

Persistence of Termiticides in Soil Inside and Outside Miniature Concrete Foundations (Isoptera)

by

J.E. Mulrooney¹, T.L. Wagner¹, B.M. Kard², & P.D. Gerard³

ABSTRACT

A cooperative study of termiticide longevity was initiated in 1990 between the Association of Structural Pest Control Regulatory Officials (ASPCRO), termiticide manufacturers, and the USDA Forest Service. By-the-label applications of seven termiticides were made to soil in trenches inside and outside miniature concrete foundations, and soil samples were collected at 1, 30, 60, 120, 180, 240, 300, 360, 734, 1057, 1420, 3650, and 4380 d after application. Termiticide residues were fitted using a logistic dose-response model. Half-lives of termiticides in soil in trenches inside and outside foundations (combined data) were: chlorpyrifos, Dursban[®] TC (1,254 d); fenvalerate, Tribute[®] (831 d); permethrin, Dragnet[®] FT (747 d); cypermethrin, Prevail[®] FT (488 d); cypermethrin, Demon[®] TC (399 d); isofenfos, Pryfon 6 (301 d); and permethrin, Torpedo[®] (138 d). Laboratory bioassays conducted 12 y after initial termiticide application showed that chlorpyrifos, applied at 1.0% AI, elicited the greatest termite mortality compared with the other termiticides. Combining inside and outside trench data for each termiticide, termites penetrated to a depth of 26.0 ± 6.3 mm through Dursban[®]-treated soil. Fenvalerate applied at 0.5% was the most effective pyrethroid at preventing penetration, restricting termite penetration to a depth of 16.1 ± 5.5 mm of the treated soil.

Keywords: termites, termiticide residue, termite mortality.

INTRODUCTION

In 1989, the Association of Structural Pest Control Regulatory Officials (ASPCRO), the National Pest Management Association (NPMA), state

¹Wood Products Insect Research Unit, USDA-Forest Service, 201 Lincoln Green, Starkville, MS 39759

²Dept of Entomology and Plant Pathology, 127 Noble Res. Center, Oklahoma State University, Stillwater, OK 74078-3033.

³Mississippi Agriculture and Forestry Experiment Station, P.O. Box 9653, Mississippi State, MS 39762.

regulators, university and government researchers, and manufacturers of termiticides recognized the need to establish a standardized soil sampling method for collecting soil treated with a termiticide. A standard method was needed to ensure consistency in soil samples collected for termiticide residue analysis. In response to these needs, ASPCRO formed the Committee on Termiticide Sampling and Concentrations in Soil that met in January 1990. The committee established the Soil Residue Data Collection Project which outlined a soil sampling study to (1) evaluate "by-the-label" termiticide applications to soil performed by pest management professionals and measure expected termiticide residue values under field conditions, (2) provide guidance to state regulatory agencies regarding how to interpret the findings of this investigation, and (3) provide guidance for implementation of study findings into respective state programs.

The study outlined by ASPCRO as part of Soil Residue Data Collection Project was conducted in Arizona, Georgia, Indiana, and Oklahoma. In each state, there were three groups of seven structures. Structures in each group were treated with one of seven registered termiticides. From the results of this study, ASPCRO established acceptable residue requirements for an effective application for each of the seven termiticides. They determined that through proper sampling procedures, it is possible to use results from soil sampling for regulatory actions if sufficient pesticide is not present following treatment.

The Committee met again in March 1990 to develop a specific soil sampling protocol, along with a termiticide soil residue study to be conducted on the Harrison Experimental Forest, 32 km north of Gulfport, MS. The termiticide soil residue study involved placing soil treated with one of seven registered termiticides in their assigned trenches inside and outside square concrete miniature foundations. Residues collected at 1.0 d after application were found to be within a 95% or better confidence interval of the theoretical amount in the soil, thus validating the sampling protocol (Kard & McDaniel 1993). McDaniel and Kard (1994) reported the results of residue analyses done at 3 y (1,057 d) after application. They reported half-lives (days) for chlorpyrifos, Dursban[®] TC (1066 d); cypermethrin, Demon[®] TC (317 d); cypermethrin, Prevail[®] FT (404 d); fenvalerate, Tribute[®] (481 d); isofenphos, Pryfon[®] 6 (207 d); permethrin, Dragnet[®] (654 d); and permethrin, Topedo[®]

(543 d). Kard (1996) reported residues from soil in trenches collected 4 y after application but did not compute half-lives.

At 10 and 12 y after application, soil samples were again collected from inside and outside the concrete miniature foundations. We combined all the data from previous residue analyses with residue analyses on the 10 and 12 y samples and report the longevity of chlorpyrifos, cypermethrin, fenvalerate, and permethrin, over 12 y. The efficacy of these compounds against subterranean termites was also determined in laboratory bioassays of soil collected from trenches 12 y after application.

MATERIALS AND METHODS

Concrete Foundations

Test plots were located on the Harrison Experimental Forest, near Saucier, MS, 32 km north of Gulfport (Kard & McDaniel 1994). Harrison County has a warm, humid climate with average temperatures ranging from 7°C in January to 35°C in July. Average rainfall is 170 cm per year. Soil type is Rumford sandy loam (70% sand, 25% silt, and 5% clay) with an average pH of 5.10.

Thirty concrete foundations were constructed to simulate vertical walls of crawl space buildings. These monolithic-poured structures were reinforced with 15.2-cm square, 9-gauge steel wire mesh, and measured 76.2 cm on a side (outside walls) × 35.6-cm high, with 5.1-cm-thick walls. They were centered within square plots arranged in a 3 × 10-plot grid, with each plot measuring 4.6 m on a side. A square trench that measured 107 cm on the outside, 36-cm wide, and 20-cm, deep was dug in each plot. After centering the structures into the trenches, soil was back-filled and compacted so that 15 cm (square cross-section) trenches remained around both the interior and exterior walls. Thus, the interior and exterior trench bottoms were separated. The remaining excavated soil was then sieved through 0.6-cm aperture mesh screening to remove roots, rocks, and debris. Individual volumes of this sieved soil measuring 0.0283 m³ (28.32 L) were sprayed with liquid termiticide at the trench label-rate volume (15.1 / 3 m) while rotated in a cement mixer for 15 min. The treated soil was placed back into the outer trench, evenly distributed, and tamped down to the level of the original soil surface. Outer trenches required 0.085 m³ of soil for each concrete foundation. This procedure was repeated for trenches inside foundations using 0.048 m³ of soil treated with termiticide

at the same rate as the outer trench soil. All structures were capped with an 81-cm square cover of 1.9-cm exterior-grade plywood that was sealed against moisture and painted glossy white to reflect heat.

The study evaluated seven termiticides (Table 1) that were marketed in the U.S. in 1990, each replicated four times, plus two water-only controls, all arranged in a completely randomized design.

Residue Analysis

Soil samples were collected with a stainless steel cylindrical coring probe (2.5-cm i.d. by 20-cm length) to a depth of 11.4 cm. One soil core was extracted from the treated soil along each exterior wall of each structure, and the top 1.3 cm discarded. The resulting four 10.2 cm cores were combined and blended in an aluminum foil-lined bag to form a single composite sample. This process was repeated for the interior trenches. Soil sample holes were plugged with sections of PVC pipe (2.5-cm diameter X 10-cm tall) to maintain integrity of the treated soil. Soil samples were collected at 1, 180, 240, 300, 360, 734, 1057, 1420, and 3650 d after initial termiticide applications.

Twelve years after application (4,380 d), soil samples were collected only from chlorpyrifos (Dursban TC), cypermethrin (Demon TC), cypermethrin (Prevail FT), fenvalerate (Tribute), and permethrin (Dragnet FT) plots. Isofenfos (Pryfon 6) and permethrin (Torpedo) were not sampled. Four 10.2-cm-deep samples and one 15.2-cm-deep sample were randomly collected from each of three reps from interior and exterior trenches in 2.5-cm diameter butyrate (Tenite[®]) sampling tubes contained within the coring probe. Three of the four 10.2-cm-deep samples were used in termite penetration bioassays, and one was used for termiticide residue analysis. The 15.2-cm-deep samples

Table 1. Termiticides and active ingredient were used to determine stratification concentration applied.

Trade Name	Active Ingredient	% Conc.
Dursban [®]	Chlorpyrifos	1.0
Demon [®] TC	Cypermethrin	0.25
Prevail [®] FT	Cypermethrin	0.30
Tribute [®]	Fenvalerate	0.50
Pryfon [®] 6	Isofenphos	0.75
Dragnet [®]	Permethrin	0.50
Torpedo [®]	Permethrin	0.50

of termiticide residues within the top 15.2 cm of soil after 12 y. All samples, other than the first ones collected 24 h after initial treatments, were collected within ± 5 d of the specified sampling day.

Analyses of samples taken during the first year were performed at the

National Monitoring and Residue Analysis Laboratory, USDA-APHIS, Gulfport, MS using their laboratory protocols for extraction of termiticides from soil (Method PR0047) and analytical methodology and quality control (Method PR0056.3). Analyses at days 734, 1,057, and 1,420 were performed at the USDA Forest Service Laboratory in Gulfport, MS, according to manufacturers' protocols (Wetters 1977, Shaw 1977) and published protocols (Sapiets *et al.* 1984, Shell Development Company 1984; Swaine & Tandy 1984). Samples collected at day 3,650 were analyzed at the Center for Urban and Structural Entomology, Texas A & M University, College Station, TX.

Samples collected at 12 y (4,380 d) were analyzed by the Wood Products Insect Research Unit, Starkville, MS, using the following methods. Soil was removed from 10.2-cm tubes and mixed thoroughly in 250-ml beakers. Termiticide residues were extracted from soil in 15.2-cm tubes that were partitioned into 0-2.5, 2.6-7.5, and 7.6-15.2 cm sections using a bandsaw. Twenty-five g samples were taken from the middle and bottom depths. Because the 0-2.5 cm layer did not contain 25 g, the amount of soil in this layer was estimated to be 14.4 g based on 25% of average dry weights of soil from 10.2-cm tubes of untreated soil. Samples from each soil layer were individually placed into individual 100-ml beakers. Chem-tube hydromatrix (Varian[®], Palo Alto, CA) was mixed with the soil to bring the volume of soil and hydromatrix to 40 ml. Beaker contents were then poured into an extraction tube. Extraction was made with an Accelerated Solvent Extractor, ASE-200 (Dionex[®], Sunnyvale, CA) using a 70/30 mixture of acetone/acetonitrile at a total volume of 50 ml. Oven temperature and pressure were 100°C and 105.4 kg/cm², respectively, with a 5 min static time. Extraction volume was reduced to 10 ml under nitrogen using a Rapid Vac (Labconco[®], Kansas City, MO). Percent recoveries of chlorpyrifos, cypermethrin, fenvalerate, and permethrin were: 107±1.8, 95.4±4.0, 101.7±2.3, and 95.2±3.5, respectively.

All termiticides were analyzed using an Agilent[®] 5990 gas chromatograph equipped with electron capture and flame photometric detectors. Cypermethrin, fenvalerate, and permethrin residues were analyzed using an electron capture detector. The parameters of the analysis method were as follows: injection volume, 1µl; carrier gas, helium; make-up gas, argon/methane; injector temperature, 250°C; detector temperature, 300°C; oven program, 225°C initial temperature with a 30°C/min ramp to 280°C for 8 min. An

Agilent® 25-m Ultra-1 methyl silicone gum phase column (I.D. 0.32 mm) with 0.52- μ m film thickness was used. Retention times of cypermethrin isomers, fenvalerate isomers, and permethrin isomers were: 21.184 – 24.478, 30.669 – 32.404, and 11.010 – 11.443 min, respectively. The concentrations reported were the sum of all isomers.

Chlorpyrifos residues were analyzed using a flame photometric detector. Chlorpyrifos analysis method parameters were: injection volume, 1 μ l; carrier gas, helium; injector temperature, 200°C; detector temperature, 250°C; oven program, 125°C initial temperature with a 25°C/min ramp to 250°C for 2 min. An Agilent® 25 m Ultra-1 methyl silicone gum phase column (I.D. 0.32 mm) with 0.52- μ m film thickness was used. Retention time of chlorpyrifos was 6.776 min.

Bioassays

Bioassays were conducted on soil samples collected 12 y after treatment. Termites from three *Reticulitermes* sp. colonies were collected from fallen pine logs that were separated from each other by at least 1000 m in the Tombigbee National Forest near Ackerman, MS, and held at ambient temperature in galvanized trashcans in the laboratory.

The bioassay method was similar to that described by Su *et al.* (1993). In our bioassay, the 10.2 cm of soil in the sample tube was reduced to 5.2 cm by pushing out the bottom 5.0 cm of soil. The tube containing the remaining top 5.2 cm of soil was connected (by a Tygon® tubing collar) to another tube containing 80 termite workers and one soldier. The 5.2-cm soil core was sandwiched between two 3.0-cm-thick agar segments with a layer of dry silica sand between the soil and the lower agar segment to reduce possible movement of termiticide from the soil to the agar. Wooden sticks of southern yellow pine and filter-paper strips provided food and harborage for termites in both the tube containing termites and the tube containing only soil, so that termites had a source of food both above and below the treated soil. The bioassay was conducted for 7 d after which mortality, as well as distance tunneled through treated soil (penetration), was determined.

Data Analysis

The experimental design was a split-plot with termiticide as whole plots and trench location (inside or outside foundation walls) as split-plots. There

were four replicates. Termiticide residues determined previously (McDaniel & Kard 1994, Kard 1996) were included with 10- and 12-year residues from the present study, and a logistic dose-response model was fitted to the data using TableCurve®2D (ver. 5.1; SYSTAT Software Inc., Richmond, CA). The equation is: $r(x) = a / [1+(x/b)^c]$, where $r(x)$ = termiticide residue (ppm) in the soil on day x after application and a , b , and c are parameters to be estimated. Parameter a represents the initial termiticide residue at day zero (y intercept) and b the number of days for half of the initial residue to dissipate. Thus, parameter b is the half-life for each termiticide. Three curve fitting techniques were employed to manage variability in the data sets and improve the overall fit of the model: (1) minimizing the sum of the squared residuals, (2) minimizing the sum of absolute values of residuals, (3) and Lorentzian minimization. The latter two methods were chosen to improve the resolution of the model at 10 and 12 years.

Concentrations of termiticides ($\mu\text{g/ml}$), determined by GC analysis, in the 0-2.5 and 2.6-7.5 cm soil depths were added together and divided by 39.4, which is the total grams of soil used in extraction, to give the amount (ppm) of termiticide in the 0-7.5 cm depth of soil. Residues at the 0-7.5 and 7.6-15.2 cm depths were compared.

Termiticide stratification and termite mortality and soil penetration at 12 y after application were analyzed using PROC MIXED (SAS Institute, 2001). LSmeans were separated using the PDIF option. Standard errors of LSmeans are model based; therefore, standard errors will be identical when replicates are equal and different when unequal.

RESULTS AND DISCUSSION

Residues

Termiticide residues in the inside and outside trench soil around foundations over 12 y are given in Table 2. Termiticide half-lives predicted by the Logistic Dose-Response model inside foundations followed the order: chlorpyrifos, Dursban TC (1113 d) > fenvalerate, Tribute (1064 d) > permethrin, Dagnet (941 d) > cypermethrin, Prevail FT (483 d) > isofenphos, Pryfon 6 (287 d) > cypermethrin, Demon TC (207 d), > permethrin, Torpedo (125 d) (Table 3). Half-lives in soil outside foundations were in the order: chlorpyrifos, Dursban TC (1357 d) > fenvalerate, Tribute (775 d) > permethrin,

Table 2. Termiticide residues (ppm) in soil inside and outside trenches around simulated miniature foundations.

Termiticide	%AI	Location	Days after Application												
			1 ^a	30 ^a	60 ^a	120 ^a	180 ^a	240 ^a	300 ^a	360 ^a	734 ^b	1057 ^b	1420 ^b	3650	4380
Chlorpyrifos (Dursban [®])	1.00	Inside	858	669	601	967	956	808	469	777	643	436	545	53	36
		Outside	990	636	654	793	822	1214	586	791	525	502	419	38	62
Cypermethrin (Demon [®])	0.25	Inside	453	188	156	357	214	239	177	147	75	42	19	10	<1
		Outside	407	200	157	326	260	232	190	110	53	38	14	2	<1
Cypermethrin (Prevail [®])	0.30	Inside	353	254	223	409	294	318	296	158	81	56	23	11	<1
		Outside	352	269	256	429	272	279	281	157	63	67	20	7	<1
Fenvalerate (Tribute [®])	0.50	Inside	692	457	424	639	349	578	751	869	334	190	225	138	57
		Outside	641	442	448	631	562	501	726	867	261	152	133	61	33
Isofenphos (Pryfon [®])	0.75	Inside	787	514	511	898	409	265	444	221	37	31	20	1	†
		Outside	778	507	396	689	563	163	491	229	12	35	5	0	†
Permethrin (Dragnet [®])	0.50	Inside	471	605	419	281	434	537	400	519	231	213	119	150	15
		Outside	465	661	389	341	307	543	446	493	172	139	56	30	5
Permethrin (Torpedo [®])	0.50	Inside	685	616	342	316	263	279	419	454	195	212	97	67	†
		Outside	497	710	388	285	157	442	280	402	168	145	49	12	†

^aPreviously published: McDaniel and Kard (1994).^bPreviously published: Kard (1996).

†Not sampled.

Table 3. Parameter estimates and r^2 values for the Logistic Dose-Response model describing termiticide residues (ppm) in soil inside and outside of trenches around miniature foundations over time (days from application). Parameter a estimates residues (ppm) on day zero, and b the half-life in days

Termiticide	Location	a	b	c	r^2
Chlorpyrifos (Dursban®)	Inside	835.87	1113.17	2.2675†	0.735
	Outside	797.77	1357.32	2.1141†	0.752
	Combined	823.78	1253.61	2.3700†	0.754
Cypermethrin (Demon®)	Inside	441.25	206.68	1.2513†	0.568
	Outside	276.88	378.49	2.9481‡	0.792
	Combined	284.23	398.61	2.2608‡	0.752
Cypermethrin (Prevail®)	Inside	318.12	483.44	2.5312†	0.868
	Outside	308.63	458.84	2.3628†	0.880
	Combined	315.75	488.27	2.6088†	0.878
Fenvalerate (Tribute®)	Inside	574.35	1064.21	3.4670‡	0.634
	Outside	600.31	774.98	4.8419‡	0.792
	Combined	585.87	830.62	2.2830†	0.676
Isofenphos (Pryfon®)	Inside	659.89	287.34	2.7467‡	0.809
	Outside	595.88	316.50	2.7790‡	0.784
	Combined	628.21	301.02	2.7304‡	0.793
Permethrin (Dragnet®)	Inside	475.62	941.57	1.8359‡	0.762
	Outside	461.44	727.32	3.5064‡	0.799
	Combined	465.02	746.94	1.9913‡	0.762
Permethrin (Torpedo®)	Inside	711.74	125.09	0.6717*	0.733
	Outside	761.51	202.25	1.3747*	0.343
	Combined	689.34	138.48	0.6605†	0.688

Parameter estimates based on:

†minimization of the sum of absolute values of residuals

‡minimization of the sum of the squared residuals

*Lorentzian minimization

Dragnet (728 d) > cypermethrin, Prevail FT (459 d) > cypermethrin, Demon TC (378 d) > isofenphos, Pryfon 6 (316 d) > permethrin, Torpedo (202 d). Predicted half-lives of cypermethrin (Prevail FT), fenvalerate (Tribute), and permethrin (Dragnet) were longer in soil inside foundations compared with those from outside foundation soil; whereas, those of chlorpyrifos (Dursban TC), cypermethrin (Demon TC), isofenphos (Pryfon 6), and permethrin

(Torpedo) were longer outside trenches compared to those inside (Table 3). McDaniel and Kard (1994) did not find large differences in termiticide concentrations between interior and exterior trenches four years after application of these treatments. Differences in chemistries and application rates should be considered when comparing the half-lives of these compounds. Isofenfos was applied at 75% of the rate of chlorpyrifos. Fenvalerate and the permethrin formulations, Dragnet and Torpedo, were applied at 50% the rate of chlorpyrifos, while the cypermethrin formulations, Prevail FT and Demon TC, were applied at 30 and 25%, respectively, of the chlorpyrifos rate.

Average half-lives (over both trench locations) estimated by McDaniel and Kard (1994) using a first-order exponential decay model 3 y after application followed the order: chlorpyrifos, Dursban TC (1,066 d) > permethrin, Dragnet (654 d) > permethrin, Torpedo (543 d) > fenvalerate, Tribute (481 d) > cypermethrin, Prevail FT (404 d) > cypermethrin, Demon TC (317 d). They did not report the half-life of isofenfos. Our estimate of the half-life of combined residues (inside and outside) of chlorpyrifos, Dursban[®] TC after 12 y was 1,254 d which is slightly higher than that of McDaniel and Kard (1994). Racke *et al.* (1994) determined half-lives of chlorpyrifos under laboratory conditions after 175, 214, 230, 335 and 1,576 days in five soils from different states. The half-life of chlorpyrifos found in our study (1,357 d) from soil outside foundations best matches a Florida urban sandy loam half-life (1,576 d) in Racke *et al.*'s (1994) test.

Estimates of half-lives of combined residues, averaged over inside and outside trenches after 12 y, for permethrin (Dragnet), fenvalerate, cypermethrin (Prevail FT), and cypermethrin (Demon TC) were 768, 831, 488, and 399 d, respectively (Table 3). These values are greater than average half-lives (654, 481, 404, and 317 d, respectively) reported by McDaniel and Kard (1994) after 3 y, while the overall average half-life for permethrin (Torpedo) after 12 y (138 d) was lower than that after 3 y (543 d).

In a similar study in 1990, Jarratt *et al.* (2004) applied six of the termiticide formulations used in our study in and around test foundations at two sites in MS, one near Starkville, and the other within 100 m of our study. They reported termiticide residues 5 y after application. Because 5 y soil samples were not collected in our study, we used the logistic dose-response model to predict these values. The 5 y residues reported by Jarratt *et al.* (2004) were

Table 4. Termiticide residues (ppm±SEM) at two depths in soil from inside and outside trenches around miniature foundations 12 y after application.

Depth (cm)	Chlorpyrifos (Dursban®)	Cypermethrin (Demon®)	Cypermethrin (Prevail®)	Fenvalerate (Tribute®)	Permethrin (Dragnet®)
Inside					
0–7.6	95.0 ± 20.4 a ²	0.3 ± 0.8	0.2 ± 0.1	156.4 ± 34.2	83.0 ± 6.7 a ²
7.7–15.2	65.5 ± 20.4 a	0.2 ± 0.8	0.3 ± 0.1	50.6 ± 41.8	10.2 ± 6.7 b
Outside					
0–7.6	30.2 ± 20.4 b	1.8 ± 0.8	0.2 ± 0.1	30.2 ± 34.2	7.4 ± 5.7 b
7.7–15.2	45.5 ± 20.4 b	0.1 ± 0.8	0.2 ± 0.1	32.7 ± 34.2	16.3 ± 5.7 b

Residues within a column followed by the same letter are not significantly different ($P>0.05$) as determined by PDIF (SAS Institute 2001).

²Main effect for location was significant ($P=0.0301$).

³Location-Depth interaction was significant ($P=0.0012$).

much higher than those generated in our study. Differences in residues between these two studies ranged from two-fold (chlorpyrifos, outside foundations and cypermethrin (Demon TC) inside foundations), to 38-fold (fenvalerate outside foundations) compared with residues in our study. Only isofenphos had similar values in both studies. The differences in residues between the two studies are entirely due to different trenching methodologies. Jarratt *et al.* (2004) applied termiticides in a trench/backfill manner using a CO₂ backpack sprayer and did not remove and sieve soil from trenches. We removed soil and applied termiticides in a cement mixer, and then returned the treated soil to its original trench. Treating soil in a cement mixer uniformly distributed termiticides in the soil, whereas Jarratt *et al.* (2004) trenched and treated in place. Jarratt *et al.* (2004) trenches were “V” shaped, this could have caused the termiticide to pool at the trench bottoms, resulting in higher localized concentrations that would degrade slower than a less concentrated, uniformly distributed termiticide.

Gold *et al.* (1996) determined persistence and toxicity of these same registered termiticides from five soils in Texas over 5 y. Termiticide residues from four of the five Texas sites were less than the five-year residues estimated by the logistic dose-response models developed from our data. Residues from

the Overton, TX, site that has a similar soil type to that in our study, came closest to matching our estimated 5-y residues. Possible reasons for the faster degradation occurring in the Texas soil are differences in rainfall, soil environment, and soil type between the two sites. Geographical differences affecting the persistence of pesticides should exist because physical, chemical, and biotic processes governing pesticide persistence in soil are positively correlated with soil temperature and moisture (Scheunert 1992).

Results of termiticide stratification in the soil after 12 y showed a significant difference ($F = 37.97$; $df = 4, 9$; $P < .0001$) among termiticides. Average residues over location (inside/outside foundations) and depth for chlorpyrifos (59.0 ± 10.2 ppm) and fenvalerate (67.2 ± 18.1 ppm) were significantly different from permethrin and both formulations of cypermethrin, but not different from each other. Average residue of permethrin (29.3 ± 3.3 ppm) was greater than cypermethrin, Demon, (0.6 ± 0.4 ppm) and cypermethrin, Prevail, (0.2 ± 0.1 ppm).

Separate analysis of each termiticide showed that location was significant for chlorpyrifos ($F = 7.99$; $df = 1, 6$; $P = 0.0301$). Residues were greater in soil in trenches inside foundations (80.2 ± 17.4 ppm) compared to those outside (37.8 ± 17.4 ppm). For permethrin, location ($F = 43.5$; $df = 1, 4$; $P = 0.0027$) and depth ($F = 40.75$; $df = 1, 4$; $P = 0.0031$) main effects as well as the location*depth interaction ($F = 66.81$; $df = 1, 4$; $P = 0.0012$) were all significant.

In general, the amount of termiticide residue was greater inside foundations at the 0-7.5-cm depth than at 7.6-15.2 cm (Table 4). The opposite condition occurred outside foundations where residues were less in the 0-7.5-cm depth than at 7.6-15.2 cm. Rainfall and greater microbial activity in the root zone of soil outside foundations could account for these differences.

Bioassay

In laboratory bioassays 12 y after termiticide applications, average termite penetration through soil was significantly different ($F = 27.03$; $df = 1, 96$; $P < 0.0001$) for trench location. However, the location, termiticide interaction was not significant ($F = 1.81$; $df = 5, 79$; $P = 0.1190$). Overall termite penetration through soil cores from interior trenches (25.2 ± 2.6 mm) was less than that through soil cores from exterior trenches (44.2 ± 2.6 mm). This is a result

Table 5. Average penetration^a (mm±SEM) by termites into 52-mm thick soil cores collected from trenches around miniature foundations 12 years after initial application (7-d bioassay).

Treatment	Penetration (mm)
Chlorpyrifos (Dursban® TC)	26.0 ± 4.5 bc
Cypermethrin (Demon® TC)	40.4 ± 4.5 a
Cypermethrin (Prevail® FT)	36.5 ± 4.5 ab
Fenvalerate (Tribute®)	16.1 ± 4.5 c
Permethrin (Dagnet® FT)	37.0 ± 4.5 ab
Control	52.0 ± 4.5 a

^aTermiticide (main effect) means, termiticide.location interaction was not significant.

of the greater concentrations of termiticides in interior trenches at 12 y after application.

Average penetration into treated soil by termites was significantly different ($F=7.62$; $df=5, 96$; $P<0.0001$) among termiticides (Table 5). Termite penetrations into soil treated with either formulation of cypermethrin or permethrin were not different from the control. Penetration of chlorpyrifos-treated soil was not significantly different compared with fenvalerate-treated soil. Residues of chlorpyrifos and fenvalerate in soil from inside trenches limited penetration by termites to 17.3 and 0.0 mm, respectively.

The interaction of treatment.location for termite mortality was significant ($F=4.07$; $df=5, 96$; $P=0.0021$); therefore, locations were analyzed separately. Termites had the greatest mortality when exposed to soil treated with chlorpyrifos (Table 6). Termite mortalities were 97% and 55% with bioassays of chlorpyrifos-treated soil from trenches inside and outside foundations, respectively. Chlorpyrifos residues associated with these mortalities ranged from 33 to 117 ppm in soil from inside trenches, whereas, residues from outside trenches ranged from 15 to 46 ppm (Table 4).

Su *et al.* (1999a) found mean termite mortality of 23.5% and mean penetration of 50 out of 50 mm, 48 mo after sand was treated with chlorpyrifos (1.0%). Su *et al.* (1999b) also reported termite mortality and penetration of 15% and 49 of 50 mm, respectively, of sand 48 mo after treatment with chlorpyrifos (1.0%). Chlorpyrifos did not remain effective as long in Florida sand in Su *et al.*'s (1999a, b) tests as it did in the sandy loam soil in our study.

Table 6. Average termite mortality in 52-mm thick soil cores collected from inside and outside trenches around simulated foundations 12 y after initial application (7-d bioassay).

Treatment	Mortality %	
	Inside	Outside
Chlorpyrifos (Dursban®)	96.9 ± 6.1 a	55.0 ± 6.1 a
Cypermethrin (Demon®)	5.8 ± 6.1 bc	8.8 ± 6.1 b
Cypermethrin (Prevail®)	10.6 ± 6.1 bc	5.6 ± 6.1 b
Fenvalerate (Tribute®)	7.6 ± 6.1 bc	12.9 ± 6.1 b
Permethrin (Dragnet®)	24.4 ± 6.1 b	8.3 ± 6.1 b
Control	9.3 ± 6.1 c	4.3 ± 6.1 b

Mortality percentages within a location followed by the same letter are not significantly different ($P < 0.05$) as determined by PDIFF (SAS Institute 2001).

Termiticide concentration dependent mortalities of termites in soil treated with pyrethroids were not as direct as that of chlorpyrifos. Pyrethroids are known to repel termites, preventing termites from tunneling in treated soil and receiving a toxic dose (Su & Scheffrahn 1990). A measure of termite penetration is a better indicator of pyrethroid efficacy than mortality; therefore, mortalities observed in our bioassays of pyrethroids had little relationship to the amount of active ingredient remaining in the soil.

Fenvalerate was the most effective pyrethroid in preventing termite penetration at 12 y after application (Table 5). Fenvalerate residue, averaged over locations and depths, 12 y after application was 67.2 ± 18.1 ppm compared with 29.3 ± 3.3 ppm for permethrin, and < 1 ppm for cypermethrin. Su & Scheffrahn (1990) developed repellency thresholds, the lowest concentration to totally stop termite penetration, for pyrethroids. These thresholds were: chlorpyrifos – 8 ppm, cypermethrin – 1-6 ppm, fenvalerate – 8 ppm, and permethrin – 0.4-0.8 ppm. They used technical grade materials that were applied to an unidentified soil type. According to their repellency thresholds, and the termiticide concentrations in soil after 12 y in this study, there should have been no penetration of soils in our bioassay. The amount of termite penetration in our tube bioassay compared with the repellency thresholds developed by Su & Scheffrahn (1990) suggests the termiticides in our study

were not as biologically available as those in Su & Scheffrahn's study, or that the termites in our study were able to avoid contact with toxic concentrations of termiticide during the construction of their tunnels. The amount of exposure of individual termites to termiticide residues in our study is unknown. Individual termites involved in tunnel construction may limit the amount of time spent exposed to toxic residues by sharing in the construction of tunnels, thus reducing uptake of the termiticide by individual termites (Jones 1990). Also, the top and bottom of the tubes used in our study are open spaces free of treated soil where termites can avoid exposure. Tunnels were constructed mostly between the soil core and the interior wall of the tube in our study, thus minimizing the amount of contact termites had with treated soil. Lastly, treatment of soil in the lab, as was done by Su & Scheffrahn (1990), is vastly different than application to soil in a forest. It is possible that soil cores collected from the study sites in the forest after 12 y contained roots and other organic matter, as well as excavations created by other soil invertebrates that could provide passage safe ways for termites through treated soil.

The USDA Forest Service has conducted field efficacy tests of chlorpyrifos, cypermethrin, fenvalerate, and permethrin in AZ, FL, MS, and SC as part of the requirements for product registration with the EPA. At the MS test site, chlorpyrifos (1%) was 100% effective at preventing termite attack on wood in concrete slab plots for 11 y; cypermethrin (0.25%), 3 y; fenvalerate (0.5%), 7 y; permethrin (0.5%, Torpedo), 1 y; and permethrin (0.5%, Dagnet), 5 y (Kard 2000, Wagner *et al.* 2003). Chlorpyrifos and fenvalerate were 100% effective longer than cypermethrin or permethrin. In our bioassays, chlorpyrifos elicited the highest mortality (96%), and fenvalerate exhibited the least overall distance penetrated (16 mm) by termites into treated soil. Overall termite penetration was limited to 31 and 50% through 52 mm-thick cores from fenvalerate and chlorpyrifos, respectively.

Our laboratory bioassay indicate that soils from trenches inside building foundations treated with chlorpyrifos or fenvalerate were both toxic and repellent to termites 12 y after application. While these tests provide some indication of termiticide performance, they cannot be considered as direct indicators of how effective these termiticides would be in preventing termite attack on wooden structures with the many possible and varied breaches of termiticide barriers that can occur around structures over a 12 y.

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REFERENCES

- Gold, R.E., H.N. Howell, Jr., B.M. Pawson, M.S. Wright, & J.C. Lutz 1996. Persistence and bioavailability of termiticides to subterranean termites (Isoptera: Rhinotermitidae) from five soil types and locations in Texas. *Sociobiol.* 28 (3): 337-363.
- Jarratt, J.H., J. Haskins, & R. Ingram 2004. Five-year soil concentrations of seven termiticides in two Mississippi soil types. *J. Entomol. Sci.* 39: 159-174.
- Jones, S.C. 1990. Effects of population density on tunneling by Formosan subterranean termite (Isoptera: Rhinotermitidae) through treated soil. *J. Econ. Entomol.* 89: 875-878.
- Kard, B.M. 1996. Gulfport termite control research. In, Proceedings of GA Pest Control Assoc. Winter Meeting, pp. 1-4 Univ. GA, Athens, Jan 10-12, 1996.
- Kard, B.M. 2000. Gulfport study welcomes new entries. *Pest Control* 69(2): 38-39, 44.
- Kard, B.M., & C. A. McDaniel 1993. Field evaluation of the persistence and efficacy of pesticides used for termite control, pp. 46-61. *In*: K.D. Racke & A.R. Leslie eds. *Pesticides in Urban Environments: Fate and Significance*. ACS Symposium Series 522, American Chemical Society, Washington, DC.
- McDaniel, C.A., & B.M. Kard 1994. The latest in termiticide degradation. *Pest Control Tech.* 22(5), 80, 84, 86, 90-91.
- Racke, K.D., D.D. Fontaine, R.N. Yoder, & J.R. Miller 1994. Chlorpyrifos degradation in soil at termiticidal application rates. *Pestic. Sci.* 42: 43-51.
- Sapiets, A., H. Swaine, & M.J. Tandy 1984. Analytical methods for pesticides and plant growth regulators, vol. XIII, Chap. 2, Cypermethrin. Academic Press, Inc.
- SAS Institute 2001. SAS User's Guide. SAS Institute, Cary, NC.
- Scheunert, I. 1992. Transformation and degradation of pesticides in soil. *In*: *Chemistry of Plant Protection: Terrestrial Behavior of Pesticides*, pp.70-140. Springer-Verlag, New York, NY.
- Shaw, H.R. 1977. Gas chromatographic method for residual of tanol and of tanol oxygen analog in soils. Chemagro Agric. Div. of Mobay Chemical Corp. Research and Development Dept. Report No. 53690, Doc. No AS79-506. 17p.
- Shell Development Company 1984. Analytical methods for pesticides and plant growth regulators, vol. XIII. Chap. 7, Pydrin: Insecticide. Academic Press, Inc.
- Su, N.Y., & R.H. Scheffrahn 1990. Comparison of eleven soil termiticides against the Formosan subterranean termite and eastern subterranean termite (Isoptera: Rhinotermitidae). *J. Econ. Entomol.* 83: 1918-1024.

- Su, N.Y., P.M. Ban, & R.H. Scheffrahn 1999a. Longevity and efficacy of pyrethroid and organophosphate termiticides in field degradation studies using miniature slabs. *J. Econ. Entomol.* 92: 890-898.
- Su, N.Y., P.M. Ban, V. Chew, & R.H. Schreffrahn 1999b. Size and edge effects of concrete plots in chlorpyrifos degradation in sub-slab sand. *J. Econ. Entomol.* 92: 409-415.
- Swaine, H. & M.J. Tandy 1984. Analytical methods for pesticides and plant growth regulators, vol. XIII. Chap. 6, permethrin. Academic Press, Inc.
- Wagner, T.L., J.E. Mulrooney, T.G. Shelton, & C.J. Peterson 2003. Reduce risk products steal spotlight. *Pest Control.* 71(2): 16-18, 20-23.
- Wetters, J.H. 1977. Determination of residues of chlorpyrifos in soils by gas chromatography. Dow Chemical U.S.A, Agric. Prod. Div., Midland, MI. ACR 77.7. 13pp.



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