

THE DISTRIBUTION OF CHLORPYRIFOS IN AIR, CARPETING, AND DUST AND ITS REEMISSION FROM CARPETING FOLLOWING THE USE OF TOTAL RELEASE AEROSOLS IN AN INDOOR AIR QUALITY TEST HOUSE

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ABSTRACT

Experiments were conducted to explore the relationships between the insecticide chlorpyrifos and its distribution into carpet, carpet dust, and reemission into air. Two total release aerosols containing 0.5% chlorpyrifos were applied in the living room and den of the EPA test house. Afterwards surface and carpet depositions, and carpet dust concentrations were determined. Portions of the contaminated carpeting were removed and placed in environmental chambers (0.053 m³). Emissions of chlorpyrifos from carpet to air were determined. Air exchange rates and airborne chlorpyrifos concentrations were measured in the test house over a 2-week period. Airborne chlorpyrifos concentrations in the treated rooms of the test house exceeded 10 µg/m³ of air following 1 hour of enhanced ventilation and declined to <1 µg/m³ by 14 days post-application. Concentrations in the untreated bedroom were 11 times lower than in the den and living room following ventilation, reached maximal levels by day 2, and declined to <0.5 µg/m³ by day 14. Surface loadings for dust were low compared to total surface deposition levels. No vertical stratification was observed within rooms. Total levels quantified from the carpet extraction and deposition coupons were similar in that levels were highest in rooms that received applications and negligible in the master bedroom. Levels of chlorpyrifos associated with house dust measured for all rooms increased immediately following the application and remained above background through day 14. Source emissions from carpet samples in chamber studies demonstrated that treated carpet is a latent source for the reemission of chlorpyrifos into air. However, carpet in an untreated room did not appear to act as a sink for chlorpyrifos. Mass balance results for the chamber and test house experiments indicate that factors other than the loss of the pesticide via outdoor air exchange must be understood to account for the decrease of pesticide in the treated carpet.

INTRODUCTION

Pesticides are applied in and around human habitations to control a variety of pests and may place toxicants in close proximity to humans and their activities. Pesticide residues may translocate from their original points of application following treatment as vapors, bound to particles, or through physical transport processes. The principal factors that influence their movement are physiochemical properties (i. e., the vapor pressure of the active ingredient and formulation type), the substrate that deposits are contacting, and the physical activities of humans and pets. Pesticides that translocate indoors are less influenced by the degrading factors such as photolysis and microbial activity that occur out-of-doors. Furthermore, residues present indoors may persist or accumulate over time and are commonly measured in residential dwellings at concentrations ranging from 10 to 100 times higher than those found out-of-doors¹. Studies have shown that carpets and associated carpet dust might serve as a latent source or reservoir of pesticide residues for months to years following application^{2,3}. Contaminants adsorbed to surfaces or bound to particles in "sinks" may disassociate as an airborne vapor or redistribute via particle movement to become available for human exposures. Exposure to indoor pollutants such as pesticides may pose risks to occupants through inhalation, dermal absorption, or ingestion.

Pilot experiments were conducted in the U. S. EPA's indoor air quality research test house in North Carolina to investigate the contribution of total release aerosols containing the insecticide chlorpyrifos to airborne residue levels, and the distribution onto carpeted surfaces, deposition coupons, and from carpet dust. In addition, carpet samples were removed from two treated rooms and placed in ventilated 0.053m³ (53 L) environmental chambers to determine reemissions of chlorpyrifos to air.

Indoor total release aerosols for chlorpyrifos were voluntarily withdrawn from the consumer market subsequent to the conduct of this experiment. Recently, all indoor consumer uses have been withdrawn. However, the data are relevant to understanding the relationships between indoor applications of semivolatile pesticides, the movement of like compounds in the indoor environment, and the potential for exposure.

MATERIALS AND METHODS

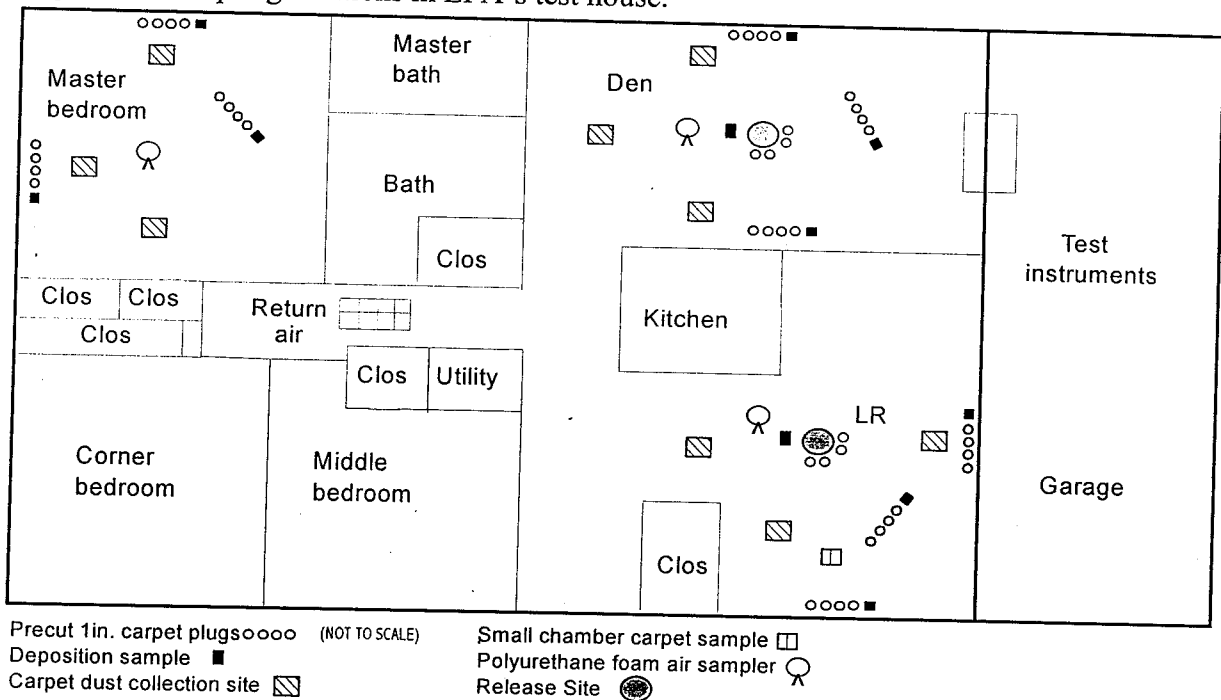
The study reported here was conducted in December 1993, in the U.S. EPA's Indoor Air Quality Research Test House. The test house is an unoccupied one-story, seven-room (three bedroom), ranch-style house located in a residential neighborhood in Cary, NC (Figure 1). The test house contains a total interior volume of 293 m³ with 122 m² of living area. All rooms are void of furniture and covered with wall-to-wall pile or shag (den) nylon carpet except the kitchen and the bathrooms. The test house is environmentally defined in that air exchange rates, temperature, and relative humidity are continuously monitored.

Two commercially available canisters of an aqueous-based total release aerosol (Real Kill, Realex Division of United Industries Corp., St. Louis, MO) formulated to contain

0.5% by weight of the insecticide chlorpyrifos [O,O-diethyl-O-(3,5,6-trichloro-2-pyridinyl) phosphorothioate] were purchased locally. Each canister was weighed before and after the application to determine the total mass released. The canisters were suspended 75 cm above the floor in the center of the den and living room with the spray tips directed upward. Note that the rooms are open to the entry hallway and the kitchen (Figure 1) and their physical separation is only partial. Prior to activation of the aerosol device, all windows and interior doors were closed, the furnace pilot light was extinguished, and the furnace fan was turned off. Upon activation, the house was vacated and reentered 2 hours later when the exterior doors and windows in the living room and den were opened and allowed to ventilate for 1 hour. Afterwards, the windows and exterior doors were closed, the furnace pilot was re-ignited, and the ceiling fans in the den and living room were turned on at low speed. The house thermostat was set to 72 °F (22 °C) for the duration of the experiment. The interior doors were closed throughout the test except for ingress and egress by the research scientists to and from the master bedroom in order to perform sampling activities.

Prior to the insecticide treatment, three or four round plugs (0.0005 m², 25.4 mm dia.) were precut in the carpet with a filter punch at selected locations. The carpet plugs were cut from the center of the living room, den, and master bedroom, from midway between the baseboards and the center and from within 0.05 m of the base boards as shown in Figure 1. A single precut carpet plug was removed from each carpet sampling location in each room prior to the treatment to determine the background chlorpyrifos levels in the carpeting. The remaining carpet plugs were left in place for collection at later sampling intervals. See Figure 1.

Figure 1. Sampling locations in EPA's test house.



Deposition coupons consisting of 1 in. square foil-backed gauze pads were affixed with safety pins to the floor adjacent to the carpet plugs. A carpet plug and a deposition coupon were removed from each carpet sampling location immediately following the ventilation period. The remaining carpet plugs were removed at the end of the 2-week test period. Both sample types were collected using clean solvent-rinsed forceps, placed in labeled glass jars, and stored in ice chests at reduced temperatures for transport.

For subsequent chamber testing, 0.09 m² (30.5 X 30.5 cm) sections of carpet were pre-cut 1-m from a corner in the den and living room. The carpet sections remained in place and were collected from the treated rooms upon entry following the air-out procedure. Upon collection, the carpet sections were sealed in Tedlar[®] bags, transported to the laboratory, immediately removed from the Tedlar[®] bags, and placed in the EPA's electropolished stainless steel, 0.053 m³ (53-L) test chambers. The chambers were held at a temperature of 23°C and supplied with purified air that was conditioned to a relative humidity of 45%. Chamber airflow was maintained at a constant rate of 0.4 L/min, providing an exchange rate of approximately 0.45 h⁻¹. Each carpet section was placed fiber side up at the bottom of each chamber. A small biscuit fan mounted in the center of each chamber and 7.4 cm from the sample maintained a nominal air speed of 5 to 10 cm/sec above the surface of each carpet section. Airborne chlorpyrifos concentrations in the chamber were collected using polyurethane foam (PUF) filters held in glass housings and vacuum pumps calibrated to a constant flow rate of 200 cm³/min. Sampling intervals varied in duration from 2 to 24 hours to provide appropriate loading on the PUF sorbents.

Vacuum-dislodged dust was collected using the high-volume sampler (HVS3) using ASTM D5438-93⁴ in the master bedroom, living room, and den. Each location consisted of an area of 1.5 m² (1 by 1.5 m) outlined with masking tape in the center of each of four designated quadrants in each room. Vacuum sweepings were collected from a single quadrant in each room just prior to the treatment, immediately following the period of enhanced ventilation and at 2 weeks post-application.

Air was monitored using PUF filters held in glass filter housings. Monitoring was conducted in the living room and den at 25, 100, and 200 cm above the floor immediately following the application and at the end of the 2-week sampling period, while all subsequent samples were collected at 100 cm above the floor. Measurements were collected for 1 hour using pumps calibrated to a continuous flow rate of 15 L/min. Following the sample collection, the PUF filters and housings were wrapped in clean aluminum foil, individually sealed in plastic bags, and stored in ice chests at reduced temperatures for transport.

The indoor/outdoor air exchange rates were monitored throughout the study using a tracer gas technique⁵. Sulfur hexafluoride (SF₆) was released every 2 hours at the furnace-return's air-vent and monitored once every 5 minutes in the master bedroom, den, and living room. The concentration of SF₆ was determined by gas chromatography with electron capture detection (HP 5890, Poropak Q, 6mm X 2 m column). To determine the air exchange rate during the flush-out period, SF₆ was released just prior to opening exterior doors and windows. The furnace fan was activated to mix the SF₆ throughout the

house. During the air-out procedure, concentrations of SF₆ were monitored only in the den to obtain a sufficient number of measurements.

The PUF filter and carpet plugs were Soxhlet extracted in 5% ethyl ether/ hexane and concentrated to a final volume of 10 mL for analysis. Vacuum sweepings were separated with a #10 sieve to obtain a fraction smaller than 150 μm. A 0.5 g aliquot of the sieved fraction was Soxhlet extracted in 5% ethyl ether/ hexane and concentrated to a final volume of 10 mL for analysis. The surrogate standard, tetrachloronaphthalene, was added to each sample prior to extraction to assess overall method performance. After the final concentration step, pentachloronitrobenzene was added to the sample extracts as an external quantitation standard. Samples were analyzed using a Varian 3700 gas chromatograph equipped with a liquid autosampler and electron capture detector. A DB-5 fused silica column (30 m X 0.32 mm) was used for quantitation. The carrier flow rate was 2.0 mL/min. The temperature program was initiated at 80°C and ramped to 300°C at 4°C/min. The capillary injector was operated in the splitless mode for 1 min. Injector and detector temperatures were 280 and 320 °C, respectively.

Label instructions for the pesticide state that each canister must be released in an area with a volume of at least 1500 ft³. With interior doors closed, the den, kitchen, living room, dining room, entry hall, and main hall leading to the bedrooms form a contiguous space with 55 m² of floor area with a volume of 158 m³ or 5600 ft³. Based upon the labeled percent active ingredient and the net weight of the containers, a total of 1700 mg of chlorpyrifos would be released. If the pesticide all deposited evenly on the contiguous floors, then predicted loading would be 31 mg/m².

Quality Control

Quality control included reagent and field blanks, reagent, PUF sampler, and carpet spikes as well as duplicate samples and replicate analyses. Chlorpyrifos was not detected (<50 ng/sample) in the nine reagent blanks or three PUF field blanks. Nine reagent blanks and three field control PUF cartridges were each fortified with 500 ng of chlorpyrifos and provided recovery efficiencies (mean ± standard deviation) of 93 ± 6%, and 90 ± 2%, respectively. Three carpet plugs were each fortified with 51 μg of chlorpyrifos and gave recoveries of 83 ± 11%. Three aliquots of carpet dust fortified with 10 μg of chlorpyrifos gave recoveries of 106 ± 4%. The surrogate standard (tetrachloronaphthalene) was added to all 149 samples of the study and provided a recovery efficiency of 100 ± 12%.

RESULTS AND DISCUSSION

The chlorpyrifos loadings measured from deposition coupons in the living room and den ranged from 28 to 73 mg/m² (Table 1). Residues for all rooms were highest nearest the spray canister (the center of the room) and decreased by approximately half at the midpoint and baseboards. Loadings measured from the master bedroom ranged from non-detectable (<0.08 mg/m²) to 0.44 mg/m². Average levels measured from the master bedroom were 2 orders of magnitude lower than those measured in the two treated rooms.

Table 1. Chlopyrifos loadings measured from deposition coupons, carpet samples, and housedust.

Location	Loading (mg/m ²)								
	Living Room			Den			Master Bedroom		
	deposition	carpet	dust	deposition	carpet	dust	deposition	carpet	dust
Preapplication									
Center	-- ^a	0.71	--	--	0.12	--	--	0.81(0.75) ^b	--
Midpoint	--	1.1	0.00012	--	0.12	0.00055	--	0.78	0.00013
Baseboard 1	--	0.78	--	--	0.20	--	--	0.73	--
Baseboard 2	--	0.71	--	--	0.10	--	--	0.61	--
Mean ±S.D.	--	0.83 ±0.19	--	--	0.14±0.04	--	--	0.62±0.23	--
Application Day									
Center	73 (120)	44 (55)	--	72 (74)	96 (83)	--	0.44	0.69	--
Midpoint	28	21	0.52	37	33	1.0 (0.95)	0.23	0.91	0.0024
Baseboard 1	29	30	--	29	22	--	<0.08	1.10	--
Baseboard 2	32	20	--	31	46	--	0.39	0.59	--
Mean ±S.D.	47±34 30±2.1 ^c	30±14 24±5.5 ^c	--	43±21 32±4.2 ^c	48±30 34±12 ^c	--	0.28±0.18	0.83±0.22	--
14 Days Post-Application									
Center	--	30	--	--	49	--	--	--	--
Midpoint	--	14	0.060	--	18	0.87(0.83)	--	0.68	0.0013
Baseboard 1	--	14	--	--	14	--	--	0.93	--
Baseboard 2	--	17	--	--	9.4	--	--	0.69	--
Mean ±S.D.	--	19±7.6 15±1.7 ^c	--	--	22±18 14±4.3 ^c	--	--	0.77±0.14	--

^a No sample

^b Results for duplicate sample

^c Calculated without center sample

A mean surface loading of 44 mg/m² for the 55-m² contiguous floor was determined from the four sampling locations in the treated rooms. A total estimated deposition of ~2420 mg for the treated rooms is ~720 mg greater than the theoretical total mass of 1700 mg emitted from the total release aerosols as determined from the label concentrations. The mean of the two deposition samples from the center of the treated rooms was 75 mg/m² compared to a mean of 31 mg/m² for all other locations. Clearly, the application resulted in a non-homogeneous distribution of chlorpyrifos on the carpet. High surface loading

immediately adjacent to the canister likely biased the mean values and overestimated the loading.

The theoretical distribution of chlorpyrifos on the treated carpeting based on the label concentration is 31 mg/m². The net weight loss from the canisters was in close agreement with the label amount. On the other hand, the concentration of chlorpyrifos in the canisters was not confirmed by independent measurement.

The aqueous-based total release aerosols produced a mist of particles in the air of the treated rooms. The deposition and carpet extract data indicate that the bulk of the active ingredients were deposited on the carpets of the contiguous rooms during the 2 hours following the release. Minimal deposition onto surfaces in the master bedroom suggests that the combination of closed doors and the heating and air-conditioning system in the off state likely minimized contamination in the untreated room.

Background levels of chlorpyrifos were detected from the total carpet extracts in all rooms (Table 1). Mean background levels were less than 1 mg/m² in all rooms and approximately 5 times greater in the pile carpets of the master bedroom and living room compared to the den. The average carpet loading for the living room and den increased above background to 30 and 48 mg/m², respectively, following the application and declined by approximately half by day 14. These loadings are similar to those measured with the deposition coupons. Concentrations measured from carpet in the untreated master bedroom show little variation throughout the test period. The carpets were not sampled for 3,5,6-trichloro-2-pyridinol, a primary metabolite of chlorpyrifos.

Low background levels of chlorpyrifos were measured from house dust collected prior to the application (Table 2). The concentrations of chlorpyrifos extracted from the pre-application dust samples were similar from room to room (0.00019, 0.00015, and 0.00023 mg/g for the living room, den, and master bedroom, respectively). On an area basis, the higher amounts seen at preapplication in the den are due to a greater amount of extractable dust in the shag carpet. Chlorpyrifos concentrations from carpet dust increased above background following the application in the den and living room and remained above background levels through day 14. Similarly, chlorpyrifos loadings in dust of the untreated master bedroom increased immediately following the application and remained above background. Prior to the application, 0.01 and 0.41% of the pesticide extracted from the carpets was found in the dust samples of the living room and den, respectively (calculated by dust loading/ average carpet loading from each room). Following the application, about 2% of the total extractable chlorpyrifos was associated with the dust fraction from these rooms. This suggests that very little of the pesticide is bound to dust particles; rather, it is in the residue form in contact with carpet material. The dust collected by the HVS3 may have been tainted from residues on the freshly treated carpet. Therefore, the dust concentrations measured following the application should be considered as upper limit estimates.

Table 2. Chlorpyrifos extracted from carpet dust fractions collected at intervals following a pesticide application using total release aerosols.

Day	Living Room		Den		Master Bedroom	
	Loading (mg/m ²)	Concentration (mg/g)	Loading (mg/m ²)	Concentration (mg/g)	Loading (mg/m ²)	Concentration (mg/g)
Preapplication	0.00012	0.00019	0.00055	0.00015	0.00013	0.00023
Application	0.52	0.68	1.0 (0.95)	0.11 (0.10)	0.0024	0.0031
Post Day 14	0.060	0.080	0.87 (0.83)	0.26 (0.25)	0.0013	0.0039

Prior to the aerosol application, airborne levels of chlorpyrifos were below detectable levels ($<0.05 \mu\text{g}/\text{m}^3$) (Table 3). Only slight differences between airborne concentrations were observed between 25 and 200 cm above the floor in the living room and den at post application day 14, when samples were collected at three elevations. Residues measured at 100 cm above the floor in the two treated rooms both reached highest levels of about $16 \mu\text{g}/\text{m}^3$ immediately following the application and decreased to about $0.4 \mu\text{g}/\text{m}^3$ by day 14. These findings agree with those of other studies⁶ where, following the use of total release aerosols containing chlorpyrifos, airborne concentrations ranged between 3 and $50 \mu\text{g}/\text{m}^3$. Conversely, airborne concentrations measured in the untreated master bedroom were determined to be $1.4 \mu\text{g}/\text{m}^3$ immediately following the application and $4.7 \mu\text{g}/\text{m}^3$ 24 hours later. The apparent delay in peak concentration in the untreated room is surprising since the air handling system mixes SF₆ evenly throughout the house within 15 min of release at the return air grill. However, sampling frequency may not have been adequate to identify peak concentrations and characterize the time concentration profiles in the treated and untreated rooms. The air concentrations declined to $<0.5 \mu\text{g}/\text{m}^3$ in the treated rooms and the master bedroom by day 14.

Table 3. Chlorpyrifos concentrations measured from the indoor air of the IAQ test house following a pesticide application using total release aerosol.

Day	Sampling Height (cm)	Living Room ($\mu\text{g}/\text{m}^3$)	Den ($\mu\text{g}/\text{m}^3$)	Master Bedroom ($\mu\text{g}/\text{m}^3$)
Preapplication	100	ND ^a	ND	NC ^b
Application	25	16	18	NC
Application	100	15 (15) ^c	17	1.4
Application	200	16	16	NC
Post Day 1	100	9.2	8.3 (7.5)	4.7
Post Day 2	100	4.1	4.0 (3.3)	NC
Post Day 3	100	2.3	2.1	NC
Post Day 7	100	0.86	1.1	0.37 (0.37)
Post Day 14	25	0.49	0.5	NC
Post Day 14	100	0.45	0.41 (0.43)	0.32
Post Day 14	200	0.48	0.38	NC

^a Not detected ($<0.05 \mu\text{g}/\text{m}^3$).

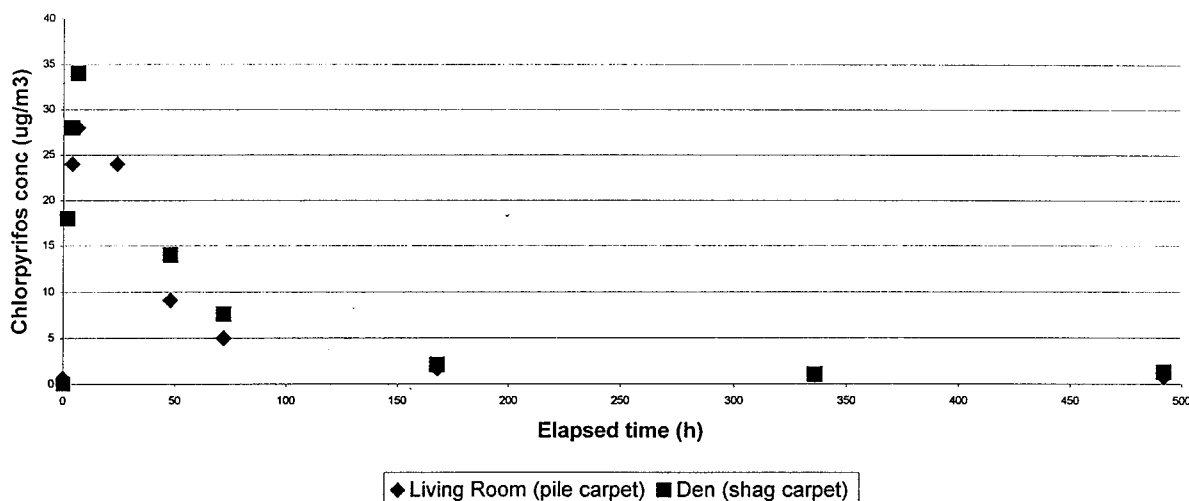
^b Not collected.

^c Result of duplicate sample analysis.

The outdoor air exchange rate for the den and dining room was determined to be 4.3 h^{-1} during the ventilation period while the exterior doors and windows were fully open. For the remainder of the test, with windows and interior doors closed, the air exchange rates for the living room, den, and master bedroom averaged 0.75 h^{-1} . The ventilation during the 1-hour air-out procedure is sufficient to exhaust >99% of the air in the den and living room at the end of the 2-hour period following the release. Thus, the air concentration in the first air sample, which accounts for less than 0.2% of the amount released, is due to reemission of chlorpyrifos from treated surfaces or resuspension of dust or droplets. The amount of chlorpyrifos leaving the house via the air during the 2-week experiment, estimated from the sum of the integrated time/concentration data (mg/m^3) multiplied by air exchange rate ($1/\text{h}$), and volume (m^3), is 100 to 130 mg. This accounts for about 10% of the net decrease of chlorpyrifos observed for the carpets over the same period, suggesting translocation of residues to other surfaces that were not sampled and/or breakdown of the parent compound.

Following the placement of carpet sections in the 53-L test chambers, the airborne chlorpyrifos concentrations increased to their highest levels by 6 h, followed by a gradual decline to $\sim 1 \text{ }\mu\text{g}/\text{m}^3$ by 492 h. Carpet loads for test chamber samples were estimated at 24 ± 6 and $34 \pm 12 \text{ mg}/\text{m}^2$ based on the average of the baseboard and midpoint carpet samples from the living room and den, respectively. The total extractable chlorpyrifos concentrations of 5.0 and $2.3 \text{ mg}/\text{m}^2$ were determined from total extracts of the carpets following 492 h in the test chambers. The mass of chlorpyrifos exiting each chamber was determined from the product of the integrated time/concentration ($\mu\text{g}/\text{m}^3$) plot times chamber flow (m^3/h) and elapsed time (h). This provided a calculated mass of 44 and 52 μg for the living room and den carpets, respectively, which accounts for only 2 to 3% of the chlorpyrifos loss from the carpets and suggests significant adsorption by chamber surfaces.

Figure 2. Chlorpyrifos concentrations in small environmental chambers.



CONCLUSIONS

The aqueous-based total release aerosol produced a heterogeneous distribution of chlorpyrifos in the treated rooms. The data indicate that most of the particles released from the canister deposit on floor surfaces within 2 hours of release, consistent with settling rates for particles $> 20 \mu\text{m}^7$. However, the findings demonstrate that the application devices also produced an area of undefined size with chlorpyrifos loadings over 2 times higher than the rest of the room. This factor may be important when considering potential human exposures to deposits of semivolatile pesticides following this type of treatment. Findings show that most chlorpyrifos deposited on carpeting in the treated rooms while little intruded into the non-target master bedroom area. Furthermore, it appears that closing doors and turning off the heating and air-conditioning system prior to the aerosol release may have reduced the movement of chlorpyrifos from the treated rooms to other rooms immediately following the application. Airborne concentrations of a semivolatile insecticide may increase for several hours after application due to reemission from treated surfaces. Clearly, and consistent with earlier observations of Leidy et al.⁸, chlorpyrifos, a semivolatile insecticide, volatilizes at measurable levels following indoor applications. The chamber studies show that chlorpyrifos continues to volatilize from the treated carpeting 3 weeks after the application. The apparent delayed increase of airborne concentrations by 24 hours in the master bedroom suggests a distribution pathway associated with passive diffusive processes or by active transport (principally the air-conditioning system). The balance of the total mass of chlorpyrifos delivered into the test house remains incomplete. A 50% decrease of chlorpyrifos from the treated carpet by 2 weeks post-application cannot be accounted for by the chlorpyrifos vapors collected or through whole-house air exchanges. Speculatively, the unaccounted for mass moved as a vapor and sorbed to household substrates (walls, ceilings, etc.) that were not measured in this study. This pilot study did not characterize adsorption/desorption to environmental surfaces, or decay rates of chlorpyrifos in indoor environmental conditions. There was no net increase observed for the total extractable chlorpyrifos in the carpet of the untreated bedroom. Thus, the carpeting in the untreated room does not appear to be a strong sink for vapor-phase chlorpyrifos over the 2-week sampling period.

Acknowledgements

Sampling at the test house and all analytical tasks were conducted by Research Triangle Institute under EPA cooperative agreement CR817083.

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Key Words

Pesticide
Chlorpyrifos
Carpet
House dust
Distribution
Reemission

NRMRL-RTP-P-533		TECHNICAL REPORT DATA <i>(Please read Instructions on the reverse before completing.)</i>	
1. REPORT NO. EPA/600/A-00/-60	2.	3. RE	
4. TITLE AND SUBTITLE The Distribution of Chlorpyrifos in Air, Carpeting, And Dust and its Reemission from Carpeting Following the Use of Total Release Aerosols in an Indoor Air Quality Test House		5. REPORT DATE	
		6. PERFORMING ORGANIZATION CODE	
7. AUTHOR(S) M. Mason (NRMRL), L. Sheldon (NERL), Z. Guo (NRMRL), and D. Stout (NERL)		8. PERFORMING ORGANIZATION REPORT NO.	
9. PERFORMING ORGANIZATION NAME AND ADDRESS U. S. EPA National Exposure Research Laboratory Human Exposure and Analysis Branch Research Triangle Park, North Carolina 27711		10. PROGRAM ELEMENT NO.	
		11. CONTRACT/GRANT NO. CR817083 (Research Triangle Institute)	
12. SPONSORING AGENCY NAME AND ADDRESS EPA, Office of Research and Development Air Pollution Prevention and Control Division Research Triangle Park, NC 27711		13. TYPE OF REPORT AND PERIOD COVERED Published paper; 10/93-6/94	
		14. SPONSORING AGENCY CODE EPA/600/13	
15. SUPPLEMENTARY NOTES APPCD project officer is Mark A. Mason, Mail Drop 54, 919/541-4835. For presentation at Engineering Solutions to IAQ Problems, Raleigh, NC, 7/17-19/00.			
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17. KEY WORDS AND DOCUMENT ANALYSIS			
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Pollution Carpets Dust Aerosols Insecticides Ventilation	Pollution Control Stationary Sources Chlorpyrifos Indoor Air	13B 11E 11G 07D 06F 13A	
18. DISTRIBUTION STATEMENT Release to Public	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES	
	20. SECURITY CLASS (This page) Unclassified	22. PRICE	