US EPA ARCHIVE DOCUMENT

Text Searchable File

US EPA ARCHIVE DOCUMENT

DATA EVALUATION RECORD

STUDY 4

CHEM 128997 Tebuconazole §164-1
CAS No. 107534-96-3
FORMULATION–06–WETTABLE POWDER

STUDY ID 44108312
Valadez, S. K., Dehart, B. A., and Lam, C. V. 1995. Terrestrial field dissipation of tebuconazole on North Carolina soil, 1991. Performing Laboratory Study ID: ETI - 1032BS01. Submitting Laboratory ID: FR022109. Miles Report No.: 106448. Unpublished study performed by Environmental Technologies Institute, Inc., Research Triangle Park, NC (in-life phase); Miles Research Park, Stilwell, KS (processing and compositing); and Miles Product Support Analytical Lab, Kansas City, MO (analytical phase); and submitted by Miles Inc., Agricultural Division, Kansas City, MO.

DIRECT REVIEW TIME =

REVIEWED BY: Andrew Glucksman	SIGNATURE:
TITLE: Staff Scientist	DATE:
EDITED BY: Dan Hunt	SIGNATURE:
TITLE: Staff Scientist	DATE:
EDITED BY: Kathleen Ferguson	SIGNATURE:
TITLE: Senior Staff Scientist	DATE:

ORG: Dynamac Corporation
Rockville, MD
TEL: 301-417-9800

APPROVED BY: Amer Al-Mudallal
TITLE: Chemist
ORG: ERB 1/EFED/OPP
TEL: 703-605-0566
SIGNATURE: AMudallal 3/13/2000

ABSTRACT

Field Dissipation - Terrestrial

Tebuconazole (LYNX 25 DF), broadcast applied three times (14-day intervals) as a spray at a nominal application rate of 0.75-0.76 lb a.i./A/application, dissipated with a registrant-calculated half-life of 340.5 days ($r^2 = 0.53$; 14-48 day data) following the third application on a bareground plot of Pocalla sand soil in North Carolina. However, the

2045880

observed half-life occurred between 58 to 90 days posttreatment. The parent compound was present in the 0- to 6-inch depth at 0.28-0.32 μg/g immediately following each of the first two applications. Following the third application, the parent compound was initially (time 0) 0.54 μg/g, was a maximum of 1.1 μg/g at 14 days following the final application, decreased to 0.66-0.68 μg/g by 28-58 days, and generally decreased to 0.27 μg/g by 548 days. The parent compound was not detected in the 6- to 12-inch depth above 0.04 μg/g, and was not detected below the 6- to 12-inch soil depth. No degradates were identified from the soil.

US EPA ARCHIVE DOCUMENT

MATERIALS AND METHODS

Tebuconazole {α-[2-(4-Chlorophenyl)ethyl]-α-(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol; LYNX 25 DF©, 26.2% a.i.; p. 14; Figure 1, p. 62} was broadcast applied three times (July 23, August 6, August 20, 1991) as a spray at a nominal application rate of 0.75-0.76 lb a.i./A/application (total application rate of 2.27 lb a.i./A; p. 14) onto a bareground plot (112.5 x 66.7 ft divided into five equal subplots, slope 0-3%; Figure 4, p. 65) of Pocalla sand soil (97% sand, 2% silt, 1.3% clay, 0.7% organic matter, pH 5.0, CEC 4.3 meq/100 g; Table 3, p. 29) in Rowland, North Carolina. Applications were made using a tractor-mounted sprayer with six Tee Jet flat-fan nozzles spaced 20 inches apart and a boom height of 18 inches above the soil surface. The depth to the water table was ≥12 feet. Each plot was treated with Round-up (glyphosate; July 8, August 16, 1991; Table 4, pp. 30-31). A five-year plot history indicated no prior use of tebuconazole or related compounds (Table 2, p. 28). Precipitation was supplemented with irrigation; total water input (93.5 inches) was 145% of the 10-year mean annual precipitation (Table 7, p. 53). Precipitation data were collected on-site. Pan evaporation data were not reported. Control plots were not mentioned.

The application rate was confirmed immediately prior to each application using six application pads (14 x 22 cm) located in each subplot (p. 15). Immediately following each application, the pads were composited by subplot. The pads were extracted by shaking with acetonitrile. Samples were shipped frozen to the analytical laboratory and analyzed by HPLC (Brownlee C 18 column, 25 x 0.4 cm) with UV (220 nm) detection (p. 19). Mean recoveries from application monitoring pads were 83%, 125%, and 79% of the expected for the first through third applications, respectively (Table 8, p. 54). Mean recoveries of the parent in the 0- to 6-inch soil depth were 67%, 38%, and 43% of the expected for the first through third applications, respectively (p. 22).

Soils were sampled 1 day prior to the first application; immediately following each application; and 1, 3, 5, 10, 14, 28, 58, 90, 182, 272, 375, 456, and 548 days posttreatment (relative to the third application; Table 4, pp. 30-31). At each sampling interval, three soil cores were randomly collected from each of the treated subplots (15 cores total; p. 15). A 0- to 6-inch depth core was collected following the first application and a 0- to 9-inch depth core was collected following the second application; 0- to 37-inch depth cores were sampled at all other sampling intervals. Samples were collected using a Giddings Sampler Device with a plastic liner (1.75 inch i.d.). Samples were transported on ice and stored frozen at the field facility until being shipped frozen to the processing laboratory. At the processing laboratory, 1/16- to 1/8-inch of the outer soil core was scraped and discarded; the core was sectioned into 6-inch increments and composited by depth. Composited samples were shipped frozen to the analytical lab for analysis. Samples were stored frozen for up to 1108 days prior to analysis (p. 20).

Soil samples were analyzed only for the parent material (p. 17). Soil samples (50 g) were extracted by refluxing for 4 hours with methanol:water (7:3, v:v). Samples were cooled and vacuum-filtered through Celite; the reflux flask was rinsed twice with solvent. 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 with ethyl acetate and the solution was filtered (0.45 μm); aliquots were analyzed by capillary GC with nitrogen-phosphorus detection (p. 18). The limit of detection was 0.01 μg/g (p. 20). Instrument operating conditions were as follows (p. 17):

Analytical Column: HP-1, 50 m x 0.32 mm id; Hewlett-Packard
Injection Port: 250°C isothermal
Nitrogen-Phosphorus 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 - 2.0 mL/minute helium; Detector make-up gas - 26 mL/minute nitrogen, 4.5 mL/minute hydrogen, and 170 mL/minute air

In a method validation study, soil samples were fortified with tebuconazole at 10, 20, and 50 μg/kg (p. 19). Mean recoveries were 87 ± 11% for the 10 μg/kg fortification, 82 ± 11% for the 20 μg/kg fortification, and 98 ± 13% for the 50 μg/kg fortification (p. 21; Appendix 1, pp. 88-90).

To determine concurrent recoveries, soil samples at each sampling interval (with the exception of samples following the initial field application) were fortified with tebuconazole at 0.05 μg/kg and 0.1 μg/kg (p. 21). Mean recoveries (across both fortifications) were 102 ± 9% (range of 85-115%).

In a transit stability study of fortified field spikes, triplicate soil samples were fortified with tebuconazole at 1 μg/g at each sampling interval (p. 15). Samples were transported and stored (up to 1108 days) in the same manner as the test samples. Mean recoveries of the parent (across all sampling intervals) were 0.90-1.2 μg/g, with the exception of 1.7 μg/g at 375 days posttreatment (Table 9, p. 55). A frozen storage stability test was

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

performed; the data indicated that the parent compound was stable when frozen for up to 566 days (tabular data not reported; p. 20). Mean recoveries from concurrent fortifications were 100 ± 5% (range of 85-105%; p. 21).

RESULTS/DISCUSSION

Tebuconazole (LYNX 25 DF), broadcast applied three times (14-day intervals) as a spray at a nominal application rate of 0.75-0.76 lb a.i./A/application, dissipated with a registrant-calculated half-life of 340.5 days ($r^2 = 0.53$; 14-48 day data; Figure 25, p. 86) following the third application on a bareground plot of Pocalla sand soil in North Carolina. However, the observed half-life occurred between 58 to 90 days posttreatment. Residue data were reported as means of three replicates. "Days posttreatment" refers to the days following the final application. The parent compound was present in the 0- to 6-inch depth at 0.28-0.32 μg/g immediately following each of the first two applications (Table 10, p. 56). Following the third application, the parent compound was initially (time 0) present at 0.54 μg/g, was a maximum of 1.1 μg/g at 14 days posttreatment, decreased to 0.66-0.68 μg/g by 28-58 days, and generally decreased to 0.27 μg/g by 548 days (Tables 11-13, pp. 57-59). The parent compound was not detected in the 6- to 12-inch depth above 0.04 μg/g, and was not detected below the 6- to 12-inch soil depth. No degradates were identified from the soil.

DEFICIENCIES/DEVIATIONS

1. The pattern of formation and decline of the degradates was not addressed. Soil samples were not analyzed for degradates of tebuconazole. 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 an aerobic soil metabolism study to determine if degradates were observed during aerobic soil metabolism.

2. Pan evaporation data were not provided. Such data are necessary to determine water balances and assess whether sufficient moisture was present in the soil to facilitate leaching of the test substance.

3. The observed and registrant-calculated half-lives were not similar. The observed half-life of the parent in the 0- to 6-inch depth occurred between 58 and 90 days (0.30-0.66 μg/g of the applied remaining) following the third application (Tables 11-13, pp. 57-59). The registrant-calculated half-life was 340.5 days ($r^2 = 0.53$; days 14-548; Figure 25, p. 86). However, the registrant-calculated half-life may be of questionable validity because it was determined by summing the residues from each depth at each sampling interval (Table 15, p. 61). The half-life should be based on the top 0- to 6-inch depth, rather than the entire soil core. The reviewer noted that the parent was not observed to leach.

4. The reviewer could not determine whether subplots were true replicate plots (separated by buffer zones; Figure 4, p. 65).

5. The formulation of the test compound was reported as "LYNX 25 DF" (p. 10). However, because no formulation code exists for the dry flowable formulation, the reviewer designated the material as a wettable powder (formulation code 06).

6. The maximum label application rate was 2.04 lb a.i./A (p. 22).

7. The reviewer noted that units for CEC values were not reported in Table 3 (p. 29). The reviewer reported units as meq/100 g. Clarification from the study authors may be required.

8. Additional terrestrial field dissipation studies were also submitted.

US EPA ARCHIVE DOCUMENT

US EPA ARCHIVE DOCUMENT

ATTACHMENT 1
Tables cited in DER

THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY
SEE THE FILE COPY

US EPA ARCHIVE DOCUMENT

ATTACHMENT 2
Excel Workbook

US EPA ARCHIVE DOCUMENT

Parent [ug/g]				
Rep 1	Rep 2	Rep 3	Average [ug/g]	ln average
0.56	0.73	0.32	0.54	-0.62238
0.39	0.6	1.46	0.82	-0.20252
0.56	0.23	0.26	0.35	-1.04982
0.52	1.78	0.21	0.84	-0.17833
1.02	1.33	0.43	0.93	-0.07616
0.43	2.12	0.77	1.11	0.10135
0.72	0.72	0.61	0.68	-0.38077
0.58	0.44	0.96	0.66	-0.41552
0.49	0.2	0.21	0.30	-1.20397
0.23	0.4	0.43	0.35	-1.04034
0.21	0.46	0.28	0.32	-1.14991
0.15	0.12	0.46	0.24	-1.41332
0.15	0.58	0.19	0.31	-1.18199
0.37	0.3	0.13	0.27	-1.3218

Half-life (days) = 346.6