Bull. Environ. Contam. Toxicol. (1993) 51:356–360

Environmental Contamination and Toxicology

Cypermethrin in the Ambient Air and on Surfaces of Rooms Treated for Cockroaches

C. G. Wright,[1] R. B. Leidy,[2] and H. E. Dupree, Jr.[1]

[1]Entomology and [2]Toxicology Departments, North Carolina State University, Raleigh, North Carolina [1]27695-7613 and [2]27695-8604, USA

Cypermethrin is one of several pyrethroid insecticides which are labeled for the control of cockroaches. In recent years cypermethrin has been used extensively in controlling the German cockroach, Blattella germanica (Linnaeus) (Blattodea: Blattellidae). Wright and Dupree (1987), Appel et al (1989) and Bohnert et al (1991), among others, reported its control effectiveness in houses and apartments. Cypermethrin also provides control of termite infestations and is used by pest control firms on a routine basis (Mix 1991). Little attention has been given to the possible presence of cypermethrin in the ambient air and/or on indoor surfaces of structures following application of the insecticide for cockroach and termite control. Therefore, the following study was done to ascertain its presence or absence in buildings when applied for control of pests.

MATERIALS AND METHODS

Twelve rooms in a vacant three-story dormitory at North Carolina State University, Raleigh NC, were used as test sites. Three treatment and three control rooms (rooms adjoining treatment rooms) on floors one and three were selected. Control rooms were used to ascertain possible cypermethrin movement following its application in the treatment rooms. Rooms contained two student desks and chairs, two dressers, two single beds, and one or two walk-in closets. Dresser and desk drawers were open and windows closed with shades drawn during the test. Rooms were closed with no heat or air conditioning. Cypermethrin (0.2%) was applied to the treatment rooms using a 3.8 L compressed air sprayer operated at 1.4 kg/cm^2. The sprayer was equipped with a Multeejet nozzle (No. 5800, manufactured by Spraying Systems, Bellwood IL) adjusted to apply the spray in a pin stream pattern directed into cracks and crevices in the rooms.

Byron Model 90 air samplers (General Monitors, Inc., El Toro, CA) with Orbo 42 tubes (Supelco, Inc., Bellefonte, PA) were used to sample the ambient air at the following times: preapplication, immediately after application and at 1, 4, 6, 8, 10, and 12 wk.

Send reprint requests to C. G. Wright at the above address.

Air samplers were calibrated to give a constant flow rate of 2.0L/min and a sample was collected for 2 h (total volume sampled = 240 L of ambient air).

After sampling front and rear portions of the Orbo 42 tube were separated and placed in separate, silylated (Sylon-CT, Supelco, Inc., Bellefonte, PA) 10-mL tubes containing 10 mL of CH_3CN. Tubes were capped and vortexed at 15 min intervals for 4 h. Following overnight refrigeration the tubes were allowed to warm to room temperature and the solvent was then transferred to a 10-mL syringe to which was attached an Acrodisc filter (0.45 ug pore size). The solution was filtered into a 12-mL test tube and concentrated to 0.2 mL under a stream of dry nitrogen at 50°C. 2-propanol (0.8 mL) was added to each tube with agitation and 250 μL aliquots were transferred from the tubes to autosampler vials equipped with 250 μL inserts (Waters Milipore, Milford, MA) for chromatography by HPLC.

An aluminum template (7.6 x 35.6 cm), with a 2.5 x 30.5 cm opening in its center, was positioned over the surface to be sampled. A sterile cotton ball was placed in a 30 mL glass bottle containing 10 mL of 2-propanol as a wetting solution. At the time of swabbing, sterile forceps were used to squeeze the cotton ball against the side of the glass bottle to remove as much of the 2-propanol as possible. The cotton ball was wiped in one direction, covering the 2.5 x 30.5 cm opening in the template, turned over and wiped in the other direction. A new pair of sterile disposal latex gloves was used when collecting each sample. The swab was returned to the glass bottle, the bottle was then sealed and labeled. Samples were taken from the following surfaces and locations in the treated rooms: two furniture surfaces (horizontal desk top and vertical side of dresser), two vertical wall surfaces (61 and 122 cm from the floor) and two horizontal floor surfaces (along the edge of the wall and at the center of the room). Control rooms were surface sampled only on the wall, 61 cm from the floor. Surface samples were taken once ambient air samples had been collected. Temperature and relative humidity were recorded at each sampling time.

The cotton ball and corresponding 2-propanol extract were transferred to a Soxhlet thimble and the sample vial was rinsed twice with 5 mL of acetone: hexane (1:1), and transferred to a Soxhlet extractor. Samples were extracted for 4.0 h with 200 mL acetone: hexane (1:1) (v/v) at 8 cycles/hr. After cooling, the solvent was reduced to 3 to 5 mL under reduced pressure at 40°C and transferred quantitatively to a silylated 12-mL tube with hexane and placed on a Florisil Sep Pak. The hexane solution was allowed to flow through the Florisil Sep Pak and the cypermethrin was eluted quantitatively with 10 mL of 25% ether in hexane. Samples were concentrated to 0.2 mL under a stream of dry nitrogen and brought to volume with 2-propanol for analysis by HPLC.

The HPLC was performed with a Waters Milipore Model 590 Pump using CH_3CN:2-propanol:H_2O (1:1:0.5) at a flow rate of 1.2 mL/min. The

column was a C_{18} (10 by 0.8 cm, 5 to 10μm particle size) contained in a Water Milipore Radial Compression 8x10 module. Detection was by UV absorption (Water Milipore Model 481 UV/VIS Detector) at 214 nm and 0.1 AUFS. The retention time of cypermethrin was 5.2 min. Analytical standards were chromatographed between every five samples for quantitation purposes using a Waters Model 710B WISP autosampler with a sample injection volume of 100 μL and a 20-min interval between injections. Three different volumes of the analytical standard were made between each sample set.

Cotton balls and ORBO tubes were fortified with various known amounts of cypermethrin, and, using the same pipet, equivalent amounts were added to 12-mL tubes and refrigerated until analyzed. One fortified sample was included with every 10 samples. Recoveries averaged 91.4% (range 86 to 96%). The data were not corrected for extraction efficiency. Residue data were analyzed by an analysis of variance (ANOVA).

RESULTS AND DISCUSSION

The amount of cypermethrin AI applied in the 3 dormitory rooms averaged 0.25 $\pm$ 0.1g. Temperature and relative humidity in the rooms during the test period ranged from 19 to 29°C and 60 to 86% RH, respectively. No significant difference in temperature or relative humidity occurred between the rooms at each sampling period. There was a significant ($P \leq 0.01$) increase in mean temperature for the rooms between the first and final temperature measurements (19 to 28°C). A significant ($P < 0.01$) increase occurred in the relative humidity, with an initial and final mean recordings of 72 and 82%, respectively. There was no discernible correlation between cypermethrin levels and the temperature or relative humidity in the rooms.

Cypermethrin was not detected in the ambient air of the rooms during preapplication sampling, but was significantly ($P \leq 0.01$) greater immediately after its application in the rooms treated and 7 d later in the adjoining, untreated rooms (Table 1). Quantities of cypermethrin were detected in the air at all postapplication sampling intervals through cessation of the test at 84 d, except in the untreated rooms immediately after it application to the treated rooms. There was a significant (($P \leq 0.05$) decline in quantities at successive sampling times. Levels detected were greater at 42 and 56 d than at 28 d in the treated rooms and at 42 d than at 28 d in the untreated rooms. The reason for this reversal is unknown. Some rooms had no detectable quantities at the last sampling times.

No cypermethrin was detected during preapplication surface sampling in the rooms (Table 2, 3). Residues were significantly ($P < 0.01$) greater on all surfaces in the cypermethrin-treated rooms immediately after its application. Several surface samples in the adjoining, untreated control rooms, contained small quantities of the insecticide immediately after application. Seven d and later postapplication levels decreased in treated and untreated rooms

Table 1. Cypermethrin ($\mu g/m^3$) detected in the air of vacant dormitory rooms following its application for cockroach control.[a]

Preapplication	Postapplication (d)					
0	7	28	42	56	70	84
Treated rooms						
ND	18.2±9.9	8.5±3.0	3.0±0.6	7.1±2.5	4.4±0.7	0.6±0.2	0.3±0.1
Untreated rooms						
ND	ND	6.4±4.6	3.9±0.2	4.2±0.9	2.1±1.5	0.2±0.1	0.1±0.1

[a]Mean with standard deviation, average of three samples. ND=Not detectable, below 0.1 μ/m^3 level. Cypermethrin levels with different superscripts are significantly ($P \leq 0.05$) different by postapplication d for treated and untreated rooms, respectively.

until at 84 d most detectable levels were <0.1 to 0.1 μg on the 100 cm^2 surface areas. Somewhat greater mean residue levels were present on horizontal (floor) than vertical (wall) surfaces, with two exceptions, although there was no significant difference due to quantity variations detected within replicates for different sites. Levels at 28 d were greater ($P \leq 0.01$) on the desk top and wall (61 cm from the floor) in the untreated rooms than in the treated rooms. The reason for this exception is not known.

Table 2. Cypermethrin ($\mu g/100cm^2$) detected on non-target, vertical surfaces in empty dormitory rooms following its application for cockroach control.[a]

Sample taken (d)	Wall			
	61 cm from floor		122 cm from floor	Dresser
	(T)	(U)	(T)	(T)
Preapplication	ND	ND	ND	ND
Post-Application				
0	1.3±0.6[a]	ND	2.5±1.3[a]	2.3±0.9[a]
7	0.8±0.9[a]	0.1±0.1[a]	1.7±0.7[a,b]	1.2±0.6[b]
28	0.9±0.9[a]	1.1±0.8[a]	1.3±0.2[a,b]	0.8±0.3[b,c]
42	0.8±0.7[a]	0.2±0.1[a]	1.2±0.4[a,b]	0.7±0.3[b,c]
56	1.9±1.3[a]	0.5±0.3[a]	1.2±0.3[a,b]	0.2±0.1[b,c]
70	0.5±0.1[a]	ND	0.4±0.1[b]	0.1±0.1[c]
84	0.2±0.1[a]	0.1±0.0[a]	0.1±0.1[b]	ND

[a]Mean with standard deviation, average of three samples. No samples taken on the wall (122 cm from floor) nor on the desk in the adjoining, untreated rooms. T=treated and U-untreated rooms. ND=Not detectable, below 0.1 $\mu/100$ cm^2 level. Cypermethrin levels with different superscripts are significantly ($P<0.05$) different by postapplication d for treated and untreated rooms.

Table 3. Cypermethrin (μg/100 cm^2) detected on non-target, horizontal surfaces in empty dormitory rooms following its application for cockroach control.[a]

Sample Taken (d)	Floor			Desk Top	
	Center		Next to Wall		
	T	U	T	T	U
Preapplication	ND	ND	ND	ND	ND
Post application					
0	3.5±2.4	0.6±0.4	3.1±3.0	2.4±1.1	0.3±0.2
7	0.4±0.2	0.1±0.1	0.3±0.2	0.3±0.2	ND
28	1.2±0.1	ND	1.1±0.3	0.6±0.4	1.4±0.7
42	0.8±0.4	ND	0.9±0.2	0.8±0.3	0.5±0.3
56	1.5±1.3	0.4±0.2	0.5±0.6	ND	0.2±0.2
70	0.2±0.1	0.1±0.1	0.4±0.2	0.5±0.1	0.1±0.0
84	0.1±0.0	0.1±0.0	0.2±0.1	0.1±0.1	ND

[a]Mean with stand deviation, average of three samples. No samples taken on the floor next to the wall in the adjoining, untreated rooms. T=treated and U=untreated rooms. (ND)=Not detectable, below 0.1 μg/100cm^2 level. Cypermetnrin levels with different superscripts are significantly ($P \leq 0.05$) different by postapplication d for treated and untreated rooms.

Cypermethrin quantities in the ambient air and on surfaces in rooms treated for cockroach control and adjoining untreated rooms were very low. Residues dissipated during the test period, yet small quantities were detected in the ambient air and on some surfaces at the last sampling time, 84 d post-application.

Acknowledgment. Use of trade names in this publication does not imply endorsement of the products named or criticism of similar ones not mentioned.

REFERENCES

Appel, AG, Sponsler RC, Strother GR (1990) Evaluation of residual spray formulations for German cockroach control, 1988. I and A Tests 15:353.

Bohnert, TJ, Reid BL, Bennett GW (1991) Field trials of insecticidal sprays, 1991. I and A Tests 17:373-374.

Mix, J (1991) Dursban TC tops termiticide survey. Pest Control 59(2):22.

Wright, CG and Dupree, Jr HE (1987) Cypermethrin sprays for German cockroach control in single-family dwellings, 1986. I and A Tests 12:360.

Received December 28, 1992; accepted March 1, 1993.