

US EPA ARCHIVE DOCUMENT

## DATA EVALUATION RECORD

## STUDY 1

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CHEM 128997	Tebuconazole	§164-1
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CAS No. 107534-96-3

FORMULATION-06-WETTABLE POWDER

STUDY ID 44108309

Valadez, S. K., B. A. Dehart, and C. V. Lam. 1994. Terrestrial field dissipation of tebuconazole on North Carolina turf, 1991. Miles Report No.: 106236. Unpublished study performed by Environmental Technologies, Inc., Research Triangle Park, NC (in-life phase); Miles Inc., Stilwell, KS (recording and compositing); and Miles Inc., Kansas City, MO (analytical phase); and submitted by Miles, Inc., Kansas City, MO.

DIRECT REVIEW TIME =

REVIEWED BY: Dan Hunt

SIGNATURE:

TITLE: Staff Scientist

DATE:

EDITED BY: Andrew Glucksman

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:



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ABSTRACT

Tebuconazole (LYNX 25 DF), broadcast applied three times (14-day intervals) at an application rate of 0.70-0.74 lb a.i./A (total application rate of 2.18 lb a.i./A) onto a plot of sand soil planted with Bermuda grass near Rowland, NC, dissipated with a registrant-calculated half-life of 100.4 days ( $r^2 = 0.35$ ; 1-119 day data) following the third application. The half-life was determined based on the calculated tebuconazole concentration for the combined grass and soil core (0- to 18-inch depth) at each sampling interval. Immediately following the first application, the parent was present in the 0- to



3-inch and 3- to 6-inch soil depths at 0.68  $\mu\text{g/g}$  and 0.04  $\mu\text{g/g}$ , respectively. Immediately following the second application, the parent was present in the 0- to 6-inch depth at 0.38  $\mu\text{g}$  and was not detected below that depth. Immediately following the third application, the parent was present at 0.59  $\mu\text{g/g}$  and 0.06  $\mu\text{g/g}$  in the 0- to 6-inch and 6- to 12-inch depths, respectively. The parent was detected in the 0- to 3-inch depth at 0.06-0.10  $\mu\text{g/g}$  at 1-5 days posttreatment, was 0.51  $\mu\text{g/g}$  at 10 days, and was 0.17-0.42  $\mu\text{g/g}$  from 14 to 119 days. The parent was detected in the 3- to 6-inch depth at 0.01-0.05  $\mu\text{g/g}$  from 1 to 119 days posttreatment. The parent was detected only once in the 6- to 12-inch depth, at 0.01  $\mu\text{g/g}$  (90 days posttreatment), and was not detected below that depth. Soil samples were not analyzed for degradates of tebuconazole.

The parent was present in the grass at 2.9  $\mu\text{g/g}$  immediately following the first application, and the parent was not analyzed immediately following the second or third applications. The parent was present in the grass at 2.3-2.9  $\mu\text{g/g}$  from 1 to 5 days posttreatment, increased to 4.1-4.5  $\mu\text{g/g}$  by 10-28 days posttreatment, and was 1.5-1.8  $\mu\text{g/g}$  from 58 to 119 days posttreatment. Grass samples were not analyzed for degradates of tebuconazole.

## MATERIALS AND METHODS

Tebuconazole { $\alpha$ -[2-(4-chlorophenyl)ethyl]- $\alpha$ -(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol; LYNX 25 DF; p. 10; Figure 1, p. 54} was broadcast applied three times (14-day intervals; July 24, August 7, and August 21, 1991) at an application rate of 0.70-0.74 lb a.i./A/application (total application rate of 2.18 lb a.i./A) onto a plot (50 x 112.5 ft divided into five equal subplots; slope 2%; Figure 4, p. 57) of sand soil (94.7% sand, 4.0% silt, 1.3% clay, 0.9% organic matter, pH 5.3, CEC 4.8 meq/100 g; Table 3, p. 27) planted with Bermuda grass near Rowland, NC (pp. 13-14); the grass was planted on May 18, 1991. Applications were made using a tractor-mounted sprayer with six Tee Jet 8008 flat-fan nozzles and a boom height of 20-22 inches. No control plot was mentioned. A three-year plot history indicated use of Round-Up<sup>®</sup> (glyphosate), Dual 8E<sup>®</sup> (metolachlor) and Bicep 6L<sup>®</sup> (atrazine plus metolachlor; Table 2, p. 26). The depth to the water table was 12 feet (p. 13). The grass was mowed periodically; clippings were left on the plots. Environmental data were collected on-site (p. 14). Precipitation was supplemented with irrigation (tripod sprinkler); total water input (22.15 inches) was 134% of the 10-year mean annual precipitation (Tables 5-7, pp. 29-35). 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. 15). 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 GC (operating conditions not specified). Mean recoveries of the parent from the application monitoring

pads were 154%, 81%, and 96% of the expected for the first, second, and third applications, respectively (Table 8, p. 36). Mean recoveries of the parent from the top soil depth were 172%, 68%, and 54% of the expected for the first, second, and third applications, respectively (p. 21).

Soil samples were collected 14 days prior to the first application, immediately following the first, second, and third applications, and at 1, 3, 5, 10, 14, 28, 58, 90, and 119 days posttreatment (relative to the third application; Table 23, p. 52). At each sampling interval, three soil samples (2.25-inch diameter) were collected randomly from each treated subplot (15 cores total; p. 16). Samples were collected using a Giddings GSR T-S sampling device equipped with a plastic liner. Soil cores were collected to a depth of 6 inches immediately following the first application, and to a depth of 18 inches at all other sampling intervals. Samples were stored frozen at the field facility until being shipped frozen to the processing laboratory. Samples were sectioned into 0- to 3-inch, 3- to 6-inch, 6- to 12-inch, and 12- to 18-inch increments and composited by depth; samples collected immediately following the second and third applications were sectioned into 6-inch increments. The composited samples were shipped frozen to the analytical laboratory. Samples were stored frozen for up to 545 days prior to analysis (p. 21). Grass samples were collected immediately following the first application and at 1, 3, 5, 10, 14, 28, 58, 90, and 119 days posttreatment; however, the methods used for sample collection and analysis were not reported.

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. 17); 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  $\mu\text{m}$ ); aliquots were analyzed by capillary GC with nitrogen-phosphorous detection. The limit of detection was 0.01  $\mu\text{g/g}$  (p. 20). 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. 19). Mean recoveries were  $82 \pm 13\%$  for the 10 ppb fortification (1 of 5

samples <70%),  $90 \pm 11\%$  for the 20 ppb fortification, and  $100 \pm 2\%$  for the 50 ppb fortification (p. 21; Figures 8-10, pp. 61-63).

To determine concurrent recoveries, soil samples were fortified with tebuconazole at 0.05 and 0.1 mg/kg (p. 21). Mean recoveries (across both fortifications) were  $100 \pm 11\%$  (range of 79 to 129%).

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. 15). Samples were transported in the same manner as the test samples and stored for up to 557 days. Data indicated that the parent was stable for up to 557 days; mean recoveries (across all sampling intervals) of the parent were 1.0-1.4  $\mu\text{g/g}$  with the exception of 0.66  $\mu\text{g/g}$  at day 119 (Table 9, p. 37).

## RESULTS/DISCUSSION

Tebuconazole (LYNX 25 DF), broadcast applied three times (14-day intervals) at an application rate of 0.70-0.74 lb a.i./A (total application rate of 2.18 lb a.i./A) onto a plot of sand soil planted with Bermuda grass near Rowland, NC, dissipated with a registrant-calculated half-life of 100.4 days ( $r^2 = 0.35$ ; 1-119 day data; Figure 52, p. 105) following the third application. However, the half-life is of questionable validity because it was determined based on the calculated tebuconazole concentration for the combined grass and soil core (0- to 18-inch depth) at each sampling interval (Tables 23-24, pp. 52-53). The reviewer was unable to calculate half-lives of the parent in soil or grass due to variability in the data over time. Data are means of three replicates. Immediately following the first application, the parent was present in the 0- to 3-inch and 3- to 6-inch soil depths at 0.68  $\mu\text{g/g}$  and 0.04  $\mu\text{g/g}$ , respectively (Table 11, p. 40). Immediately following the second application, the parent was present in the 0- to 6-inch depth at 0.38  $\mu\text{g}$  and was not detected below that depth (Table 12, p. 41). Immediately following the third application, the parent was present at 0.59  $\mu\text{g/g}$  and 0.06  $\mu\text{g/g}$  in the 0- to 6-inch and 6- to 12-inch depths, respectively (Table 13, p. 42). The parent was detected in the 0- to 3-inch depth at 0.06-0.10  $\mu\text{g/g}$  at 1-5 days posttreatment, was 0.51  $\mu\text{g/g}$  at 10 days, and was 0.17-0.42  $\mu\text{g/g}$  from 14 to 119 days (Tables 14-22, pp. 43-51). The parent was detected in the 3- to 6-inch depth at 0.01-0.05  $\mu\text{g/g}$  from 1 to 119 days posttreatment. The parent was detected only once in the 6- to 12-inch depth, at 0.01  $\mu\text{g/g}$  (90 days posttreatment; Table 21, p. 50), and was not detected in the 12- to 18-inch depth. Soil samples were not analyzed for degradates of tebuconazole.

The parent was present in the grass at 2.9  $\mu\text{g/g}$  immediately following the first application and was not analyzed for immediately following the second or third applications (Tables 11-13, pp. 40-42). The parent was present in the grass at 2.3-2.9  $\mu\text{g/g}$  from 1 to 5 days posttreatment, increased to 4.1-4.5  $\mu\text{g/g}$  at 10-28 days posttreatment, and was 1.5-1.8

$\mu\text{g/g}$  from 58 to 119 days posttreatment (Tables 14-22, pp. 43-51). Grass samples were not analyzed for degradates of tebuconazole.

#### 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 registrant-calculated half-life of the parent is of questionable validity because the half-life was determined based on calculated tebuconazole concentrations for the combined soil and grass samples at each sampling interval (Tables 23-24, pp. 52-53), rather than using the top 6 inches of soil. The reviewer was unable to calculate half-lives for the parent in soil or grass because data were variable over time.
4. The method for the collection and analysis of grass samples was not reported. Additionally, method validation and concurrent recovery data were not reported for grass samples.
5. The study authors stated that the total application rate (2.18 lb a.i./A) was 109% of the current label rate (2.0 lb a.i./A), and that the minimum multiple application interval specified on the label is 14 days (p. 14).
6. The formulation of the test compound was reported as "LYNX 25 DF." However, because no formulation code exists for dry flowable formulations, the reviewer reported the formulation as a wettable powder (formulation code 06).
7. The reviewer noted that the subplots were not true replicate plots (separated by buffer zones; Figure 4, p. 57).
8. The soil series name was not reported.
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 that additional terrestrial field dissipation studies were also submitted.