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ENVIRONMENTAL MONITORING OF THE 1978 SPRUCE BUDWORM SPRAY PROGRAM IN NEW BRUNSWICK, CANADA — FIELD SAMPLING AND AQUATIC TOXICITY STUDIES WITH FISH

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ENVIRONMENTAL MONITORING OF THE 1978 SPRUCE BUDWORM SPRAY PROGRAM IN NEW BRUNSWICK, CANADA - FIELD SAMPLING AND AQUATIC TOXICITY STUDIES WITH FISH

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ABSTRACT

Monitoring of the New Brunswick, Canada, spruce budworm spray program in 1978 consisted of sampling shellfish and sediments in the field for fenitrothion content, and further aquatic toxicity studies with rainbow trout and concentrates and formulations of the carbamate insecticide, aminocarb.

Fenitrothion was not detected in shellfish collected before and after the spray operations in several locations in New Brunswick in 1978. Fenitrothion was either not present or detected in trace amounts (up to 0.05 µg/g) in aquatic sediments after the spray operations in several locations in New Brunswick in 1978. Indirect transport of fenitrothion to sediments in water bodies not directly sprayed can occur.

The 1976 aminocarb concentrate (Matacil®), containing 18% aminocarb, had a 4-day LC50 to rainbow trout of 0.75-0.96 mg/ℓ (nominal concentration) or 0.13 (0.08-0.27)^{R} mg/ℓ, expressed as mg aminocarb per liter of water. The 1976 aminocarb formulation, containing 5.4% aminocarb, had a 4-day LC50 of 3.0 mg/ℓ (nominal concentration), or 0.16 mg/ℓ (meas. conc.), expressed as mg aminocarb per liter of water. Test solutions of the concentrate and the formulation aged in the laboratory for 3 days did not change in acute toxicity. Those solutions aged in the laboratory for 10 days became less toxic (the concentrate) or did not change significantly in toxicity (the formulation). Test solutions of the formulation aged for 10 days outdoors in the dark showed little decrease in aminocarb content but became non-lethal, suggesting an alteration of other components of the formulation that substantially contributed to its original toxicity.

The 1976 formulation contained aminocarb (5.4%), No. 2 and 4 fuel oil, and emulsifiers (composition unknown to us) as the main constituents. The 1978 formulation contained aminocarb (4.4%), nonylphenols, and insecticide diluent fuel oil 585, as the main constituents. The aquatic toxicity of major constituents of the aminocarb spray formulation needs to be studied individually and collectively, under laboratory and natural conditions, before the fate and effects of the formulation in aquatic systems will be adequately assessed and understood.

Résumé

En 1978, le contrôle des pulvérisations antitordeuses au Nouveau-Brunswick a consisté à cueillir, en plusieurs endroits, des échantillons de mollusques, de crustacés et de sédiments en vue de connaître leur teneur en fénitrothion, ainsi qu'à étudier, sur la truite arc-en-ciel, la toxicité en milieu aquatique des concentrés et préparations d'aminocarbe, insecticide de la famille des carbamates.

On n'a décelé aucune trace de fénitrothion chez les crustacés recueillis avant et après les pulvérisations. Dans les sédiments aquatiques, le fénitrothion était soit complètement absent, soit présent sour forme de traces (jusqu'a 0,05 µg/g). Cet insecticide est parfois transporté au-dessur de nappes d'eau non soumises aux pulvérisations et il se dépose dans leurs sédiments.

Le concentré d'aminocarbe à 18% de 1976 (Matacil®) avait, pour la truite arc-en-ciel, une CL50 après 4 jours de 0.75 à 0,96 mg/L (concentration nominale) ou de 0,13 $(0,08\text{-}0,27)^R$ mg/L, exprimée en mg d'aminocarbe par L d'eau. Quant à la préparation d'aminocarbe à 5, 4% de 1976, elle avait une CL50 après 4 jours de 3,0 mg/L (concentration nominale), ou de 0,16 mg/L, exprimée en mg d'aminocarbe par L d'eau. La toxicité aigue des solutions du concentré et de la préparation que l'on avait laissées reposer en laboratoire pendant 3 jours n'a pas changé. Les solutions qu'on avait laissées reposer pendant 10 jours ont, dans le cas du concentré, perdu en toxicité alors que, dans le cas de la préparation, elles sont restées pratiquement aussi toxiques. La teneur en aminocarbe de solutions de préparation laissées à l'extérieur dans l'obscurité pendant 10 jours n'a guère diminué, mais les solutions elles-mémes sont devenues non léthales, ce qui donne à penser que d'autres composants contributant à leur toxicité initiale se sont altérés.

La préparation de 1976 contenait essentiellement de l'aminocarbe (5,4%), du mazout n^{os} 2 et 4 et des émulsifiants (de composition inconnue). Les principaux composants de la préparation de 1978 étaient de l'aminocarbe (4,4%), des nonylphénols et du mazout 585, ce dernier servant à diluer l'insecticide. On devra étudier in vivo et in vitro la toxicité en milieu aquatique des principaux composants des préparations, pris ensemble et séparément, avant de pouvoir évaluer et bine comprendre leur devenir et leurs effets dans les systèmes aquatiques.

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1 INTRODUCTION

The 1978 Spray Program to combat the spruce budworm in New Brunswick covered a total area of 3.8 million acres, 2 million acres of which were sprayed with fenitrothion and 1.8 million acres with aminocarb.

Field activities undertaken by the Environmental Protection Service during the 1978 spray program consisted of visits to airstrips, and the collection of sediments and molluscs (both freshwater and estuarine) for analysis of contamination by parent fenitrothion. This was a continuation of previous work, reported by Lord et. al. (1978). The laboratory program throughout 1977 and 1978 consisted of a continued assessment of the acute lethal toxicity of an aminocarb concentrate and formulation to fingerling rainbow trout (*Salmo gairdneri*), and chemical analyses in support of the field and toxicity studies. Toxicity studies were on aminocarb because of the obvious paucity of such data on this pesticide (Wells et al., 1978). This report briefly describes major findings of this work.

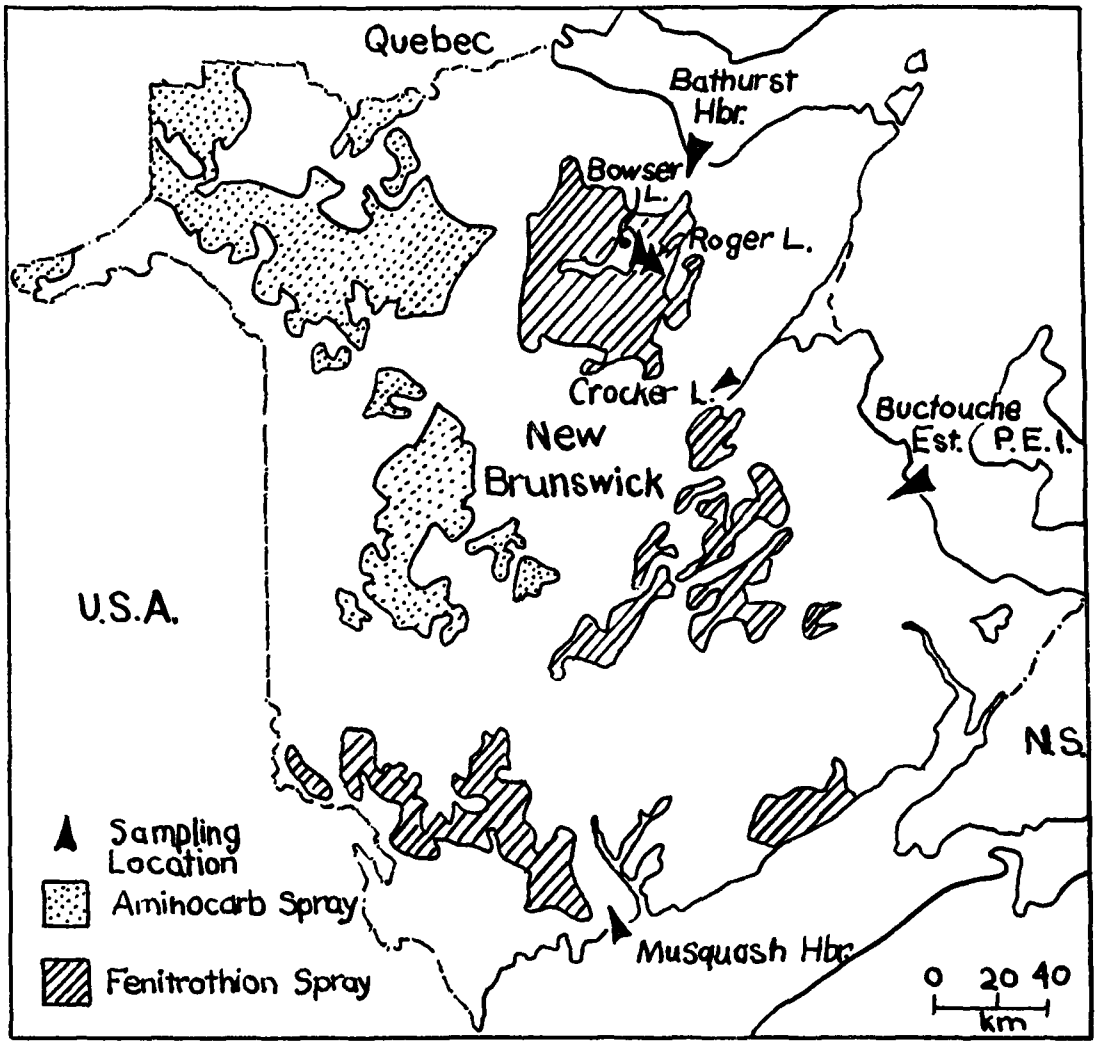
2 FIELD SAMPLING

2.1 Airport Monitoring

Four airstrips (Juniper, Blissville, Sevogle and Charlo) were visited during operational spray periods. Generally, practices at airstrips were well controlled, a very similar situation to 1977. No serious problems of pesticide contamination of the airstrip grounds were encountered.

2.2 Fenitrothion in Sediments and Mollusc Tissue

Six sites were selected for sampling. Their locations and the reasons for their selection are shown in Figure 1 and Table 1, respectively.



N.B. 1978 SPRAY PROGRAM & SAMPLING LOCATIONS

FIGURE 1

TABLE 1 SUMMARY OF LOCATIONS, SELECTION CRITERIA AND SAMPLES FOR 1978 NEW BRUNSWICK SPRAY MONITORING PROGRAM

LOCATION	SELECTION CRITERIA FOR LOCATION	SAMPLES
FRESHWATER:		
Bowser Lake - Shallow Lake	In area sprayed directly	Sediment Shellfish
Crocker Lake	Control site, 50 km from nearest spray block	Sediment
Roger Lake	In area to be sprayed, but lake not to be sprayed	Sediment
ESTUARINE AND MARINE		
Buctouche Estuary	Not sprayed in 1978, but watershed sprayed in 1976 and 1977, and data are available for these years	Sediment Shellfish
Bathurst Harbour	Nepisiquit drainage - much of Nepisiquit drainage basin sprayed in 1978	Sediment Shellfish
Musquash - River & Estuary	Almost entire watershed sprayed in 1978	Sediment Shellfish

For sediment cores, the top 2 cm of each sample was separated for analysis. Grab samples were composited and a subsample was separated for analysis. For molluscs, entire animals (excluding shells) were homogenized together and a subsample analyzed. Analyses of samples were for parent fenitrothion (Matheson and Pelly, 1978).

Results for the 6 sampling locations (Table 2) show that:

(i) Sediment and shellfish samples that were collected during June (6th, 7th and 8th), which coincided with the start of spraying, showed no detectable levels of fenitrothion.

(ii) Post-spray samples collected in freshwater environments (Bowser, Crocker and Roger Lakes) showed minor amounts (just above detection levels) of fenitrothion in sediments from both Bowser Lake and Roger Lake. No fenitrothion was detected in sediments from Crocker Lake.

(iii) Bowser and Roger Lakes were in spray zones while Crocker Lake was 50 km from the nearest spray block. Sediment uptake of pesticide is not a major problem in these lakes as fenitrothion levels were either extremely low or non-detectable. However, it is clear that indirect transport (e.g. aerial drift, stream flow) results in the occurrence of detectable amounts of fenitrothion in bodies of water not directly sprayed, such as Roger Lake.

(iv) Post-spray (July 11th and 12th) samples, collected from estuarine and marine environments (Buctouche, Bathurst and Musquash), showed no detectable levels of fenitrothion in either sediment or shellfish.

TABLE 2 FENITROTHION IN SEDIMENTS AND MOLLUSCS - 1978 NEW BRUNSWICK SPRAY PROGRAM

LOCATION	DATE OF SAMPLING	TYPE OF SAMPLE	FENITROTHION CONC.* (µg/g)
Bowser Lake	June 6	Freshwater Mussels (*Anodonta spp.*)	<0.005**
	June 6	Grab - Sediment	<0.01
	June 6	Core - Sediment	<0.07
	July 11	Grab - Sediment	0.03; 0.05
	July 11	Core - Sediment	<0.04
Crocker Lake	June 7	Grab - Sediment	<0.02
	June 7	Core - Sediment	<0.04
	July 12	Grab - Sediment	<0.01
	July 12	Core - Sediment	<0.03
Roger Lake	June 6	Grab - Sediment	<0.02
	June 6	Core - Sediment	<0.07
	July 11	Grab - Sediment	0.03; 0.02
	July 11	Core - Sediment	<0.04
Buctouche			
a) Buctouche Bay	June 5	Oysters (*Crassostrea virginica*)	<0.005
b) Riviere Principale	June 5	Oysters (*Crassostrea virginica*)	<0.005
a) Buctouche Bay	July 19	Oysters (*Crassostrea virginica*)	<0.005
b) Riviere Principale	July 19	Oysters (*Crassostrea virginica*)	<0.005
Bathurst Harbour	June 7	Core - Sediment	<0.07
	June 7	Grab - Sediment	<0.01
	July 10	Core - Sediment	<0.01
	July 10	Grab - Sediment	<0.01
	July 10	Clams (*Mya arenaria*)	<0.005
Musquash Estuary	June 8	Core - Sediment	<0.07
	July 17	Grab - Sediment	<0.01
	July 17	Mussels (*Anodonta spp.*)	<0.005

* Sediment - µg/g Dry Weight
a) Grab Samples - composited and a 15-20 g sample separated for analysis
b) Core Samples - top 2 cm portion analyzed
Tissue - µg/g Wet Weight, a pool of 6 specimens used in all cases

** Sample detection limits vary due to variations in sample masses used for extraction and/or daily variations in instrumental detection limits.

3 AQUATIC TOXICITY STUDIES

The long-term objectives of the studies were to determine the sources of acute toxicity to fish of the various components of aminocarb formulations, to determine the degree of persistence of both toxicity and aminocarb in test solutions, and to evaluate the degree of risk imparted to non-target aquatic species of aminocarb, its metabolites, and other key constituents of the formulations. The studies reported below were conducted in 1977 and 1978. This is a brief report of major findings.

3.1 Methods

A number of aquatic bioassays were conducted on fresh and aged (3d and 10d) test solutions of an aminocarb oil soluble concentrate (Matacil®) and of an aminocarb formulation, both collected at airports in New Brunswick during the 1976 spray program. The samples tested were:

(1) aminocarb concentrate (Matacil®), presumed to be 1.68 D oil soluble Matacil® concentrate, containing 18% aminocarb based on our analyses;

(2) aminocarb formulation containing 5.4% aminocarb, the remainder being No. 2 and 4 fuel oil and emulsifiers as the main constituents.

All 4-day and 6-day bioassays were non-aerated, static 15°C tests with fingerling rainbow trout (*Salmo gairdneri* Richardson), following methods described in Wells et al. (1978).

Three 16-day experiments were conducted to examine the influence of aging and different light-temperature regimes on the acute toxicity of test solutions of the formulation (5.6 mg/ℓ (nominal)). Aging was 0 d and 10 d prior to a 6-day bioassay. Light/temperature regimes were laboratory

(12L:12D)/15°C and ambient outdoors (May, June, September), light-dark, constant dark, and temperature. All combinations of these factors were included in the experimental design.

Aminocarb was isolated from aqueous bioassay samples by chloroform extraction. A gas chromatograph equipped with an alkali flame detector was utilized for identification and quantitative analysis. The concentrate and formulation were appropriately diluted and analyzed in a similar manner.

3.2 Results

3.2.1 Four-Day Bioassays - Aminocarb Concentrate and Formulation

The aminocarb concentrate (Matacil®) had 4-day LC50's ranging between 0.75 - 0.96 mg/ℓ (nominal concentration); test solutions aged for 3 days in the laboratory did not change in acute toxicity but those aged for 10 days became less toxic. Concentrates collected in 1976 and 1977 had the same lethal toxicity. Measurements of aminocarb in the test solutions showed that levels during the tests remained stable at 13-17% of total nominal concentration; a more accurate estimate of the 4-day LC50 for the aminocarb concentrate may be 0.13 $(0.08 - 0.27)^{RANGE*}$ mg/ℓ, expressed as mg aminocarb per liter of water.

The aminocarb formulation had a 4-day LC50 of 3.0 mg/ℓ (nominal concentration), slightly but significantly less toxic than the aminocarb concentrate. Test solutions of the formulation aged for 3 days in the laboratory did not lose their acute toxicity.

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* Range of concentrations causing 0% and 100% mortalities.

3.2.2 Sixteen-Day Experiments - Aminocarb Formulation

During the 10 days of aging, only those test solutions aged outdoors and exposed to ambient photoperiods showed a significant decline in aminocarb (Table 3). In all treatments (aged and non-aged), there was little change of aminocarb content over the 6-day bioassay period.

The acute toxicity (expressed as median lethal times (h)) of the formulation aged in the laboratory changed only slightly in comparison with test solutions prepared just prior to toxicity testing (LT50's: fresh solution = 9-18 h; 10 d aged solution = 22-31 h). However, outdoor aged test solutions were all non-lethal (LT50: 10 d aged solutions >144 h), even though the solutions aged outdoors in the dark showed little decrease in aminocarb content compared to fresh test solutions (Table 3). This latter observation suggests that under our test conditions, the content of the aminocarb is not the only, and perhaps not even the major, contributor to the acute lethal toxicity of the freshly prepared and laboratory aged test solutions of formulation. Alterations of formulation components other than the active ingredient aminocarb probably resulted in the non-lethality of the outdoor aged test solutions.

3.3 Discussion

Based on the above results, it is possible to express the acute lethality to fish of the aminocarb concentrate (Matacil®) and aminocarb formulation in terms of the aminocarb present:

4-day LC50 aminocarb concentrate = 0.13 $(0.08\text{-}0.27)^{R}$ mg/ℓ*

4-day LC50 formulation = 5.4% x 3.0 mg/ℓ = 0.16 mg/ℓ*

LT50 of 5.6 mg/ℓ formulation = 9-18 h at 0.30 mg/ℓ*

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* mg aminocarb/liter of water.

TABLE 3 LEVELS OF AMINOCARB, IN mg/ℓ, DURING SIXTEEN-DAY EXPERIMENTS WITH TEST SOLUTIONS OF THE 1976 AMINOCARB FORMULATION AGED IN THE LABORATORY AND OUTDOORS

LOCATION	REPLICATE	DAY OF EXPERIMENT				
		AGING PERIOD ←		BIOASSAY PERIOD* →		
		0	10	13	16	20
LABORATORY-FRESH	(1)	-	0.29	-	-	-
	(2)	-	0.16	0.14	-	0.14
	(3)	-	0.32	-	-	-
LABORATORY-AGED	(1)	0.27	0.27	0.27	-	-
	(2)	0.14	0.14	0.13	-	0.12
	(3)	0.30	0.26	0.26	-	-
OUTDOORS (LIGHT)-AGED	(1)	-	0.25	0.21	-	0.17
	(2)	0.14	0.09	0.08	-	0.08
	(3)	0.32	0.11	0.12	-	0.10
OUTDOORS (DARK)-AGED	(1)	-	0.30	-	-	0.24
	(2)	0.15	0.14	0.12	-	0.11
	(3)	0.31	0.26	0.25	-	0.23

* Only bioassay data of first 6 days were analyzed; test tanks were continued until day 20.

The three results agree well with each other but we have assumed that the active ingredient aminocarb is primarily responsible for the acute lethal toxicities of the concentrate and formulation. However, the 16-day experiments showed that this assumption is most likely erroneous, as it ignores contributions to toxicity of other constituents. In addition, D. Lamb in a talk at Moncton, New Brunswick, in August, 1978, reported the acute lethal toxicity of technical grade aminocarb to rainbow trout to be 8.9 mg/ℓ (4-day LC50). The concentrate was 18% aminocarb, and the formulation was 5.4% aminocarb in the present study, yet their toxic levels are much lower than Lamb's reported value. Although a direct comparison of our data and that of Lamb cannot be without error, it again suggests a significant contribution to acute toxicity of components other than the active ingredient aminocarb, or of the aminocarb in combination with these components.

The sources and degree of aquatic toxicity in each spray formulation* and the potential for this toxicity to appear under natural field conditions will only be understood if all known major constituents (aminocarb, oil, nonylphenols) of the formulation are studied individually and collectively, for toxicity, under laboratory and natural conditions. This has been and still is the main objective of our small toxicity program. Results and hypotheses from such studies should then be verified under field spray conditions, where concentrations of the constituents in water and tissue, and immediate biological effects, can be

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* In 1978, the spray formulation contained a new oil-Insecticide Diluent 585, a low aromaticity fuel oil-resulting in the need to completely re-evaluate the formulation. To the best of our knowledge, the concentrates from 1976 to 1978 contained aminocarb, fuel oil, and emulsifiers (nonylphenols in the 1978 formulation - D. Lamb, pers. comm.) as the major constituents.

measured. Until such information is obtained, published, and considered in the light of recent conclusions of the AIBS Task Group on the Aquatic Hazards of Pesticides (Anon, 1978), an understanding of the interaction of aminocarb formulations with important non-target aquatic species, such as fish, crustaceans and insects, will continue to be extremely limited.

4 SUMMARY AND CONCLUSIONS

1. Fenitrothion was not detected in shellfish collected before and after the spray operations in several locations in New Brunswick in 1978.

2. Fenitrothion was either not present or detected in trace amounts (up to 0.05 µg/g) in aquatic sediments after the spray operations in several locations in New Brunswick in 1978. Indirect transport of fenitrothion to sediments in water bodies not directly sprayed can occur.

3. The 1976 aminocarb concentrate (Matacil®), containing 18% aminocarb, had a 4-day LC50 to rainbow trout of 0.75-0.96 mg/ℓ (nominal concentration) or 0.13 (0.08-0.27)^{R} mg/ℓ, expressed as mg aminocarb per liter of water.

4. The 1976 aminocarb formulation, containing 5.4% aminocarb, had a 4-day LC50 of 3.0 mg/ℓ (nominal concentration), or 0.16 mg/ℓ, expressed as mg aminocarb per liter of water.

5. Test solutions of the concentrate and the formulation aged in the laboratory for 3 days did not change in acute toxicity. Those solutions aged in the laboratory for 10 days became less toxic (the concentrate) or did not change significantly in toxicity (the formulation).

6. Test solutions of the formulation aged for 10 days outdoors in the dark showed little decrease in aminocarb content but became non-lethal, suggesting an alteration of other components of the formulation that substantially contributed to its original toxicity.

7. The 1976 formulation contained aminocarb (5.4%), No. 2 and 4 fuel oil, and emulsifiers (composition unknown to us), as the main constitutents. The 1978 formulation contained aminocarb (4.4%), nonylphenols and insecticide diluent fuel oil 585 as the main constituents. The aquatic toxicity of major constituents of the aminocarb spray formulation must be studied individually and collectively, under laboratory and natural conditions, before the fate and effects of the formulation in aquatic systems will be adequately assessed and understood.

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