3-11-2002

DATA EVALUATION RECORD

STUDY 11

CHEM 120603 Tetraconazole §164-1
CAS No. 112281-77-3
FORMULATION–12–EMULSIFIABLE CONCENTRATE

STUDY ID 44865406

Bieber, W. D. and W. Jonas. 1999. Field study: Dissipation of tetraconazole from four agricultural field soils in Germany. NATEC Study No. NA 92 9822/2. Unpublished study performed by NATEC Institut, Hamburg, Germany; and submitted by Sipcam Agro USA, Inc., Roswell, GA.

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ABSTRACT

Field Dissipation - Terrestrial

1. This study is scientifically valid and provides supplemental information that tetraconazole ((±)-2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)-propyl-1,1,2,2-tetrafluoroethylether; M 14360 10 EC; 10.86% a.i.), applied once at a nominal rate of 0.128 kg a.i./ha to bareground plots of loamy sand (Bad Oldesloe/ Pölitz), silty sand (Klein-Offenseth), strongly loamy sand (Hamburg-Moorfleet), and loamy silt (Uslar/Verliehausen) soils in

Germany, dissipated with EFED-calculated first order (overall) linear half-lives of 577 (r^2 = 0.29; nonlinear $t_{1/2}$ = 340 days and r^2 = 0.28), 800 (r^2 = 0.11; nonlinear $t_{1/2}$ = 377 days and r^2 = 0.15), 265 (r^2 = 0.79; nonlinear $t_{1/2}$ = 189 days and r^2 = 0.66), and 182 (r^2 = 0.90) days, respectively. Initialy, however, tetraconazole dissipated rapidly in three of the sites (Bad Oldesloe/ Pölitz, Klein-Offenseth, and Hamburg-Moorfleet, see Attachment) with the initial half-lives of 18.6 (0-27 days) and 31.8 (0-29 days) days in loamy sand soil and strongly loamy sand soil, respectively. The initial $t_{1/2}$ could not be calculated for the silty sand soil due to insufficient data points in the initial degradation phase. After 365 days, at least 30 to 40 µg/kg of the parent compound was detected at the field site, and had the potential to carryover into the following season. Tetraconazole was not detected in the 10- to 20-cm soil depth with the exception of a single detection in the silty sand soil (at 14.3 µg/kg DS at 67 weeks PTT). However, a high water deficit during and after application could result in low soil moisture content and thus prohibit the compound from leaching below 10 cm depth. Soil samples were not analyzed for degradates of tetraconazole.

In the **silty sand soil** from Klein-Offenseth, tetraconazole was detected in the 0- to 10-cm soil depth at 141.3 µg/kg at 0 days posttreatment, 30.1-52.1 µg/kg at 1-48 weeks, and 28.1-31.9 µg/kg at 58-67 weeks.

In the **loamy silt soil** from Uslar/Verliehausen, tetraconazole was detected in the 0- to 10-cm soil depth at 65.0-71.4 µg/kg at 0-4 weeks posttreatment, 42.6-46.2 µg/kg at 8-16 weeks, 28.6-33.1 µg/kg at 24-49 weeks, and was last detected at 16.3 µg/kg at 58 weeks.

In the **loamy sand soil** from Bad Oldesloe/Pölitz, tetraconazole was detected in the 0- to 10-cm soil depth at 138.0 µg/kg at 0 days posttreatment, 72.3 µg/kg at 1 week, and varied from 36.1 to 49.9 µg/kg at 3-67 weeks with no discernable pattern of decline.

In the **loamy sand soil** from Hamburg-Moorfleet, tetraconazole was detected in the 0- to 10-cm soil depth at 102.8 µg/kg at 0 days posttreatment, 49.6-55.1 µg/kg at 1-8 weeks, 26.4-29.5 µg/kg at 23 to 58 weeks, and 15.9 µg/kg at 67 weeks posttreatment.

2. This study does not meet Subdivision N Guidelines for the fulfillment of EPA data requirements on terrestrial field dissipation for the following reasons:

A. The study was not conducted domestically;

B. Only a single application was tested;

C. European liquid formulation was tested instead of formulation being registered for uses in the U.S.A.;

D. Water deficit could prohibit tetraconazole from leaching;

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E. Soil was not characterized according to the USDA textural classification;

F. Degradates were not studied;

G. The soil cores were not analyzed to depths below the deepest detect;

H. The storage stability study results were not provided;

I. The water table depth was not reported; and

J. Pan evaporation data were not provided.

3. The registrant conducted three other terrestrial field dissipation studies out of which two were conducted on the bare ground plots in GA and CA (MRID 44865405). The linear tetraconazole half-lives were 91 weeks ($r^2 = 0.62$; nonlinear $t_{1/2} = 90$ weeks and $r^2 = 0.59$) and 222 weeks ($r^2 = 0.18$; nonlinear $t_{1/2} = 198$ weeks and $r^2 = 0.22$) in GA and CA, respectively. The domestic field dissipation data indicate that tetraconazole is very persistent and will accumulate in the soil environment under field conditions. Although the German field dissipation study initially showed rapid tetraconazole dissipation in the first 8 to 30 days, it persisted in the soil unchanged after 30 days indicating seasonal carryover.

In a third study conducted on bare ground in Salerano sul Lambro, Italy (MRID 44865404), tetraconazole applied as the Eminent 40EW (Emulsifiable Concentrate, 40 g a.i./L) formulation dissipated with a linear half-life of 128 days ($r^2 = 0.86$; nonlinear $t_{1/2} =$ 41 days and $r^2 = 0.71$). Similar to German field studies, the initial tetraconazole dissipation was rapid in the first 7 days and after that progressed slowly.

Both studies conducted in Germany and Italy are alternative terrestrial field dissipation studies which raised an issue of different tetraconazole formulations being less persistent. Although the studies were considered, they were not accepted for the purpose of tetraconazole risk assessment as a part of Eminent 125SL registration process because different liquid formulation of the product, the soil type, climate, and site variables contributed to high uncertainty of the studies' results.

4. No further information is needed on the terrestrial field dissipation study of tetraconazole at the present time. However, if any of tetraconazole degradate was of the human health or/and ecotoxicological concern, additional terrestrial field dissipation fate data for this degradate may be required.

MATERIALS AND METHODS

Tetraconazole [(±)-2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)-propyl-1,1,2,2-tetrafluoroethylether; batch FCF/T/109-92; p. 12], as the formulation M 14360 10 EC (10.86% a.i.), was applied at a nominal application rate of 0.128 kg a.i./ha to four bareground plots (25 m^2 each) in Germany (p. 14). The application rate was not confirmed. Soil parameters were as follows (Table 12, p. 30):

Parameter	Location			
	Klein-Offenseth	Bad Oldesloe/ Pölitz	Hamburg-Moorfleet	Uslar/ Verliehausen
Texture (as listed in the study)	silty sand	loamy sand	strongly loamy sand	loamy silt
Particle size distribution (%)				
<0.002 mm	0.1	12.0	12.1	16.5
0.002-0.006 mm	9.5	6.3	9.2	7.9
0.006-0.020 mm	5.6	9.3	7.1	18.9
0.020-0.063 mm	11.3	16.6	3.9	45.9
0.063-0.200 mm	33.2	26.0	14.1	5.1
0.200-0.630 mm	35.6	21.2	45.1	3.8
0.630-2.0 mm	4.7	8.6	8.5	1.9
pH	4.2	6.8	6.7	5.8
Organic carbon (%)	2.24	3.11	2.80	1.24
Microbial biomass (mg C/100 g soil)	11-15	35-78	34-63	26-42

The test plots were not irrigated during the study period. At all field sites, precipitation was less than the long-term average during the first two months of the study (p. 7). Total precipitation at each test site (1042-1312 mm) was 98-118% of the long-term average precipitation during the study period. Air temperatures at the four sites reportedly ranged from 2.0 to 19.7°C. Pan evaporation data were not reported.

Soil samples were collected from each test site prior to application and at 0, 1, 4, 8, 16, 24, 48, 58, and 68 weeks posttreatment (targeted sampling intervals; p. 14). At each sampling interval, 20 soil samples were collected from each test site using a Coremaster-zero contamination core handle with an inserted plastic tube; samples were collected diagonally across the test plots. Samples were placed in frozen storage (-20°C) within 8 hours of sampling and stored frozen in the dark until analysis. Samples were divided into 0- to 10-cm and 10- to 20-cm depth increments (p. 15). Samples were composited by sampling interval and depth, and homogenized. One subsample (50 g) from the homogenate was used for analysis.

Soil subsamples were Soxhlet-extracted with acetone:water (5:1, v:v) and the acetone was removed by rotary evaporation (p. 16; Figure 1, p. 17). Following the addition of water and acetone, the aqueous solution was then extracted three times with dichloromethane and the extracts were filtered through Na_2SO_4. Following rotary evaporation, the residues were dissolved in cyclohexane:ethyl acetate (1:1, v:v) and cleaned up using a gel permeation chromatography column. The eluent (100-160 mL fraction) was collected and evaporated by rotary evaporation. The residue was redissolved in n-hexane prior to GC analysis; the limit of detection was 7.97 µg test substance/kg soil (Tables 5-8, pp. 23-26). Samples were co-chromatographed with an analytical standard containing tetraconazole and the internal standard, Endosulfanesulfate. GC conditions were as follows (p. 16):

Model	HP 58 90
Autosampler	HP 76 73 B
Injector	275°C, splitless
Injection volume	2 µL
Septum purge flow	2 mL/min
Column	J & W Scientific Products; DB 5, 60 m (length) x 0.25 mm (inner diameter) x 0.25 µm (film thickness)
Column pressure	21 psi
Temperature program	100°C for 1 min, 15 K/min to 200°C (1 min), 3 K/min to 260°C (0 min), 40 K/min to 30°C, 30 min isotherm
Detector temperature	325°C
Detector	Electron Capture Detector

To determine method recoveries, wet soil samples (50 g) were fortified with tetraconazole, in acetone, at 6.73, 22.4, 67.3, and 224 µg/kg (p. 18). The soil was mixed and the acetone was allowed to evaporate for at least 24 hours prior to analysis. Recoveries of tetraconazole (across all soil types) were 60.9-86.2% for the 6.73 µg/kg fortification (n = 2), 76.7-118.5% for the 22.4 µg/kg fortification (n = 12), 86.9-95.9% for the 67.3 µg/kg fortification (n = 2), and 70.0-101.7% for the 224 µg/kg fortification (n = 10; Table 2; p. 21); recoveries were corrected for control samples. The overall mean recovery (across all fortifications and soil types) was 89.0 ± 16.0% (Table 3, p. 22).

RESULTS/DISCUSSION

Tetraconazole (M 14360 10 EC), applied once at a nominal application rate of 0.128 kg a.i./ha to single bareground plots of silty sand and loamy silt soil and two plots of loamy sand soil in Germany, dissipated with respective registrant-calculated half-lives (reported as DT_{50}s) of 6.19

(5)

weeks (0-16 week data), 5.78 weeks (0-24 week data), and 6.56-8.91 weeks (0-16 or 0-23 week data; biphasic model; Table 11, p. 29). Based on the reported data, the observed half-lives occurred between 0 and 8 days for the silty sand soil, between 112 and 169 days for the loamy silt soil, and between 7 and 29 days for the two loamy sand soils. At each sampling interval, a single subsample from the composited sample was analyzed for tetraconazole. Data were not corrected for concurrent recoveries. Soil samples were not analyzed for degradates of tetraconazole.

In the loamy sand soil from Bad Oldesloe/Pölitz, tetraconazole was initially present in the 0- to 10-cm soil depth at 138.0 µg/kg, was 72.3 µg/kg at 1 week, and varied from 36.1-49.9 µg/kg at 3-67 weeks posttreatment (Table 5, p. 23). Tetraconazole was not detected in the 10- to 20-cm soil depth.

In the silty sand soil from Klein-Offenseth, tetraconazole was initially present in the 0- to 10-cm soil depth at 141.3 µg/kg and varied from 28.1-52.1 µg/kg at 1-67 weeks posttreatment (Table 6, p. 24). Tetraconazole was not detected in the 10- to 20-cm soil depth with the exception of 14.3 µg/kg at 67 weeks posttreatment.

In the loamy sand soil from Hamburg-Moorfleet, tetraconazole was initially present in the 0- to 10-cm soil depth at 102.8 µg/kg, was 49.6-55.1 µg/kg at 1-8 weeks, 26.4-29.5 µg/kg at 23-58 weeks, and 15.9 µg/kg at 67 weeks posttreatment (Table 7, p. 25). Tetraconazole was not detected in the 10- to 20-cm soil depth.

In the loamy silt soil from Uslar/Verliehausen, tetraconazole was present in the 0- to 10-cm soil depth at 65.0-71.4 µg/kg at 0-4 weeks, was 42.6-46.2 µg/kg at 8-16 weeks, was 28.6-33.1 µg/kg at 24-49 weeks, and was last detected at 16.3 µg/kg at 58 weeks (Table 8, p. 26); tetraconazole was not detected in the 10- to 20-cm soil depth.

COMMENTS

1. The study was carried out according to the BBA-Guideline Part IV, 4-1, section 2, and did not meet US EPA Subdivision N Guidelines, Series 164-1, for the following reasons:

   - The study was not conducted domestically. Instead, the study was conducted at four locations in Germany. The environmental conditions under which the study was conducted may not be sufficiently similar to those in the United States to allow the study results to be extrapolated to US growing conditions.

   - European formulation (an emulsifiable concentrate: M 14360 10 EC (10.86% a.i.)) was tested instead of formulation being registered for uses in the US (Eminent 125 SL: 11.6% a.i.(1 lb ai/gal)). Different formulations may dissipate with different rates under actual use conditions.

- June 1992 was an extremely dry month and during July through September precipitation was less than the long-term average for all sites. The high water deficit during and after application could result in low soil moisture content and thus prohibit the compound from leaching below 10 cm depth. The author noted that the temperature during that time was remarkably above the long-term average. The test plots were not irrigated at any of the four field sites.

- Soil was not adequately characterized using the USDA classification scheme. Soils were classified based on the following particle size distributions: <0.002 mm, 0.002-0.006 mm, 0.006-0.020 mm, 0.020-0.063 mm, 0.063-0.200 mm, 0.200-0.630 mm, and 0.630-2.0 mm. Because the particle size distribution that was used is not comparable to that used in the US, the soils could not be accurately reclassified.

- Patterns of formation and decline of degradates of tetraconazole were not established. Soil samples were not analyzed for degradates of tetraconazole. For example, HPLC chromatograms of selected soil extracts, presented in Figures 6-12 (pp. 38-44), indicated that additional compounds were present in the soil extracts.

- Subdivision N guidelines specify that the depth of leaching should be established by analyzing the soil core two depths below the deepest detection. The study design did not permit this since the soil cores were divided into 0 to 10 cm and 10 to 20 cm depths. In the silty sand soil, tetraconazole was detected in the 10 to 20 cm depth.

- The test plots were not replicated, and replicate soil samples were not analyzed to establish sampling variability. At each test site, all 20 soil cores collected at each sampling interval were composited by depth and homogenized to provide a single sample for analysis. Test plots should consist of at least three replicated plots, separated by buffer zones.

- A storage stability study was not conducted. Table 4 (pp. 32-33) indicated that samples were stored for up to approximately 20-21 months. Storage stability studies should be conducted using samples collected from each test site, fortified separately with the parent and degradates, and stored for a length of time equal to the longest storage interval utilized for the test samples.

- Samples were not selected randomly. Instead, samples were collected in a diagonal manner across the test plots.

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- It was unclear whether the test plots were treated with other pesticides prior to and during the study because the study author blends the discussion of the test plots with history of the surrounding area (p. 11). It was certain that the Bad Oldesloe/Pölitz and Uslar/Verliehausen sites received no pesticide treatment before and during the study. In 1991, the Klein-Offenseth site was treated with a mixture of 2,4-D and MCPA salts and the Hamburg-Moorfleet site was treated with demeton-S-methyl, methomyl, and desmetryn.

- The depth to the water table was not reported. Pan evaporation data were not reported.

2. Application monitoring pads or similar devices were not used to monitor the application rate at any of the test sites.

3. The study was not conducted in accordance with Good Laboratory Practice standards as defined by the US EPA. The study was carried out in compliance with the OECD Principles of Good Laboratory Practice, Germany.

ATTACHMENT 1
Data Critical to the Study Interpretation

THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY
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FER-Dissipation of Tetraconazole from Agr. Field Sols. ... MRID 44856406

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