

with its oxidation product [i.e., PBacid (5)] represented the major ^{14}C residues (up to 49% of the applied dose). As with fenvalerate (Mikami et al., 1980), PPAnitrile (9) was a significant aqueous photoproduct (up to an 8% yield).

Conclusions. When fluvalinate is exposed to light in organic solvent, on glass, on soil, or in aqueous solution, it is readily degraded. In natural sunlight, the half-life of fluvalinate is typically 1 day (or less). The alcohol portion of fluvalinate yields most of the same photoproducts (Figure 1) as similar pyrethroids such as cypermethrin, deltamethrin, and fenvalerate although free radical derived photoproducts (e.g., CO_2 elimination adducts like 12) are considerably less abundant from fluvalinate. The acid portion of fluvalinate is converted by sunlight mostly into haloaniline 3 and anilino acid 2 although two previously uncharacterized degradation products from 1 were identified also (formanilide 8 and oxamic acid 10).

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Persistence of Aminocarb in Balsam Fir Foliage, Forest Litter, and Soil after Aerial Application of Three Formulations

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One water-based (180 FE) and two oil-based (180 FO and 180 D) aminocarb formulations were applied twice by a fixed-wing aircraft, each at 70 g of a.i./ha, to a coniferous forest near Bathurst, New Brunswick. The highest concentration of aminocarb in foliage was 2.76 ppm (fresh weight), detected 1 h after the second application of formulation 180 D. The residues decreased rapidly within 1 or 2 days after spray application but persisted at lower concentrations thereafter. Twenty-one days after the second spray application, the concentrations of aminocarb in foliage ranged from 0.14 to 0.64 ppm (fresh weight). Only low levels of residue were detected in forest litter and soil. The highest concentrations in litter and in soil were 0.216 ppm (fresh weight) and 0.044 ppm (fresh weight), respectively, detected 2 h after the second application of formulation 180 D. Residues in litter were higher and persisted longer than in soil. There was 0.013 ppm (fresh weight) present in litter 21 days after the second application of formulation 180 D but none were detected in soil after 8 days.

Aminocarb [4-(dimethylamino)-*m*-tolyl *N*-methylcarbamate, Matacil), a broad-spectrum insecticide, was first field tested against spruce budworm [*Choristoneura fumiferana* (Clem.)] in 1970 in New Brunswick and was first used in operational spray programs in 1975. Since then, a total of about one million kg of aminocarb has been applied to the forests of Newfoundland, New Brunswick, Quebec, and Ontario (Nigam, 1980). The formulation

Matacil 180 D, which has been used extensively in spray operations, contains by weight 19.5% aminocarb, 30.0% Shell insecticide diluent 585, and 50.5% nonylphenol (National Research Council of Canada, 1982). It has been shown that Matacil 180 D is toxic to juvenile Atlantic salmon, *Salmo salar*, and some species of marine and freshwater invertebrates. The formulation is even more toxic than the pure active ingredient because nonylphenol itself is extremely toxic to these animals (McLeese et al., 1980). In order to minimize the toxicity of the aminocarb formulation, Chemagro Ltd., marketers of Matacil in Canada, has developed a flowable suspension, Matacil 180 F, containing air-milled particles of aminocarb (2-3- μm diameter) suspended in oil, which can be applied either as a water-based (180 FE) or an oil-based (180 FD) for-

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mulation for aerial spray. In 1981 the Forest Pest Management Institute conducted a field trial to evaluate the efficacy and the environmental impact of conventional Matacil 180 D and the new Matacil 180 F. The findings on the distribution and persistence of aminocarb on various compartments of the forest environment are presented in this paper.

MATERIALS AND METHODS

Experimental Plots. Four 50-ha (1000 m × 500 m) plots in a mixed, mature coniferous forest, about 80 km southwest of Bathurst, New Brunswick, Canada, were selected for the field trial. They were approximately 2 km apart; three served as treatment plots, and one served as the control.

Most of the trees in the plots showed evidence of moderate to severe defoliation from past spruce budworm outbreaks. Samples of foliage, forest litter, and soil were collected before and at various intervals after spray application for the analysis of aminocarb.

Spray Application. Three aminocarb formulations were evaluated: (1) 180 FE containing by volume of 25.9% Matacil 180 F, 1.3% Atlox 3409F (an emulsifier supplied by Atlas Chemical Industries), 0.5% Rhodamine B (a tracer dye supplied by Allied Chemicals), and 72.3% water; (2) 180 FO containing by volume of 25.9% Matacil 180 F, 2.0% Automate B Red (a tracer dye supplied by Morton Williams, Ltd.), and 72.1% Shell insecticide diluent 585 (supplied by Shell Canada Ltd.); (3) 180 D containing 25.9% Matacil 180 F, 2.0% Automate B Red, and 72.1% Sunspray 6N oil (supplied by Sun Oil Co.). The three formulations were applied twice at 70 g of a.i./ha to the experimental plots separately by a Cessna 188 aircraft equipped with four Micronair AU 3000 atomizers. The flight speed was 160 km/h at a height of 25–30 m depending upon the terrain. The emission rate of the four atomizers was calibrated to deliver 24.5 L/min, which provided a total emission of 1.5 L/ha. The swath width was about 60 m.

Sampling and Analysis. Twelve balsam fir trees, *Abies balsamea* (L.) Mill, about 14 m in height and 16.5 cm in DBH (diameter at breast height) were randomly selected in each plot for foliage sampling. They were tagged with plastic tape for identification. Ground vegetation surrounding each sampling tree was cleared up to a radius of 5 m to enhance exposure to the spray cloud. At each sampling, one branch, approximately 25 cm long, was taken from the midcrown of each tree. The cut-up branches were kept in plastic bags, stored in plastic-foam coolers with dry ice, and transported to the field laboratory, where they were pulverized in a Hobart meat grinder and then stored at -20 °C until analyzed.

Two fully exposed areas, about 5 m × 2.5 m, were selected for sampling forest litter and soil, respectively, in each plot. All fallen branches and small rocks were cleared from these areas. In the soil sampling area, moss and organic detritus were also removed to expose the underlying soil layer. Litter was collected from an area of 240 cm² at a depth of 1 cm by driving a metal frame (15.5 cm × 15.5 cm) into the ground and lifting the contents with a spatula. Soil was collected as 2.5-cm diameter cores, and 25 cores were randomly taken from the top 1-cm layer. All samples were wrapped in aluminum foil and processed as described previously for foliage.

Residues of aminocarb in foliage, forest litter, and soil were determined according to the GLC method of Szeto and Sundaram (1980).

Spray Deposit Assessment. Two glass slides (7.5 cm × 5.0 cm) and two Kromekote cards (10 cm × 10 cm)

Table I. Weather Conditions during Aerial Applications of Three Aminocarb Formulations near Bathurst, New Brunswick, in 1981

formulation	180 FE		180 FO		180 D	
	1st spray	2nd spray	1st spray	2nd spray	1st spray	2nd spray
date of application	June 12	June 18	June 12	June 18	June 13	June 18
time of application, h	1945	0622	2100	0720	2035	2023
wind speed, km/h	0.25	1.0	0	5	0	1.5
wind direction	E	W		W		W
temperature, °C	13.0	10.3	10.0	13.8	16.5	22.3
relative humidity, %	80	100	96	73	73	58
precipitation	nil	nil	nil	nil	nil	nil
cloud cover	1/10	0/10	0/10	0/10	0/10	0/10

Table II. Spray Deposit from Aerial Application of Various Aminocarb Formulations at 70 g of a.i./ha near Bathurst, New Brunswick, in 1981

formulation	application	drops/cm ²	range of droplet size, μm (diameter)	deposite rate, g of a.i./ha
180 Fe	1	6.0 ± 6.0	7–73	2.0 (2.5) ^a
	2	0.5 ± 0.4	7–73	0.3 (0.9)
180 FO	1	13.0 ± 6.0	16–85	6.0 (7.5)
	2	3.0 ± 1.0	4–85	0.9 (1.0)
180 D	1	16.0 ± 6.0	4–105	11.7 (10.0)
	2	13.0 ± 6.0	4–105	13.5 (12.5)

^a Values without parentheses were determined by gas-liquid chromatography, and values with parentheses were determined by spectrophotometry of the tracer dye.

mounted on a collection unit were used for spray deposit assessment (Randall, 1980). One-half hour prior to spray application, four units were placed in each corner of the litter and soil sampling station; three units were placed around each sampling tree, one at the upwind side under the tree, one at the downwind side under the tree, and one in the open area of the upwind side of the tree. They were collected 1 h after spray application and transported immediately to the field laboratory where the deposits on the glass slides were removed by washing them 3 times with 5 mL of pesticide-grade ethyl acetate. The washings were stored in amber glass bottles at 0 °C until analyzed by spectrophotometry and gas-liquid chromatography. The Kromekote cards were examined under magnification to determine the droplet density and the droplet spectrum of each application.

RESULTS AND DISCUSSION

Spray Deposition. Weather conditions at time of spraying are indicated in Table I. Spray deposit data from aerial application of various aminocarb formulations are presented in Table II. When compared with the two oil-based formulations, 180 FO and 180 D, the water-based formulation, 180 FE, gave a narrower spectrum (7–73 μm in diameter) and a lower droplet density (0.5 ± 0.4 droplet/cm²) at ground level, probably due to its higher volatility. Exceptionally low ground deposit was obtained in the second application of 180 FE (0.5 ± 0.4 droplet/cm²) and of 180 FO (3.0 ± 1.0 droplets/cm²). There was no apparent reason to which the low depositions observed in these two applications could be attributed. The light wind and the smaller droplet size suggest drift may have occurred in these two sprays and therefore resulted in lower deposit rates. Higher deposit rates and larger droplets were obtained with 180 D than with 180 FO probably due to the lower volatility of Sunspray 6N than Shell insecticide diluent 585. We determined the evaporation of Sunspray 6N and Shell insecticide diluent 585 at 20 °C

Table III. Aminocarb Residues in Balsam Fir Foliage Treated Twice with Various Formulations at 70 g of a.i./ha near Bathurst, New Brunswick, in 1981

time after spraying	aminocarb residues, ppm (fresh wt) ^a		
	180 FE	180 FO	180 D
First Application			
0.5 h	2.41	2.27	0.77
1.0 h	1.86	1.98	1.30
4.0 h	1.54	1.67	1.15
12.0 h	1.37	1.45	1.01
15.0 h	1.12	1.36	0.96
1 day	0.88	1.31	0.87
2 days	0.48	1.14	0.72
3 days	0.35	0.79	0.68
4 days	0.27	0.57	0.61
5 days	0.20	0.38	0.52
Second Application			
0.5 h	0.75	0.85	2.35
1.0 h	0.96	1.79	2.76
4.0 h	0.85	1.44	2.69
12.0 h	0.70	1.16	2.04
15.0 h	0.66	1.12	1.92
1 day	0.68	1.06	1.68
2 days	0.63	1.02	1.59
3 days	0.58	0.98	1.43
4 days	0.55	0.88	1.36
5 days	0.51	0.69	1.23
6 days	0.45	0.58	1.19
8 days	0.35	0.51	0.97
10 days	0.29	0.48	0.92
12 days	0.24	0.44	0.84
21 days	0.14	0.32	0.64

^aThe moisture content of balsam fir foliage ranged from 58 to 62%.

in the laboratory. No significant evaporation occurred with Sunspray 6N, whereas 90% by volume of Shell insecticide diluent 585 was lost through evaporation.

Residues in Balsam Fir Foliage. Aminocarb residues in foliage at various intervals after spray applications are presented in Table III. The spray deposit levels determined at ground level did not correlate with the aminocarb residues found in foliage shortly after spraying (Tables II and III). For example, 1.79 ppm of aminocarb was found in foliage 1 h after the second application of 180 FO whereas the ground deposit level was 0.9 g of a.i./ha as determined by gas-liquid chromatography. By comparison, the insecticide concentration was only 1.30 ppm in foliage 1 h after the first application of 180 D but the deposit level at ground level was 11.7 g of a.i./ha. It has been suggested (Joyce and Beaumont, 1978) that high levels of deposit at ground level could mean low rates of deposit on target trees because of the high rate of sedimentation of larger droplets due to gravitational pull. However, our data did not support this suggestion. The highest concentration of aminocarb in foliage was at 2.76 ppm, detected 1 h after the second application of 180 D when the deposit rate at ground level was 13.5 g of a.i./ha, the highest value obtained among all the spray applications of the present study. It is apparent that there is no simple relationship between spray deposit on the target trees and spray deposit on the ground since various physical and environmental factors such as morphology of the forest canopy, vertical temperature gradient, channelization of wind, micrometeorological conditions in and beneath the canopy, etc. would affect movement of the spray cloud (Yates and Akesson, 1973; Cramer and Boyle, 1976).

The disappearance of aminocarb from foliage appeared to be biphasic. The dissipation was relatively rapid within 1-2 days after spray application; thereafter, the residues

Table IV. Aminocarb Residues in Forest Litter and Soil Treated Twice with Various Formulations at 70 g of a.i./ha near Bathurst, New Brunswick, in 1981

time after spraying	aminocarb residues, ppm (fresh wt) ^a					
	180 FE		180 FO		180 D	
	L	S	L	S	L	S
First Application						
0.25 h	0.018	0.004	0.038	0.008	0.132	0.024
0.50 h	0.023	0.008	0.054	0.014	0.159	0.032
1.0 h	0.022	0.006	0.086	0.018	0.178	0.050
2.0 h		0.005	0.080	0.016	0.188	0.051
3.0 h	0.023	0.006	0.077	0.010	0.160	0.046
5.0 h	0.018	trace ^b	0.074	0.011	0.146	0.037
12.0 h	0.015	ND ^c	0.072	0.007	0.098	0.024
1 day	0.018	ND	0.068	0.004	0.085	0.011
2 days	0.015		0.064	ND	0.078	0.007
3 days	0.017	ND	0.052	ND	0.074	0.004
4 days	0.014		0.045		0.069	trace
5 days	0.015	ND	0.034	ND	0.061	trace
Second Application						
0.25 h	0.016	trace	0.042	0.004	0.126	0.008
0.50 h	0.018	0.003	0.044	0.005	0.144	0.016
1.0 h	0.015	trace	0.046	0.010	0.206	0.034
2.0 h		ND	0.044	0.005	0.216	0.044
3.0 h	0.016	ND	0.049	0.004	0.215	0.038
5.0 h	0.016	ND	0.036	trace	0.196	0.030
12.0 h	0.014		0.040	ND	0.180	0.022
1 day	0.012		0.026	ND	0.126	0.017
2 days	0.012		0.019	ND	0.110	0.011
3 days	0.010	ND	0.017	ND	0.098	0.006
4 days	0.007		0.017		0.081	0.004
5 days	0.006	ND	0.016	ND	0.074	trace
6 days	0.007		0.015		0.061	trace
8 days	0.005		0.014		0.049	ND
10 days	0.006		0.013		0.035	ND
12 days	trace		0.010		0.029	ND
21 days	ND		ND		0.013	

^aThe moisture content ranged from 12 to 34% in forest litter (L) and from 36 to 43% in forest soil (S). ^bTrace = less than 0.005 ppm (fresh weight). ^cND = not detectable at the limit of 0.003 ppm (fresh weight).

persisted at lower levels (<1.0 ppm fresh weight) for 21 days (Table III). The exception was in the second application of the water-based 180 FE; the highest postspray 1 h foliar concentration detected was 0.96 ppm, which decreased to 0.51 ppm in 5 days and 0.14 ppm in 21 days. The dissipation of aminocarb by volatilization may have been the primary cause of the rapid decrease in aminocarb residues in foliage within the first few days after spray application. As the residues were gradually absorbed by the lipophilic terpenoids of the foliage, they degraded relatively slowly. Little is known about the degradation of aminocarb by plant enzymes and living organisms. More research in these areas is necessary to elucidate the degradation of the chemical in the environment. Depending upon the initial concentrations of aminocarb in foliage following first application, the residue levels decreased by 50% in approximately 1-5 days. The rates of aminocarb residue disappearance observed in our study are well within the range found earlier for white spruce [*Picea glauca* (Moench) Voss] foliage (Sundaram et al., 1976; Sundaram and Hopewell, 1977).

Residues in Forest Litter and Soil. Concentrations of aminocarb in forest litter and soil at various times after spray application are presented in Table IV. Residues found in litter and soil within the first few hours after spray application generally correlated with the spray deposit at the ground level (Table II). The residue levels in litter were generally low; the highest was 0.216 ppm (fresh weight), detected 2 h after the second application of 180 D. The residues persisted in litter for 12-21 days (Table

IV). Concentrations of aminocarb in soil, as observed previously (Sundaram et al., 1976), were much lower than those in the corresponding litter samples. The highest was 0.051 ppm (wet weight), detected in soil samples collected 2 h after the first application of 180 D (Table IV). The residues persisted in soil for about 5 days. Aminocarb was not detected in any of the soil samples collected 8 days after the second application. The difference in persistence of aminocarb in litter and in soil could be partly explained by the difference in acidity of the two matrices. The litter, which was composed of fallen needles, twigs, and organic detritus, was definitely acidic (pH 5.4), whereas the soil (sandy loam) had mild acidity with a pH of 6.3. It has been shown that the hydrolysis of aminocarb is pH dependent and the hydrolysis rate is much higher at elevated pH. According to the studies of Murphy et al. (1975) and Tessier et al. (1978), the estimated half-lives for aminocarb in aqueous buffers at 20 °C were 28.5 days at pH 7.0 and 90.2 days at pH 5.0. Since the pH of the soil was higher than that of the litter, hydrolytic degradation of aminocarb in soil may have been at a faster rate than that in litter.

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Bound Residues of Deltamethrin in Bean Plants

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Deltamethrin [(S)- α -cyano-3-phenoxybenzyl (1R,3R)-*cis*-2,2-dimethyl-3-(2,2-dibromovinyl)cyclopropanecarboxylate] labeled with ^{14}C at the methyl or benzylic position formed unextractable (bound) ^{14}C residues in bean plant shoots. The amount of bound ^{14}C residues formed was higher in the benzylic label deltamethrin treated plants. Deltamethrin and a number of metabolites, present in the plant as bound ^{14}C residues, were released and identified with the high temperature distillation technique followed by thin-layer and gas chromatographic analysis, whereas a major portion of the unextractable products remaining was of unknown composition. A small proportion of the bound ^{14}C residues from plant tissue was released after incubation with enzymes.

Deltamethrin [(S)- α -cyano-3-phenoxybenzyl (1R,3R)-*cis*-2,2-dimethyl-3-(2,2-dibromovinyl)-2,2-cyclopropanecarboxylate] is a new synthetic pyrethroid insecticide that is active against numerous species of insects when applied on field crops. Recent interest in deltamethrin metabolism in plants has centered primarily on the fate of this insecticide in cotton and bean plants (Ruzo and Casida, 1979; Cole et al., 1982). A large number of extractable metabolites have been successfully isolated and characterized after application of deltamethrin to cotton and bean leaves.

However, a portion of the insecticide or its products were unextractable (bound), and greater quantities of these bound residues were present in outdoor samples (Ruzo and Casida, 1979). These bound and usually chemically unidentified residues may, however, be important. For example, they might become released on digestion of the contaminated food or their accumulation may be of significance to crops growing in the treated soils. The present study reports the formation of bound residues in bean plants when treated with radiolabeled deltamethrin and attempts to identify some of these residues. Crop material containing bound residues from the treated plants was also subjected to enzyme hydrolysis to determine the release of bound radioactivity.

MATERIALS AND METHODS

Chemicals. Deltamethrin (^{14}C labeled and unlabeled) was a gift from Roussel-Uclaf-Procida through its subsidiary Hoescht of Canada, Ltd. The position of labeling,

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