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Abamectin in the Ambient Air, on Surfaces, and in Food of Dining Facilities Treated for Cockroaches

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Abamectin (PT 310 Avert™, Whitmire Research Laboratories, Saint Louis MO) was registered in the United States for cockroach control in 1990 (Anon 1990). The chemical represents a new class of insecticides, the avermectins, of which avermectin B_{1a} has good insecticidal activity, being a toxicant and reproductive inhibitor (Putter et al 1981 and Glancy et al 1982). Cochran (1985) reported that newly emerged adult female German cockroaches, Blattella germanica (L.) (Blattodea: Blattellidae), exhibited high mortality at 10 d after feeding on avermectin B_{1a} food bait at concentrations of 6.5 ppm and that survivors failed to reproduce. Abamectin baits gave good control of German cockroach infestations in houses during 8 and 12 wk test periods. (Wright and Dupree 1988, 1990). Since no data have been published on the presence of abamectin in the ambient air, on surfaces or in exposed food in structures following their treatment with abamectin for cockroach control the following study was done.

MATERIALS AND METHODS

Food-preparation, serving and dining areas of three similar-size dining facilities at Ft. Bragg NC were used in the test. These buildings were selected because they operated on a seasonal basis and were closed during the test period (September through December 1989). All food-storage and holding areas, except for one cooler, contained no food. Doors and windows remained closed during the test. Abamectin, supplied as a ready-to-use 0.05% dust type bait and contained in a flexible plastic tube with an opening through an extruded tip, was applied into cracks and crevices in the dining facilities. The amount applied per mess hall was recorded. Temperature and relative humidity were taken at each monitoring time. There was no heat or air-conditioning in the facilities during the test. The ambient air, various surfaces in each dining facility and representative food samples were monitored for abamectin. Sample preparation and analyses were conducted by one of the authors (RBL) at the Piedmont Analytical Corporation, Fuquay-Varina NC using a slightly modified method reported in Anon (1987).

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Bryon Model 90 air samplers (Bryon Instruments, Inc., Raleigh, NC) with Orbo 42 tubes (SUPLECO, Inc., Bellefonte, PA) were used to sample the ambient air of the dining areas for abamectin at the following times: preapplication, immediately after application and postapplication 1, 3, 7, 30, 60, and 90 d. Air samplers were calibrated to give a constant flow rate of 2 L/min, and each sample was collected for 2 hr (240 L of ambient air).

Front and rear portions of the Orbo 42 tube were separated and placed in separate silylated (Sylon-CT, Supleco, Inc.) 10- mL tubes containing 10 mL of CH_3CN. Tubes were capped and vortexed at 15 min intervals for 2 hr. Following overnight refrigeration the tubes were allowed to warm to room temperature and the solvent removed from the adsorbent with a Pasteur pipet and transferred to a 12- mL silylated tube (tube filled with Cylon CT, allowed to sit 30 min, then rinsed with successive 10-ml portions of toluene, acetone and methanol and allowed to air dry. This prevented the adsorption of abamectin to the tube.) An additional 2 mL were added to the adsorbent, vortexed and transferred to the appropriate 12- mL tube and the solvent blown to dryness under a stream of dry nitrogen at 50°C.

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, precleaned 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 were used for taking each sample. The swab was returned to the glass bottle, the bottle sealed and labeled. Samples were taken from the following surfaces and locations: horizontal surface of a stainless steel countertop in the food-preparation area, vertical surface of a stainless steel food-serving cabinet on the serving line and the upper (horizontal) surface of a formica table in the dining area following the same schedule as that for the air samples.

The cotton ball and 2-propanol were transferred to a Soxhlet thimble, placed in a Soxhlet apparatus and extracted for 2.0 hr with 200 mL of CH_3CN at 6 turnovers per hr. After cooling, the solvent was reduced to 2 to 3 mL under reduced pressure at 60°C and transferred quantitatively to a silylated 10- mL tube with CH_3CN. The solvent was blown to dryness with a stream of dry nitrogen at 50°/C.

Lettuce and bread were used as representative foods. Preapplication food samples were taken as follows. Prior to abamectin application one slice of bread and 1/4 head of lettuce were placed on adjacent, separate paper plates on the food-serving counter. Like-size samples were removed from a loaf of bread and head of lettuce, immediately put into plastic bags and placed on

ice for use as unexposed food sample checks. Two hr later the exposed samples were put in separate plastic bags and placed on ice. All samples were homogenized, placed in 950 mL glass jars and frozen (-20°C) until analyzed. Postapplication food samples were taken as follows. Immediately prior to abamectin application three slices of bread and two-1/4 heads of lettuce samples were put on individual paper plates on the food-serving counter in each dining facility. One slice of bread was removed immediately after and at 1 and 3 d postapplication, respectively. Single 1/4-head lettuce samples were removed immediately after and 1 d postapplication. Each sample was placed in a plastic bag, put on ice, and placed in a freezer until analyzed. Other single slice bread and 1/4-head lettuce samples were taken from loaves of bread and heads of lettuce at the same times as the exposed samples were secured. These were used as untreated control samples and handled in the same way as the exposed samples. The procedures were replicated in the three dining facilities.

A 10.0 g subsample was tared into a 950- mL blender jar and homogenized two times at medium speed for 15 min with 150 mL of CH_3CN. The extracts were combined in a 500 mL boiling flask and an additional 25 mL of CH_3CN were used to rinse the blender jar and added to the flask. The solvent was reduced to ca. 5 mL and transferred to a 25- mL cylinder where the volume was adjusted to 10 mL with CH_3CN. The solvent was transferred to a 125- mL separatory funnel and the cylinder rinsed with 2-20 mL aliquots of HPLC-grade water and added to the flask. The aqueous phase was extracted three times with 25 mL of hexane, filtered through ca. 5.0 g of acetone-washed NA_2SO_4 (anhyd) into a 500- mL boiling flask. An additional 25 mL of hexane were used to rinse the funnel and the combined extracts were evaporated to 3 to 5 mL under reduced pressure at 40°C, transferred quantitatively to a 10- mL silylated tube, the volume adjusted to 10 mL with hexane and filtered through a 0.45-μm Acrodisc into another 10- mL silylated tube. Samples were blown down to 1 mL with a stream of dry nitrogen at 50°C and 0.5 mL were taken for derivatization as described below.

Samples were blown to dryness under a stream of dry nitrogen at 50°C. Abamectin residues were trifluoroacetylated by adding 0.2 mL of the derivatizing reagent [0.4 mL of 1-methylimidazole added to 3.6 mL of N, N-dimethylformamide to which, after cooling in an ice bath, 0.6 mL of redistilled trifluoroacetic anhydride (BP: 39.0-39.5°C) were added and then vortexed], mixing and placing the stoppered tube in a 40°C water bath for 1.0 hr. Tubes were removed from the bath and 0.1 mL of methanolic ammonium hydroxide (0.2 mL of ammonium hydroxide added to 3.0 mL of methanol and vortexed) was added, mixed thoroughly, and placed in a 40°C water bath for 30 min. After removal from the bath, 4.0 mL of $CHCl_3$ were added to the tubes and the contents placed on a 500 mg Bond-Elut silica cartridge (Analytichem International, Harbor City, CA) which had been prerinsed with 5.0 mL of $CHCL_3$. Cartridges were connected to a Sep-Pak Rack with Tygon tubing, vacuum was applied by aspiration, and the eluate was collected in a silylated tube.

Additional $CHCl_3$ was added to the cartridge to give a final volume of ca. 12 mL. Volume was reduced to ca. 1.0 mL under a stream of dry nitrogen in a water bath at 50°C, 5.0 mL of methanol were added and sample volumes were adjusted with methanol for chromatography.

Samples were analyzed using a Shimadzu Model LC-G HPLC equipped with a Model 535 Fluorescent Detector set as follows: excitation energy, 365 nm; emission, 418 nm; range, X8, medium response and high sensitivity. The solvent system was methanol: water (95:5)(v:v) at a flow rate of 2.0 mL/min. The column was a Waters C_{18} reversed phase (10 x 0.8 cm) (5.0 to 10.0 um diameter packing) contained in a Waters Millipore RCM 8 x 10. A 10 mV recorder operated at 0.25 cm/min was used to record chromatograms.

Data were quantified against a standard curve containing 5 different concentrations of abamectin (varying from 2 to 400 ng) which were derivatized with each set of samples. Seventeen sets of standard curves were run with the samples. In addition, samples were quantitated against analytical standards of the derivatized abamectin standard. Fortified untreated samples were analyzed with each sample set to determine the efficiency of the analytical method. Data were analyzed using an ANOVA procedure and the Waller-Duncan K-ratio t test for comparison of means (SAS User's Guide: Statistics, Version 5 Edition, Cary, NC; SAS Institute, Inc. 1985. 956 p.).

RESULTS AND DISCUSSION

The recovery of residues of abamectin from various matrices is shown in Table 1. There were few differences when overall recovery averages were compared between the sample matrices, although the recovery for lettuce was highest. A number of lettuce samples apparently had some underlying colluting peak, particularly at the low fortification levels, which was diluted out at higher fortification levels, thus reducing recovery values to below 100%.

Standard curves run with each sample set showed excellent linearity in the range of 2.0 to 900 ng. The trifluoroacetylated derivative of abamectin was detectable at 0.02 ng. The analytical method is quite involved, and it was found that the trifluoroacetic anhydride had to be redistilled in order to effect good derivatization.

The amount of abamectin AI applied in the 3 dining facilities was 32, 45, and 37 mg, respectively.

Temperatures and relative humidities in the dining facilities during the test period ranged from 18 to 39 °C and 44 to 80% RH, respectively. No significant differences occurred between the 3 dining facilities during the sampling times. There was a significant ($P<0.01$) decrease in temperature in all dining

facilities during the months following abamectin application (September through December). No significant change occurred in the relative humidity.

Table 1. Recovery of abamectin from various sample matrices[a]

Matrix	No.	Amount added (ng)	Amount recovered (ng)	Avg recovery (%)
Orbo 42[b]	3	3.0	3.0	100
	3	6.0	4.7-5.9	86
	1	9.0	7.0	78
	2	25.0	18-22	79
	2	36.0	35.9	98
	2	50.0	44-47	91
Bread	2	2.0	1.6-1.8	86
	2	4.0	3.1-3.6	83
	1	8.0	7.7	96
	2	10.0	9.0-10.9	100
	1	15.0	14.7	98
	2	30.0	26.7-28.7	92
	2	40.0	32.7-36.7	87
Cotton Ball[c]	3	7.0	6.4-7.3	100
	2	14.0	11.2-14.0	90
	4	28.0	26.1-29.1	98
	1	45.0	40.4	90
	2	130.0	110.8-133.6	94
	2	250.0	206.2-231.7	88
	2	400.0	344.4-360.4	88
Lettuce	2	3.0	2.0-2.3	72
	3	6.0	6.5-71	118
	2	9.0	8.3-9.1	97
	3	18.0	16.5-17.3	93
	2	36.0	34.7-36.0	98

[a] Various amounts of abamectin added to untreated sample matrices and extracted with actual sample sets. Samples were allowed to sit for 2 hr to allow solvent evaporation, extracted and derivatized. Using the same pipet, an equivalent amount of abamectin was added to a 10 mL sylated tube, and refrigerated until those samples and fortified sample matrices were analyzed. Recovery values were based upon the value of these "spikes".
[b] Orbo 42 tubes used to collect air samples.
[c] Cotton balls used to collect surface samples.

Before abamectin application no cockroaches were detected in the dining facilities by visual inspection. However, at 3 d post-application a few dead German cockroaches, Blattella germanica (L.) (BLATTODEA: Blattellidae), were observed on the floor in one facility. At 7 d there were a few dead German cockroaches on the floor in a second facility and in the third facility several dead American, Periplaneta americana (L.) (BLATTODEA: Blattidae), and smokybrown, P. fuliginosa (Serville) (BLATTODEA: Blattidae),

cockroaches were noted. These cockroaches indicated a light infestation existed in the dining facilities prior to abamectin application.

Abamectin was not detected in the ambient air of mess halls during preapplication sampling (Table 2). Immediately after application and at 1 and 3 d postapplication there were significant ($P<0.01$) levels of abamectin in the ambient air of the dining facilities. Total amounts detected decreased significantly at each of the 3 sampling times. Monitoring at 7, 30, 60 and 90 d showed quantities no different from preapplication levels.

Table 2. Abamectin (ng/m^3) detected in the air of vacant dining facilities at Fort Bragg NC following its application for cockroach control

Preapplication	Postapplication (d)						
	0	1	3	7	30	60	90
<0.5	901±85	308±79	100±4	2±1	<0.5	<0.5	1.0±0

[a] Mean with standard deviation. Lowest detection level was 0.5 ng/m^3. Average of three replications.

Residues were significantly ($P<0.01$) greater on the horizontal stainless steel surfaces in the food-preparation area immediately following abamectin application (Table 3). One d after application and thereafter levels decreased to non-detectable levels on this surface type. Levels of abamectin on vertical stainless steel surfaces in the food-serving areas and the horizontal table tops in the dining areas were lower than horizontal surfaces, ranging from a mean of 3 ng to non-detectable on the 100 cm^2 surface area.

Table 3. Abamectin ($ng/100\ cm^2$) detected on non-target surfaces after its application in vacant dining facilities at Fort Bragg NC for cockroach control.[a]

Preapplication	Postapplication (d)						
	0	1	3	7	30	60	90
Horizontal stainless steel surface in food preparation area							
<0.02	42±32	3±2	3±1	3±1	<0.02	<0.02	<0.02
Vertical stainless steel surface in food serving area							
<0.02	<0.02	1±0	<0.02	<0.02	<0.02	<0.02	<0.02
Horizontal formica table top in dining area							
<0.02	3±1	1±0	<0.02	<0.02	<0.02	<0.02	<0.02

[a] Mean with standard deviation. Lowest detection level was 0.02 $ng/100cm^2$. Average of three replications.

Uncovered bread and lettuce samples, placed in the facilities and prior to abamectin application, and sampled immediately and 1 d after application for both foods and at 1 and 3 d after application for the bread contained significant abamectin (Table 4). There was more abamectin on both food samples analyzed immediately after application than at later periods. No differences occurred between levels for the bread and lettuce on the d of treatment and at d 1 after application, respectively.

Table 4. Abamectin (ppb) detected in uncovered and wrapped foods after being exposed during application to vacant Fort Bragg NC dining facilities for cockroach control.

Preapplication	Postapplication (d)		
	0	1	3[b]
		Uncovered Bread	
<0.2	8.4±4.2	3.6±0.6	3.3±0.5
		Uncovered Lettuce	
<0.2	6.9±1.2	2.6±1.4	

[a]Mean with standard deviation. Lowest detection level was 0.2 ppb. Average of three replications.
[b]No lettuce sampled at 3 d postapplication.

Quantities of abamectin present in the ambient air, on surfaces and in food in mess halls following abamectin application for cockroach control were very low. The data obtained from this study indicate that residues of abamectin on surfaces rapidly dissipate to non-detectable levels by 7 d yet small quantities were detected in air 90 d postapplication.

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

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