DISTRIBUTION, PERSISTENCE AND TRANSLOCATION OF ORTHENE® IN SPRUCE TREES AFTER SIMULATED AERIAL SPRAY APPLICATION

by

K.M.S. Sundaram and W.W. Hopewell,

Chemical Control Research Institute,

Canadian Forestry Service - Environment Canada,

25 Pickering Place, Ottawa, Ont., Canada

K1A 0W3

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ABSTRACT

A sensitive GLC analytical technique has been developed and described for the extraction of Orthene® (acephate) and its metabolite Ortho 9006 from conifer foliage. Using this method, the dissipation rate of the insecticide on spruce foliage was determined after a simulated aerial spray application. Applied dosage of 0.28 kg AI/ha (4 oz AI/acre) yielded a mean initial deposit of 55.15 ppm on the foliage which decreased rapidly to 2.92 ppm within 5 days and disappeared completely after 32 days. The metabolite was found only in traces and the mechanism of dissipation is primarily through physical processes. There was no indication of any translocation of the chemical into the untreated parts of the foliage.

RESUME

Une technique utilisant la chromatographie en phase gaseuse a été développée et décrite pour l'extraction de l'Orthene® (acephate) et son métabolite Ortho 9006, du feuillage de conifère. En employant cette méthode le taux de dissipation de l'insecticide sur les feuillages d'épinettes fut déterminé après plusieurs arrosages aériens, le dosage appliqué de 0.28 kg /ha (4 oz AI/acre) donne un dépot initial de 55.15 ppm sur le feuillage pour ensuite diminuer rapidement à 2.92 ppm en moins de 5 jours et enfin disparaitre complètement après 32 jours. Le mètabolite fut trouvé seulement en quantités traces. Le mécanisme de disparition est du principalement aux processus physiques. Il n'y avait pas d'indication d'aucune translocation du composé dans les parties non traitées du feuillage.

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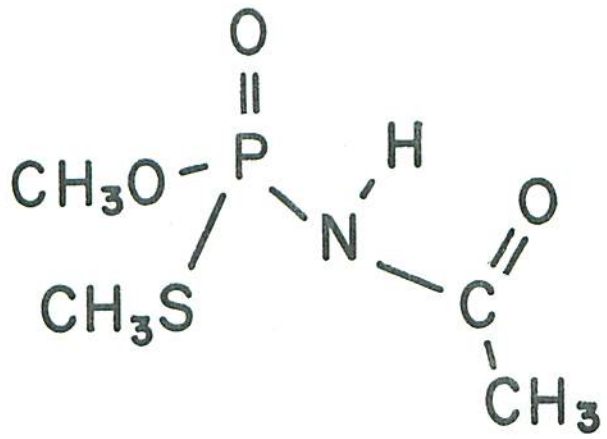
INTRODUCTION

Organophosphates have already gained an important place among the insecticides used for forest protection in Canada (Fettes 1968, Fettes and Buckner 1972, Fettes 1975, Anonymous 1975). Acephate (O,S-dimethyl acetylphosphoramidothioate) with the following structure:



and marketed under the trade name Orthene® * by the Chevron Chemical Company, is currently used on an experimental basis for the control of spruce budworm, *Choristoneura fumiferana* (Clemens), (Armstrong and Nigam 1975). Nigam and Hopewell (1973) have shown that field applications of Orthene on budworm infested individual white spruce [*Picea glauca* (Moench) Voss] trees at the rate of 0.36 kg AI/ha (5.2 oz AI/acre) resulted in 76% mortality of the insects. Hopewell (1975) under the same test conditions observed 93% budworm mortality at deposits of 0.3 kg AI/ha (4.25 oz AI/acre). Ground applications of Orthene to white spruce

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* Use of the abbreviation "Orthene" in this report refers to Orthene® Insecticide.

plantations at 0.56 kg AI/ha (8 oz AI/acre) gave a budworm population reduction of 98% after an interval of 10 days (DeBoo 1974). Recent studies (Armstrong and Nigam 1975) showed that budworm population reduction and foliage protection of white spruce was possible at a dosage of 0.21 kg AI/ha (3.0 oz AI/acre) in a volume of 4.67 ℓ/ha (0.5 gal (US)/acre).

Among the different criteria used in evaluating a candidate material for spruce budworm control, the important ones are the amount of the toxicant deposited on the coniferous needle, its persistence and toxicity. If an insecticide is labile and does not retain its activity for a minimum of about 3 days, it is considered to have too short a life to be effective in controlling forest insect pests and is seldom used in large operational spray programs (Armstrong and Nigam 1975).

The study reported here was designed with the following two objectives in mind:

(1) To obtain data on deposit and residue levels of Orthene and its persistence and rate of dissipation after application to white spruce trees. Such a study would enable personnel involved in pest management programs and other regulating agencies to evaluate and assess the overall efficacy and safety of the material in a correct perspective, and

(2) To examine the reported systemic nature of the insecticide as observed earlier by Lyon (1973) in his evaluation studies which, if confirmed in conifer foliar sprays under normal weathering conditions, would be an added advantage in forest

pest control.

MATERIALS AND METHODS

1. Experimental Design

The research work was conducted on a tree farm near Shawville, Quebec. Five spruce trees including one control selected for the experiment were of near uniform size and shape (2.0 to 2.5 m in height and 7.5 to 8.0 cm d.b.h.) with plenty of foliage. Trees numbered 1, 3, 4 and 5 were selected for the spray application and tree # 2 served as the untreated check. Four midcrown branch tips, 45 cm in length, one in each quadrant of each treated tree, were protected from direct spray deposit during application by enclosing them in plastic bags which were removed immediately after the treatment. Samples of exposed and protected foliage were taken at regular intervals after treatment for study of the possible systemic qualities of the chemical.

2. Insecticide Formulation

Two formulations of Orthene were tested. Both were made up to contain 10% (wt/vol) of active ingredient as follows:

1. Orthene technical (90% AI) 11.1 g made up to 100 ml in a solvent of water: ethylene glycol 9:1 by volume. One millilitre rhodamin B dye solution was added as a tracer for deposit and droplet density determinations.
2. Same as 1 above with 0.2% Atlas G 1249 (now Atplus® 555) spreader and sticker added.

Each formulation was tested in duplicate, i.e. two trees for each. Trees 3 and 5 were treated with solution 1 whereas trees 1 and 4

were sprayed with solution 2.

3. Application of the Insecticide

The application of the simulated aerial spray was carried out as described by Hopewell (1973) and Hopewell and Nigam (1974). A portable shelter enclosing an area of 2.1 x 2.1 m (7 x 7 ft) was placed around each tree to shelter it during application. Application was made at a nominal rate of 2.9 ℓ/ha (40 fl oz/acre) [0.28 kg AI/ha or 4 oz AI/acre] i.e. 1.3 ml of the formulation emitted over the 4.55 m^2 enclosed area.

Treatments were applied on May 15, 1975 from 0910 to 2130 hours. Budworm development was predominantly L_2 and L_3 in needle mining stage. Bud development had not at that time reached the point where larvae could feed on new foliage.

4. Sampling

Deposit samples were taken from each quadrant on glass slides and Kromekote® cards for the determination of deposited volume (ℓ/ha) and the droplet density (number/cm^2) respectively. Samples of the sprayed and covered foliage were taken at prespray, 0, 5, 10, 21, 32 and 53 days post treatment. A sample from each treated tree was a composite of clipped twigs from each quadrant to give *ca* 100 g foliage, one from sprayed and one from covered foliage, put in plastic bags and taken immediately in polystyrene coolers to the pesticide laboratory at the Chemical Control Research Institute (CCRI) and stored in deep freeze until time for analysis.

5. Analytical Methodology

a. Extraction

The foliage samples were cut into small pieces by scissors,

macerated in a Hobart chopper and 20 g aliquot of each sample was homogenized twice in a Sorvall blender for 5 minutes at speed 6 with 100 ml of ethyl acetate as extractant. The homogenate was filtered under suction using "S and S sharkskin" filter paper. After washing the residue with 20 ml portions of the extractant, it was discarded. The extracts of individual samples were pooled separately. passed through columns of Na_2SO_4 (50 g), which were rinsed with 2 x 25 ml of the solvent and flash evaporated to *ca* 10 ml. The concentrated extracts were transferred quantitatively to 250 ml separatory funnels along with 100 ml of CH_3CN and equilibrated with 3 x 25 ml of hexane. The non-polar phases were discarded and the polar ones were evaporated gently to dryness under vacuum using a rotary evaporator.

b. Cleanup Procedure

The chromatographic adsorbent column cleanup of the samples was carried out by dissolving each sample residue in 4 x 25 ml of diethyl ether and passing it through a Shell type glass column (26 cm x 2 cm) containing 15 g of E. Merck silica gel (0.05 - 0.22 mm extra pure 70-325 mesh ASTM for column chromatography) sandwiched between 10 g Na_2SO_4 and prewashed with 50 ml ether. The columns were first eluted with 100 ml 5% CH_3OH in ether and all the eluates collected to this point were discarded. The final eluations were done by using 250 ml of 10% CH_3OH in ether which removed the residues

of Orthene and its metabolite Ortho 9006 ® + from the column. These eluates were flash evaporated gently to dryness, the residues were dissolved in methyl isobutyl ketone* (MIK) and analysed by GLC. An aliquot (10 g) was taken for moisture determination (AOAC 1955).

c. Gas-liquid Chromatographic (GLC) Conditions

GC	:	HP Model 810
Detector	:	FPD (P-mode)
Column	:	46 x 0.64 cm (I.D.) coiled Teflon, 1% Reoplex - 400 on Gaschrom Q (100/200 mesh)
Temp (°C)		
(Program)		
Oven-initial	:	130 (Hold 1 min)
Oven-final	:	180 (Hold 4 min)
Rate of increase	:	15 / min
Elapsed time/sample	:	8 min 20 s
Detector	:	200
Inlet	:	200
Carrier gas (ml/min)	:	Nitrogen - 40
Gas Flow (ml/min)	:	H_2 - 150
	:	Air - 75
	:	O_2 - 12

+ O,S-dimethyl phosphoramidothioate. Ortho 9006 is the registered trade name of Chevron Chemical Co. for the material.

* Later it was found ethyl acetate to be a better solvent than MIK in GLC analysis.

Attenuation	:	32
Range	:	10^3
Recorder	:	Linear Instruments
	:	Span 1 MV
Chart Speed (cm/hr)	:	41
Retention time (min)	:	Ortho 9006 4.28
	:	Orthene 5.91
Relative retention time	:	Ortho 9006 0.72
	:	Orthene 1.00

d. Standardization of GLC

The gas chromatograph was standardized on the same day as the foliage samples were analysed by injecting aliquots (2-4 µl) of freshly prepared standard solutions in ethyl acetate containing Orthene and its metabolite, measuring the peak heights and preparing a calibration curve by plotting peak heights (in centimetres) *vs* weight (in nanograms) of the materials. The foliar extracts were either diluted with MIK or concentrated to bring the amount of the insecticides injected within the linear range of the detector. Quantitative results of the extracted samples were obtained by measuring each of the peak heights after injections (2-4 µl) under the same operating conditions and reading the concentrations from the calibration curves.

The chromatograms of Orthene and its metabolite, O,S-dimethyl phosphoramidothioate, (Ortho 9006), were narrow and symmetrical with good reproducibility and absence of additional peaks.

e. Recovery Studies

The method described has been used to extract and quantify spiked spruce foliage samples containing the insecticide and its metabolite. The average percent recoveries for both compounds from 20 g aliquots of foliage were satisfactory and the results are given below.

Fortification Level (ppm)		Average % Recovery*		Coefficient of variation %	
Orthene	Ortho 9006	Orthene	Ortho 9006+	Orthene	Ortho 9006
0.25	0.25	87	82	6	4
0.50	0.50	92	85	6	3

6. Solvents and Chemicals

All solvents used were of pesticide grade. The silica gel** adsorbent was of chromatographic grade (70-325 mesh) obtained from E. Merck, A.G. Darmstadt (Germany). The anhydrous sodium sulphate (Fisher, S-421) was of reagent grade, heated at *ca* 150°C overnight and stored in a glass-stoppered bottle.

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* Each value represents the average of three analytical replicates.

\+ About 5-10% of the fortified Ortho 9006 was lost in the first column eluate (100 ml 5% CH_3OH in ether).

** The commercial sample was found to contain *ca* 2.5% water and used as received.

Table I

Spray Deposit and Droplet Density Found on Samples of Deposit from each Test Tree

Tree No.	Formulation Used*	Average+ Spray Deposit		Average Number Drops/cm^2	Calc. Avg. Drop Diam. (μ)
		ℓ/ha	(fl. oz/acre)		
1	Solution # 2	2.8	(38)	30	121
3	Solution # 3	2.5	(34)	6.5	194
4	Solution # 2	1.3	(18)	12	103
5	Solution # 1	0.9	(12)	3.2	141

* See under "Materials and Methods" - Insecticide Formulations.

\+ Average for the four quadrants.

Table II

Orthene and Ortho 9006 Residues in Foliage Samples from Tree # 1

Days After Application	Moisture Content %	Residue Concentration (ppm)					
		As Sampled			Oven Dried		
		Orthene	Ortho 9006	Total	Orthene	Ortho 9006	Total
Prespray	42	-	-	-	-	-	-
0*	43	38.5	0.10	38.6	67.54	0.17	67.71
5	33	3.43	0.16	3.59	5.11	0.17	5.28
10	54	0.19	N.D.	0.19	0.42	N.D.	0.42
21	64	0.06	N.D.	0.06	0.16	N.D.	0.16
32	64	0.01	N.D.	0.01	0.03	N.D.	0.03
53	59	N.D.	N.D.	-	N.D.	N.D.	-

* Sample taken two hours after application.

N.D.= Not Detectable

Table III

Orthene and Ortho 9006 Residues in Foliage Samples from Tree # 3

Days After Application	Moisture Content %	Residue Concentration (ppm)					
		As Sampled			Oven Dried		
		Orthene	Ortho 9006	Total	Orthene	Ortho 9006	Total
Prespray	42	-	-	-	-	-	-
0*	40	26.00	0.06	26.06	43.33	0.09	43.42
5	29	2.97	0.09	3.06	4.18	0.13	4.31
10	59	0.09	N.D.	0.09	0.22	N.D.	0.22
21	63	0.04	N.D.	0.04	0.12	N.D.	0.12
32	59	0.01	N.D.	0.01	0.03	N.D.	0.03
53	53	N.D.	N.D.	-	N.D.	N.D.	-

* Sample taken two hours after application.

N.D. = Not Detectable.

Table IV

Orthene and Ortho 9006 Residues in Foliage Samples from Tree # 4

Days After Application	Moisture Content %	Residue Concentration (ppm)					
		As Sampled			Oven Dried		
		Orthene	Ortho 9006	Total	Orthene	Ortho 9006	Total
Prespray	44	-	-	-	-	-	-
0 *	47	33.00	0.07	33.07	62.26	0.13	62.39
5	26	0.63	0.11	0.74	0.65	0.11	0.76
10	49	0.14	N.D.	0.14	0.27	N.D.	0.27
21	61	0.06	N.D.	0.06	0.14	N.D.	0.14
32	67	T	N.D.	T	T	N.D.	T
53	57	N.D.	N.D.	-	N.D.	N.D.	-

* Sample taken two hours after application.

N.D. = Not Detectable.

T = Trace (< 0.01 ppm).

Table V

Orthene and Ortho 9006 Residues in Foliage Samples from Tree # 5

Days After Application	Moisture Content %	Residue Concentration (ppm)					
		As Sampled			Oven Dried		
		Orthene	Ortho 9006	Total	Orthene	Ortho 9006	Total
Prespray	40	-	-	-	-	-	-
0*	41	28.00	0.07	28.07	47.45	0.11	47.56
5	41	1.03	0.06	1.09	1.74	0.11	1.85
10	63	0.19	N.D.	0.19	0.31	N.D.	0.31
21	63	0.05	N.D.	0.05	0.12	N.D.	0.12
32	60	0.01	N.D.	0.01	0.01	N.D.	0.01
53	56	T	N.D.	T	T	N.D.	T

* Sample taken two hours after application.

N.D. = Not Detectable.

T = Trace (< 0.01 ppm).

Table VI

Total Insecticide (Orthene + Ortho 9006) Levels Found in Foliage Samples from the Four Treated Trees

Days After Application	Residue Concentration - ppm & (% of original) Total as Sampled					Residue Concentration - ppm & (% of original) Total Oven Dried				
	Tree 1 +	Tree 3	Tree 4 +	Tree 5	Mean	Tree 1 +	Tree 3	Tree 4 +	Tree 5	Mean
Prespray	-	-	-	-	-	-	-	-	-	
0 *	38.60	26.06	33.07	28.07	31.45	67.71	43.42	62.39	47.56	55.27
5	3.59 (9.3)	3.06 (11.7)	0.74 (2.2)	1.09 (3.9)	2.12 (6.7)	5.28 (7.8)	4.31 (9.9)	0.76 (1.2)	1.85 (3.9)	3.05 (5.5)
10	0.19 (0.5)	0.09 (0.3)	0.14 (0.4)	0.19 (0.7)	0.15 (0.5)	0.42 (0.6)	0.22 (0.5)	0.27 (0.4)	0.31 (0.6)	0.31 (0.6)
21	0.06 (0.16)	0.04 (0.15)	0.06 (0.18)	0.05 (0.18)	0.05 (0.16)	0.16 (0.24)	0.12 (0.28)	0.14 (0.22)	0.12 (0.25)	0.14 (0.25)
32	0.01 (0.02)	0.01 (0.04)	T	0.01 (0.04)	0.01 (0.03)	0.03 (0.04)	0.03 (0.07)	T	0.01 (0.02)	0.02 (0.04)
53	-	-	-	T	-	-	-	-	T	-

* Sample taken 2 hours after application.

\+ Atplus 555 in formulation.

T = Trace (< 0.01 ppm).

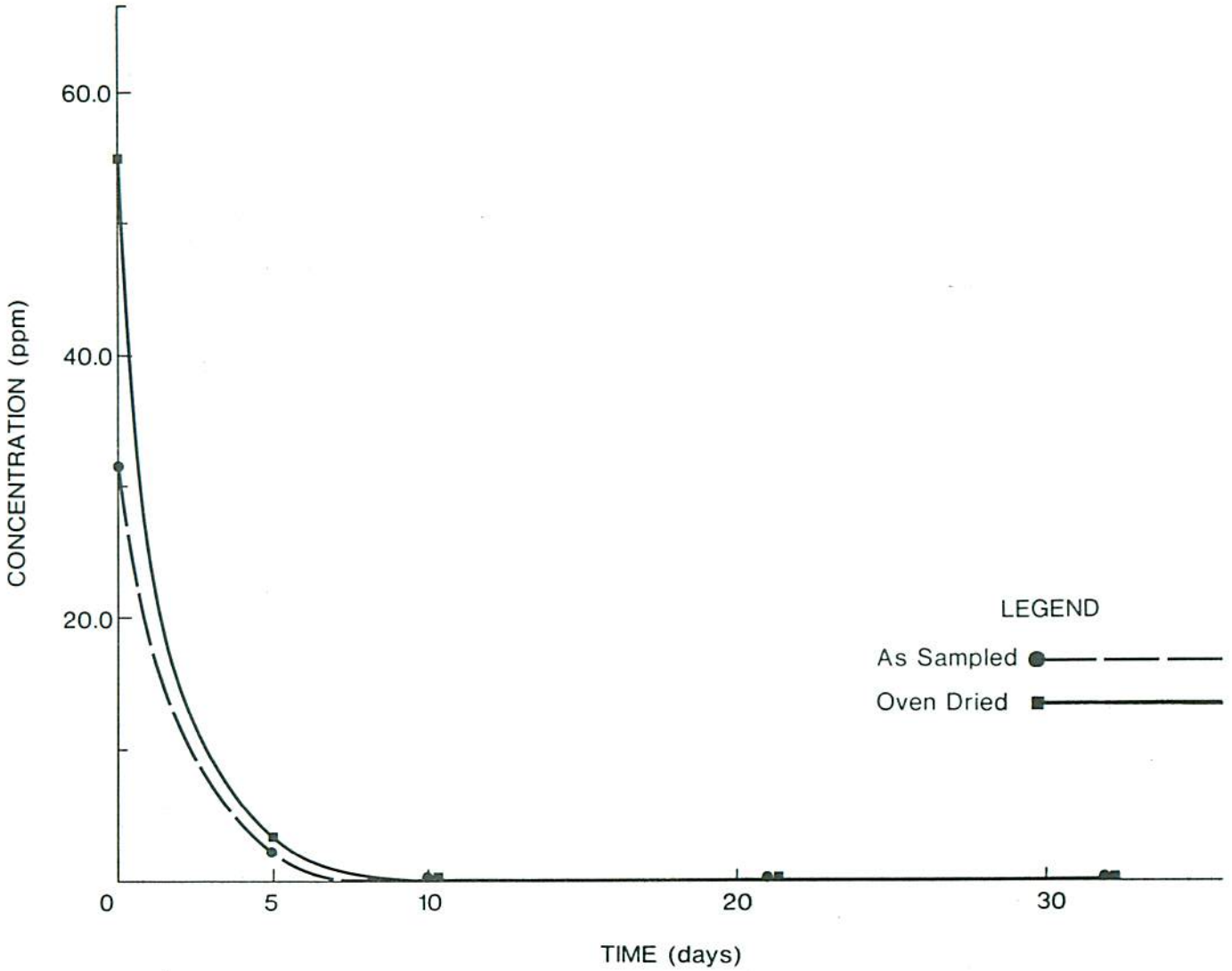

Fig. 1 Dissipation of Orthene residues in spruce foliage after simulated aerial application.

RESULTS AND DISCUSSION

1. Analytical Methodology

Recoveries of Orthene and Ortho 9006 in spruce foliage obtained from the check tree and spiked with the known amounts of analytical grade materials were 90±6 and 84±4% respectively with a minimum detection limit of 0.01 ppm for 20 g substrate. The low recovery of the metabolite was due to its loss (5-10% of the fortified amount) in the first column eluate (100 ml of 5% CH_3OH in ether). The GLC responses to the standards were sharp, symmetrical and well defined with retention times of 4.28 and 5.91 minutes, for Ortho 9006 and Orthene, respectively. The background interference in the chromatogram of the foliage fortified with the toxicants was small, showing that the extraction, partition, column cleanup and final quantitation methods employed in this study were excellent. Frequent replacement of the Reoplex column was found to be necessary to maintain the analytical precision, and the temperature programming panel of the HP 810 instrument often failed to respond properly, creating considerable delay in the overall analysis. Apart from these minor difficulties, the program went smoothly and yielded useful data.

2. Distribution of Spray Deposit and Droplet Density

The average spray deposit and droplet density found on the sampling units are given in Table I. The deposit levels ranged from 0.9 to 2.5 ℓ/ha (12 to 34 fl. oz/acre) averaging 1.86 ℓ/ha (25.5 fl. oz/acre). The values are unusually low (average deposition level 64%) for a nominal emission rate of 2.9 ℓ/ha (40 fl. oz/acre) probably due to overshadowing of the sampling units by foliage. It is worthwhile to note that the second formulation containing the adjuvant Atlas G.1249 (now Atplus ®

555) sprayed on trees 1 and 4 gave 22% more deposition on the sampling units than the first one. Similarly, a higher average droplet density, i.e. 4.9 *vs* 21 (finer droplets) was observed from solution 2 containing the adjuvant as shown by the more efficient coverage in drops per square centimetre. The additive, being a surfactant, reduced the surface tension of the formulation, thereby reducing the droplet size and increasing the droplet density.

3. Distribution of Orthene Residues

Amounts of Orthene residues in ppm found in the foliage of sprayed spruce trees are recorded in Tables II to VI. The concentration levels are expressed in ppm units on moist weight (as sampled) basis including the variables such as water and volatile components for the purpose of ecological interpretations under actual field conditions and also on oven-dry weight basis representing samples containing less water and readily volatile components so as to standardize residue levels for easy comparison between different foliage samples analysed. The oven-dry results which are more consistent are used in the present study to facilitate comparisons and interpretations of the residue data. The data presented in Tables II to VI do not include correction for % recovery from the spiked samples mentioned earlier.

The check and prespray foliage samples as well as the samples taken from the protected branches of the sprayed trees by enclosing in plastic bags did not contain any detectable insecticide residues and are not recorded in this publication.

The results in Tables II to V show that after 2 hours of spray application, the concentration of Orthene and Ortho 9006 in the spruce foliage ranged from 43.33 to 67.54 ppm (average 55.15 ppm) and 0.09 to

to 0.17 ppm (average 0.12 ppm) respectively. The average concentration of the metabolite found in the day zero samples compared to the parent material was extremely low (*ca* 0.22%). After an interval of 5 days, the concentration of Orthene and the metabolite in the substrate ranged from 0.65 to 5.11 ppm (average 2.92 ppm) and 0.11 to 0.17 ppm (average 0.13 ppm) respectively. During this interval, the Ortho 9006 concentration remained nearly constant and after 5 days, the transient metabolite apparently due to its low persistence in spruce needles, disappeared completely from all the foliage samples analysed. It is also interesting to note that within a short interval of 5 days, the average initial concentration of the insecticide decreased rapidly from 55.15 to 2.92 ppm, showing a loss of 94.7% and persisted only in detectable quantities at 0.31, 0.14 and 0.02 ppm respectively on 10, 21 and 31 day intervals.

Table VI shows the total insecticide residue (Orthene + Ortho 9006) present in or on the foliage of the four treated trees at various times post-treatment. Trees 1 and 4, treated with solution # 2 containing 0.2% Atplus 555, and trees 3 and 5 treated with solution # 1, all showed approximately the same rate of loss of insecticide.

The foliage of trees 1 and 4 sprayed with the solution containing the adjuvant Atplus 555 received a better coverage and higher deposition (65.05 *vs* 45.49 ppm or 43% more) of the active material than the foliage of the other two trees. Distribution of spray drops and deposits of active ingredients retained on foliar surfaces are directly related to stability of formulations, amount and nature of surfactants and droplet size of spray mixture.

A preliminary laboratory test performed for the measurement of these properties could be useful in enhancing the efficacy of the chemical before the field trials are conducted.

The average deposit level (55.27 ppm) found on the spruce foliage by the simulated aerial spray application technique is approximately 16 times higher than that resulting from the conventional aerial application techniques used for other organophosphorus insecticides (Yule and Duffy 1972, Sundaram 1974, Varty and Yule 1976). The variation of the deposit levels among the individual trees sprayed by the technique developed by Hopewell (1973) is minimal compared to aerial applications and the method is extremely useful in evaluating the efficacy of different insecticide materials under forest environmental conditions with minimum interference from existing meteorological conditions at the time of application.

4. Persistence of Orthene Residues in Spruce Foliage

Orthene residues in the foliage samples decreased very rapidly with time. More than half the amount was lost within a day, and after 32 days it had diminished to negligible amounts. The Ortho 9006 metabolite was found in small amounts (*ca* 0.12 ppm) up to 5 days after the spray application (Tables II-V; the maximum concentration being *ca* 0.2% of the parent compound. Magee (1974) also reported that the formation of Ortho 9006 was found to be low (3.7 to 4.8%) when the insecticide was topically applied to pinto bean leaves.

A plot of the average concentration (ppm) of the insecticide residues present in foliage *vs* time (days) showed (Fig. 1) a uniform but steep curvilinear decrease. Taking the initial concentration as 55 ppm,

the half-life for the insecticide from the dissipation curve was *ca* 0.8 day. The extremely rapid loss of Orthene and the absence of significant amounts of the metabolite Ortho 9006 indicates that the effective mechanism of dissipation of the insecticide in spruce foliage under the experimental conditions studied, was primarily due to various environmental factors such as temperature (volatilization, photodegradation) and weathering action of humidity, rain and wind rather than metabolic breakdown by plant micro-organisms. However, no rain was recorded in the interval before the 5 day sample. In addition to these factors, the chemical characteristics of the insecticide including its over-all stability either as the parent material or the metabolite (Ortho 9006), its volatility, solubility, formulation and the method and site of application also influence its persistence. The rapid dissipation of Orthene compared to other organophosphates (Yule and Duffy 1972, Sundaram 1974, Varty and Yule 1976) suggests that in addition to physical, and to a lesser extent, metabolic degradation, the physico-chemical and structural properties of Orthene are exerting considerable influence on its rapid dissipation from the substrate thus increasing its usefulness as an ecologically acceptable insecticide.

Since the insecticide is rather labile, for a satisfactory budworm population reduction and foliar protection, a higher dosage level e.g. 0.56 kg/ha (8 oz AI/acre) used by DeBoo (1974) would seem to be useful in spray programs for efficient forest pest management. At that level, the possibility of the chemical being present at the site of action in sufficient quantities and for a sufficient length of time to provide effective control would be high. Magee (1974) reported that the material is effective on crops at 0.56 to 1.12 kg AI/ha (8 oz to 1 lb. AI/acre). Possibly the addition of various adjuvants, e.g. surfactants, U.V. light

protectors, stickers etc. also might prolong its activity and increase its effectiveness in insect control operations. In general, insect mortality depends on the magnitude of the concentration level deposited on foliage, the toxicity of the compound and the duration of persistence of the insecticide exposed to the target organism.

5. Translocation of Orthene

Lyon (1973) reported Orthene to be systemic in its action in certain cases, e.g. when applied to soil or as a basal trunk spray. In the present investigation, all the covered foliage samples collected at different intervals of time after the foliar spray application were analysed for residues of Orthene and Ortho 9006. None of the samples contained any detectable levels (0.01 ppm) of the insecticide indicating that no translocation of the material had occurred when the chemical was applied as a foliar spray at a dosage level of 0.28 kg AI/ha (4 oz AI/acre) under normal weathering conditions. In addition to the rapid dissipation of the material, the toxicant molecules probably did not penetrate the waxy barrier present on the cuticular surface into the foliage to reach the xylem-phloem transport stream for adequate translocation of the material. Current investigations undertaken by the authors using C-14 labelled Orthene will throw more light on the systemic properties of the chemical in coniferous trees under forest environmental conditions.

SUMMARY AND CONCLUSIONS

Orthene® (O,S-dimethyl acetylphosphoramidothioate) insecticide was applied at a dosage of 0.29 kg AI/ha (4 oz AI/acre) to white spruce [*Picea glauca* (Moench) Voss] trees as simulated aerial spray under normal

weathering conditions. Foliage samples were collected from the trees at various time intervals for residue analysis.

A sensitive gas-liquid chromatographic (GLC) method is reported for determining the residues of Orthene and its metabolite Ortho 9006® (O,S-dimethyl phosphoramidothioate). Homogenization of foliage and extraction by ethyl acetate yielded good recovery of the residues. The cleanup procedure involved hexane partition, concentration followed by silica gel column chromatography and quantitation by flame photometric GLC in phosphorus mode. Average recoveries of Orthene and Ortho 9006 from spiked foliage samples were above 80% with minimum detection limit of 0.01 ppm.

Distribution and persistence of Orthene and Ortho 9006 in spruce foliage collected at intervals after the simulated aerial application were studied. The average zero day concentration of the insecticide was 55.15 ppm; being labile, 95% of it was lost within 5 days. The residues persisted in small but detectable amounts up to 32 days. From the dissipation curve, the approximate time for 50% disappearance (half life) of the insecticide was found to be 0.8 day. The Ortho 9006 metabolite was found only in traces for a short period. The mechanism of dissipation of the insecticide appeared to be due to physical factors (volatilization, weathering, decomposition by light and hydrolysis) rather than metabolic processes. Under the experimental conditions, the material was not found to be systemic.

Addition of Atplus ^{R} 555 spreader/activator adjuvant to the formulation (0.2%) had no apparent effect on increasing absorption of Orthene into foliage or influencing its rate of dissipation; however, it did

improve coverage by breakup of the spray into smaller drops under identical conditions of application.

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