan-Agricultural Laboratories, Inc., Madera, CA and Twin City Testing Corporation, St. Paul, MN and submitted by Ciba-Geigy Corporation, Greensboro, NC. 272 pp.

DIRECT REVIEW TIME = 5

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CONCLUSIONS:

Aquatic Field Dissipation

1. EFGWB concludes that this study is scientifically valid and provides supplemental information that shows metalaxyl to dissipate from rice paddy water with a calculated half-life of 20 days and from soil with a half-life of 24 days. The study is not acceptable because:

   Soil samples containing metalaxyl and its major degradate, CGA-62826, were stored frozen for up to 259 days before analysis; however, a storage stability experiment with soil was performed for only 105 days. Therefore, the registrant will have to provide data that shows metalaxyl and CGA-62826 are stable when stored frozen for up to 259 days.

METHODOLOGY:

The field study was conducted on an Alamo clay (fine, montmorillonitic, thermic, Typic Duraquolls, Table I) at Pan-Agricultural Laboratories, Inc., Madera, CA (Figure 2) during 1990-91. Metalaxyl was applied preflood at the rate of 0.50 lb ai/A to the soil of three experimental 0.1 acre (50 feet by 100 feet) rice paddies. Rice paddy water was sampled at the day of flooding (1 day after application), and at 1, 3, 7, 14, 21, 30, 62 and 92 days after application (DAA). Sampling of the soil/hydrosoil to a depth of 12 inches occurred on the same schedule as water, except no soil samples were collected at 21 DAA, and continued after draining the field at 122, 154, 178 and 259 DAA. An untreated control plot was sampled like the treated plots.

In order to verify the application rate, 50 ml samples were collected from two tank mixes for determination of metalaxyl. In addition, four glass petri plate 9-cm bottoms were placed in each of the treated paddies just prior to application. After application the petri plates were collected and analyzed for metalaxyl. The rice plots were a closed system and water was not circulated through or between treated plots. The water levels in the plots was maintained at a depth of about 4 to 6 inches by a float valve interfaced with a water meter.

All samples were kept frozen within a temperature range of -15 to 24 °F prior to shipping and analysis. In order to determine stability of metalaxyl and CGA-62826, during transport, control soil and well-water were fortified with a known concentration of each.

Residues of metalaxyl and CGA-62826 were extracted from the acidified water with methylene chloride:ethyl acetate and concentrated to dryness. The residue was transferred to a test tube using methylene chloride, evaporated to dryness and derivatized with BF_3-butanol reagent to convert the CGA-62826 into the butyl ester derivative (Figure 1). The derivatized CGA-62826 and unreacted metalaxyl were extracted into hexane, taken to dryness, dissolved in isooctane, and analyzed by GC with a nitrogen-phosphorous detector (NPD).

Residues of metalaxyl and CGA-62826 in soil were first extracted with methanol:water and then treated as above for water samples. The limit of quantitation for both analytes was 1 and 10 ppb in water and soil, respectively.

Concentration of metalaxyl in spray solutions was determined by dilution with isooctane and analysis by GC with NPD. Determination of metalaxyl residue in the petri dishes was performed by quantitative transfer with isooctane and analysis by GC with NPD.

Method validation was accomplished by fortifying water and soil prior to sample analysis. Water samples were fortified with 1, 5, 20, 100 and 500 g/l

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of metalaxyl and CGA-62826; while soil samples were fortified with 10, 50 and 500 ng/g. Parent and degradate were analyzed as noted above.
A storage stability experiment was established by spiking well water and control soil with metalaxyl or CGA 62826 and then analyzed at storage intervals of about 1, 2 and 3 months.

When appropriate, least squares regression analysis was applied to natural log (ln) transformed residue data over time in order to determine if decline in residues followed a 1st-order degradation rate. If linear correlation values were >0.7, a 1st-order degradation rate was assumed and a half-life ($t_{1/2}$) was calculated.

STUDY AUTHORS' DATA SUMMARY:

All results were corrected for procedural recoveries and percent moisture, where appropriate.

Application Verification:

Metalaxyl concentration in the top 3 inches of soil on the day of application averaged 347 ppb (Table III) which is 69% of the theoretical concentration of 500 ppb. Metalaxyl concentration in the two spray solution samples averaged 2.6 g/l which is 87% of the theoretical concentration of 3 g/l (Table VI). The petri plates averaged 314 g metalaxyl/plate or 88% of the theoretical concentration of 358 g/plate (Table VII). These data, along with the documented calibration, mixing and application (Appendix D), verify the application rate of 0.5 lb ai/A.

Freezer Stability:

Recovery of metalaxyl fortified-water after 34, 62 and 97 days of freezer storage averaged 86, 86 and 90%, respectively. At the same storage intervals, the recovery of CGA-62826 averaged 108, 87 and 105%, respectively (Table 11 from Appendix B).

Recovery of metalaxyl from fortified soil stored for 33, 61 and 105 days in a freezer averaged 83, 68 and 93%, respectively; while recovery of CGA-62826 for the same intervals averaged 117, 104 and 107%, respectively (Table 12 from Appendix B).

Recoveries of water samples fortified in the field, and shipped to the laboratory by overnight carrier or freezer truck averaged $\geq$90% (Table 7), soil samples fortified in the field averaged 84% (overnight carrier) and 104% (freezer truck) and recoveries of CGA-62826 averaged 107% (Table 8 from Appendix B).

The study author concludes that the data supports the stability of metalaxyl and CGA-62826 in water and soil during transport to the laboratory, and subsequent freezer storage for three months. All the water samples were extracted within 20 to 70 days of sampling. Soil samples were extracted within 62 to 259 days of sampling. The study authors report that an ongoing storage stability study will be submitted at a later date to provide details on soil samples stored longer than 3 months.

Metalaxyl Residues in Water:

The results of the paddy water sample analyses are shown in Table II. Means and total residues are present in Table IV. The maximum concentration of metalaxyl in water (68 ppb) occurred at 3 DAA. Decline was thereafter rapid, with concentrations below limit of quantitation (LOQ = 1 ppb) in two paddies and at 2.6 ppb in the third paddy just before draining by evaporation. The theoretical half-life ($t_{\frac{1}{2}}$) of metalaxyl in rice paddy water was 20 days (Figure 6 and 7, r=0.82). Peak concentration of CGA-62826, averaging 5.5 to 6.4 ppb occurred in water at 3, 21 and 30 DAA. Residues declined to an average of 2.9 ppb just before drainage.

Metalaxyl Residues in Soil:

The results of the soil sample analyses are shown in Table III. Means and total residues are presented in Table V. Generally, metalaxyl was not detected at or above the LOQ (10 ppb), below the top 3 inches of soil, with the exception of a few near LOQ detections in the 3-6 and 6-9 inches depth increments on the day of application, and then 1, 3, 7 and 14 DAA. (The study authors concluded from this that residues of metalaxyl below the top 3 inches on the day of application were the result of contamination). Decline of metalaxyl in the top 3 inches of soil was rapid, with a theoretical half-life of 24 days (Figure 6 and 8, r=0.85). Residues of CGA-62826 were detected below the top 3 inches, starting 14 DAA and throughout the 0-12 inch sampling depth by 92 DAA. Residues declined in all depth increments to essentially the LOQ by 122 to 154 DAA.

Conclusions:

The overall results of this study indicate that the dissipation of metalaxyl should be fairly rapid with a $t_{\frac{1}{2}}$ = <30 days in the water plus soil of water-seeded rice in California. Some apparent mobility of CGA-62826 in the soil occurred in the study; however, the concentration found below the top 6 inches generally remained near the LOQ of 10 ppb.

REVIEWER'S COMMENTS:

1. The study authors indicated that the storage stability study is continuing and will provide details on stability of soil samples stored longer than 3 months. This additional information is needed to indicate whether the pesticide and its degradates in the soil samples from the test plots, stored longer than 3 months, are degrading during handling and storage, and if so, whether it is possible to normalize the results to account for the amount of change during storage.

2. EFGWB notes that in the anaerobic metabolism study (MRID 104494) CGA-62826 was the only major metabolite found, besides CO_2; while, nonextractable residues were 8.4% of the applied.

METALAXYL

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