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DATA EVALUATION RECORD

STUDY 2

CHEM 128997 Tebuconazole §164-1

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Valadez, S. K., B. A. Dehart, and C. V. Lam. 1994. Terrestrial field dissipation of tebuconazole on Georgia soil, 1991. Miles Report No.: 106446. Unpublished study performed by Miles Research Farm, Tifton, GA (in-life phase); Miles Research Park, Stilwell, KS (recording and compositing); and Miles Product Support Analytical Laboratory, Kansas City, MO (analytical phase); and submitted by Miles, Inc., Kansas City, MO.

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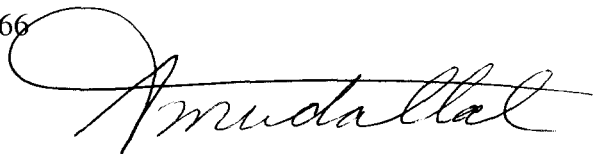
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ABSTRACT

Tebuconazole (FOLICUR 3.6 F), broadcast applied three times (14-day intervals) at a nominal application rate of 0.25 lb a.i./A/application and once (14 days following the third application) at a nominal application rate of 0.60 lb a.i./A (total application rate of 1.35 lb a.i./A) onto a plot (peanuts were planted six days prior to the fourth application) of Lakeland sand soil near Tifton, GA, dissipated with a registrant-calculated half-life of 349.4 days ($r^2 = 0.73$) following the fourth application. The parent was present in the 0- to 6-inch depth at 0.10, 0.21, and 0.20 $\mu\text{g/g}$ immediately following the first, second, and

third applications, respectively, and was not detected above 0.02 $\mu\text{g/g}$ in any replicate below the 0- to 6-inch depth. Following the fourth application, the parent was present in the 0- to 6-inch depth at 0.35-0.38 $\mu\text{g/g}$ from 0 to 3 days posttreatment, was a maximum of 0.46 $\mu\text{g/g}$ at 10 days, was 0.24-0.37 $\mu\text{g/g}$ from 14 to 273 days, and was 0.11-0.13 $\mu\text{g/g}$ from 455 to 546 days. The parent was only detected once in the 6- to 12-inch depth above 0.01 $\mu\text{g/g}$, at 0.12 $\mu\text{g/g}$ (single replicate) at 3 days posttreatment, and was not detected below that depth. Soil samples were not analyzed for degradates of tebuconazole. Peanut plants were not analyzed for the parent or degradates.

MATERIALS AND METHODS

Tebuconazole { α -[2-(4-chlorophenyl)ethyl]- α -(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol; FOLICUR 3.6 F; p. 15; Figure 1, p. 83} was broadcast applied three times (14-day intervals; July 16, July 30, and August 13, 1991) at a nominal application rate of 0.25 lb a.i./A/application and once (14 days following the third application; August 27, 1991) at a nominal application rate of 0.60 lb a.i./A (total application rate of 1.35 lb a.i./A) onto a plot (100 x 108 ft divided into five equal subplots; slope 2%) of Lakeland sand soil (90.7% sand, 8.0% silt, 1.3% clay, 0.9% organic matter, pH 7.0, CEC 5.5 meq/100 g; Table 3, p. 30) near Tifton, GA; peanuts were planted on August 21, 1991. Applications were made using a trailer-mounted conventional sprayer with twelve Tee Jet 8002 flat-fan nozzles and a delivery height of 19 inches. No control plot was mentioned. Round-Up[®] (glyphosate, 1.0 lb a.i./A) plus the wetting agent Penetrator 3[®] was applied to the plot on July 18 and August 16, 1991. A complete plot history was not reported (Table 2, p. 29). The depth to the water table was >150 feet. Environmental data were collected on-site (p. 17). Precipitation was supplemented with irrigation (overhead sprinkler); total water input (82.39 inches) was 107% of the 10-year mean annual precipitation (Tables 5-7, pp. 33-57). Pan evaporation data were not reported.

The application rate was confirmed using six application pads placed in each subplot immediately prior to each application (p. 16). Immediately following each application, the pads were composited by subplot and extracted by shaking with acetonitrile. Samples were shipped frozen to the analytical laboratory and analyzed by HPLC (Brownlee C18 column) using an isocratic mobile phase of acetonitrile:water (4:1, v:v) and equipped with a UV detector (wavelength not specified; p. 20). Mean recoveries of the parent from the application monitoring pads were 82%, 46%, 84%, and 69% of the expected for the first, second, third, and fourth applications, respectively (Table 8, p. 58). Mean recoveries of the parent from the soil (all depths) were 71%, 82%, 49%, and 51% of the expected for the first, second, third, and fourth applications, respectively (p. 24).

Soil samples were collected 4- to 6-days prior to the first application, immediately following the first, second, third, and fourth applications, and at 1, 3, 6, 10, 14, 28, 63, 91, 190, 273, 364, 455, and 546 days posttreatment (relative to the fourth application; Table

29, p. 82). At each sampling interval, three soil samples (2 3/8-inch diameter for samples collected following the first application; 1 7/8-inch diameter for all other samples) were randomly collected from each treated subplot (15 cores total; p. 17). Samples were collected using a Giddings sampler device equipped with a plastic liner. Soil cores were collected to a depth of 10 inches immediately following the first application, and to a minimum depth of 31 inches at all other sampling intervals. Samples were stored frozen at the field facility until being shipped frozen to the processing laboratory. At the processing laboratory, 1/16th-1/8th inch of the outer soil cores was shaved and discarded; samples were sectioned into 6-inch increments and composited by depth. The composited samples were shipped frozen to the analytical laboratory. Samples were stored frozen for up to 409 days prior to analysis (p. 22). Peanut plants were not collected for analysis.

Samples were analyzed only for the parent compound. Soil samples were extracted by refluxing for four hours with methanol:water (7:3, v:v; p. 18); the samples were cooled and vacuum-filtered through Celite. The filtrate was concentrated by rotary evaporation and partitioned three times with methylene chloride. The organic phase was filtered through sodium sulfate, which was rinsed three times with methylene chloride. The organic phase was concentrated by rotary evaporation and evaporated to dryness under nitrogen. The residue was reconstituted in ethyl acetate and the solution was filtered (0.45 μm); aliquots were analyzed by capillary GC with nitrogen-phosphorous detection. The limit of detection was 0.01 $\mu\text{g/g}$ (p. 21). Instrument operating conditions were as follows:

Analytical Column: HP-1; 50 m x 0.32 mm

Injection Port: 250°C isothermal

Nitrogen-Phosphorous Detector: 300°C isothermal

Column Oven Temperature Program: 180°C for 1 minute, 180°C to 230°C at 10°C per minute, hold at 230°C for 20 minutes

Flow Rates: Carrier gas - 2 mL/minute helium; Combustion make-up gas - 26 mL/minute nitrogen, 4.5 mL/min hydrogen, and 170 mL/min air

In a method validation study, soil samples were fortified with tebuconazole at 10, 20, and 50 ppb (p. 20). Mean recoveries (across both fortifications) were $92 \pm 15\%$ (3 of 20 samples outside 70-120%; p. 22; Figures 7-9, pp. 89-91).

To determine concurrent recoveries, soil samples were fortified with tebuconazole at 0.05, 0.1, and 0.5 $\mu\text{g/g}$ (p. 23). Mean recovery (across all fortifications) of the parent was $103 \pm 10\%$ (range of 93 to 137%).

In a transit stability study of fortified field spikes, triplicate soil samples were fortified with tebuconazole at 1.0 $\mu\text{g/g}$ at each sampling interval (p. 16). Samples were transported in the same manner as the test samples and stored for up to 559 days (p. 22).

Data indicated that the parent was stable for up to 559 days; mean recoveries (across all sampling intervals) of the parent were 1.0-1.4 $\mu\text{g/g}$ with the exception of 3.9 $\mu\text{g/g}$ at day 364 (Table 9, pp. 60-62). Mean recovery from concurrent fortifications was $103 \pm 12\%$ (range of 90 to 137%; p. 23).

RESULTS/DISCUSSION

Tebuconazole (FOLICUR 3.6 F), broadcast applied three times (14-day intervals) at a nominal application rate of 0.25 lb a.i./A/application and once (14 days following the third application) at a nominal application rate of 0.60 lb a.i./A (total application rate of 1.35 lb a.i./A) onto a plot (planted with peanuts six days prior to the fourth application) of Lakeland sand soil near Tifton, GA, dissipated with a registrant-calculated half-life of 349.4 days ($r^2 = 0.73$; Figure 67, p. 149) following the fourth application. Data are means of three replicates. The parent was present in the 0- to 6-inch depth at 0.10, 0.21, and 0.20 $\mu\text{g/g}$ immediately following the first, second, and third applications, respectively, and was not detected above 0.02 $\mu\text{g/g}$ in any replicate below the 0- to 6-inch depth (Tables 11-13, pp. 64-66). Following the fourth application, the parent was present in the 0- to 6-inch depth at 0.35-0.38 $\mu\text{g/g}$ from 0 to 3 days posttreatment, was a maximum of 0.46 $\mu\text{g/g}$ at 10 days, was 0.24-0.37 $\mu\text{g/g}$ from 14 to 273 days, and was 0.11-0.13 $\mu\text{g/g}$ from 455 to 546 days (Tables 14-27, pp. 67-80). The parent was only detected once in the 6- to 12-inch depth above 0.01 $\mu\text{g/g}$, at 0.12 $\mu\text{g/g}$ (single replicate) at 3 days posttreatment (Table 16, p. 69), and was not detected below that depth. Soil samples were not analyzed for degradates of tebuconazole. Peanut plants were not analyzed for the parent or degradates.

DEFICIENCIES/DEVIATIONS

1. The pattern of formation and decline of degradates of tebuconazole were not addressed. Soil samples were not analyzed for degradates of the parent. One of the primary purposes of a terrestrial field dissipation study is the determination of the pattern of formation and decline of major degradates of the parent. The reviewer did not have access to metabolism studies of tebuconazole.
2. Pan evaporation data were not reported. Such data are necessary to determine water balances and to assess whether sufficient moisture was present to facilitate leaching of the test substance.
3. The peanut plants were not analyzed for the parent or degradates. It is necessary that total residues in the crop be monitored in order to accurately determine the routes of dissipation of the test compound. The study authors did not state whether the peanuts were harvested or remained on the plot throughout the study period.

4. The study authors stated that the half-life of the parent was “determined by summing residues at each sampling interval ($\mu\text{g/g}$) from each depth,” rather than using data from only the top 6 inches. The reviewer noted that the parent was not observed to leach.
5. The reviewer was unable to determine whether related compounds were applied to the test plot within the previous three years because a complete plot history was not reported (Table 2, p. 29).
6. The reviewer noted that the subplots were not true replicate plots (separated by buffer zones).
7. The study authors stated that the “total desired rate applied was 150% of the current label rate of 0.90 lb a.i. per acre for peanuts” (p. 24). The use of exaggerated dose rates may effect the degradation rate of the chemical relative to the degradation rate that would occur under normal use rates. However, the reviewer noted that recoveries from application monitoring pads were 46-84% of the expected.
8. The formulation of the test compound was reported as “FOLICUR 3.6 F.” However, because the reviewer was unable to determine the formulation, the reviewer reported the formulation as not identified (formulation code 90).
9. Units were not reported for CEC values reported in Table 3 (p. 27); the reviewer reported the units as meq/100 g.
10. The reviewer noted a discrepancy. On page 16, the study authors stated that extracts from the application monitoring pads were analyzed by GC; however, it was stated on page 20 that samples were analyzed by HPLC.
11. The reviewer noted that additional terrestrial field dissipation studies were also submitted.

ATTACHMENT 1
Tables cited in DER

THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY
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ATTACHMENT 2
Excel Workbook

0-6 inch depth

Sampling interval (days)	Parent (ug/g)	ln parent (ug/g)
0	0.48	-0.733969
0	0.35	-1.049822
0	0.31	-1.171183
1	0.31	-1.171183
1	0.35	-1.049822
1	0.39	-0.941609
3	0.40	-0.916291
3	0.28	-1.272966
3	0.43	-0.84397
6	0.31	-1.171183
6	0.21	-1.560648
6	0.24	-1.427116
10	0.56	-0.579818
10	0.39	-0.941609
10	0.44	-0.820981
14	0.42	-0.867501
14	0.36	-1.021651
14	0.32	-1.139434
28	0.41	-0.891598
28	0.29	-1.237874
28	0.29	-1.237874
63	0.29	-1.237874
63	0.24	-1.427116
63	0.18	-1.714798
91	0.34	-1.07881
91	0.30	-1.203973
91	0.29	-1.237874
190	0.30	-1.203973
190	0.32	-1.139434
190	0.26	-1.347074
273	0.26	-1.347074
273	0.26	-1.347074
273	0.29	-1.237874
364	0.20	-1.609438
364	0.20	-1.609438
364	0.18	-1.714798
455	0.15	-1.89712
455	0.10	-2.302585
455	0.14	-1.966113
546	0.09	-2.407946
546	0.12	-2.120264
546	0.12	-2.120264

Half-life (days) = 346.6

