DISTRIBUTION AND PERSISTENCE OF AMINOCARB IN TERRESTRIAL COMPONENTS OF THE FOREST ENVIRONMENT AFTER SEMI-OPERATIONAL APPLICATION OF TWO MIXTURES OF MATACIL® 180F

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ABSTRACT

Two spray mixtures, one oil-based (180FO) and one water-based (180FE), of Matacil® 180 flowable (Matacil® 180F) were applied twice by fixed-wing aircraft at a dosage rate of 70 g AI/ha to selected plots in a mixed mature coniferous forest near Fredericton, New Brunswick. Spray distribution and deposition varied considerably between two applications of the same spray mixture and between the two mixtures. The average amount of aminocarb deposited and the percent deposition at the forest floor were 12.4 g/ha and 17.7%, respectively. The highest concentration of aminocarb in balsam fir *[Abies balsamea* (L.) Mill] foliage was 1220 ng/g (fresh wt.), detected 1 h after the second application of 180FE. With both spray mixtures the residues were lost in an exponential decay pattern with time, and the half-lives ranged from 78 to 143 h. The post spray samples collected 12 days after the second application contained only about 20% (as sampled) of the initial concentration of the material, showing that the chemical is short lived. In forest litter and soil environments, aminocarb was present in amounts as low as the detection limit (10 ng/g), indicating that at the dosage rate used, aminocarb should not pose any undue hazard to the soil microorganisms.

RÉSUMÉ

Deux préparations de Matacil® 180F, une à base d'huile (180FO) et une à base aqueuse (180FE), ont été épandues par avion à deux reprises, à la dose de 70 g/ha d'ingrédient actif, dans des parcelles choisies dans une forêt mixte de conifères à maturité, près de Frédéricton au Nouveau-Brunswick. La dispersion et le dépôt des produits ont varié considérablement entre les deux arrosages de la même préparation et entre les deux préparations. La quantité moyenne d'aminocarbe s'étant déposé et le pourcentage de dépôt à la surface du sol se sont élevés respectivement à 12,4 g/ha et 17,7%. La plus forte concentration d'aminocarbe mesurée sur le feuillage du sapin baumier *(Abies balsamea* [L.] Mill.) a été de 1 220 ng/g (masse à l'etat frais) et a été obtenue 1 h après le deuxième arrosage de la préparation 180FE. Avec chacune des deux préparations, la disparition des résidus en fonction du temps a suivi une courbe d'affaiblissement exponentielle et les périodes (demi-vie) s'étendaient de 78 à 143 h. Les échantillons recueillis 12 jours après le deuxième arrosage renfermaient seulement 20% de la concentration maximale initiale du produit, indiquant que celui-ci avait une courte durée de vie. Dans la litière forestière et le sol, les concentrations étaient présentes en quantités aussi faibles que le taux de détection (10 ng/g), indiquant qu'à la dose utilisée, l'aminocarbe ne devrait représenter aucun risque indu pour les micro-organismes du sol.

INTRODUCTION

Aminocarb (Trade name: Matacil®) [4-dimethylamino-m-tolyl N-methylcarbamate] has been used for spruce budworm *[Choristoneura fumiferana* (Clem.)] control in eastern Canada, first experimentally and then operationally, since 1970, and to date approximately 1.0 x 10^6 kg of the material have been sprayed over 10 x 10^6 ha of forest (Sundaram and Sundaram 1981). The insecticide is usually applied by aircraft, at 2 x 0.07 kg AI/ha, as a homogeneous oil formulation (Matacil® 180D) containing (wt. %) aminocarb 19.5, Shell insecticide diluent (I.D.) 585 (Shell Canada Ltd., Toronto, Ont., Canada) 30.0, and nonylphenol (Rohm and Haas Canada Ltd., West Hill, Ont., Canada) 50.5. Since nonylphenol, the major adjuvant in the formulation, was found to be toxic to juvenile Atlantic salmon, *[Salmo salar]* (McLeese et al. 1980), Chemagro Chemical Company, Toronto (the marketers of Matacil), introduced a flowable suspension, Matacil® 180F. This suspension, containing air-milled particles of aminocarb (2-3 µm diameter) suspended in oil, can be used either as a water-based emulsion (Matacil® 180FE) containing Atlox® 3409F (a mixture of polyoxyethylene ethers and dodecylbenzene sulphonate) (Atlas Chemical Industries, Brantford, Ont.) emulsifier or as an oil-based (Matacil® 180FO) formulation with Shell I.D. 585 (a petroleum distillate) for aerial application.

Before a chemical can be used operationally for forest insect control in Canada, its environmental fate, distribution and persistence must be evaluated according to the set protocols established by the registration agency, Agriculture Canada. The present study is intended to address these problems, and to provide the necessary data base on the new formulation, Matacil 180F, for use in the registration process.

During the 1982 field season, the Forest Pest Management Institute (FPMI) conducted a field trial to study the efficacy, environmental impact and environmental chemistry of Matacil® 180F formulations containing ID-585 (180FO) and Atlox® 3409F (180FE). The distribution, persistence and fate of aminocarb in some terrestrial components of the forest environment are presented in this report.

MATERIALS AND METHODS

Experimental plots

Three experimental plots were selected within a mixed mature coniferous forest in the province of New Brunswick (N.B.) for this experimental study. Two 250 ha plots designated as P82 (46°08'N and 66°49'W) and P86 (45°42'N and 66°49'W) were located approximately 30 km northeast and southwest, respectively of Fredericton; the check plot was situated about 20 km west of P82. Most of the trees in the plots showed evidence of moderate to severe defoliation caused by 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

Two aminocarb spray mixes were evaluated. The composition of each tank mix (vol. %), dosage, application rate, and the formulation each plot received are shown in Tables 1 to 3. Aerial applications were conducted by Forest Protection Limited (FPL). Plots P82 and P86 were each sprayed twice at 70 g AI/ha during an interval of about 5 days using TBM Avenger aircraft equipped with twenty-four 1010 Flatfan Teejet® nozzles. Aircraft type, spray parameters, emission rates, and meteorological conditions that existed during the spray operations are given in Tables 3 and 4.

Sampling of substrates

i) Balsam fir foliage: Seven dominant balsam fir *[Abies balsamea* (L.) Mill] trees *(ca.* 15 m in height and 15 cm DBH) with fully developed crowns, ample growing space and exposure to sunlight were selected randomly in both the spray and check blocks prior to spray application and marked with

Table 1. Pesticide formulation, diluent oil and surfactant used in the study

Name	Abbreviation used	Source
Matacil® 180F*	Mat-180F	Chemagro Ltd. (Mississauga, Ont., Canada)
Insecticide diluent 585@	ID-585	Shell (Toronto, Ont., Canada)
Atlox® 3409F	Atlo-3409	Atkemix (Brantford, Ont., Canada)

* A flowable suspension of air-milled particles of aminocarb of 2-3 μm diameter in a highly viscous paraffinic oil, containing 180 g AI/litre.
@ A petroleum oil of low viscosity with a distillation range from 210°C (10% recovery) to 288°C (90% recovery).

Table 2. Percentage composition of ingredients in spray mixtures

Abbreviation of spray mixtures	Plot No. sprayed	Percentage composition (v/v)
180FO	P82	Mat-180F 26.7 / ID-585 73.3
180FE	P86	Mat-180 26.7 / Atlo-3409 1.3 / water 72.0

Table 3. Aircraft and spray application details

Spray plot size:	250 ha
Aircraft type:	TBM Avenger
Atomizer units:	Twenty four 1010 Flatfan Teejet® nozzles
Aircraft speed:	260 ± 20 km h^{-1}
Spray height:	30 - 35 m above canopy
Spray pressure:	240 - 270 kPa
Swath width:	133 m
Dosage rate:	70 g AI/ha
Volume rate of application:	1.5 L/ha
Emission rate:	80 L/min
Sampling trees:	7 per plot
Ground sampling units (no./plot):	15, consisting of two glass slides each of 7.5 cm x 7.5 cm

Table 4. Application dates, meteorological conditions and deposit characteristics of aminocarb spray mixtures, following broadcast treatment in New Brunswick in 1982

Measurements	Plots and spray mixtures			
	P82 (180FO)		P86 (180FE)	
	1st spray	2nd spray	1st spray	2nd spray
Date of application (1982)	June 4	June 9	May 31	June 8
Time of application (ADT) (h)	0630	0550	1908	0554
Windspeed (km/h) (3 m above ground)*	8 ± 2	3 ± 1	4 ± 2	12 ± 3
Temperature (°C) (3 m above ground)*	15	18	15	10
Relative humidity (%) at 1 m above ground*	77	76	90	87
Deposit on glass plate (g AI/ha)	20.9	7.2	15.7	28.6
Percent deposited	29.9	10.4	22.4	40.9

* Meteorological measurements were carried out inside the spray plots and the data were given in Sundaram and Sundaram (1983).

surveyor's tape. Ground vegetation and trees neighboring each sample tree were cleared up to a radius of 5 m to enhance their exposure to the spray cloud. At each sampling, 1 branch 30-cm long was taken at mid-crown from each quadrant of the tree. All new growths from the current year were removed and the old foliage was processed for analysis. The cut-up branches were kept in plastic bags and promptly stored in styrofoam coolers equipped with ice packs for immediate transport to the field laboratory. In the field laboratory, the needles and associated small twigs were clipped from the branches, mixed thoroughly and each sample was stored at -20°C in sealed plastic bags until analyzed.

ii) Forest litter: A fully exposed litter plot (*ca.* 20 m^2) was established within each plot. All small objects such as fallen branches, twigs, and small stones were cleared from the sampling area. Litter was collected, at the same frequency as foliage, from an area of 240 cm^2 at a depth of 1 cm by driving a metal frame (15.5 cm x 15.5 cm) into the ground and removing the contents with a trowel. The samples were wrapped in aluminum foil and subsequently processed as described for foliage.

iii) Forest soil: A fully exposed soil plot (*ca.* 15 m^2) was established near the litter plot. The overlying litter, moss and other organic detritus were removed to fully expose the underlying soil layer to the spray cloud. All small objects such as fallen branches, twigs, roots, stones, etc. were also removed. Soil samples (sandy loam, pH 6.1 to 6.3) were taken randomly from the top 1 cm layer as 2.5 cm diameter cores (20 cores per sample) using an auger. The samples were wrapped in aluminum foil and processed as described for foliage.

iv) Spray deposit assessment: Two glass slides (7.5 cm x 7.5 cm) mounted on a folding aluminum collection unit were used

for deposit assessment (Randall 1980). One-half hour prior to spray application, 15 such collection units were placed randomly around the litter and soil plots and around some of the trees selected for foliage sampling. The glass slides were collected 1 h after the application, kept in styrofoam coolers at 0°C and transported to the field laboratory for deposit analysis. The active ingredient was recovered from the slides by washing them with ethyl acetate (5 x 2 mL). The ethyl acetate extracts were stored in amber-colored reagent bottles at 4°C until analyzed by gas liquid chromatography (GLC).

Sample preparation, extraction and column clean-up

i) Forest litter: Small stones, twigs, etc. were removed from each sample, which was then thoroughly mixed with a spatula. Forty gram aliquots of litter were macerated for 5 min with 150 mL ethyl acetate (Caledon Chemicals) using a Waring blender. The blender was rinsed with the solvent (2 x 25 mL) and the rinse was added to the macerated extract. This extract was then passed through a 3 cm layer of anhydrous granular sodium sulphate over Whatman No. 1 filter paper in a Buchner funnel, then rinsed with the solvent (3 x 10 mL). The volume of the crude extract was reduced to 40 mL by flash evaporation (Buchii Rotovapor®) so that 1 mL of extract was equivalent to 1 g of sample.

One mL aliquots of crude extract, in duplicate, were transferred to minicolumns (10 cm x 8 mm, i.d.) (Pasteur pipettes--Fisher 13-678-8) containing successively from the bottom a small wad of silane-treated glass wool, 3 cm layer of a mixture of neutral charcoal (Nuchar SN®) and cellulose powder (Whatman CF-11®) (4:10) covered with 1 g of anhydrous Na_2SO_4. The minicolumns were attached by Tygon® tubing to glass tubes (10 cm x 8 mm, i.d.) to serve as solvent reservoirs. As the aliquot passed into the Na_2SO_4 layer, a solution containing 35 mL of glass distilled CH_3OH and ethyl acetate (20 + 80) was added to the column. The eluate was allowed to percolate through the column and was then collected in pre-cleaned containers.

The eluate was concentrated to approximately 5 mL using a Buchii Rotovapor, quantitatively transferred to a graduated centrifuge tube and concentrated to a known volume under a stream of dry N_2.

ii) Forest soil: Forty grams of thoroughly mixed sample, free from stones, twigs, etc., were macerated for 5 min with 150 mL pesticide grade ethyl acetate using a Waring blender. The blender was rinsed with the solvent and the macerate was filtered under suction. The crude filtrate was passed through a column of anhydrous sodium sulphate and flash evaporated to 40 mL so that 1 mL of aliquot corresponded to 1 g of soil sample. The clean-up was done as described under litter.

iii) Foliage: The bulk foliage sample was macerated in a food chopper. Forty grams of chopped sample were then homogenized for 5 min with 150 mL of ethyl acetate using a Brinkmann Polytron® blender. The blender was rinsed with solvent and the homogenate was filtered through a column of Na_2SO_4. The filtrate was concentrated under low pressure, processed and cleaned as described for soil.

Moisture contents of litter, soil, and foliage samples were determined by drying 10 g duplicates of each sample at 105°C for 18 h in a thermostatic oven.

Gas chromatographic analysis

A Hewlett Packard HP5840A gas chromatograph fitted with a 6 ft x 4 mm glass column packed with 1.5% OV-17 + 1.95% OV-210 on Chromosorb W HP (80/100 mesh) and coupled to an N-P specific detector was used in the analysis. A carrier gas flow (He) of 40 mL/min and an oven temperature of 185°C were maintained. Aminocarb concentrations found in foliage, litter, and soil samples are given in Tables 5, 7 and 8, respectively.

Table 5. Aminocarb residues (ppb)* in balsam fir foliage

Time after spraying	P82	P86
	1st application	
1 h	790 (1470)	1220 (2190)
3 h	790 (1470)	1190 (2100)
6 h	1080 (2000)	1100 (2030)
12 h	720 (1280)	1340 (2450)
1 d	890 (1610)	920 (1670)
2 d	500 (900)	730 (1510)
5 d	360 (640)	400 (750)
7 d		290 (550)
	2nd application	
1 h	760 (1380)	930 (1670)
3 h	580 (1140)	1210 (2190)
6 h	510 (930)	1030 (1860)
12 h	690 (1260)	1005 (1920)
1 d	940 (1750)	985 (1801)
2 d	720 (1450)	960 (1700)
3 d	300 (630)	900 (1650)
5 d	380 (690)	480 (900)
12 d	150 (325)	220 (480)

* Values without parentheses = residues in terms of "as sampled" weight.
Values with parentheses = residues in terms of oven-dry weight.

Spikes and blanks were introduced into each batch of analytical samples, especially at the column cleanup step, to assess analytical recovery and the effect(s), if any, of reagent changes. The recoveries from spiked samples on average were 85 ± 6%. None of the prespray and check samples (balsam fir foliage, forest soil, and litter) contained any detectable levels of aminocarb.

The analytical limit of detection for the insecticide was 0.01 µg/mL of final extract for injection. This in turn was equivalent to 0.01 µg/g of the same on the "as sampled" basis. Due to variations in the moisture contents of different varieties of substrates (foliage, litter, and soil) studied, the calculated limit of detection (MDL) on a dry weight basis varied usually from 0.01 µg/g to 0.05 µg/g.

RESULTS AND DISCUSSION

Spray deposition

Spray deposit data from the aerial application of the two aminocarb formulations are presented in Table 4. The quantities of aminocarb that deposited in all 4

Table 6. Curvilinear decay equations for dissipation of aminocarb with time 't'

Plot No.	Formulation	Application No.	Type of sample	Regression equation	R^2 (%)	B	C	$T_{\frac{1}{2}}$(h)
P82	180FO	1st	Fresh	$Y = 871e^{-0\ 00693\ t}$	87.8	871	0.00693	100
			Oven-dry	$Y = 1585e^{-0\ 00686\ t}$	86.4	1585	0.00686	101
		2nd	Fresh	$Y = 676e^{-0\ 00534\ t}$	79.6	676	0.00534	130
			Oven-dry	$Y = 1259e^{-0\ 00486\ t}$	79.7	1259	0.00486	143
P86	180FE	1st	Fresh	$Y = 1202e^{-0\ 00875\ t}$	98.8	1202	0.00875	79
			Oven-dry	$Y = 2188e^{-0\ 00841\ t}$	99.2	2188	0.00841	82
		2nd	Fresh	$Y = 1096e^{-0\ 00564\ t}$	94.8	1096	0.00564	123
			Oven-dry	$Y = 1995e^{-0\ 00500\ t}$	93.2	1995	0.00500	139

Table 7. Aminocarb residues (ppb) in forest litter

Time after spraying	P82	P86
	1st application	
1 h	40 (100)	70 (110)
3 h	<10	20 (30)
6 h	<10	<10
12 h	<10	<10
1 d	<10	<10
2 d	<10	<10
3 d	<10	10 (20)
5 d	<10	50 (110)
7 d		50 (70)
	2nd application	
1 h	40 (60)	20 (30)
3 h	20 (30)	10 (15)
6 h	<10	<10
12 h	<10	<10
1 d	<10	<10
2 d	<10	40 (60)
3 d	<10	<10
5 d	<10	<10
7 d	<10	<10
12 d	<10	<10

See footnotes in Table 5.

applications varied enormously, ranging from 7.2 g/ha to 28.6 g/ha ($\bar{x}$ = 12.4 g/ha). Physical factors such as droplet size spectra, rate of evaporation, and existing climatic conditions could have contributed to this variation. The average percent of the released material (AI) deposited on the forest floor was low (17.7%). This was probably because a large proportion of the spray cloud was in small droplets, which must have impacted on the tree crown instead of the glass plates on the forest floor level.

Aminocarb residues in conifer foliage

It is evident from Table 5 that at five days after treatment, 33 and 40% of the maximum AI (as sampled) had been retained in the foliage in P82, and 30 and 40% in P86. In all cases, the active material was rapidly lost during the initial stages. Then, there followed a gradual and curvilinear decrease with time, which indicated a tendency for the chemical to persist in the needles. The dissipation of aminocarb followed an exponential decay pattern according to the equations (1) to (3):

$$Y = A + Be^{-C\,t} \qquad (1)$$

$$\log (Y - A) = \log B - \frac{C}{2.303}\,t \qquad (2)$$

$$Y = A \text{ (when } t = \infty) \qquad (3)$$

Table 8. Aminocarb residues (ppb) in forest soil

Time after spraying	P82	P86
	1st application	
1 h	<10	<10
3 h	<10	<10
6 h	40 (50)	<10
12 h	<10	<10
1 d	<10	<10
2 d	<10	<10
3 d	<10	<10
5 d	<10	<10
	2nd application	
1 h	<10	<10
3 h	<10	<10
6 h	<10	<10
12 h	<10	<10
1 d	<10	<10
2 d	<10	<10
3 d	<10	<10
5 d	<10	<10
12 d	<10	<10

See footnotes in Table 5.

If we assume that eventually all residues would be lost from the foliage, at the infinite time "t ∞" equations (1) to (3) become (4) to (6)

$$Y = Be^{-Ct} \quad (4)$$

$$\log Y = \log B - \frac{C}{2.303} t \quad (5)$$

$$Y = 0 \text{ (when } t = \infty) \quad (6)$$

The half-lives ($T_{\frac{1}{2}}$) of the exponential decay can be calculated from equation (7):

$$T_{\frac{1}{2}} = \frac{2.303 \log 2}{C} \quad (7)$$

The regression coefficients B and C, and half-lives of decay are given in Table 6 along with regression equations. The rate constant C represents the rapidity with which the residues were lost from the foliage, i.e., the greater the value, the faster the rate of loss. Consequently, it appears that aminocarb was lost at a faster rate from foliage treated with 180FE than with 180FO, since the C values were higher for 180FE. This is again shown in the $T_{\frac{1}{2}}$ values for the two formulations, which are consistently higher for 180FO than for 180FE. This would mean that, the higher the foliage residues, the faster the rate of dissipation.

The half-lives observed in the present study ranged from 78 to 143 h, and these values are well within the range found in earlier studies (Sundaram and Hopewell 1977, Sundaram 1981).

The dissipation of aminocarb from the leaves must have been caused by volatilization, photolysis, weathering action of humidity, rain and wind (codistillation), at the initial stages after spray application. As the residues were gradually absorbed by the lipophilic terpenoids of the foliage, they seemed to be degraded in a relatively slow manner, partly through chemical and biological means, and partly through dilution.

Aminocarb residues in forest litter

Residues of aminocarb present in forest litter from the two spray plots are given in Table 7. The maximum levels ranged from 10 ppb to 70 ppb (as sampled), at 1 h post-spray and never exceeded the detection limit (10 ppb) afterwards, with a few exceptions in P86. The low concentration levels found in litter are difficult to explain, especially when the concentrations of aminocarb collected on the glass slides (Table 4) were much above the detection limits. Evidently, no noticeable correlation existed between the two receptors. It is possible that differences in microturbulence between the two neighboring sample sites could have caused such variations in deposits.

The residue levels in P86 increased after the 2-day post-spray period after the first application (Table 7). The reason for this could be the foliar leaching and run-off by rain onto the forest floor. The litter sample contained fallen needles and organic detritus (polyglucuronic acids, humic acids, fulvic acids, amino acids, phenolic and carboxylic compounds), and was found to be acidic (pH 5.2 to 5.6). In such cases, as observed earlier (Sundaram et al. 1978, 1979), if sufficient concentrations of AI have reached the forest floor, one would normally expect that aminocarb molecules would be adsorbed onto litter particulates and persist as aryldimethylammonium cations (Figure 1) resisting degradation. In a similar study conducted recently (Sundaram 1981), the initial maximum concentrations found in litter ranged from 18 to 216 ppb and persisted for 12 to 21 days.

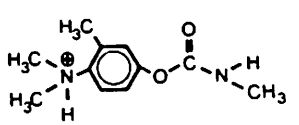

Figure 1. Cationic form of aminocarb.

Residues in forest soil

The forest floor is usually considered a major receptor of aerially applied sprays. As observed earlier, only minimum levels of aminocarb ($\leq$10 ppb) (Table 8) reached the soil. This result compares favorably with the low values (8 to 51 ppb) obtained in an earlier study (Sundaram 1981).

Mechanisms of disappearance of aminocarb from the forest floor include volatilization, leaching through the soil profile by water, and degradation by various physico-chemical processes including sunlight and biological means. Among these, a combination of chemical and bacterial degradations and volatilization from the soil surface probably played vital roles. In addition, various soil factors such as soil type (sandy loam), organic matter, moisture content, pH (6.1 to 6.3) and temperature might have had a profound influence on the fate and persistence of aminocarb. Under forestry conditions, canopy cover, relative humidity and rainfall would also have played significant roles in insecticide persistence.

Pesticide-soil interactions, especially focussing on persistent organo-chlorines, have been studied extensively and documented. The present study has shown that, when used at field application rates little aminocarb reaches the forest floor. Any that did reach the forest floor did not

remain very long. Because of its extremely short life in the lithosphere, it is very unlikely that the chemical would pose any significant hazard to soil micro-organisms (bacteria, fungi, etc.) and soil invertebrates.

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