DATA EVALUATION RECORD

STUDY 8

CHEM 128997 Tebuconazole §164-1
CAS No. 107534-96-3
FORMULATION–90–FORMULATION NOT IDENTIFIED

STUDY ID 44108316

Nonne, N. G., G. C. Mattern, and D. L. Green. 1996. Terrestrial field dissipation of tebuconazole (LYNX) on Wisconsin soil, 1993. Bayer Study No.: FR022112. Bayer Report No.: 107332. Unpublished study performed by AGSTAT, Verona, WI (in-life phase); Bayer Environmental Fate Analytical Laboratory, Stillwell, KS (analytical phase); QC, Inc., Southampton, PA (analytical phase); and Writers, Inc., Wilmington, DE (report preparation); and submitted by Bayer Corporation, Kansas City, MO.

DIRECT REVIEW TIME =

REVIEWED BY: Dan Hunt
TITLE: Staff Scientist
SIGNATURE:
DATE:

EDITED BY: Andrew Glucksman
TITLE: Staff Scientist
SIGNATURE:
DATE:

EDITED BY: Kathleen Ferguson
TITLE: Senior Staff Scientist
SIGNATURE:
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

Tebuconazole (LYNX 25), broadcast applied three times (14-day intervals) at nominal application rates of 0.75 lb a.i./A, 1.5 lb a.i./A, and 0.75 lb a.i./A, respectively (total application rate of 3.0 lb a.i./A) onto a plot (planted with grass seed five days following the third application) of Dickinson sandy loam soil near Belleville, WI, dissipated with a registrant-calculated half-life of 216.3 days ($r^2 = 0.86$) following the third application. Residue data were not reported for samples following the first application. It was not

specified whether the 0- to 6-inch depth soil core included the turf layer. Immediately following the second application, the parent was present in the 0- to 6-inch depth at 1.1 μg/g, was detected in the 6- to 12-inch depth at 0.02 μg/g, and was not detected below the 6- to 12-inch depth. Immediately following the third application, the parent was present in the 0- to 6-inch depth at 1.3 μg/g, was a maximum of 1.6 μg/g at 3 days posttreatment, decreased to 0.73-0.87 μg/g by 14-300 days, was 0.20-0.28 μg/g from 363-609 days, and was 0.06 μg/g at 790 days. The parent was detected sporadically in the 6- to 12-inch, 12- to 18-inch, and 18- to 24-inch depths at ≤0.06 μg/g (individual replicates). Soil samples collected below 24 inches were only analyzed at 300 and 363 days posttreatment; the parent was detected once in the 24- to 36-inch depth at 0.03 μg/g (single replicate) at 363 days posttreatment and was not detected below that depth. Following the third application, the degradate 1,2,4-triazole was detected in the 0- to 6-inch depth, at ≤0.02 μg/g (individual replicates) at 29 and 62 days posttreatment, and was not detected at any other sampling interval or depth.

MATERIALS AND METHODS

Tebuconazole {α-[2-(4-chlorophenyl)ethyl]-α-(1,1-dimethylethyl)-1*H*-1,2,4-triazole-1-ethanol; LYNX 25, 25% a.i.; p. 12; Figure 1, p. 90} was broadcast applied three times (14-day intervals; July 16, July 30, and August 13, 1993) at nominal application rates of 0.75 lb a.i./A, 1.5 lb a.i./A, and 0.75 lb a.i./A, respectively (total application rate of 3.0 lb a.i./A) onto a plot (78 x 133 ft divided into five equal subplots; slope ≤1%; Figure 4, p. 93) of Dickinson sandy loam soil (77.2% sand, 14.4% silt, 8.4% clay, 0.91% organic matter, pH 5.5, CEC 4.6 meq/100 g; Table 3, p. 41) near Belleville, WI (pp. 17-18); grass seed was planted on the test plot on August 18, 1993. Applications were made using a tractor-mounted boom sprayer with six flat-fan 8003 nozzles and a delivery height of 20 inches. A control plot (29 x 18 ft) was located >200 feet from the treated plot (Figure 3, p. 92). A three-year plot history indicated no prior use of tebuconazole or related compounds (Table 2, p. 40). Roundup (glyphosate, 1qt/A) was applied four days following the first application to control annual weeds (Table 4, p. 42). The depth to the water table was >5 feet (p. 17). The grass was mowed as necessary (Table 4, p. 42). Environmental data were collected on-site (p. 20). Precipitation was supplemented with irrigation (above-ground sprinkler); total water input (81.44 inches) was 108% of the 30-year mean annual precipitation (Tables 5-6, pp. 44-73). 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. 19). 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 (Microsorb C18 column; p. 27) using an isocratic mobile phase of acetonitrile:water (80:20, v:v), and equipped with a UV (220 nm) detector. Mean recoveries of the parent from the

application monitoring pads were 51%, 108%, and 57% of the expected for the first, second, and third applications, respectively (p. 30; Table 7, p. 74). Mean recoveries of the parent from the 0- to 6-inch soil depth were 129% and 102% of the expected for the second and third applications, respectively (p. 31); results were not reported for the first application.

Soil samples were collected from the treated plot 4 days prior to the first application, immediately following the first, second, and third applications, and at 1, 3, 5, 10, 14, 29, 62, 97, 249, 300, 363, 456, 609, and 790 days posttreatment (relative to the third application; p. 19); samples were collected from the control plot at 1, 249, 363, 609, and 790 days posttreatment. At each sampling interval, three soil samples were collected randomly from each treated subplot (15 cores total); samples were collected using a Concord probe equipped with an acetate plastic liner. Soil cores were collected to a depth of 6 inches (2.25-inch diameter) immediately following the first application, and to a minimum depth of 48 inches at all other sampling intervals; it was not specified whether the 0- to 6-inch depth soil cores included the turf layer. Samples were collected in two phases; 6- to 48-inch depth samples had a core diameter of 1.75 inches. Samples were shipped frozen to the processing laboratory. At the processing laboratory, soil samples were sectioned into 6-inch increments and composited by depth. The composited samples were shipped frozen to the analytical laboratory. Samples analyzed for tebuconazole and 1,2,4-triazole were stored frozen for up to 639 and 915 days prior to analysis, respectively (p. 28).

Soil samples were analyzed for the parent compound (p. 21). Samples were extracted by refluxing for four hours with methanol:water (7:3, v:v); 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 μg/g (p. 26). 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

Soil samples were analyzed for the degradate 1,2,4-triazole (p. 22). Samples were extracted with 0.01 M potassium phosphate buffered-water (pH 7.0), centrifuged, and the

supernatants were decanted through glass wool. The extracts were purified by passing through a column containing copper-activated Chelex 100. The extracts were derivatized with 2,4-dinitrofluorobenzene and partitioned with methylene chloride. Extracts were concentrated, reconstituted in toluene, and passed through a glass column plugged with glass wool, packed with activated silicic acid, and topped with anhydrous granular sodium sulfate. The extracts were concentrated, reconstituted in acetone:toluene (1:1, v:v), and analyzed by GC with a thermionic specific detector optimized for nitrogen. The limit of detection was 0.01 μg/g (p. 26). Instrument operating conditions were as follows:

Analytical Column: Restek Rtx; 30 m x 0.53 mm
Injection Port: 210°C isothermal
Detector: 300°C isothermal
Column Oven Temperature Program: 150°C for 5 minutes, 150°C to 230°C at 25°C per minute, hold at 230°C for 13 minutes
Flow Rates: Carrier gas - 4 mL/minute helium; Combustion make-up gas - 4.0 mL/min hydrogen and 175 mL/min air

In a method validation study, soil samples collected from the control plot were fortified separately with tebuconazole and 1,2,4-triazole at 0.01, 0.02, and 0.05 ppm (p. 26). Mean recoveries of the parent were 111 ± 11% for the 0.01 ppm fortification (1 of 5 samples >120%), 97 ± 12% for the 0.02 ppm fortification, and 108 ± 7% for the 0.05 ppm fortification (p. 29; Appendix 1, p. 157). Mean recoveries of 1,2,4-triazole were 106 ± 9% for the 0.01 ppm fortification, 100% for the 0.02 ppm fortification (single replicate), and 98% for the 0.05 ppm fortification (single replicate).

To determine concurrent recoveries, soil samples were fortified separately with tebuconazole and 1,2,4-triazole at 0.02 and 0.1 μg/g (p. 29). Mean recoveries of tebuconazole and 1,2,4-triazole (across both fortifications) were 95 ± 8% (range of 84 to 110%) and 91 ± 13% (range of 75 to 125%), respectively.

In a transit stability study of fortified field spikes, duplicate soil samples were fortified separately with tebuconazole and 1,2,4-triazole at 1.0 ppm at 5, 29, 62, 97, 249, 300 (tebuconazole only), 363 (tebuconazole only), 456, and 609 days posttreatment (p. 19). Samples were transported and stored (up to 412 days for tebuconazole and up to 819 days for 1,2,4-triazole; p. 28) in the same manner as the test samples. Data indicated that the parent was stable for up to 412 days; mean recoveries (across all sampling intervals) of the parent were 0.98-1.1 μg/g (Table 10, p. 77). Data indicated that the degradate 1,2,4-triazole was not stable during transport and storage; mean recoveries were 0.59-0.62 μg/g for samples stored for 248-595 days, 0.46-0.88 μg/g for samples stored for 746-767 days, and 0.29-0.47 μg/g for samples stored for 800-819 days posttreatment (Tables 9, 11; pp. 76, 78).

RESULTS/DISCUSSION

Tebuconazole (LYNX 25), broadcast applied three times (14-day intervals) at nominal application rates of 0.75 lb a.i./A, 1.5 lb a.i./A, and 0.75 lb a.i./A, respectively (total application rate of 3.0 lb a.i./A) onto a plot (planted with grass seed five days following the third application) of Dickinson sandy loam soil near Belleville, WI, dissipated with a registrant-calculated half-life of 216.3 days ($r^2 = 0.86$; p. 32; Figure 66, p. 155) following the third application. Data are means of three replicates. Residue data were not reported for samples following the first application. It was not specified whether the 0- to 6-inch depth soil core included the turf layer. Immediately following the second application, the parent was present in the 0- to 6-inch depth at 1.1 µg/g, was detected in the 6- to 12-inch depth at 0.02 µg/g, and was not detected below the 6- to 12-inch depth (Table 12, p. 79). Immediately following the third application, the parent was present in the 0- to 6-inch depth at 1.3 µg/g, was a maximum of 1.6 µg/g at 3 days posttreatment, decreased to 0.73-0.87 µg/g by 14-300 days, was 0.20-0.28 µg/g from 363-609 days, and was 0.06 µg/g at 790 days (Tables 12-15, pp. 79-82). Following the third application, the parent was detected sporadically in the 6- to 12-inch, 12- to 18-inch, and 18- to 24-inch depths at ≤0.06 µg/g (individual replicates). Soil samples collected below 24 inches were only analyzed at 300 and 363 days posttreatment; the parent was detected once in the 24- to 36-inch depth at 0.03 µg/g (single replicate) at 363 days posttreatment and was not detected below that depth.

One degradate was isolated from the soil:

1,2,4-triazole

Following the third application, 1,2,4-triazole was detected in the 0- to 6-inch depth, at ≤0.02 µg/g (individual replicates) at 29 and 62 days posttreatment, and was not detected at any other sampling interval or depth (Tables 16-19, pp. 83-86).

DEFICIENCIES/DEVIATIONS

1. Storage stability data for the degradate 1,2,4-triazole were inadequate. Mean recoveries from field fortified spikes (Tables 9, 11; pp. 76, 78) indicated that 1,2,4-triazole was not stable during frozen storage over the time period which test samples were stored for.

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. It was unclear whether the 0- to 6-inch soil core included the turf layer. It is necessary that total residues in the turf be monitored in order to accurately determine the routes of dissipation of the test compound.

4. The study authors stated that the registrant-calculated half-life of the parent was determined by summing residues at each sampling interval from each depth, rather than using data from only the top 6 inches (p. 28). The reviewer noted that the parent was not generally observed to leach.

5. The study authors stated that the rate of the first and third applications (0.75 lb a.i./A) was 1.1 times the maximum label rate, and that the second application was made at an exaggerated rate (1.5 lb a.i./A) to "ensure that adequate test compound was reaching the soil" (p. 12). The reported maximum label rate for LYNX 25 is 2.04 lb a.i./A/year (p. 18).

6. Residue data were not reported for samples following the first application. The study authors stated in a footnote to Table 12 (p. 79) that samples were missing.

7. The formulation of the test compound was reported as "LYNX 25." However, because the reviewer was unable to determine the formulation, the reviewer reported the formulation as not identified (formulation code 90).

8. The reviewer could not determine whether subplots were true replicate plots (separated by buffer zones; Figure 4, p. 93).

9. 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
SEE THE FILE COPY

ATTACHMENT 2
Excel Workbook

0-6 inch depth

Sampling interval (days)	Parent ug/g	ln parent (ug/g)
0	1.51	0.41211
0	1.26	0.231112
0	1.04	0.039221
1	0.91	-0.094311
1	1.02	0.019803
1	1.43	0.357674
3	1.72	0.542324
3	1.14	0.131028
3	1.81	0.593327
5	1.46	0.378436
5	1.47	0.385262
5	1.43	0.357674
10	1.57	0.451076
10	1.37	0.314811
10	1.12	0.113329
14	0.87	-0.139262
14	0.79	-0.235722
14	0.96	-0.040822
29	0.78	-0.248461
29	0.72	-0.328504
29	0.96	-0.040822
62	0.62	-0.478036
62	0.88	-0.127833
62	0.68	-0.385662
97	0.77	-0.261365
97	0.92	-0.083382
97	0.54	-0.616186
249	0.75	-0.287682
249	0.68	-0.385662
249	0.88	-0.127833
300	0.97	-0.030459
300	0.77	-0.261365
300	0.76	-0.274437
363	0.30	-1.203973
363	0.28	-1.272966
363	0.27	-1.309333
456	0.20	-1.609438
456	0.19	-1.660731
456	0.23	-1.469676
609	0.12	-2.120264
609	0.25	-1.386294
609	0.24	-1.427116
790	0.06	-2.813411
790	0.06	-2.813411
790	0.07	-2.65926

Half-life (days) = 203.9