# Laboratory Toxicity of Aqueous Formulations of Fenitrothion and Aminocarb Containing the Emulsifiers Triton® X100 and Triton® X114 Against Spruce Budworm Larvae

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B.V. Helson

J. McFarlane

D. Comba

Forest Pest Management Institute

Canadian Forestry Service

Environment Canada

Sault Ste. Marie, Ontario

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## Introduction

As part of the CFS/FPMI Action Plan to investigate several new aqueous formulations of fenitrothion and aminocarb, the toxicity of several formulations containing the emulsifiers Triton® X100 or Triton® X114 to spruce budworm larvae, Choristoneura fumiferana, was determined in the laboratory and compared to standard formulations containing the emulsifier Atlox 3409F.

## Methods and Materials

1. Insecticides and Formulations

The insecticides, formulations and concentrations used in contact toxicity tests were:

1.1. Fenitrothion (Sumithion® technical, 99.8% AI w/w)

1.1.1. Sumithion® (0.7% AI by w.) Triton® X100 (14.5% by v.) + water (to 100% v.).

1.1.2. Sumithion® (0.7% AI by w.) + Triton® X100 (3% by v.) + Cyclosol 63 (24% by v.) + water (to 100% v.).

1.1.3. Sumithion® (0.7% AI by w.) + Triton® X114 (14.5% by v.) + water (to 100% v.).

1.1.4. Sumithion® (0.7% AI by w.) + Triton® X114 (3% by v.) + Cyclosol 63 (24% by v.) + water (to 100% v.).

1.1.5. Sumithion® (0.7% AI by w.) + Triton® X114 (10% by v.) + Canola Oil (10% by v.) + water (to 100% v.).

The standard formulation for comparison was: Sumithion® (0.7% AI by w.) + Atlox 3409F (1.05% by v.) + Dowanol TPM (1.05% by v.) + water (to 100% v.).

1.2. Aminocarb (Matacil® 180F, 180 g AI/L).

1.2.1. Matacil® 180F (0.1% AI by w.) + Triton® X100 (3% by v.) + water (to 100% v.).

1.2.2. Matacil® 180F (0.1% AI by w.) + Triton® X100 (3% by v.) + Cyclosol 63 (24% by v.) + water (to 100% v.).

1.2.3. Matacil® 180F (0.1% AI by w.) + Triton® X114 (3% by v.) + water (to 100% v.).

1.2.4. Matacil® 180F (0.1% AI by w.) + Triton® X114 (14.5% by v.) + water (to 100% v.).

The standard for comparison was: Matacil® 180F (0.1% AI by w.) + Atlox 3409F (1.5% by v.) + water (to 100% v.).

The water added to each formulation contained ca. 0.2% Rhodamine B dye for colorimetric assessment of insecticide deposits.

The residual toxicity of the following fenitrothion and aminocarb formulations were also tested in 1982:

1.3.1. Sumithion® (2.0% AI w.) + Triton® X100 (14.5% v.) + water (to 100% v.).

1.3.2. Sumithion® (2.0% AI w.) + Triton® X100 (3% v.) + Cyclosol 63 (24% v.) + water (to 100% v.).

1.3.3. Sumithion® (2.0% AI w.) + Triton® X100 (10% v.) + Canola Oil (10% v.) + water (to 100% v.).

1.3.4. Matacil® 180F (2.0% AI w.) + Triton® X100 (3% v.) + water (to 100% v.).

Difficulties were experienced in obtaining the required deposits with formulations containing Atlox 3409F in these residual toxicity tests. Consequently the following 'standard' formulations were used for comparison: Sumithion® (2.0% AI w.) + Standard Solvent Solution (to 100% v.). SSS = 80% Ethylene Glycol + 20% Dowanol TPM Matacil® 180F (2.0% AI w.) + ID585 (to 100% v.).

2. Contact Toxicity Tests

The methods and equipment for the contact toxicity tests were developed by P.C. Nigam and have been described by him (Nigam 1975). Fifth-instar laboratory stock spruce budworm larvae were anaesthetized with $CO_2$, placed on 9 cm diameter filter papers and sprayed in a modified Potter's tower with 6 different dosages of each formulation corresponding to ca. 1.12, 2.24, 4.48, 6.72, 8.96 and 11.2 L/ha by varying the spray times. The tower was calibrated prior to treatment to deliver the volumes necessary to give these dosages by colorimetric assessment of dye deposits on filter papers in comparison with a colour standard for each dyed insecticide formulation using a Baush and Lomb Spectronic® 70 or 100 Spectrophotometer. Actual dosages in μg AI/$cm^2$ applied during an experiment were also determined colorimetrically by measuring the dye deposits on the filter papers, which had held the insects. Three groups of 10 insects were sprayed with each dosage in an experiment and the same number were not treated to serve as control. After spraying, each group was placed on larch foliage in a paper cup and held in an environmental incubator at 20°C, 60% RH + 16 hour photoperiod. Mortality was assessed at 24, 48 and 72 hours after treatment. A minimum of 3 such experiments were conducted with each formulation. The mortality data was corrected by Abbott's method for any control mortality and subjected to probit analyses to obtain $LD_{90}$ and $LD_{95}$ values.

3. Residual Toxicity Tests

The procedures for determining the residual toxicity of insecticides against spruce budworm are also described by Nigam (1975). These tests are designed to determine the crawling contact and/or stomach toxicity of a material on foliage at selected intervals after treatment under natural weathering conditions. Two potted 4-5 year old balsam fir and 2 white spruce trees were sprayed with 11.2 L/ha of a formulation in a spray chamber. The chamber was calibrated to deliver this dosage using colorimetric techniques similar to those in the contact toxicity tests.

After the deposit had dried ca. 4 hours after treatment, unweathered fresh buds were clipped from the trees and placed in plastic cups. Fifteen field-collected fifth-instar spruce budworm larvae were put in each cup. The cups were kept in a controlled environment incubator at 20°C, 60% RH and 16 hour photoperiod, and the larvae were checked for mortality 24 hr, 48 hr and 72 hr after exposure to the foliage. Two such cups were prepared for each tree, giving a total of 4 cups and 60 larvae for each tree species. Two untreated trees of each species were also handled in the same fashion.

All trees were then placed outdoors fully exposed to natural weathering conditions. At 1, 3, 5 and 10 days after treatment, larvae were again exposed to clipped foliage as above. Temperature, dew point, precipitation and sunshine data during the weathering periods were obtained from the Sault Ste. Marie airport ca. 12 km from the location of the trees.

The mean percent mortality on each tree species was calculated for each interval after treatment and corrected for any control mortality by Abbott's method.

## Results and Discussion

### 1. Contact Toxicity

The results with fenitrothion formulations are presented in Table 1. The median lethal doses for these formulations after 72 hours were similar. The 72 hour $LD_{50}$ value for the most toxic formulation, Triton® X100 + Cyclosol 63 was only 1.37 times more potent than the least toxic one, Triton® X114 + Canola Oil. The relative toxicities of the formulations containing Triton® emulsifiers ranged from 0.86 to 1.18 compared with the standard Atlox formulations.

With the exception of the Triton® X114 + Canola Oil formulation, the 72 hour $LD_{95}$ values for these formulations were also comparable. Relative toxicities ranged from 0.83 to 1.14 compared with the Atlox formulation. The 72 hour $LD_{95}$ for the Triton® X114 + Canola Oil formulation was 2.23 x higher than the standard. Difficulties were experienced in obtaining high mortalities with this formulation. A Canola Oil and Triton® X100 fenitrothion formulation was also tested at 0.7% AI but mortality was so low, probit analysis could not be performed on the data. Problems with the separation of ingredients may have occurred in the testing apparatus with these formulations.

The Triton® X100 and Triton® X114 formulations of technical Sumithion® with Cyclosol 63 were slightly more toxic than the formulations with these emulsifiers alone. The reduction in the concentration of the emulsifiers in these Cyclosol 63 formulations (3% vs. 14.5%) may have increased their toxicity.

The contact toxicities of Matacil® formulations are presented in Table 2. With the exception of the 14.5% Triton® X114 formulation the 72 hour median lethal doses for the formulations containing Triton® X100 or Triton® X114 were very similar and all were slightly more toxic than the Atlox formulation. Although somewhat greater differences between 72 hour

$LD_{95}$ values were observed these formulations were again more toxic than the Atlox formulations. Interestingly, the toxicity of a 14.5% Triton® X114 formulation was less than the corresponding 3% formulation and this difference was greater than between other formulations containing different ingredients. The 3% formulation was the one recommended for testing. The effects of varying the proportions of adjuvants on the toxicity of formulations is being investigated further.

2. Residual Toxicity

The results obtained on the residual toxicity of some of these aqueous formulations are presented in Table 3. These results are very preliminary. Due to limited resources and a restricted testing period only one test with each formulation was conducted, usually during different periods and hence under different weathering conditions. For some formulations, the 0 and 1 day evaluations could not be performed because sufficient field insects were not available. No residual toxicity tests were performed with Triton® X114 since it was introduced into the testing scheme after the period when such tests are normally performed.

Keeping these limitations in mind, the aqueous fenitrothion formulations containing Triton® X100 usually provided mortalities comparable with or higher than the standard formulation. Although the residual toxicity of the Matacil® formulation containing Triton® X100 appeared to be slightly less than that of the corresponding ID585 formulation particularly on the third day after treatment, rain fell during the first 5 days of the testing period of the former formulation but not the latter (3, 5 and 10-day trees). This residual toxicity of the Triton® X100 formulation can still be considered as good. Although no significant problems with the residual toxicity of these Triton® X100 formulations are foreseen, firm conclusions cannot be made until further tests directly comparing these formulations with standard formulations including aqueous ones are performed.

## Reference

Nigam, P.C. 1975. Chemical insecticides. In Aerial Control of Forest Insects in Canada, M.L. Prebble, Ed., Dept. of the Environment, Ottawa, Canada.

Table 1. The contact toxicity of technical Sumithion® aqueous formulations to spruce budworm larvae.

| Formulation | No. expts | Hr post | Slope | µg AI /cm² | | | |
|---|---|---|---|---|---|---|---|
| | | | | $LD_{50}$ | FL | $LD_{95}$ | FL |
| Triton® X100 | 6 | 24 | 3.48 | .686 | .578-.898 | 2.036 | 1.353-5.003 |
| | | 48 | 5.95 | .485 | .465-.505 | .916 | .851-1.002 |
| | | 72 | 6.11 | .465 | .446-.484 | .865 | .807- .941 |
| Triton® X100 + Cyclosol 63 | 3 | 24 | 4.49 | .460 | .430-.493 | 1.071 | .939-1.273 |
| | | 48 | 7.59 | .355 | .333-.375 | .586 | .543- .650 |
| | | 72 | 7.48 | .345 | .322-.365 | .572 | .529- .636 |
| Triton® X114 | 4 | 24 | 4.17 | .579 | .540-.622 | 1.436 | 1.226-1.796 |
| | | 48 | 5.45 | .466 | .437-.495 | .935 | .851-1.055 |
| | | 72 | 5.81 | .456 | .428-.482 | .875 | .804- .974 |
| Triton® X114 + Cyclosol 63 | 3 | 24 | 4.77 | .518 | .480-.566 | 1.147 | .993-1.421 |
| | | 48 | 7.10 | .385 | .358-.410 | .657 | .603- .740 |
| | | 72 | 7.32 | .377 | .350-.401 | .633 | .582- .712 |
| Triton® X114 + Canola Oil | 3 | 24 | 2.64 | .636 | .451-1.659 | 2.672 | 1.228-93.800 |
| | | 48 | 2.85 | .509 | .458-.576 | 1.924 | 1.445-2.979 |
| | | 72 | 2.94 | .471 | .331-.785 | 1.707 | .934-23.260 |
| Atlox 3409F + Dowanol TPM | 3 | 24 | 4.64 | .491 | .458-.529 | 1.112 | .963-1.356 |
| | | 48 | 5.18 | .411 | .384-.439 | .854 | .766- .982 |
| | | 72 | 5.83 | .400 | .291-.486 | .766 | .602-1.479 |

Table 2. The contact toxicity of Matacil® 180F aqueous formulations to spruce budworm larvae.

| Formulation | No. expts | Hr post | Slope | μg AI/cm² | | | |
|---|---|---|---|---|---|---|---|
| | | | | $LD_{50}$ | FL | $LD_{95}$ | FL |
| Triton® X100 | 3 | 24 | 6.14 | .058 | .055-.062 | .108 | .096 .128 |
| | | 48 | 6.43 | .054 | .051-.057 | .097 | .088- .111 |
| | | 72 | 6.12 | .052 | .049-.055 | .096 | .087- .110 |
| Triton® X100 + Cyclosol 63 | 3 | 24 | 5.07 | .071 | .066-.076 | .149 | .131- .178 |
| | | 48 | 5.71 | .059 | .055-.063 | .114 | .103- .131 |
| | | 72 | 6.11 | .058 | .054-.061 | .107 | .097- .122 |
| 3% Triton® X114 | 4 | 24 | 3.66 | .075 | .070-.081 | .213 | .181- .261 |
| | | 48 | 4.22 | .064 | .060-.068 | .157 | .139- .183 |
| | | 72 | 4.24 | .057 | .053-.061 | .139 | .124- .160 |
| 14.5% Triton® X114 | 4 | 24 | 3.76 | .106 | .082-.544 | .291 | .152-58.390 |
| | | 48 | 3.75 | .096 | .087-.113 | .265 | .197- .444 |
| | | 72 | 3.78 | .094 | .085-.109 | .256 | .192- .418 |
| Atlox 3409F | 4 | 24 | 5.44 | .096 | .089-.105 | .192 | .161- .251 |
| | | 48 | 4.80 | .082 | .077-.088 | .180 | .154- .226 |
| | | 72 | 5.00 | .073 | .069-.078 | .155 | .136- .186 |

Table 3. The residual toxicity of aqueous formulations of technical Sumithion® and Matacil® 180F to spruce budworm larvae, 1982.

| Insecticide/ formulation | Host | Treat. date | Hr. exposure on foliage | % corrected mortality after indicated weathering period of treated plants | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | 0 | 1 | 3 | 5 | 10 Days |
| Sumithion®/ Triton® X100 | Balsam fir | 2.6 | 24 | - | - | 33 | 0 | 2 |
| | | | 48 | - | - | 80 | 0 | 8 |
| | | | 72 | - | - | 97 | 5 | 18 |
| | White spruce | | 24 | - | - | 15 | 0 | 0 |
| | | | 48 | - | - | 36 | 0 | 2 |
| | | | 72 | - | - | 51 | 0 | 1 |
| | Mean, Bf and Ws | | 72 | - | - | 74 | 3 | 10 |
| | Mean temp. (°C) | | | | | 7.1 | 9.9 | 11.7 |
| | Total rain (mm) | | | | | Nil | Nil | 27.3 |
| | Total sun (hr) | | | | | 36.8 | 65.2 | 108.2 |
| Sumithion®/ Triton® X100/ Cyclosol 63 | Balsam fir | 29.5 | 24 | 98 | 24 | 0 | 11 | 26 |
| | | | 48 | 100 | 84 | 21 | 36 | 36 |
| | | | 72 | 100 | 95 | 60 | 61 | 46 |
| | White spruce | | 24 | 93 | 19 | 2 | 3 | 0 |
| | | | 48 | - | 41 | 3 | 5 | 0 |
| | | | 72 | 100 | 51 | 5 | 5 | 0 |
| | Mean, Bf and Ws | | 72 | 100 | 73 | 33 | 33 | 23 |
| | Mean temp. (°C) | | | | 18.2 | 15.1 | 11.7 | 11.9 |
| | Total rain (mm) | | | | Nil | 1.7 | 5.8 | 9.1 |
| | Total sun (hr) | | | | 2.0 | 10.6 | 32.8 | 92.5 |
| Sumithion®/ Triton® X100/ Canola Oil | Balsam fir | 1.6 | 24 | - | - | 33 | 0 | 0 |
| | | | 48 | - | - | 64 | 4 | 0 |
| | | | 72 | - | - | 91 | 6 | 0 |
| | White spruce | | 24 | - | - | 7 | 21 | 0 |
| | | | 48 | - | - | 27 | 43 | 0 |
| | | | 72 | - | - | 31 | 71 | 0 |
| | Mean Bf and Ws | | 72 | - | - | 61 | 39 | 0 |
| | Mean temp. (°C) | | | | | 6.8 | 8.4 | 11.2 |
| | Total rain (mm) | | | | | 4.1 | 4.1 | 31.4 |
| | Total sun (hr) | | | | | 29.2 | 57.4 | 101.0 |
| Sumithion®/ Ethylene Glycol/ Dowanol TPM | Balsam fir | 15.6[1] 1.6[2] | 24 | 83 | 46 | 9 | 10 | 1 |
| | | | 48 | 100 | 81 | 38 | 29 | 6 |
| | | | 72 | 100 | 89 | 66 | 52 | 10 |
| | White spruce | | 24 | 64 | 5 | 0 | 0 | 0 |
| | | | 48 | 92 | 13 | 0 | 0 | 0 |
| | | | 72 | 98 | 21 | 0 | 0 | 0 |
| | Mean Bf and Ws | | 72 | 99 | 70 | 33 | 26 | 5 |
| | Mean temp. (°C) | | | - | 10.9 | 6.8 | 8.4 | 11.2 |
| | Total rain (mm) | | | - | 2.7 | 4.1 | 4.1 | 31.4 |
| | Total sun (hr) | | | - | 0 | 29.2 | 57.4 | 101.0 |
| Matacil®/ Triton® X100 | Balsam fir | 29.5 | 24 | 97 | 61 | 15 | 23 | 32 |
| | | | 48 | 100 | 82 | 43 | 49 | 74 |
| | | | 72 | 100 | 95 | 80 | 73 | 92 |
| | White spruce | | 24 | 85 | 37 | 17 | 16 | 13 |
| | | | 48 | 100 | 78 | 45 | 20 | 22 |
| | | | 72 | 100 | 92 | 60 | 52 | 26 |
| | Mean Bf and Ws | | 72 | 100 | 94 | 70 | 63 | 59 |
| | Mean temp. (°C) | | | | 18.2 | 15.1 | 11.7 | 11.9 |
| | Total rain (mm) | | | | Nil | 1.7 | 5.8 | 9.1 |
| | Total sun (hr) | | | | 2.0 | 10.6 | 32.8 | 92.5 |
| Matacil®/ ID585 | Balsam fir | 15.6[1] 2.6[2] | 24 | 98 | 98 | 82 | 29 | 62 |
| | | | 48 | 100 | 98 | 98 | 51 | 87 |
| | | | 72 | 100 | 100 | 100 | 88 | 97 |
| | White spruce | | 24 | 92 | 82 | 73 | 18 | 2 |
| | | | 48 | 100 | 100 | 83 | 33 | 5 |
| | | | 72 | 100 | 100 | 95 | 52 | 8 |
| | Mean Bf and Ws | | 72 | 100 | 100 | 98 | 70 | 53 |
| | Mean temp. (°C) | | | | 10.9 | 7.1 | 9.9 | 11.7 |
| | Total rain (mm) | | | | 2.7 | Nil | Nil | 27.3 |
| | Total sun (hr) | | | | 0 | 36.8 | 65.8 | 108.2 |

[1] 0 and 1 day trees treated on this date.
[2] 3, 5 and 10 day trees treated on this date.