DATA EVALUATION RECORD

STUDY 7

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Nonne, N. G., G. C. Mattern, and D. L. Green. 1996. Terrestrial field dissipation of tebuconazole (LYNX) on Wisconsin turf, 1993. Bayer Study No.: FR022701. Bayer Report No.: 107331. Unpublished study performed by AGSTAT, Verona, WI (in-life phase); Bayer Environmental Fate Analytical Laboratory, Stillwell, KS (analytical phase); QC, Inc., Southampton,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.

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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 turf plot of Dickinson sandy loam soil near Belleville, WI, dissipated with a registrant-calculated half-life of 163.2 days ($r^2 = 0.96$) following the third application. Residue data were not reported for samples following the first application. The 0- to 6-inch depth soil cores included the turf layer. Immediately

following the second application, the parent was present in the 0- to 3-inch depth at 2.3 μg/g and was not detected above 0.06 μg/g (individual replicates) below the 0- to 3-inch depth. Following the third application, the parent was present in the 0- to 3-inch depth at a maximum of 2.6 μg/g at 1 day posttreatment, decreased to 1.2-1.4 μg/g by 62 to 97 days, was 0.57 μg/g at 239 days, and was 0.07 μg/g at 789 days. Following the third application, the parent was not detected above 0.06 μg/g (individual replicates) below the 0- to 3-inch depth. Following the third application, the degradate 1,2,4-triazole was detected sporadically in the 0- to 3-inch depth at ≤0.02 μg/g (individual replicates), and was not detected below that 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. 13; 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 turf plot (78 x 103 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); the sod was laid on the plot on June 28, 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 10.5 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). The depth to the water table was >5 feet (p. 17). The grass was mowed prior to the first and second applications, and every 1-3 weeks, as needed, following the third application. Environmental data were collected on-site (p. 20). Precipitation was supplemented with irrigation (overhead sprinkler); total water input (85.39 inches) was 114% 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 detector (wavelength not specified). Mean recoveries of the parent from the application monitoring pads were 52%, 111%, and 55% 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 3-inch depth were 139% and 94% 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 2 days prior to the first application, immediately following the first, second, and third applications, and at 1, 3, 5, 10, 14, 29, 62, 97, 239, 364, 452, 602, and 789 days posttreatment (relative to the third application; p. 19); samples were collected from the control plot at 1, 239, 364, 602, and 789 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 24 inches at all other sampling intervals; the 0- to 6-inch soil cores included the turf layer. Samples were collected in two phases; lower depth samples (below 6-inches) had a core diameter of 1.75 inches. Samples were shipped frozen to the processing laboratory. At the processing laboratory, 1/8th inch of the outer soil core was shaved and discarded; 0- to 6-inch samples were sectioned into 0- to 3-inch and 3- to 6-inch increments, and 6- to 24-inch samples were sectioned into 6- to 12-inch and 12- to 18-inch increments. Samples were 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 465 and 893 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 methanol: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-5; 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 99 ± 18% for the 0.01 ppm fortification (1 of 8 samples >120%), 108 ± 30% for the 0.02 ppm fortification (1 of 5 samples >120%), and 88 ± 14% for the 0.05 ppm fortification (p. 29; Appendix 1, p. 160). Mean recoveries of 1,2,4-triazole were 100 ± 12% for the 0.01 ppm fortification, 110% for the 0.02 ppm fortification (single replicate), and 112% for the 0.05 ppm fortification (single replicate; Appendix 2, p. 191).

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 all fortifications) were 94.5 ± 11.1% (range of 70 to 112%) and 90.3 ± 13.6% (range of 70 to 120%), 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 and 29 days (tebuconazole only), and at 62, 97, 239, 364, 452, and 602 days posttreatment (p. 19). Samples were transported and stored (up to 452 days for tebuconazole and up to 804 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 452 days; mean recoveries (across all sampling intervals) of the parent were 0.93-1.3 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 0.84-0.90 μg/g for samples stored for 273-422 days, 0.71-0.72 μg/g for samples stored for 509-633 days, and 0.51-0.53 μg/g for samples stored for 770-804 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 turf plot of Dickinson sandy loam soil near Belleville, WI, dissipated with a registrant-calculated half-life of 163.2 days ($r^2 = 0.96$; p. 32; Figure 69, p. 158) following the third application. Data are means of three replicates. Residue data were not reported for samples following the first application. The 0- to 6-inch depth soil cores included the turf layer. Immediately following the second application, the parent was present in the 0- to 3-inch depth at 2.3 μg/g and was detected at ≤0.06 μg/g (individual replicates) in the 3- to 6-inch, 6- to 12-inch, and 12- to 18-inch soil depths (Table 12, p. 79). Immediately following the third application, the parent was present in the 0- to 3-inch depth at 2.3 μg/g, was a maximum of 2.6 μg/g at 1 day posttreatment, decreased to 1.2-1.4 μg/g by 62 to 97 days, was 0.57 μg/g at 239 days, and was 0.07 μg/g at 789 days (Tables 12-15, pp. 79-82). Following the third application, the parent was detected at ≤0.08 μg/g (individual replicates) in the 3- to 6-inch, 6- to 12-inch, and 12- to 18-inch depths.

One degradate was isolated from the soil:

1,2,4-triazole

Following the third application, 1,2,4-triazole was detected sporadically in the 0- to 3-inch depth at ≤0.02 μg/g (individual replicates), and was not detected below that 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 for which test samples were stored.

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 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 0- to 6-inch soil cores included the turf layer. The reviewer noted that the parent was not observed to leach.

4. 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 substance was reaching the soil" (p. 12). The reported maximum label rate for LYNX 25 is 2.04 lb a.i./A/year (p. 18).

5. 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.

6. 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).

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

8. The reviewer noted that additional terrestrial field dissipation studies were also submitted.

ATTACHMENT 1
Tables cited in DER

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ATTACHMENT 2
Excel Workbook

0-6 inch depth

Sampling interval (days)	Parent ug/g	ln parent (ug/g)
0	2.60	0.955511
0	2.19	0.783902
0	2.20	0.788457
1	2.50	0.916291
1	2.55	0.936093
1	2.72	1.000632
3	1.92	0.652325
3	1.70	0.530628
3	2.13	0.756122
5	2.25	0.81093
5	1.55	0.438255
5	1.96	0.672944
10	1.84	0.609766
10	1.70	0.530628
10	1.45	0.371564
14	1.85	0.615186
14	1.66	0.506818
14	2.38	0.8671
29	1.64	0.494696
29	1.78	0.576613
29	1.53	0.425268
62	1.26	0.231112
62	1.12	0.113329
62	1.23	0.207014
97	1.27	0.239017
97	1.57	0.451076
97	1.21	0.19062
239	0.61	-0.494296
239	0.53	-0.634878
239	0.57	-0.562119
364	0.29	-1.237874
364	0.39	-0.941609
364	0.26	-1.347074
452	0.20	-1.609438
452	0.17	-1.771957
452	0.20	-1.609438
602	0.18	-1.714798
602	0.18	-1.714798
602	0.14	-1.966113
789	0.08	-2.525729
789	0.09	-2.407946
789	0.05	-2.995732

Half-life (days) = 157.5