SESSION VIII

NEW DEVELOPMENTS, INCLUDING NEW COMPOUNDS, NOVEL FORMULATIONS, OR NEW APPLICATION METHODS

Chairman: J. T. Martin
Long Ashton Research Station

INSECTICIDE GROUP

SYSTEMIC MIGRATION AND INSECTICIDAL ACTIVITY OF DIMETHOATE APPLIED ON TREE TRUNKS

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Introduction

Defending plants from pests by endotherapic means is a problem which researchers have been trying to solve for a long time; but the answer has not yet been found despite the discovery of many organic insecticides with systemic properties. With the exception of the application to seeds and to the soil (methods which can be used only with vegetable crops), these substances are applied in relatively low concentration by the traditional technique of spraying or dusting the foliage of trees. The systemic properties are therefore exploited only partially for the active ingredient reaches a sufficient dosage only in certain parts of the leaves, in the less deep layers of the endocarp of the fruit, and in some portions of the bark of the branches and twigs (de Pietri-Tonelli and Barontini, 1961a, d). Because systemic insecticides migrate in plants mostly through the xylem, that is in an acropetal direction, a complete and rational utilization of the systemic properties would necessitate the application of the product to the roots or trunk.

Bypassing the application to the roots, which especially in the case of trees presents some disadvantages, let us examine the trunk application. The vast literature on this subject has been reviewed, among others, by Craighead and St. George (1938), Ripper (1957) and more recently, by Mitchell et al. (1960). The following are the advantages (partly already indicated by Jeppson et al. (1952) which might derive from the unusual application of the insecticide to the trunk of trees:

a) the possibility of distributing the product, by internal translocation, to every part of the plant, even to parts which can hardly be reached by other means;

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b) a simplification of the application technique and, possibly, independence of the availability of water and of certain meteorological conditions which are unfavourable with other means of application;
c) a reduction of antithesis between chemical and biological control owing to the absence of external toxic residues on large vegetable surfaces;
d) a reduction of the danger of contaminating cover crops and a lesser risk for the operator.

Although there are a few negative aspects or such that might be considered so (such as the difficulty in establishing easily the quantities of the product to apply to each plant; difficulties of the application technique; fear that the toxic residue variation may be unpredictable) we believe that the fundamental obstacle is created by the insufficient insecticidal activity of the products tested, or more often, by adverse effects on plants.

For the last few years, we have been making a basic study of the effects of the application of insecticides to the trunk and stem of plants (de Pietri-Tonelli and Barontini, 1961c; Santi, in prep.) and although we have achieved no conclusions of immediate application we are now reporting on the results of our work in the hope that they may contribute to the solution of the problem.

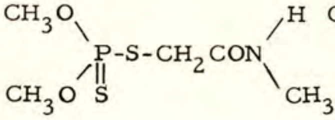
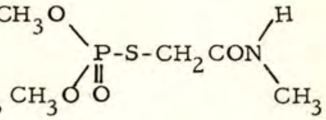
Materials and Methods

In this work we employed dimethoate (OO-dimethyl S-methylcarbamoylmethyl phosphorodithioate) and, in many experiments, also its P=O derivative (Table 1) which is its principal active metabolite (Santi and Pietri-Tonelli, 1959; 1960; Dauterman et al., 1959; 1960).

Unless otherwise indicated, the two products were used at 50% (w/v) in tri-n-butylphosphate (TBF) a solvent which permits highly concentrated solutions of the active ingredient. The solutions were applied to the plants by painting the tree trunk for varying widths (from 20 to 40 cm.) downward from the branches. Since it was necessary to carry out the experiments under natural conditions, with particular regard to the size of the plant, researches were conducted on trees in open fields, except in certain tests for phytotoxicity.

In order to determine the concentration of the product applied and of its possible metabolites having insecticidal properties, samples of leaves and fruit were bioassayed with Drosophila melanogaster at various intervals of time after application (de Pietri-Tonelli and Barontini, 1961b). In some cases P^{32}-labelled dimethoate (initial specific activity 1.43 mc/mM) was applied to the plants, which made it possible to determine, through radioanalysis and autoradiography the concentration of some of the transferred substances and the distribution of all of those which had in common the radioactive phosphorus of dimethoate (de Pietri-Tonelli and Barontini, 1961 a, c, d).

Table 1. Chemical, physical and biological properties of dimethoate and its P=O derivative.

	DIMETHOATE	P=O DERIVATIVE
Structural formula		
Water solubility	2.5 : 100	∞
Hydrolysis at 21° C Half life at pH 9	140 hours	7 hours
" " " pH 4	no hydrolysis after 100 hours	no hydrolysis after 100 hours
LD 50 - mouse orally (mg/Kg)	200-250	27
I 50 - bovine cholinesterase	7.63×10^{-3} M.	3.91×10^{-5} M.
I 50 - fly-head cholinesterase	1.50×10^{-4} M.	1.71×10^{-7} M.

Effects of the Treatment on Insects and Mites

Citrus - Both dimethoate and the P=O derivative were applied at various dates and localities, on lemon, orange and tangerine trees, all heavily infested with scales in all stages. Both the leaves and the fruit were freed from Chrysomphalus dictyospermi, Mytilococcus Beckii and the fruit from Pseudococcus citri. Only a partial effect was obtained on Coccus oleae and a very slight effect on Aspidiotus haederae (Table 2). The application of the two products was completely effective on lemon plants badly infested with Tetranychus telarius (Table 3). However both products were practically useless against Aceria sheldoni on lemons, and exerted an inadequate control of adults of T. telarius which had been artificially transferred on leaves from orange trees previously treated (Table 3).

In Sicily where the two insecticides were applied to orange plants while the fruit was still green, we noticed that all the newborn larvae of Ceratitis capitata (which in Sicily die before penetrating the pulp, but which in other localities do irreparable damage to the pulp) died before leaving the egg-cavities in the outer portion of the peel (flavedo), while on the untreated plants the larvae penetrated into the inner colorless portion of the peel (albedo) (Table 4).

Olive - The treatment was effective on *Dacus oleae* larvae when the fruit was artificially infested in two different seasons (Table 5). An excellent control was obtained in November on leaf mining larvae of *Prays oleellus* (Table 5), while winter applications, when the larvae had already started to damage the leaves, were completely ineffective.

Apple - A rather inefficient control of *Metatetranychus ulmi*, and an incomplete effect on *T. telarius* were obtained with both products on leaves artificially infested with mites and taken from trunk treated plants in two experiments carried out on the same days in different localities (Table 6). A preblossom trunk application of dimethoate was completely ineffective against *M. ulmi*.

Table 2. Citrus. Toxicity of leaves and fruit to citrus scales and mealybugs after trunk applications of dimethoate and P=O derivative.

Plants	Active Ingredient (grams per tree)	Dates of applications and localities	Days after treatment	Average No. of Live Scales					
				Check		dimethoate		P=O derivative	
				per leaf	per fruit	per leaf	per fruit	per leaf	per fruit
Lemon[a]	10	Sept. 16 Catania	<u>Aspidiotus hederae</u> (adult females)						
			40	9.5	4.8	12.1	5.6	6.5	0.4
			80	44.0	48.4	20.1	4.0	16.1	0.0
Lemon[a]	20	Sept. 22 Catania	<u>Pseudococcus citri</u> (all stages)						
			4	-	30.5	-	15.5	-	21.5
			16	-	45.6	-	5.3	-	3.2
			32	-	32.3	-	0.4	-	0.1
Orange[a]	20	Aug. 17 Catania	<u>Chrysomphalus dictyospermi</u> (adult females)						
			32	38.2	-	0.9	-	0.3	-
			42	58.2	> 1,800	0.2	0.0	0.1	0.0
			69	44.2	> 1,600	0.0	0.0	0.0	0.0
			74	42.6	> 2,000	0.0	0.0	0.0	0.0
Orange[b]	5	Sept. 8 Orbetello	<u>Mytilococcus Beckii</u> (adult females)						
			29	6.5	42.5	0.0	0.0	1.3	0.0
			51	6.1	76.0	0.0	0.0	1.5	0.0
Orange[b]	2.5	Oct. 7 Orbetello	<u>Coccus oleae</u> (2nd and 3rd instar)						
			22	22.9	78.8	0.9	0.1	0.7	0.0
Tangerine[a]	25	Sept. 15 Catania	<u>Pseudococcus citri</u> (all stages)						
			8	-	145	-	8.3	-	1.2
			18	-	176	-	0.2	-	0.0
			32	-	67	-	0.0	-	0.0
Tangerine[b]	2.5	Oct. 7 Orbetello	<u>Mytilococcus Beckii</u> (adult females)						
			22	15.2	24.4	0.1	0.1	0.0	0.0
			Coccus oleae (2nd and 3rd instar)						
			22	18.5	21.6	13.4	8.8	0.6	0.4

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[a] - Average height : 3 meters

[b] - Average height : 1.5 meters

Table 3. Lemons and oranges. Toxicity of leaves and fruit to _Tetranychus telarius_ after trunk applications of dimethoate[a] and P = O[a] derivative.

Plants	Dates (1959) of applications and localities	Days after treatment	Effect on Mites			
			dimethoate		P=O derivative	
			leaves	fruit	leaves	fruit
			Percent reduction of infested leaves on fruit			
Lemon[b]	Sept. 22 Catania	4	58	0	85	50
		16	99.5	100	100	100
		32	100	100	100	100
			Percent mortality of mites[c]			
Orange[b]	Aug. 17 Catania	4	98.5	-	99.0	-
		8	98.8	-	99.0	-
		16	99.8	-	96.5	-
		32	32.0	-	60.0	-

[a] - 20 grams per tree

[b] - Average height : 3 meters

[c] - On detached leaves infested with adult females of _T. telarius_

Table 4. Oranges. Toxicity of fruit to newly hatched larvae of _Ceratitis capitata_ after trunk applications of dimethoate[a] and P = O derivative[a].

Treatment[b]	Percent Dead Larvae in the Ripe Fruit[c]		
	flavedo (in the egg cavities)	albedo	total
Dimethoate	100	0	100
P = O derivative	100	0	100
Check, no treatment	39.1	54.8	93.9

[a] - 8 grams per tree; average height : 2.5 meters

[b] - Date and locality of application : October 13, 1959, at Catania.

[c] - 32 days after treatment.

Table 5. Olives. Toxicity of fruit and leaves to largae of Dacus oleae and Prays oleellus respectively, after trunk or branch applications of dimethoate and P=O derivative.

Active Ingredient (grams)	Dates (1959) of applications and localities	Days after treatment	Percent Dead Larvae in the Fruit or in the Leaves	
			dimethoate	P=O derivative
			Dacus oleae[c] (in the fruit)	
10 (per branch of the same tree[a])	Jul. 27	44	99	87
	Grosseto	64	100	43
		85	51	5
			Dacus oleae[c] (in the fruit)	
20 (per tree[b])	Sept. 7	11	76	95
	Firenze	18	78	100
		23	81	100
		29	100	100
		37	89	98
		44	83	100
		66	77	92
			Prays oleellus (in the leaves)	
20 (per tree[b])	Nov. 21	32	65	96
	Firenze	76	99.5	100

[a] - Average height : 3.50 meters; "Ascolana" variety (eating-olives).

[b] - Average height : 3.50 meters; "Frantoio" variety (oil-olives).

[c] - On fruit samples picked and artificially infested with D. oleae.

Table 6. Apples. Toxicity of leaves to Tetranychus telarius and Metatetranychus ulmi after trunk applications of dimethoate and P=O derivative

Active Ingredient (grams per tree)	Dates (1959) of applications and localities	Days after treatment	Percent Dead Mites (adult females) on Detached Leaves	
			dimethoate	P=O derivative
			Tetranychus telarius	
30[a]	May 26 Bologna	8	48	80
		15	32	63
			Metatetranychus ulmi	
		8	70	83
		15	48	46
			Metatetranychus ulmi	
10[b]	May 27 Firenze	3	25	-
		5	30	-
		12	90	-
		36	10	-
			Tetranychus telarius	
		5	90	-
		12	100	-
		36	81	-

[a] - Average height of trees : 6 meters.
[b] - Average height of trees : 3 meters.

Pear - Dimethoate, applied on the trunk, inhibited the development of the C. capitata larvae in artificially infested fruit.

Cherry - The application of dimethoate when the fruit was still green (Table 7) resulted in a great reduction of cherries infested by Rhagoletis cerasi larvae. This control seemed better than that obtained with later application when the cherries were changing color and the infestation had already started (Table 7). On the other hand, we had negative results on leaves, from the same plants, infested with T. telarius.

Peach - Following repeated positive results from preliminary tests on C. capitata carried out by means of artificial infestations, we tried an experiment under natural conditions. When applied while the fruit was green (about 24 days before ripeness) dimethoate was able to inhibit almost completely the development of the larvae in the peaches (Table 8). The results of this Table show also, for each plant, the effects of different dosages of insecticide, and the erratic performance of one of them. The results on Hyalopterus pruni (Table 9) were completely positive with both compounds though on the same leaves T. telarius survived. Dimethoate appeared quite ineffective against Quadraspidiotus perniciosus and Pseudaulacaspis pentagona.

Considering the results in toto (Table 10), we can arrive at the following conclusions:

For certain species of pest there is a considerable variation in the results depending on dosage and date of application. The most susceptible pests were certain species of scales (which feed on the sap from the leaves and from the epicarp of citrus fruit); aphids, (feeding on the sap from leaves of the apple, cherry and peach trees); dipterous larvae, (which develop in the mesocarp of peaches, cherries and olives); and microlepidopterous larvae, (which, like leaf miners, feed on the parenchyma of the leaves of the olive).

Table 7. Cherries. Toxicity of fruit to larvae of Rhagoletis cerasi after trunk applications of dimethoate.

Dimethoate (grams per tree[a])	Percent Infested Fruit	Percent Dead Larvae in the Ripe Fruit[b]				Percent Reduction of Infested Fruit[c]
		first instar	second instar	third instar	Total	
		Treatment: May 17, 1960 (green fruit)				
12	96.5	90.7	0.5	0	91.2	80.3
24	97.0	94.8	0.5	0	95.3	86.2
48	99.5	88.5	0.5	0	89.0	92.7
		Treatment: May 31, 1960 (fruit changing colour)				
12	97.0	48.5	1.0	1.0	50.5	70.6
48	98.5	33.5	21.3	5.1	59.9	77.8
		Check, no treatment				
-	99.0	1.0	0	0	1.0	-

[a] - Average height of trees (Durello variety) : 8 meters.
[b] - 200 cherries per tree examined.
[c] - 2000 cherries per tree examined.

Table 8. Peaches. Toxicity of fruit to larvae of <u>Ceratitis capitata</u> after trunk applications of dimethoate.

Trees[a] No.	Days after treatment[b]	Percent Infested Fruit	Percent Dead Larvae in the Fruit			
			first instar	second instar	third instar	Total
			dimethoate: 8 grams per tree			
1	6	50	33	0	0	33
	10	60	72	0	0	72
	17	100	100	0	0	100
	24	100	95.2	4.8	0	100
2	6	0	0	0	0	0
	10	70	39.3	0	0	39.3
	17	90	47.5	5.4	0	52.9
	24	100	85.7	3.8	6.6	96.1
			dimethoate: 20 grams per tree			
3	6	40	36	0	0	36
	10	80	64.7	0.8	0	65.5
	17	80	100	0	0	100
	24	100	91.4	8.6	0	100
4	6	40	25.4	0	0	25.4
	10	70	52.6	0.6	0	53.2
	17	100	86.0	14.0	0	100
	24	100	100	0	0	100
			check: no treatment			
5	6	30	0	0	0	0
	10	50	0	0	0	0
	17	100	0	2.8	0	2.8
	24	100	0	14.8	0	14.8

[a] - Average height of trees ("Hale" variety) : 3 meters.

[b] - Treatment : July 13, 1960 at Catania; fruit was completely green.

Table 9. Peaches. Toxicity of leaves to Hyalopterus pruni and Tetranychus telarius after trunk applications of dimethoate[a] and its P=O[a] derivative.

Days After Treatment[b]	Effect on Aphids and Mites	
	dimethoate	P=O derivative
	Hyalopterus pruni (percent reduction of infested leaves)	
1	0	10
3	34	94
4	90	96
6	100	100
13	100	100
29	100	100
60	100	100
	Tetranychus telarius (percent mortality of adult females)	
4	41	90
13	22	56
30	8	8

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[a] - 5 grams per tree ("Alexander" variety); average height: 3 meters.
[b] - treatment : May 26, 1959, at Florence.
[c] - on detached leaves.

Table 10. Relation of effectiveness and phytotoxicity of dimethoate and its P=O derivative to several species of pests and trees and to dosage applied.

Plants	Pests	dimethoate						P=O derivative				
		dosage: rating value[c]										
		1.5x	2x	3x	4x	5x	8x	1.5x	2x	3x	4x	5x
Lemon	P. citri		△++						△++			
	A. haederae		△o						△o			
	T. telarius		△++						△++			
	A. sheldoni	□o	△+					□o	△+			
Orange	C. dictyospermi				△++						△++	
	M. Beckii	□+		△++				□+		□+		
	C. oleae	□+		△+				□+		□+		
	C. capitata				△++						□++	
	T. telarius[d]				△+						△+	
Tangerine	M. Beckii	□+						□+				
	P. citri			△++						△++		
	C. oleae	□+						□+				
Olive	D. oleae[e]				□+	○++					□+	○++
	P. oleellus		◎		◎	○++	◎					○++
Apple	Y. plantaginea				□++							
	A. pomi			□++						□++		
	M. ulmi		◎		□o	◎			◎			
	T. telarius[d]		◎		□+				◎			
Pear	C. capitata[e]		□++									
Cherry	M. cerasi			□++								
	R. cerasi		□+		□+	△++	△++					
	T. telarius[d]			□o						□o		
Peach	Y. pruni	○++										
	P. pentagona			◎								
	Q. perniciosus	◎										
	C. capitata	◎□+		△++		○++○+		○+○++			○++	○++

Effect on Pests[a] and Trees[b]

[a] - Effectiveness. Rating values: ++ complete control; + fairly good control; o = inadequate or no control.

[b] - Phytotoxicity. Rating values: △ severe injury; □ moderate injury; ○ = slight or no injury.

[c] - Dosage. Rating values: Times (1.5x ...8x) that the dosage of active ingredient applied on the tree trunk is superior to the dosage which could be applied by full coverage spray.

[d] - on detached leaves. [e] - fruit artificially infested. [f] - Insecticides applied on the branches.

The most unsuccessful results, on the other hand, were to two species of scales (P. pentagona and Q. perniciosus) which feed on the cortical tissues of the branches and trunk, and three species of mites which feed on the content of first layers of cells below the epidermis of the leaves (M. ulmi and T. telarius) or of the buds (A. sheldoni).

The activity of the two insecticides does not differ substantially, but it was noted that the P=O derivative acted in many cases with greater rapidity than dimethoate.

Distribution of the Substances Translocated into the Plants

The concentration of the insecticide in leaves and fruit was assessed at various intervals of time, by means of bio-assays, on the same plants used for the insect and mite tests described above.

The results are shown, in graphic form, as follows: lemons (Fig. 1 and 2 above), tangerines (Fig. 2 below), oranges (Fig. 3), olives (Fig. 4), cherries and apples (Fig. 5). In order to facilitate comparison, the two variables (time and concentration) are plotted on semi-logarithmic paper: segments of lines having equal slope with respect to the abscissa, represent, therefore, equal relative variations. Despite the changes in the values and relative variations in the concentrations of insecticide in the leaves and in the fruit (depending on kind of plant, time of application and product applied), there are some uniform tendencies which can be summarized as follows:

The maximum concentration of insecticide which reaches the leaves is notably higher than that in the fruit (Figs. 1, 2, 3 below, 5). There was only one exception (Fig. 3 above).

In citrus plants, the insecticide accumulates much more in newly formed leaves than in old leaves (Figs. 2 above, 3 below).

For the whole or greater part of the experiment, maximum concentrations of insecticide are found in the peel of citrus fruit, which are higher than those found in the pulp (Figs. 1, 2, 3). Moreover in ripe fruit the concentration is less than that found in green fruit (Fig. 1).

Considering the variations with time, it seems that the concentration of insecticide having reached a maximum in leaves and fruit (more frequently first in the leaves and then in the fruit), diminishes in both. In the leaves this happens more rapidly in most cases than in the fruit (Figs. 1, 2, 3, 5). The curves which express the insecticide content in the vegetative tissue, at different times, are almost always asymetric: the maximum concentration occurs in a short period of time, while a variable but longer period is necessary to achieve the minimum values (less than 0.1 p.p.m.) which can be regarded as having no pesticidal and toxicological meaning.

Even though the behavior of the products is similar in a general way, several differences distinguish dimethoate from the P=O derivative (Figs. 1, 2, 3, 4). The difference between the concentrations of the insecticide in the leaves and in the fruit of the plants treated with the P=O derivative is less than in those treated with dimethoate. Furthermore the maximum concentration of the insecticide is attained in a shorter time with the P=O derivative, and the decreases are more rapid. In particular the decrease in the peel of citrus fruit takes place faster than in the pulp, so that some time after the application of the P=O derivative, the concentration in the pulp is higher than in the peel.

More detailed aspects of the distribution of compounds translocated into the leaves and fruit are shown in an experiment made by applying P^{32}-labelled dimethoate to the trunk of a peach tree and determining: a) the concentration of radioactive phosphorus percent as dimethoate and its degradation products (total P^{32}) and b) the concentration of radioactive phosphorus which can be extracted by means of chloroform (Santi, in prep.) and present as dimethoate and its P=O metabolite ($P^{32}/CHCl_3$).

The graphs in Fig. 6 again show that the concentration of both total P^{32} and $P^{32}/CHCl_3$ reaches and maintains higher levels in the leaves (greater in the terminal leaves) than in the fruit. Moreover, while the total P^{32} content in leaves reaches a maximum and then very slowly decreases and, in peaches, the increase continues until ripeness, the concentration of $P^{32}/CHCl_3$ after having reached its maximum in the leaves and a little later in the fruit, decreases rapidly in both. Further, even though the $P^{32}/CHCl_3$ content of terminal peaches is not always greater than that of fruit picked near the base of twigs, the same substances (mainly dimethoate and the P=O metabolite) appear more concentrated in the half of the fruit near the stem. This pattern of distribution, which is due to the penetration of the radioactive compounds through the stem end, has been confirmed by autoradiograms of the section of peaches (Fig. 7), which differ substantially from that obtained from fruit sprayed with P^{32}-labelled dimethoate (Fig. 7). The autoradiograms of peach leaves (Fig. 8) show that the substances containing labelled phosphorus, which penetrate through the petiole and the midrib, distribute themselves initially in the tissues between the secondary veins and then localize progressively along the borders of the leaf blade.

Adverse Effects on Plants

The adverse effects following the application of the two insecticides dissolved in TBF are shown by symptoms which generally begin to appear many weeks or months after the date of treatment. They appear both at that part of the trunk where the product was applied (lesions in the outer layers of the bark, cortical necrosis, injuries to the conducting tissues) and in the foliage (slowing-down of growth, chlorosis, phylloptosis). The nature of the symptoms and the slowness with which they become apparent, in contrast to the rapidity of damage when excessive dosage of the same insecticides is used as a spray, leads to the hypothesis that the cause of adverse effects after trunk application can be traced to lesions on the treated part of the trunk with results, in the most severe cases, which can be compared to those caused by girdling.

A summary of the phytotoxic effect to various kinds of plants, many months and even almost two years after the date of treatment, is depicted in Table 10, in which are also shown the relative dosages of the product used and the corresponding insecticidal and acaricidal activity. Some uniformities are evident: within the limits of the dosages tested, it would seem that the adverse effects are more closely related to the botanic species than to the dosage of the product. In fact, although we noted a certain variance in results on the same species of plants (with regard to dosage and other factors not easily ascertainable), citrus trees were badly damaged even with relatively low dosage; cherry, pear and even more so apple trees reacted less to the same dosage; peach trees were even more tolerant with only two exceptions and, finally, olive trees were the only plants to tolerate dosages of the two insecticides which were heavy enough to control D. oleae and P. oleellus effectively.

Of the two products dimethoate appears to be more phytotoxic, especially to citrus, than does the P=O derivative.

Influence of the Formulation on the Effect on Pests and Plants

In reviewing the results of the experiments with dimethoate and its P=O derivative in TBF from the practical viewpoint, two positive aspects emerge: the effective control of several species of pests and, which is peculiar, the minimum residue of insecticide in the fruit. However, a negative aspect has to be mentioned: the absence of an adequate and constant margin of safety, in many of the plants studied, between the effective insecticidal dose and that causing damage.

Tests with P^{32}-labelled dimethoate in TBF, applied to the stem of potted sour orange seedlings, have shown that, by varying the concentration of active ingredient in the formulation and varying consequentially, the amount of trunk surface to be painted, neither the phytotoxicity nor the speed of translocation of the penetrated substances changes appreciably, provided that the same amount of active principle is used.

We continued with further experiments varying the type of solvent. Tests, carried out with potted apple trees infested with Aphis pomi (Fig. 9), demonstrated that the speed, with which both dimethoate and its P=O derivative control aphids, is a function of the solvent, the greater speed being attained with TBF. The same conclusion was reached with the application of dimethoate in different solvents on peaches infested with H. pruni (Fig. 9). But while on peaches none of the formulations tried gave adverse effects, on the apples treated with both products in TBF, the painted portion of the bark of the stem appeared severely injured.

There is evidence, therefore, of a correlation between the speed of appearance of insecticidal effects and phytotoxicity.

More extended tests were made on sour orange seedlings in pots which proved particularly susceptible to phytotoxic effects. The results showed that while none of the ten solvents tested could itself be blamed for the adverse effects, the addition of dimethoate (Table 11) caused injury, the severity of which was dependent on the solvent and the dosage of the active ingredient. Heavy damage was caused by dimethoate in TBF at all dosages tested, while lower doses of the same product diluted with other solvents (cyclohexanone, methyl cellosolve, methanol, methyl phosphate) were practically innocuous. Further experiments, by means of radiometric and autoradiographic techniques, have established that even the least damaging among the quoted solvents, permits the transfer of P^{32}-labelled dimethoate and its metabolites from the trunk to the leaves.

We can, therefore, conclude that the seriousness of the effect is positively related to the speed of translocation of the active ingredient and to its maximum concentration in the leaves. This conclusion represents a starting point for further studies.

Table 11. Phytotoxicity of several formulation of dimethoate (30%) with different solvents (70%) applied on the stem of sour orange potted seedlings.

Solvents	Phytotoxicity[a]					
	dosage applied (μg)					
	750		1500		3000	
Butyl phosphate	△	△	△	△	△	△
Ethyl phosphate	o	o	△	□	◫	△
Methyl phosphate	o	o	o	□	△	□
Propyl phosphate	o	△	△	◫	△	△
iso-Propyl phosphate	△	□	△	□	△	△
iso-Butyl phosphate	o	o	△	△	△	△
Methanol	o	o	o	o	□	△
Dioxane	o	◫	□	□	△	△
Methyl cellosolve	o	o	o	o	△	o
Cyclohexanone	o	o	o	o	△	△

[a] - Phytotoxicity. Rating values: △ = severe injury or dead plant; ◫ = heavy injury; □ = moderate injury; o = slight or no injury. Results were checked 5 months after treatment.

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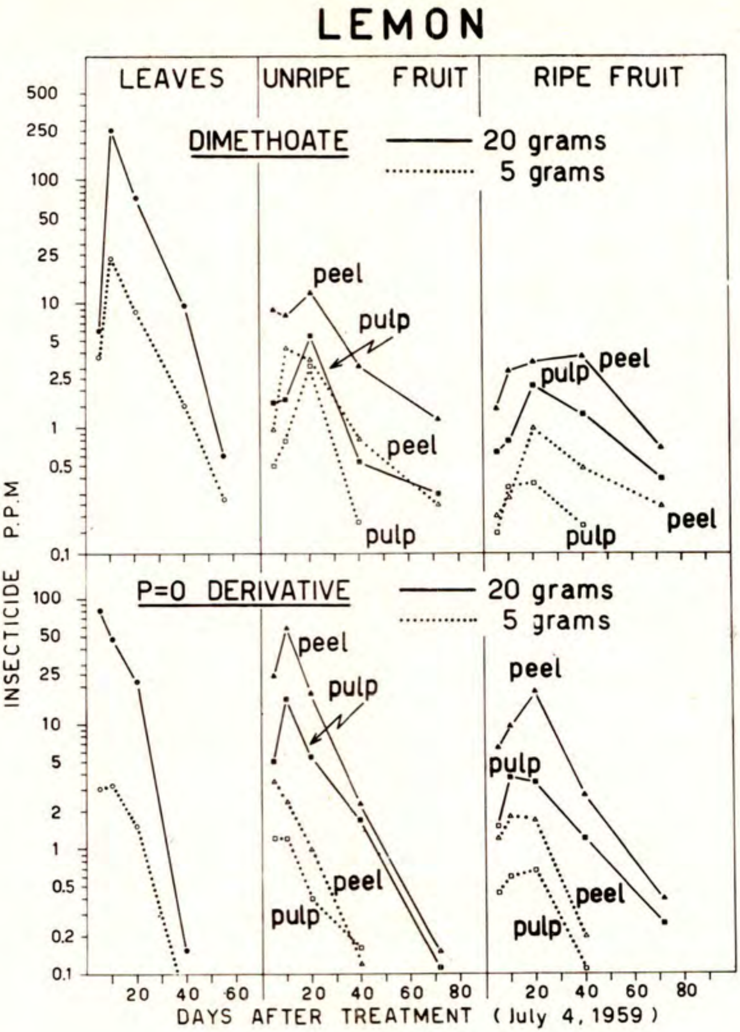

Fig. 1. Insecticide concentrations in the leaves and fruit of lemon trees after trunk applications of dimethoate and P=O derivative.

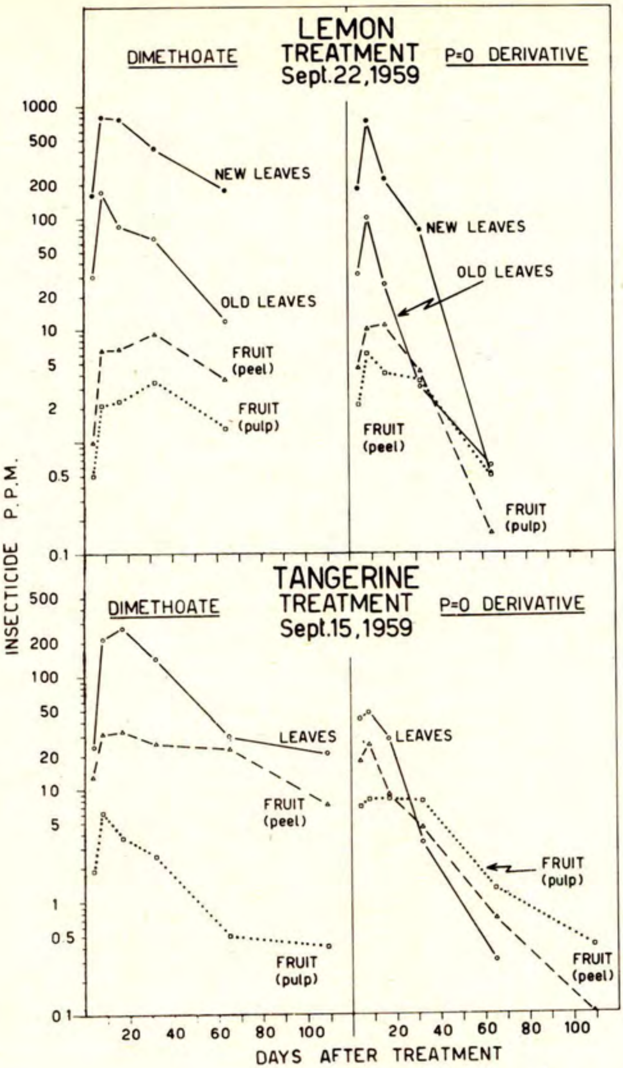

Fig. 2. Insecticide concentrations in the leaves and fruit of lemon (above) and tangerine (below) trees after trunk applications of dimethoate and P=O derivative.

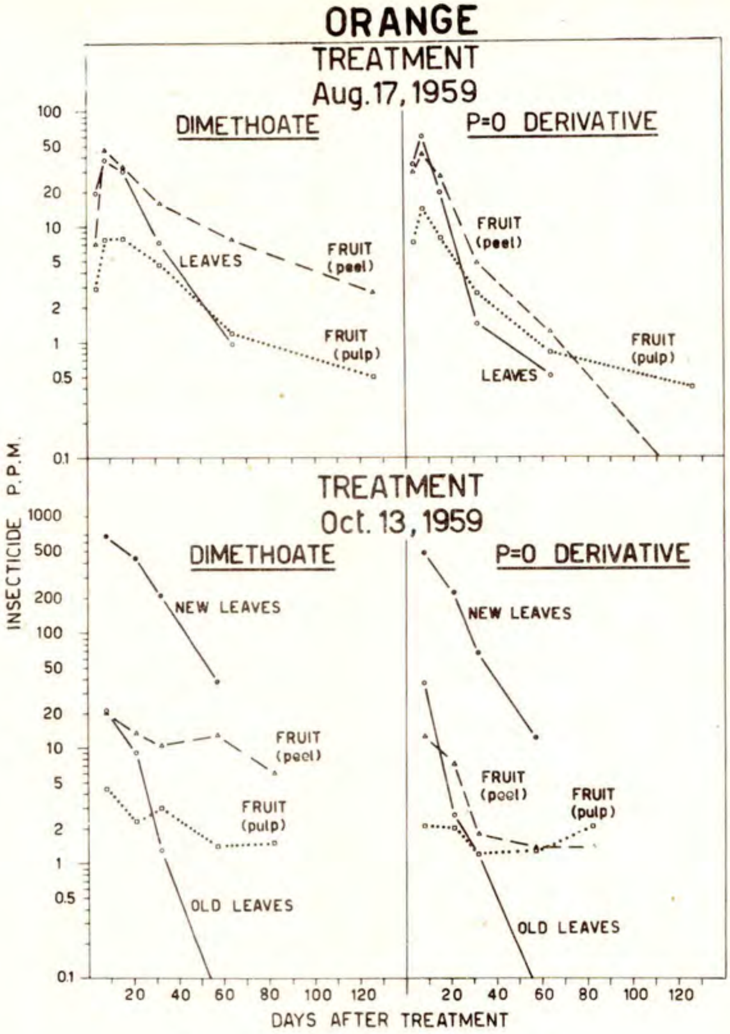

Fig. 3. Insecticide concentrations in the leaves and fruit of orange trees after trunk applications of dimethoate and its P=O derivative at two different dates.

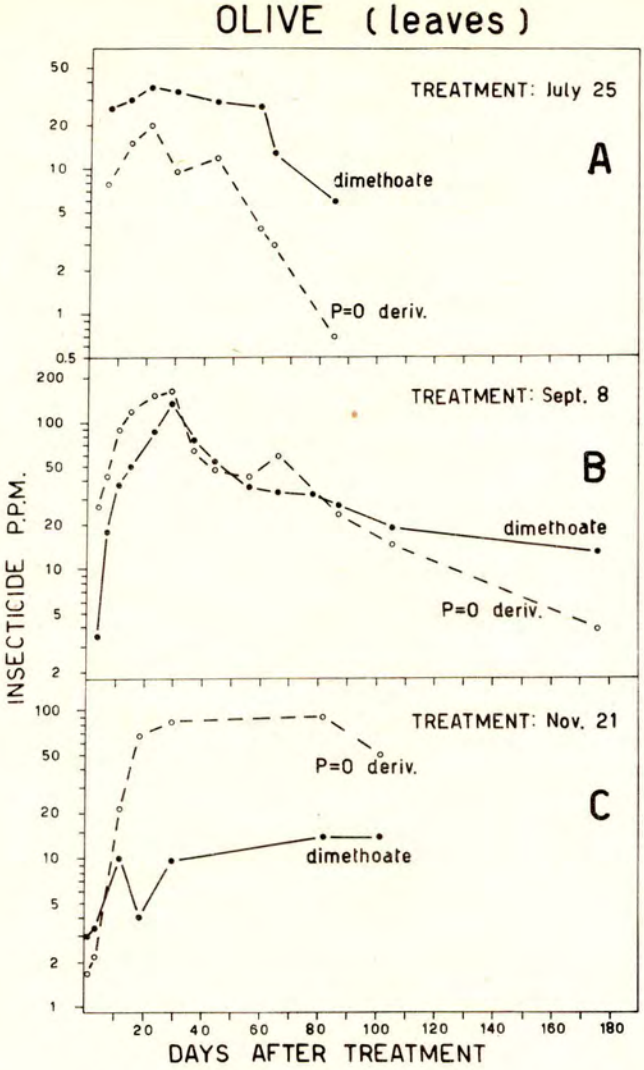

Fig. 4. Insecticide concentrations in the leaves of olive trees after branch (A) or trunk (B, C) applications of dimethoate, at three different dates.

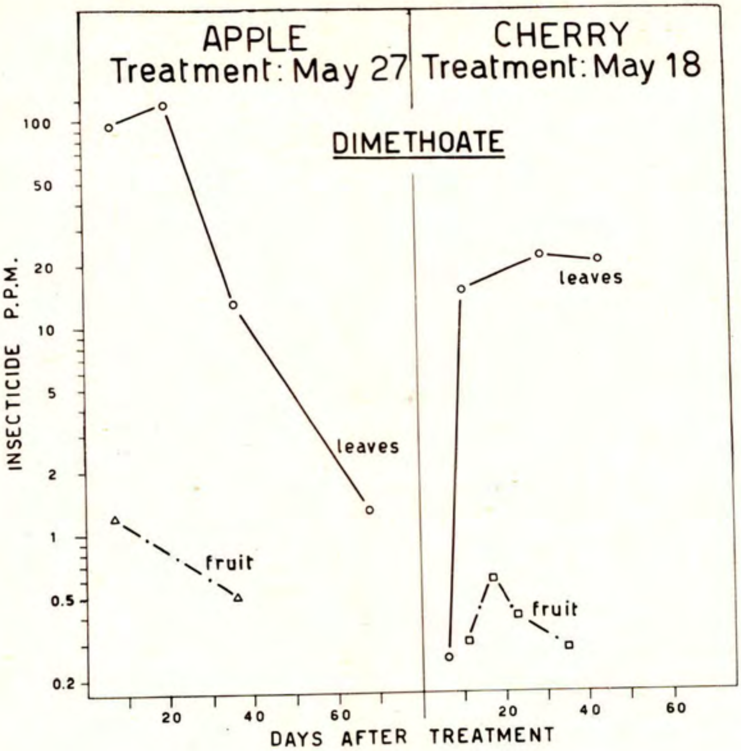

Fig. 5. Insecticide concentrations in the leaves and fruit of apple and cherry trees, after trunk applications of dimethoate.

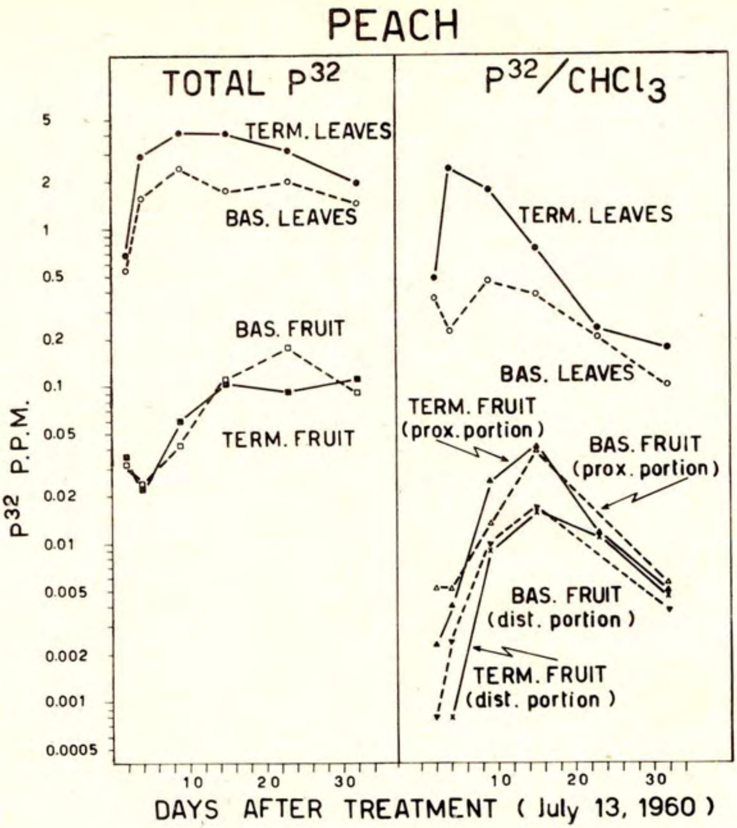

Fig. 6. Concentrations of total radioactive phosphorus (total P^{32}) and chloroform soluble radioactive phosphorus ($P^{32}/CHCl_3$) in the leaves and fruit of a peach tree after trunk application of P^{32}-labelled dimethoate.

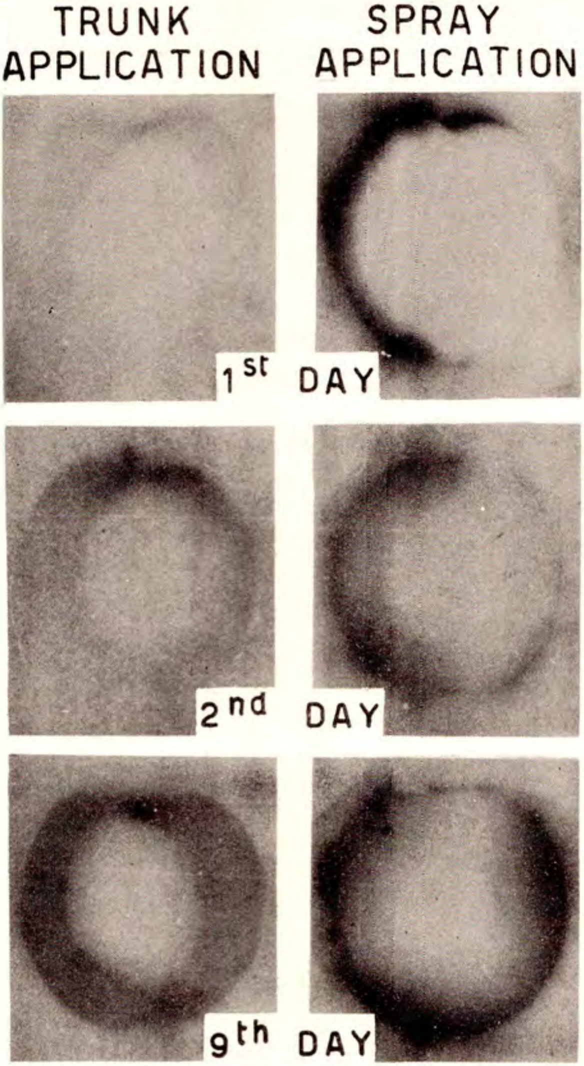

Fig. 7. Autoradiograms of longitudinal sections through peach fruits picked 1; 2; 9 days after trunk and spray applications of P^{32}-labelled dimethoate.

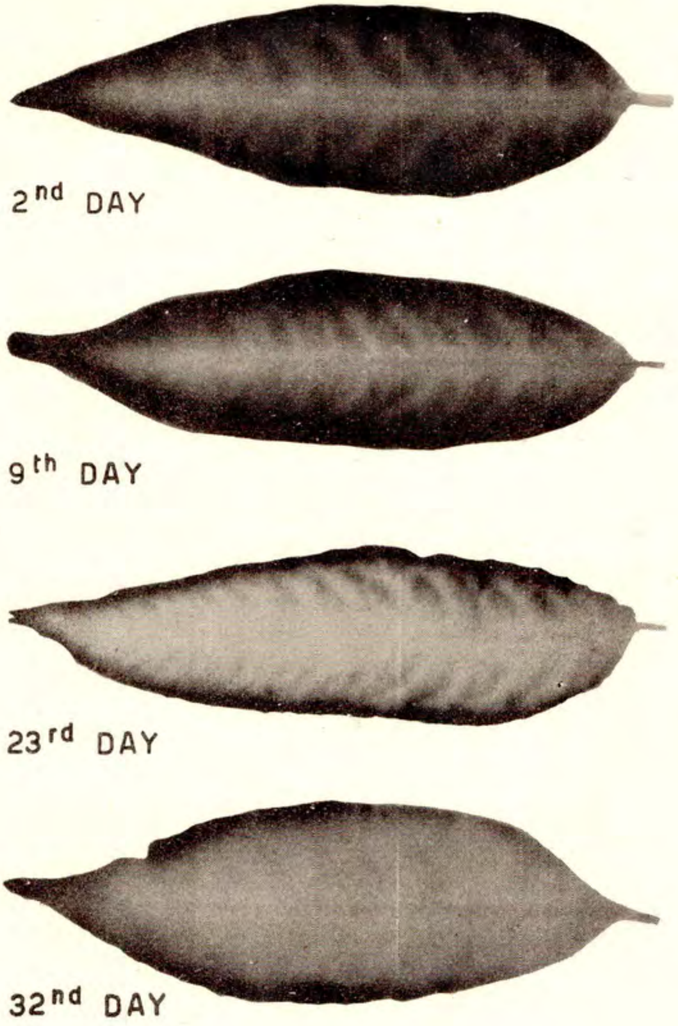

Fig. 8. Autoradiograms of peach leaves samples 2; 9; 23 and 32 days after trunk applications of P^{32}-labelled dimethoate.

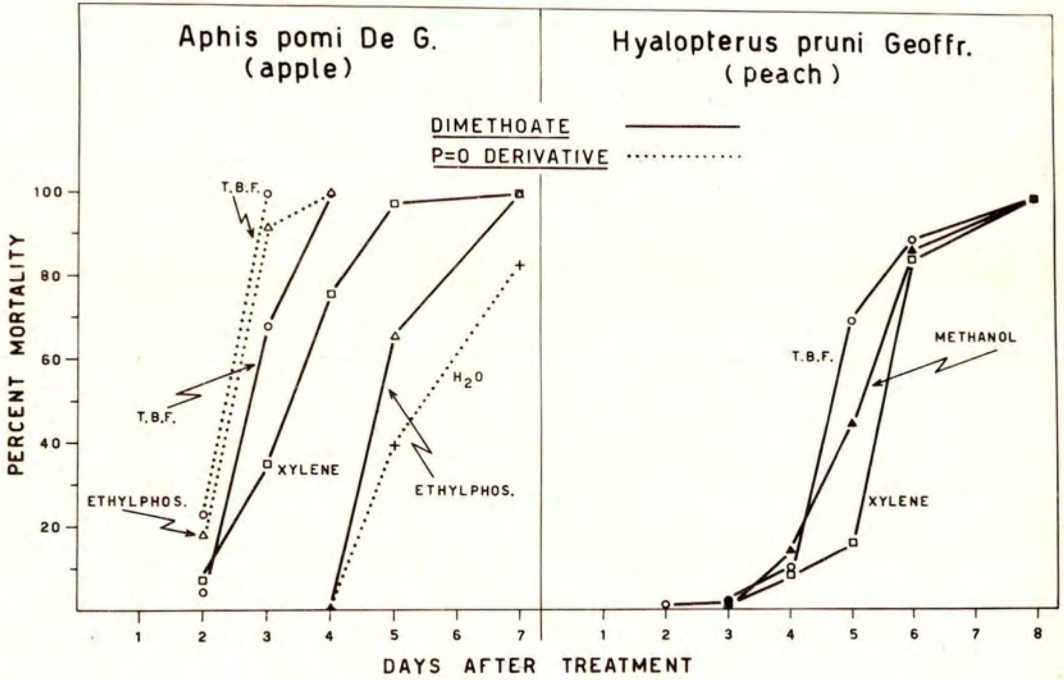

Fig. 9. Speed of action on *Aphis pomi* (apple tree) and *Hyalopterus pruni* (peach tree) of dimethoate and P=O derivative after trunk applications of the solution with different solvents: butylphosphate (T.B.F.), ethylphosphate, xylene, water, methanol.

MENAZON: DEVELOPMENT OF A SELECTIVE SYSTEMIC APHICIDE

by D. Price Jones
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Introduction

Recent trends in aphid control have been conditioned by two apparently conflicting requirements: 1, The need in some cases for a quick clean-up before harvest, with emphasis on a rapid reduction in residues of the aphicide and/or its metabolites; 2, The need in other cases for a more prolonged effect, with emphasis on the persistence of the aphicide for a few weeks with subsequent reduction of residues to safe levels.

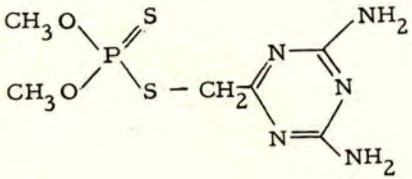
The former requirement applies for instance to the treatment of infested lettuce a few days before harvest and is not concerned with the prevention of damage. The latter requirement - persistence - is commercially the more important and applies to crops such as sugar beet and potatoes where the main objectives are the limitation of aphid populations and, often more important, the restriction of virus spread.

Menazon promises to meet the requirements of the second category. Its properties are discussed below and suggestions are made for the exploitation of these properties.

Chemical and Physical Properties of Menazon

The following account is intended to provide a suitable basis for the discussion of the biological properties. More information on chemical aspects may be found in a preliminary report by Calderbank, Edgar and Silk (1961).

Menazon is the BSI accepted common name for S-(4, 6 diamino - 1, 3, 5 triazin-2-yl) methyl OO-dimethyl phosphorothiolothionate:



It is a white crystalline material with a melting point of 160° C. It has an extremely low - as yet undertermined - vapour pressure and, as a consequence, no fumigant properties. The technical material, in bulk, has a mercaptan like odour which is not apparent in aqueous spray dilutions.

Menazon is soluble in water to about 0.024 g/100 ml at 20 - 25° C. At 4 oz a.i./acre, it should be largely soluble in 100 gal water/acre, given a sufficient interval between mixing and spraying. At lower volume rates, e,g, 3 gal/acre as in aerial spraying, very little of the menazon would be present in solution. Menazon is moderately soluble in hydroxylic solvents and only very slightly in other organic solvents.

The crystalline solid is stable at temperatures up to 50° C. The formulated material is stable on leaf surfaces and is also sufficiently stable in most soils to warrant its use as a soil or seed treatment. It is compatible with most commonly used insecticides and fungicides but should not be mixed with strongly alkaline materials.

Biological Properties

Mammalian Toxicity

As the development of a new material is inevitably governed by its mammalian toxicity, it is pertinent to compare menazon with other aphicides in current use (Table 1). In this comparison on rats, the only one readily available, menazon is shown to be one of the least toxic.

Table 1. Acute oral toxicity of important aphicides

Aphicide	Acute oral LD 50 mg/kg, rat
Menazon	1950
Malathion	1400-5800
Dimethoate	600
Lindane	125
Demeton-methyl	85-120
Nicotine	50-60
Parathion	6-15
Demeton	9
Mevinphos	7
Phorate	4
TEPP	2

Sources of toxicity data: Menazon: Imperial Chemical Industries; Dimethoate: Fison's Pest Control; Demeton-methyl: Baywood Chemicals; for other materials see Gunther and Jeppson (1961).

Investigations into other toxicological aspects, although not yet complete, are sufficiently advanced to establish criteria for commercial usage. Information has been submitted to the Advisory Committee on Poisonous Substances used in Agriculture and Food Storage and a clearance has been obtained for the use of menazon in 1962 on a number of crops, including sugar beet, potatoes, brussels sprouts, beans, hops, apples and strawberries, with a minimum interval of three weeks.

Mode of Action

Menazon is capable of killing aphids either by contact or by systemic action, the latter being in general the more important. Detailed physiological and biochemical investigations have yet to be undertaken. Present information is that menazon is a weak inhibitor of locust cholinesterase.

Spectrum of Activity

Menazon can fairly be described as a selective aphicide. While this selectivity is enhanced by systemic activity, it is in fact an inherent feature of the material as shown in direct contact toxicity tests in the absence of the host plant. It is important however to note that this selectivity is shown only at normal field dosage rates; at rates in excess of these, some kill of other insects may be achieved. The results of laboratory tests using menazon at different concentrations are given in Table 2. At the normal high volume concentration used on field crops, about 0.025%, only aphids suffered a total kill. However, Tetranychus, Dysdercus and Musca larvae were affected to some extent. At the highest concentration tested Phaedon and Musca adults were also affected.

These laboratory indications of selectivity are confirmed by observations in the field. Most aphids on field crops are amenable to control at spray concentrations of about 0.025% menazon at 100 gal/acre, or at equivalent low volume rates. Certain species of mites are also susceptible at this dosage level. Otherwise all the indications are that the vast majority of insects are relatively resistant to menazon.

Since the selectivity of menazon is not absolute, some kill of beneficial insects may be expected under some circumstances. This has been demonstrated by Way (1960, 1961) and has since been confirmed at Jealott's Hill for syrphids. Results of toxicity tests on bees are given in Table 3. Regarded pessimistically, these results show that a significant mortality among syrphid larvae and bees may be expected under conditions of commercial usage. Examined more critically, they reveal considerable progress in the development of a selective aphicide. Whether reformulation can increase the selectivity still further remains to be seen. It is essential in any case to adopt a method of using menazon which will fully exploit the degree of selectivity inherent in the material.

The significance of broad spectrum insecticides in the production of 'flare-ups' and also in inducing insecticide resistance in aphids in closed ecological communities has been discussed by Stern and Reynolds (1958). The integration of chemical control with biological control of the alfalfa aphid has been admirably appraised by Stern, Smith, van den Bosch and Hagen (1959).

Systemic Properties and Persistence

Menazon can be absorbed either through the roots or through the leaves and can move systemically to the upper parts of plants. Absorption into the leaf is relatively slow and is apparently influenced by the nature of the leaf surface, being apparently slower on waxy leaves. Penetration

Table 2. Menazon selectivity related to spray concentration

Pest Type of test	% Kill at concentration (ai) indicated				
	0.3%	0.1%	0.05%	0.025%	0.005%
Aphis fabae	100	100	100	100	100
DC, RC, Sy					
Acyrthosiphon pisi	100	100	100	100	100
RC, Sy					
Tetranychus telarius					
Walking stages	-	100	-	95	20
DC, RC, St, Sy					
Eggs	0	0	0	0	0
DC					
Dysdercus fasciatus	92	25	35	35	13
RC, Sy					
Phaedon cochleareae	40	0	5	0	0
RC, St					
Sitophilus granarius	0	0	0	0	0
RC, St					
Plutella maculipennis					
Caterpillars	0	0	0	0	0
DC, RC, St					
Musca domestica					
Adults	30	30	0	0	0
DC, RC, St					
Larvae	100	70	-	25	-
DC, RC, St					

Type of test: DC - direct contact, RC - residual contact,
St - stomach, Sy - Systemic

into the leaf may also be influenced by rainfall. Absorption into the plant by this route is very clearly dependent upon the amount of foliage present. Thus control of oat apple aphid is poor at bud burst, much better at green cluster and excellent in late summer. A similar progression has been noted with Myzus persicae on peach. Absorption through the roots, as with other systemics, leads to a more effective distribution within the aerial part of the plant, particularly into the new growth.

Little is known as yet about the mechanics of translocation and redistribution, although some interesting suggestions are beginning to emerge. For example, it is now becoming apparent that menazon deposited on the leaf surface forms a local reservoir from which active material can move into the leaf. The chemical stability of menazon and the resistance to weathering conferred by certain formulations, coupled with relatively slow absorption, can lead to a marked persistence of effect. A most striking example is given by the ability of a spray applied to apples on August 19th to give control of return migrants of the oat apple aphid even as late as 24th October. It is in fact, becoming apparent that the persistence of the aphicidal action of menazon is dependent upon the creation of such a reservoir on the leaf surface or in the soil. The half-life in the plant, judging by the results of recent biochemical work on young, actively growing tomatoes and beans, is only about 7 days. An external reservoir is therefore essential.

The redistribution of menazon is believed to be somewhat slower than that of certain other commonly used aphicides. Its performance is therefore likely to be relatively better against those aphids which show a preference for the older leaves. It does not appear to be capable of downward translocation in aphicidally significant amounts. It can however move from leaf to stem and thence to higher leaves on, for instance, broad bean plants. In comparison with demeton-methyl, such redistribution is slower but more persistent. There is a possibility that some redistribution on brussels sprouts is achieved by the movement of water from the leaves down the grooved petioles to the sprouts.

Phytotoxicity

Menazon has been tested in the field on a wide range of plants under varied climatic conditions. The general picture which has emerged so far is one of very low phytotoxicity. In particular, as a foliage spray it has proved safe at all normal dosage rates (4 - 8 oz per acre) on all the main crops, about thirty in all, including potatoes, sugar beet, beans, brassicas, cereals, apples, blackcurrants and peaches, and on a variety of ornamental plants, including roses.

As a seed dressing, it is harmless to most seeds even at relatively high dosage rates. For example no phytotoxicity has been noted in sufar beet at 2 and 4% W/W menazon on the seed, field beans and broad beans at 0.2 and 0.4%, alfalfa at 4%, cauliflower at 4%, cotton at 4%. On barley in one laboratory trial very slight tip scorch was produced presumably by the high concentration of menazon in the guttation droplets. On lettuce a

Table 3. Toxicity of systemic aphicides to honey bees

Aphicide	% Kill after 48 hours	
	Stomach poison (1)	Contact sprayed foliage (2)
Menazon	0	53
Demeton-methyl	10	77
Dimethoate	100	100
Control	0	0

(1) Bees fed on sugar syrup containing 5 p.p.m. a.i.
(2) Bees confined on dry foliage freshly sprayed at about 6 oz a.i./acre.

slight delay in emergence has been noted at 4% but in carrots phytotoxicity has occurred at 2%. On potatoes some slight delay in emergence has been noted in three trials but it is not known yet to what extent this is due to phytotoxicity or to mechanical damage to sprouts during the extra handling of the seed during treatment.

Granules applied to the soil at $\frac{1}{2}$ - 2 lb menazon/acre were not injurious to hops as a hill treatment, sugar beet and beans as a drill treatment or potatoes as a furrow treatment.

Drenches have been applied to the soil around hop hills at 4 lb menazon/acre, to beans in pots at 2 lb/acre, tobacco at transplanting at $\frac{1}{2}$ and 1 lb/acre and roses at indefinite but high rates, in all cases without indications of phytotoxicity.

Odour and Low Taint Potential

The technical material is somewhat malodorous in bulk but is relatively inoffensive when formulated and the spray fluid at normal concentrations is quite innocuous.

Tasting tests have been conducted with fruit and vegetables, including apples, peaches, strawberries, potatoes, brussels sprouts, lettuces and beans, so far without a hint of flavour changes. The short life of menazon within the plant and the even shorter life of its main metabolite suggest that the taint potential is very low. Nevertheless, tasting tests will continue for some time to come.

Development of Menazon

The development of menazon has already been in progress for about three years and a considerable amount of information has now been acquired about the control of specific aphids. Nevertheless with new formulations, further information on biochemical aspects and the emergence of new political attitudes towards the use of chemicals in the field, the true position of menazon is only now becoming clear. This position is reviewed below with only brief reference to the results of recent trials. Full results will be reported elsewhere.

Seed Dressings

In laboratory tests, seed treatment with menazon has given protection against aphids for varying periods to alfalfa, barley, beans, carrots, cauliflowers, cotton, lettuce and sugar beet.

In the field good protection of beans against *Aphis fabae* for two months after spring plantings has been obtained with dosage rates of 0.2 and 0.4% menazon W/W. Likewise delay in the spread of viruses, mainly pea leaf roll has been obtained in beans, presumably through the control of pea aphid.

In sugar beet, Bonnemaison (1960) has reported up to 22 days protection against *Myzus persicae* with dosages of 0.8 - 1.6% W/W. In the U.K. in one trial, virus yellows incidence in August was reduced to half that in the control by seed treatment at 2% menazon W/W.

Foliage Sprays

Encouraging results have been obtained against aphids on an extensive series of crops, using in the majority of cases a dosage rate of about 4 oz menazon/acre on field crops and 8 - 12 oz on top fruit. The following are the more important crops and aphids, on which good, and in some cases excellent, results have been obtained: potatoes: Various leaf aphids; beans: *Aphis fabae*; sugar beet: *Aphis fabae* and *Myzus persicae*, with consequent reduction in yellow virus; brussels sprouts: *Brevicoryne brassicae*; groundnuts: *Aphis craccivora*; cotton: *Aphis gossypii*; sorghum: *Longuinguis sacchari*; tobacco: *Myzus persicae*; apple: *Aphis pomi*, *Eriosoma lanigerum*; cherry: *Myzus cerasi*; peach: *Myzus persicae*; citrus: *Toxoptera aurentii*.

In a different category are a few crops, with their attendant aphids, where results as yet have not been satisfactory. On hops for instance, menazon sprays have not produced a sufficiently quick initial kill although their persistence has resulted in the depression of aphid populations for long periods, compared with demeton-methyl. On strawberries aphid control has been rather poor. It has been suggested that the outer leaves shield the inner parts of the plants and that very little translocation to the centre occurs with any of the systemic compounds. However some of them, e.g. demeton-methyl, are apparently sufficiently volatile to have a fumigant effect not obtainable with menazon. As already mentioned, control of oat apple aphid at bud burst does not seem to be readily achievable, because of the limited absorptive surface.

Despite the slow initial action of menazon, excellent control of virus has been obtained on sugar beet. Nevertheless the slow initial action does underline the need for early application of menazon in certain cases, e.g. on brussels sprouts.

The further development of menazon in foliage applications is likely to be conditioned to a large extent by its selectivity of action. On the one hand its use on crops such as apples and cotton will be restricted by the occurrence of pest complexes. On the other hand, its use on large areas of crops such as sugar beet, potatoes, beans and alfalfa will be favoured by its minimal toxicity to beneficial insects.

Acknowledgements

Acknowledgements are due to the many colleagues who have participated in this work and particularly to Mr. E. C. Edgar and Dr. A. Calderbank who have been responsible for most of the biological and biochemical work, respectively.

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MENAZON: CONTROL OF POTATO APHIDS AND APHID-TRANSMITTED VIRUSES BY TUBER TREATMENT

by J. F. Newman
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Introduction

In the last thirty years British potato stocks have greatly improved in their freedom from virus diseases. This has been achieved by certification regulations for seed produced on higher ground in the more northerly parts of the country. At the present time, about two-thirds of the ware potato acreage is planted with one of the grades of certified seed and most of the remaining area is planted with once grown seed. Cost of certified seed represents about one quarter of the total production cost of main crop potatoes, and about one third of the cost of growing earlies. Over the last ten years, experiments have been done, notably by Broadbent and others at Rothamsted Experimental Station, on the use of insecticidal foliage sprays to control the aphid vector of the important virus diseases and so to facilitate the production of virus free seed, and even to extend seed production into areas which would otherwise be unsatisfactory. This work has shown that a programme of foliage sprays with an efficient insecticide, starting at the time of 80% emergence of the sprouts and continued at fortnightly intervals into July can largely stop the spread of leaf roll and check the spread of virus Y. (Broadbent et al. 1956, 1960).

In 1960, menazon (Calderbank, Edgar and Silk, 1961) became available for field trial work. This compound shows high systemic aphicidal activity of long persistence, together with a low mammalian toxicity (approx. 2000 mg/kg to rats) and low phytotoxicity. Pot tests done early in 1960 having given favourable indication, field experiments were started to investigate the use of menazon treatments against potato aphids, and in particular to find the extent to which potatoes could be protected against virus disease spread by a single application of menazon used as a tuber dressing.

Experimental

Replicate plot trials

In 1960 replicate plot trials were done at Jealott's Hill, Berkshire and at Fernhurst, Sussex. In 1961, in addition to further trials at Jealott's Hill and Fernhurst, a similar trial was laid down at Rippingale Fen, Lincolnshire. In each of these trials five treatments were used in a Latin Square plan, with square plots, 1/40 acre in area, of side lengths equivalent to 16 rows of potatoes. The plots were separated on all sides by untreated guard areas of width equivalent to 9 rows. Twelve virus infected tubers, 6 leaf roll and 6 virus Y, were planted at regularly spaced intervals in each plot, giving approximately 1.7% of each virus.

Tuber treatments were done by spraying with dispersions containing, for the 1 lb/ton treatment, 2% or 4% of menazon at rates of 2 pints or 1 pint per cwt of seed respectively. The menazon content of the dispersion was adjusted appropriately for the other treatments. Where chitted seed was used, the application was made to the tubers in the normal chitting trays, loaded at 3 trays/cwt. Bagged seed was spread on the floor, sprayed in the same way and allowed to dry before being rebagged. Tubers were generally treated shortly before planting.

Granular treatments, using granules containing 0.5% of menazon on an inert base, were applied in the open furrows at the time of planting. Tins were cut to size to hold sufficient granules to treat 100 ft. row and the granules were shaken by hand from these tins into the rows.

Foliage spray treatments were applied by an eight nozzle hand boom sprayer, held by two persons, and operated through a pressure regulating valve from a pressurised knapsack sprayer. In all cases, the spray liquid was applied at 40 gal/acre, using Allman No. 00 jets at 20 lb/in^2.

Observations on aphid infestation were made by the method of Hollings (1955). A top, a middle and a lower leaf were examined for the presence of apterae on each of 20 plants per plot. Any plant on which an aphid was found was counted as infested. In some cases, counts were also made of the total number of apterous aphids on the sampled leaves, and these figures are shown bracketed in the tables.

At harvest time, samples of 300 tubers from each plot were taken and stored for growing on in the following year for assessment of virus disease. Observations on plants from the 1960 trials tuber samples were made in July 1961, and similar observations on the 1961 samples will be made in 1962.

Strip Trials

In 1961, in addition to the replicate plot trials, strip trials of greater area were done at several sites in Lincolnshire, Pembrokeshire, and Berkshire. In these trials, no attempt was made to assess virus disease spread, but aphid population observations were made as in the replicate plot trials. In one trial, at Grazeley Green, Berkshire, a mechanical planter was used, and a granule treatment was applied through the fertilizer section of the machine, the fertilizer having been applied previously broadcast.

Results

Results are shown in detail in the tables. Figures for aphid population in all cases refer to total potato aphids. Where the date of an observation is the same as a spray application date, in all cases the count was made before the spray was applied. In the trials in Pembrokeshire, no aphid attack occurred and no results were obtained.

Tuber Treatments

Tuber treatments were used at 1 lb/ton in 1960 and at ½, 1 and 2 lb/ton in 1961. All treatments resulted in substantial aphid control extending into July in the case of main crop trials or until the potatoes were lifted in early crops. In the replicate plot trials where the comparison is available the degree of control maintained through the season seems similar to that produced by three foliage spray applications of demeton methyl or of menazon. In the strip trials at Threekingham and Folkingham the tuber treatments applied in the preceding autumn exerted considerable control until the crops were harvested on 23rd June and 7th July respectively.

The figures for the replicate plot trials at Jealott's Hill and Fernhurst, where early aphid attacks occurred, provide an interesting comparison between the tuber treatments and spray treatments in their ability to control an early attack. The first spray treatment in each trial was applied at the stage of approximately 80% emergence of the sprouts, at which time many aphids were present on the controls and on the plants to be sprayed, while on the tuber treatments the aphids were already well controlled. The control of early attack is most important in the limitation of virus spread and it is evident that the protection provided by tuber treatments right from the time the sprouts emerge from the soil is a considerable advantage arising from this application method.

Granule Treatments

Granule applications were made in 1961 in two replicate plot trials (Jealott's Hill, Fernhurst) and in two strip trials (Threekingham and Grazeley Green). In all these trials, the ultimate residual aphicidal action was similar to that produced by tuber dressings at comparable rates, but the granule treatments were somewhat inferior in the early protection of the emerging sprouts. The granules were scattered more or less evenly along the rows at planting, and hence the acquisition by the plant of a protective concentration of menazon in the sprout is presumably dependent upon sufficient root development to reach enough granules. The dry weather conditions in the early summer of 1961 may also have had some effect in limiting the release of menazon from the granules in the soil. It is clear that further experiments in the use of granules should aim to investigate the use of placement treatments in which the granules are grouped more closely around the seed tuber.

Spray Treatments

In the replicate plot trials, foliage spray treatments with demeton methyl, demeton S methyl and menazon were included mainly as standards for comparison with the soil systemic treatments. The menazon spray treatments at 4 oz/acre appeared to produce aphid control similar to that given by other established spray treatments.

Control of virus disease spread

Samples of tubers from the 1960 Jealott's Hill trial proved to be heavily infected with leaf roll and virus Y when grown on in 1961. It is clear that the intensity of attack by virus infected aphids completely swamped any differences in spread within the crop.

Results for the Fernhurst 1960 trial are shown in Tables 9, 10 and 11. Infection in these samples was not heavy, but no significant differences occur between any of the treatments and the control. This result indicates that the infection arose from viruliferous winged aphids migrating into the crop from outside, a process which no insecticidal treatment applied to the crop can affect, and that little spread took place within the crop even in the absence of any insecticide treatment. A marked positional effect is noticeable in the incidence of leaf roll in this trial (Table 9), indicating arrival of infected aphids from a south-westerly direction. It should be noted that the test conditions used were severe, with minimal plot sizes in a small total trial area, and the gradient of infection indicates that more satisfactory virus control might be expected in large potato fields, particularly when isolated from untreated potato crops carrying virus infection.

These results, however, illustrate the fact that insecticide treatments alone are not likely to enable good quality seed potatoes to be produced for many years without regard to location and hence are not likely greatly to threaten the carefully established seed potato industries of Scotland and Northern Ireland and elsewhere. Insecticide treatments will rather, in due course, be of value as an adjunct to ecological and cultural practices in seed production.

Residues

Samples of ware tubers were taken at harvest from the 1960 replicate plot trials and subjected to chemical analysis for residues of menazon, using a method capable of determining 0.05 p.p.m. of menazon. No residues were detectable in any samples. Analyses were also done on tubers from plants sprayed with menazon at 8 oz/acre at either 8 weeks or $3\frac{1}{2}$ weeks before harvest. No residues were detectable.

Acknowledgements

Mr. G.H. Stock, Mr. E.C. Edgar and Mr. N.W.R. Crockford, of Jealott's Hill Research Station have collaborated with the author in carrying out the experiments. Dr. L. Broadbent, of the Glasshouse Crops Research Institute has provided valuable advice and assistance, particularly in the assessment of virus infected plants. Thanks are also due to the various farmers who have accommodated trials on their land, often at some inconvenience to themselves, and to members of the NAAS staff in the trial areas for considerable help and advice.

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Table 1. JEALOTT'S HILL - Replicate Plot Trial - 1960
Planted 13th April 1960 : Variety - Majestic

Treatments	Aphid observations at various dates			
(All rates refer to active ingredients)	2/6	20/6	6/7	14/7
Tuber treatment - Menazon 1 lb/ton + foliage spray - Menazon 6 oz/acre on 7/6	11	24	73 (309)	25 (67)
Menazon foliage spray) - 4 oz/acre x 2 applications on 7/6 and 15/6	35	13	64 (204)	34 (72)
Menazon foliage spray) - 4 oz/acre x 3 applications on 26/5, 7/6 and 15/6	37	22	61 (207)	29 (52)
Demeton methyl foliage spray) - 6 oz/acre x 3 applications on 26/5, 7/6 and 15/6	32	17	80 (306)	4 (5)
Control	83	92	100 (2482)	92 (739)

Note: Aphid observation - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 2. FERNHURST - Replicate Plot Trial - 1960
Planted 28th April 1960 : Variety - Majestic

Treatments (All rates refer to active ingredients)	Aphid observations at various dates			
	21/6	30/6	12/7	19/7
Tuber treatment - Menazon 1 lb/ton + foliage spray - Menazon 4 oz/acre on 12/7	9	34 (108)	35 (66)	4 (4)
Menazon foliage spray - 4 oz/acre x 2 applications on 1/6 and 22/6	76	46 (101)	48 (109)	20 (27)
Menazon foliage spray - 4 oz/acre x 3 applications on 1/6, 22/6 and 12/7	76	39 (87)	54 (115)	7 (11)
Demeton methyl - 6 oz/acre x 3 applications on 1/6, 22/6 and 12/7	69	28 (51)	51 (93)	1 (1)
Control	79	96 (739)	82 (409)	44 (121)

Note: Aphid observation - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 3. JEALOTT'S HILL - Replicate Plot Trial - 1961
Planted 21st April 1961 : Variety Majestic

Treatments (All rates refer to active ingredients)	Aphid observations at various dates						
	30/5	2/6	14/6	16/6	30/6	4/7	14/7
Tuber treatment - Menazon ½ lb/ton + 1 spray at 4 oz/acre on 30/6	12 (36)	16 (55)	52 (144)	63 (152)	73 (246)	24 (41)	12 (12)
Tuber treatment - Menazon 2 lb/ton	2 (7)	7 (12)	37 (58)	27 (63)	55 (148)	33 (61)	12.5 (15)
Granule treatment - Menazon granules in rows at planting at 2 lb/acre	25 (88)	18 (87)	34 (74)	31 (66)	66 (196)	47 (87)	10 (22)
Demeton S methyl foliage spray 3 oz/acre x 3 applications on 30/5, 14/6 and 30/6	72 (411)	4 (4)	67 (189)	6 (6)	58 (142)	3 (3)	7 (7)
Control	70 (343)	79 (405)	88 (464)	89 (651)	95 (735)	76 (287)	17 (17)

Note: Aphid observations - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 4. FERNHURST - Replicate Plot Trial - 1961
Planted 18th April 1961 - Variety - Majestic

Treatments	Aphid observations at various dates				
(All rates refer to active ingredients)	6/6	19/6	27/6	5/7	25/7
Tuber treatment - Menazon 2 lb/ton	21 (30)	41 (61)	38 (76)	31 (42)	10 (10)
Tuber treatment - Menazon 1 lb/ton	28 (30)	48 (82)	42 (83)	40 (64)	0 (0)
Menazon foliage) spray) - 4 oz/acre x **3** applications on 6/6, 19/6 and 5/7	57 (140)	46 (80)	17 (30)	19 (27)	8 (8)
Granule treatment - Menazon granules in rows at planting at 2 lb/acre	39 (85)	45 (82)	42 (115)	48 (95)	13 (13)
Control	59 (116)	51 (100)	72 (204)	70 (223)	0 (0)

Note: Aphid observation - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 5. RIPPINGALE FEN - Replicate Plot Trial - 1961
Planted 28th May 1961 :
Variety - Record.

Treatments	Aphid observations at various dates			
(All rates refer to active ingredients)	6/6	22/6	11/7	24/7
Tuber treatment - Menazon $\frac{1}{2}$ lb/ton	1 (1)	41 (122)	18 (24)	0 (0)
Tuber treatment - Menazon 1 lb/ton	0 (0)	34 (72)	16 (28)	0 (0)
Tuber treatment - Menazon $\frac{1}{2}$ lb/ton + 1 spray 4 oz/acre on 11/7	3 (15)	52 (152)	26 (39)	0 (0)
Demeton methyl foliage spray - 6 oz/acre x 3 applications on 6/6, 22/6 and 11/7	3 (3)	73 (334)	38 (60)	0 (0)
Control	4 (5)	51 (155)	35 (49)	0 (0)

Note: Aphid observations - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 6. FOLKINGHAM - Strip Trial - 1961
Planted 14th March 1961 : Variety - Arran Pilot

Treatment	Aphid observations at various dates				
(All rates refer to active ingredient)	25/4	17/5	6/6	22/6	12/7
Tuber treatment - Menazon 1 lb/ton at planting	0	0	3	45 (165)	100 (430)
Tuber treatment - Menazon ½ lb/ton at planting	0	0	5	55 (315)	100 (1117)
Tuber treatment - Menazon 1 lb/ton applied in chitting house on 15th November 1960	0	0	10	63 (654)	100 (558)
Control	0	0	22	96 (3860)	100 (4449)

Note: Aphid observations - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 7. THREEKINGHAM - Strip Trial - 1961
Planted 9th March 1961 : Variety - Arran Pilot

Treatment	Aphid observations at various dates			
(All rates refer to active ingredient)	25/4	17/5	6/6	23/6
Tuber treatment - Menazon 1 lb/ton at planting	0	0	15	90 (277)
Granule treatment - Menazon granules in rows at planting at rate of 1 lb/acre	0	1	44	96 (674)
Tuber treatment - Menazon 1 lb/ton applied in chitting house on 16th November 1960	0	1	31	98 (527)
Control	0	3	36	100 (1039)

Note: Aphid observations - First figure gives % infested plants
Bracketed figure gives number of aphids per 300 leaves

Table 8. GRAZELEY GREEN - Strip Trial - 1961
Planted 12th/13th May 1961 :
Variety - Majestic

Treatment	Aphid observations at various dates			
(All rates refer to active ingredient)	13/6	16/6	4/7	18/7
Tuber treatment - Menazon 1 lb/ton	5	31 (71)	40 (85)	12.5 (15)
Tuber treatment - Menazon ½ lb/ton	15	38 (84)	65 (192)	25 (30)
Granule treatment - Menazon granules in rows at planting at 2 lb/acre	30	37 (70)	50 (77)	30 (48)
Control.	65	74 (442)	80 (260)	25 (48)

Note: Aphid observations - First figure gives % infested plants
Bracketed figure gives numbers of aphids per 300 leaves

Virus disease spread in 1960 Fernhurst trial

Squares show 1960 layout. Figures show numbers of virus infected plants, secondary infections, appearing in plants from 300 tubers sampled from plots and grown on in 1961.

Table 9

Leaf Roll

16 A	17 B	17 C	8 D	2 E
11 C	3 D	9 E	5 A	7 B
16 B	11 C	7 A	6 E	9 D
25 E	14 A	3 D	2 B	13 C
16 D	9 E	2 B	6 C	10 A

Treatments as in Table 11

Table 10

Virus Y

4 A	3 B	1 C	1 D	1 E
8 C	4 D	0 E	2 A	3 B
0 B	2 C	2 A	1 E	0 D
2 E	0 A	1 D	3 B	9 C
2 D	1 E	1 B	0 C	7 A

Table 11

Treatments	Mean % disease	
	Leaf Roll	Virus Y
A Menazon foliage spray - 4 oz/acre x 2 applications	3.3	0.7
B Demeton - methyl foliage spray - 6 oz/acre x 3 applications	2.5	0.5
C Control - unsprayed	3.8	0.9
D Menazon tuber treatment - 1 lb/ton, followed by 1 Menazon foliage spray at 4 oz/acre	2.4	0.4
E Menazon foliage spray - 4 oz/acre x 3 applications	3.0	0.3
Significant difference between treatment means	Not significant	Not significant

EXPERIENCES WITH 5-AMINO-3-PHENYL-1-BIS(DIMETHYLAMIDO) PHOSPHORYL-1, 2, 4-TRIAZOLE, A NEW FUNGICIDE CONTROLLING POWDERY MILDEW*

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Summary

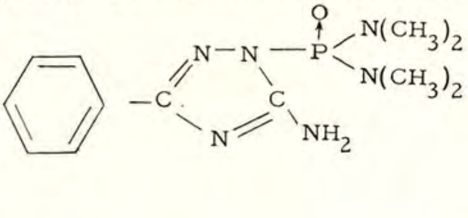
A review of the results with 5-amino-3-phenyl-1-bis(dimethylamido) phosphoryl-1, 2, 4-triazole is given. This compound, indicated as WP 155, when applied as a leaf spray, has a considerable effect on powdery mildew infection. WP 155 is a poisonous substance with cholinesterase-inhibiting properties.

In laboratory tests a systemic fungicidal effect was demonstrated by applying the substance to the roots of plants. In a number of countries WP 155 is now applied in practice for the control of powdery mildew on a number of ornamental plants, such as roses, Crataegus, Acer, Begonia and Cineraria. Its use on apples is promising and tests are being made on a number of other crop plants.

Apart from fungicidal properties WP 155 also has, under practical conditions, a useful effect on aphids, and some effect on spider mites. The activity against other insects is still under investigation. Honey bees are fairly resistant.

Introduction

This paper presents a review of the results obtained to date by the author and his colleagues with 5-amino-3-phenyl-1-bis(dimethylamido) phosphoryl-1, 2, 4-triazole. This compound will for convenience be designated WP 155, which was the code number generally used during the investigations. WP 155 is one of a series of amino-triazole derivatives with fungicidal, insecticidal and acaricidal properties described by v. d. Bos, Koopmans and Huisman (1960). Its chemical structure is (v. d. Bos, 1960):



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[*] Communication no. 64 of the Agrobiological Laboratory "Boekesteyn".

The melting point of the pure compound is 167° - 168°. It is soluble in most organic solvents, but only slightly in water. The compound is stable under neutral and basic conditions and rather unstable in the presence of strong mineral acids.

WP 155 has, as is common with other organophosphate insecticides, cholinesterase-inhibiting properties. The acute oral LD 50 for female albino mice appeared to be 20 mg/kg, and the corresponding value for male mice was 32 mg/kg. After intraperitoneal administration, values for the acude LD 50 of 8 and 9 mg/kg for female and male albino mice respectively were found. The acute dermal LD 50 for rabbits lies between 1500 and 3000 mg/kg. WP 155, applied to the skin of rabbits for 20 days in dosages of up to 10 mg/kg, did not cause any mortality, nor any differences with respect to behaviour, food consumption or weight. These dosages caused some, probably reversible, cholinesterase inhibition. Scarification of the skin did not influence the results in this experiment. Cumulative feeding studies are in progress.

Fungicidal activity

In laboratory trials, WP 155 and a number of related compounds, when sprayed on the leaves, gives good protection to plants against attack by members of the Erisyphaceae. As far as is known at present, there are differences in the degree of activity of, but no principal differences in, the type of action between these substances, which, surprisingly, are able to protect the plant against powdery mildew infection when they were administered to the roots (Koopmans, 1960).

An illustration of this systemic effect is given in Table 1.

Table 1. Infection by Podosphaera leucotricha of apple seedlings in WP 155 solutions.

Initial concentr. p.p.m.	Average infection after 14 days		Number of newly formed leaves per plant
	On existing leaves in % of untreated leaves	On newly formed leaves Evaluation of infection *	
31.5	5	0.3	1.1
10	17	1.1	2.3
0	100	2.8	1.5

* 0 = no infection; 1 = light infection; 2 = moderate infection; 3 = severe infection.

The exact mode of the systemic action is not known. Koopmans observed well developed germination tubes with an appressorium after the application of considerable overdosages of WP 155 that completely arrested any mycelial development. Probably the development can only be arrested when a penetration hypha has been formed and - if this is true - the point of action is obviously situated in the epidermal cells. Treatment of a grape leaf with WP 155 does not result in protection of neighbouring leaves against powdery mildew infection, when the treated leaf is undamaged, but if the leaf is bruised before treatment, then no powdery mildew develops on neighbouring leaves.

Since 1957 field experiments have been carried out with WP 155. On apples a wettable powder containing 25% of active ingredient and an 8.9% water-miscible solution were used. It appeared that the fungicidal effect of WP 155 against apple powdery mildew under practical conditions was the same or slightly better than the effect of dinocap when applied in equal dosages.

The standard concentration for WP 155 is at present considered in the Netherlands to be 0.025% active ingredient, the concentration for dinocap being 0.03%. The usual spray interval with these concentrations has to be some 12 days. Shorter spray intervals - about 1 week with half the normal concentration - usually result in better control and quite a number of growers favour this practice with dinocap despite the increased spraying costs. On apples one can often observe that treatments with WP 155 cause shoots - grown from infected buds and normally completely covered by powdery mildew - to produce healthy top leaves again. Experience so far is that on apples, the interval between WP 155 applications should not be shorter than those employed for sulphur and dinocap, the older mildew control materials.

In most of the trials 25 shoots from each plot were chosen at random and marked. The degree of attack was assessed for each leaf on these shoots. There were as a rule three replications. The assessment was repeated in the course of the growing season on the same shoots. The leaves were graded into four classes: (0) free from mildew lesions; (1) slight attack: one or two small lesions; (3) moderate attack: more than one or two lesions, but less than half the leaf area covered with mycelium; (5) heavy attack: more than 50% of the leaf area covered with mycelium. The degree of attack was obtained by multiplying the percentages of slightly, moderately and heavily infected leaves with the arbitrarily chosen factors 1, 3 and 5 respectively and adding the products. The degree of control achieved by the treatments, as compared to the untreated plots, seems only moderate. This is mainly because the sprays had sometimes to be timed too far apart and pruning, which is a very valuable additional control measure, was omitted.

Table 2. Variety Jonathan. Treatments: 30/4, 9/5, 20/5, 3/6, 14/6.

	% infected leaves						Degree of attack	
	17/6			4/7			17/6	4/7
	light	moderate	heavy	light	moderate	heavy		
WP 155 25% w.p. 0.1%	9	0	0	21	0	0	9	21
dinocap 25% w.p. 0.12%	14	1	1	21	1	3	22	39
Untreated	45	5	1	48	8	2	65	82

Table 3. Variety Jonathan. Treatments: 24/4, 4/5, 16/5, 29/5
10/6, 20/6, 4/7, 16/7, 1/8, 15/8.

	% infected leaves												Degree of attack			
	3/6			18/6			17/7			20/8						
	l	m	h	l	m	h	l	m	h	l	m	h	3/6	18/6	17/7	20/8
WP 155 25% w.p. 0.1%	13	0	0	31	1	0	31	1	1	50	4	2	13	34	39	72
dinocap 25% w.p. 0.12%	10	0	0	23	2	0	27	2	0	48	3	2	10	29	33	67
Untreated	33	1	0	44	4	2	40	5	6	55	8	9	36	66	85	124

Table 4. Variety Lombarts Calville. Treatments: 14/4, 25/4, 16/5, 24/5, 8/6, 20/6, 12/7, 31/7, 18/8.

	% infected leaves						Degree of attack	
	13/6			5/7			13/6	5/7
	light	moderate	heavy	light	moderate	heavy		
WP 155 8.9% e.c. 0.25%	8	1	1	11	0	0	16	11
dinocap 25% w.p. 0.12%	10	1	1	14	1	0	18	17
Untreated	37	6	5	34	9	3	80	76

Table 5. Variety Jonathan. Treatments: 4/5, 13/5, 25/5, 6/6, 12/6, 24/6, 6/7.

	% infected leaves									Degree of attack		
	29/5			25/6			13/8					
	l	m	h	l	m	h	l	m	h	29/5	25/6	13/8
WP 155 25% w.p. 0.1%	10	1	0	35	8	2	41	11	13	13	69	139
dinocap 25% w.p. 0.12%	14	1	1	39	11	4	45	9	13	22	92	137

Table 6. Variety Cox' Orange Pippin.
Treatments: 24/4, 13/5, 5/6, 7/7, 24/7.

	% infected leaves						Degree of attack	
	8/7			25/7				
	light	moderate	heavy	light	moderate	heavy	8/7	25/7
WP 155 25% w.p. 0.12%	30	7	4	34	11	7	71	102
dinocap 25% w.p. 0.12%	36	8	3	39	11	4	75	92
Untreated	40	14	12	33	16	17	142	166

Table 7 gives the results of an experiment in four replications in France. Here the leaves were graded in the same way as described above, but the factors 1, 2 and 3 respectively were chosen by the investigator. The results are expressed as the mean of these values for all the leaves of a treatment. 20 shoots were examined per plot.

Table 7. Variety Jonathan. Treatments: 2/4, 16/4, 30/4, 14/5, 11/6, 27/6, 14/7, 1/8.

	Degree of attack	
	17/5	6/7
WP 155 8.9% e.c. 0.25%	0.51	0.18
dinocap 25% w.p. 0.3%	0.90	0.63
Untreated	1.38	1.02

Attempts to control apple powdery mildew by administering WP 155 to the trunks and roots of the trees were made but these treatments were generally too phytotoxic.

Damage to apple leaves has been observed a number of times in the Netherlands but in other countries phytotoxicity has seldom been reported. The injury manifested itself by a narrow yellowing band along the edges of part of the leaves, usually the older ones. In the few more severe cases this extended into a slight irregular yellowing over the whole leaf area or sometimes a necrosis of the leaf edges. These symptoms occurred particularly on the Jonathan variety, and then only after three or more treatments in spring. There are also numerous instances where the normal or even higher concentrations (e.g. 0.2% of the 25% w.p.) gave no discernible symptoms under otherwise similar conditions. Perhaps the growth conditions of the tree or climatic conditions are of importance, but up to now there is no evidence to support these assumptions. Thus phytotoxicity may well restrict the use of WP 155 on Jonathan in the Netherlands. With Golden Delicious marked increases in yield and quality are often noticed, as shown in Table 8.

Table 8. Variety Golden Delicious
Treatments: 1 & 2 : 28/4, 12/5, 29/5, 7/6, 21/6, 3/7, 15/7, 29/7, 14/8.
No. 3 on same days and in addition on 6/5, 18/5, 31/5, 15/6, 28/6, 10/7, 22/7, 7/8.

	kg/150 trees	% of fruits		
		glossy	light russet	russet
WP 155 25% w.p. 0.1%	3400	74	3	23
WP 155 8.9% e.c. 0.25%	3980	59	7	34
dinocap 25% w.p. 0.06%	2620	16	26	58

Scab control with captan on same dates as 1 & 2.

Residue studies on apple fruit are in progress. The results so far indicate a waiting period of 4 to 6 weeks between last treatment and harvest. On average the residues on apple fruits decrease rapidly after a few days and are less than 0.1 p.p.m. within about three weeks or less.

On roses, both greenhouse and outdoor, WP 155 is highly effective against powdery mildew. Treatment with WP 155 as a water-miscible solution (0.2 - 0.25% concentration) is now an accepted practice among rose growers in the Netherlands, Belgium and West-Germany. The treatments appear to be quite safe even on sensitive varieties like Baccarat. The wettable powder cannot be used on roses grown for flowers, because of the disfiguring residue, and in any case it is also somewhat less effective than the liquid formulation. Curative effects have been reported by Betgem (1960). A remarkable shoot growth of roses is often seen after WP 155 treatment in contrast to the results obtained after dusting with, or vaporization of, sulfur.

Other plants on which powdery mildew can effectively be controlled with WP 155 include Crataegus spp., Acer spp., Begonia, Chrysamthemum and Cineraria. In several countries it is at present being used for these crops. On other plants, including cucumber, delphinium and oaks, the effect of WP 155 against powdery mildew is considerable, but not quite good enough to compete with existing materials.

Tables 9, 10 and 11 illustrate the results obtained in roses and Crataegus.

Table 9. Rose: Variety Nymph. Treatment: 7/7.

	Mean evaluation mildew infection* on 16/7
WP 155 8.9% e.c. 0.25%	8
WP 155 8.9% e.c. 0.5%	9
dinocap 25% w.p. 0.1%	7½
Untreated	7

* 10 = no infection
0 = maximal infection

Table 10. Crataegus monogyna.
Treatments: 17/6, 28/6, 11/7, 20/7, 3/8, 19/8.

	Mean evaluation mildew infection				
	7/7	11/7	3/8	19/8	20/9
WP 155 8.9% e.c. 0.15%	6.3	4.6	6.3	7.3	7.6
WP 155 8.9% e.c. 0.2%	7.0	7.0	7.6	7.6	8.3
WP 155 8.9% e.c. 0.25%	8.3	7.0	7.6	8.6	8.6
dinocap 25% w.p. 0.1%	8.0	6.0	7.3	7.0	8.6
Untreated	4.6	3.6	4.0	5.5	4.6

Table 11. Crataegus monogyna.
Treatments: 20/6, 28/6, 10/7, 26/7, 3/8, 30/8, 12/9.

	% mildew-free shoots	
	19/9	29/9
Dinitro-sec. butylphenyl dimethylacrylate 25% w.p. 0.05%	23	31
WP 155 8.9% e.c. 0.25%	44	41
Untreated	18	23

Insecticidal and acaricidal activity

Meltzer (in the press) described the insecticidal properties of this group of compounds in general, and of WP 155 in particular. WP 155 has considerable contact and systemic aphicidal and acaricidal activity. The systemic effect, however, has only been investigated after the application of WP 155 to the roots of plants. WP 155 was found to have no activity towards the common housefly, whether by contact or in the vapour phase. Honey bees were able to tolerate WP 155 in a concentration of 10 p.p.m. without harmful consequences; 100 p.p.m., however, was lethal. Under practical conditions the effect of WP 155 on aphids, at concentrations required to control mildew, is sometimes satisfactory after a single spray and is in any case adequate if treatment against mildew is given regularly. The effect on spider mites is generally less pronounced, although definitely present. Table 12 represents a case in which the effect on an unspecified spider mite on strawberries was obvious.

Table 12. Strawberries, variety Deutsch Evern.
Treatments: 21/5, 30/5, 14/6.

	Number of spider mites per 100 leaves	
	11/6	17/6
WP 155 25% w.p. 0.1%	208	114
WP 155 25% w.p. 0.2%	86	96
dinocap 25% w.p. 0.1%	684	420
Control	444	964

There are indications that on apples the activity of WP 155 on capsids and sawflies is of practical importance. The effect on woolly aphid in the concentration used against mildew is probably negligible.

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TRIPHENYL TIN HYDROXIDE, A FUNGICIDE FOR THE CONTROL OF PHYTOPHTHORA INFESTANS ON POTATOES, AND SOME OTHER FUNGUS DISEASES

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Summary

A survey is given of laboratory investigations into the fungicidal activity of some organotin compounds. Results are given of field trials with some representatives of the triphenyl tin compounds, especially for the control of *Phytophthora infestans* in potatoes. Excellent results were obtained particularly with formulations of triphenyl tin hydroxide. Favourable results were also obtained in the control of *Cercospora beticola* on sugar beets. Results of tests against *Phytophthora infestans* and *Cladosporium fulvum* on tomatoes are promising.

In 1950 the Institute for Organic Chemistry, T.N.O. at Utrecht, was requested by the International Tin Research Council in London to carry out scientific and field investigations in the field of organotin compounds. van der Kerk and Luijten (1954) described the high fungicidal activity *in vitro* of alkyl tin compounds. The fungicidal activity appeared to increase in this direction:- $RSnX_3 \rightarrow R_2SnX_2 \rightarrow R_4Sn \rightarrow R_3SnX$

Table 1. Influence of the number of alkyl groups directly attached to the tin atom on the fungicidal properties of tehyl tin compounds.

		Minimum inhibiting conc. (p.p.m.)			
		Botrytis allii	*Penicillium italicum*	*Aspergillus niger*	*Rhizopus nigricans*
Tetraethyltin	$(C_2H_5)Sn$	50	>1000	100	100
Triethyltin chloride	$(C_2H_5)_3SnCl$	0.5	2	5	2
Diethyltin dichloride	$(C_2H_5)_2SnCl_2$	100	100	500	200
Ethyltin trichloride	$C_2H_5SnCl_3$	>1000	>1000	>1000	>1000
Stannic chloride	$SnCl_4$	>1000	>1000	>1000	>1000
Stannous chloride	$SnCl_2.2\ H_20$	>1000	>1000	>1000	>1000

(after Van Der Kerk and Luijten (1954))

Only the triethyl tin chloride appeared to be strongly fungicidal. The main influence on the fungicidal effect is exerted by the number of ethyl groups. The nature of the fourth group is of less importance in the triakyl tin compounds for the chloride, sulphate, hydroxide, acetate, etc. showed all practically the same activity. Variation of the alkyl group yielded the results of Table 2, also taken from the publication by van der Kerk and Luijten.

Table 2. Influence of the nature of the group R on the fungicidal properties of compounds $R_3Sn0.C0.Ch_3$

	Minimum inhibiting concentration (p.p.m.)			
	Botrytis allii	Penicillium italicum	Aspergillus niger	Rhizopus nigricans
Trimethyltin acetate	20	20	200	200
Triethyltin acetate	1	2	5	2
Tri-n-propyltin acetate	0.1	0.1	1	1
Triisopropyltin acetate	0.1	0.1	1	0.5
Tri-n-butyltin acetate	0.1	0.1	0.5	0.5
Tri-n-hexyltin acetate	1	10	20	100
Tri-n-octyltin acetate	>100	>100	>100	>100
Triphenyltin acetate	2	1	0.5	10
Phenylmercuric acetate	0.5	0.5	0.5	5

The order of increasing fungicidal activity appeared to be as follows: octyl → methyl → ethyl → propyl ≈ butyl.

For the sake of comparison, the well-known mercury fungicide phenyl mercuric acetate was included in the tests.

It is clear that, in the group of the trialkyl compounds, the effect of the tripropyl, triisopropyl and tributyl compounds is sufficient to place the products in the category of powerful fungicides. Though these fungicides may be considered for industrial application, the investigations regarding their effect on agricultural crops have led to disappointment. At concentrations required to control the disease, all the above mentioned fungitoxic trialkyl compounds appeared to be too phytotoxic.

As seen from Table 2, the triaryl compound, triphenyl tin acetate, has a fungicidal effect which is 10 to 20 times less than that of the best trialkyl compounds, but this substance could nevertheless still be looked upon as a powerful fungicide. Tests revealed that this substance is well tolerated by many plants, even in concentrations considerably higher than those required for fungus control.

The high fungicidal activity of the trialkyl compounds in vitro was not revealed under field conditions whereas the triaryl tin compounds, appeared to be powerful fungicides under these conditions. As a consequence particular attention was paid to the triphenyl tin compounds. It soon became clear that the X in the general formula R_3SnX exerted a considerable influence, if not on the fungitoxic, then on the phytotoxic characteristics of the product, which deferred the use of several compounds. On the basis of laboratory experiments, the following sequence of decreasing phytotoxicity was found:- triphenyl tin acetate, triphenyl tin hydroxide, triphenyl tin p-toluene sulfonamide. Fungitoxic tests on Fusarium culmorum, P. infestans and Venturia inaequalis led to this sequence of increasing activity:- triphenyl tin oxalate, triphenyl tin hydroxide, triphenyl tin acetate. On the basis of these and other data, obtained in the laboratory, it was decided to arrange larger field trials with triphenyl tin acetate, triphenyl tin oxalate, triphenyl tin hydroxide and triphenyl tin p-toluene sulfonamide.

All these compounds gave a better control of P. infestans on potato tubers than copper oxychloride. The leaf infestation was always better controlled by the tin compounds than by copper oxychloride, though the differences were smaller towards the end of the season than at the beginning.

To illustrate the point, results are given of two trials on potatoes (Tables 3 and 4). In Table 3 the various preparations were sprayed at identical dosages. A total of 6 spraying was carried out between June 24 and August 27. The seasonal averages of the leaf infestation were based on 6 observations between August 14 and September 6. The scale used was: 10 = free from Phytophthora, and 0 = completely dead.

After statistical analysis it appears that triphenyl tin acetate, and triphenyl tin hydroxide yielded the best control, but that their mutual differences were statistically not significant at a level $P = 0.05$. Triphenyl tin oxalate, copper oxychloride and triphenyl tin p-toluene sulfonamide, though statistically highly significantly better at a level $P = 0.01$ than untreated lagged behind the products of the first-mentioned group.

With reference to phytotoxicity, 10 stands for "no injury" and 0 = completely dead. Much injury, particularly in the later growth occurred with triphenyl tin acetate. Only slight injury was caused by the other compounds. Triphenyl tin p-toluene sulfonamide and triphenyl tin hydroxide were the safest and even better than copper oxychloride.

Practically all treatments led to a higher yield of healthy tubers than copper oxychloride. However, attention should be drawn particularly to the percentage of tubers infected by Phvtophthora. In all the tin treatments, except triphenyl tin p-toluene sulfonamide, the percentage of infected tubers is significantly lower than with copper oxychloride.

Table 3. Niew Vennep (clay, variety Bintje)

	Spraying dates and dosages in kg/ha							Seasonal averages leaf infestation 1)		Average injury figures 3)		Average yield of 50 plants			
	24/6	3/7	15/7	25/7	6/8	18/8	27/8		Transf. 2)	18/8	Transf. 2)	Healthy in kg	copper oxychl. = 100	% diseased	Transf. 2)
1. Untreated								2.4	2.75	10	5.74	40.2	81	19.9	26.41
2. Copper oxychloride 50%	7	8	9	10	10	10	10	5.6	4.29	8.1	5.15	49.7	=100	8.6	16.80
3. TPT-acetate 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	2.4	7.4	4.94	6.6	4.64	53.1	107	2.6	8.75
4. TPT-oxalate 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	2.4	6.1	4.46	8.3	5.20	53.4	107	4.9	12.67
5. TPT-hydroxide 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	2.4	7.1	4.84	9.4	5.55	52.4	105	3.1	10.03
6. TPT-p-toluene sulfonamide 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	2.4	4.4	3.76	9.6	5.61	49.4	99	11.9	19.95
P 0.05									0.49		0.31	4.7			4.64
P 0.01									0.74		0.47	7.2			7.03

1) 10 = free from Phytophthora; 0 = completely dead.
2) Transformation applied: bg sin $\sqrt{\frac{x}{10}}$
3) 10 = no injury.

Table 4. Boekesteyn (sand, variety Bintje)

	Spraying dates and dosages in kg/ha on:						Seasonal averages leaf infestation 1)		Average injury figures 3)		Average yield of 50 plants			
	16/6	26/6	4/7	15/7	25/7	13/8		Transf. 2)	30/7	Transf. 2)	Healthy in kg	copper oxychl. = 100	% diseased	Transf.
1. Untreated							2.8	3.02	0	0.40	43.9	108	3.6	10.63
2. Copper oxychloride 50%	7	8	9	10	11	11	5.6	4.31	0.9	3.46	40.7	=100	10.6	18.59
3. TPT-acetate 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	6.3	4.55	2.9	5.93	42.5	104	4.4	12.19
4. TPT-oxalate 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	5.2	4.15	1.1	3.78	45.4	112	3.4	10.75
5. TPT-hydroxide 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	6.4	4.59	1.8	4.55	44.6	110	6.2	14.49
6. TPT p-toluene sulfonamide 20% w. p.	1.8	2.0	2.2	2.4	2.4	2.4	4.0	3.61	0.3	1.09	42.5	104	4.5	12.37
P 0.05								0.34		0.96				3.55
P 0.01								0.51		1.45	N.S. 4)			5.38

1) 10 = free from Phytophthora; 0 = completely dead
2) applied transformation: $\text{bg}\sin\sqrt{x}$
3) 0 = no injury; 1, 2, 3 = light, moderate and heavy injury, respectively.
4) N.S. = Statistically not significant.

It appears, from Table 4, that the control of Phytophthora in the haulm obtained by the hydroxide and the acetate was better than that obtained by copper oxychloride, though the differences were statistically not significant. In this table phytotoxicity is expressed by the following figures: 0 = no injury; 1 = slight injury; 2 = moderate injury; 3 = heavy injury. The injury was lowest for triphenyl tin p-toluene sulfonamide; then came copper oxychloride, triphenyl tin oxalate, triphenyl tin hydroxide. Triphenyl tin acetate produced very serious injury. As compared with copper oxychloride, all the treatments gave higher yields; the best in this respect were triphenyl tin oxalate and triphenyl tin hydroxide. The percentage of diseased tubers was in all the tin treatments significantly lower than with copper oxychloride.

In view of the results obtained, it was decided to continue the field investigations mainly with triphenyl tin hydroxide and triphenyl tin acetate. Unfortunately, however, very little Phytophthora occurred the next year, so that little information was obtained on the control of the disease. Important data were obtained, on the other hand, on phytotoxicity, as shown in Table 5.

Table 5

	Dosage in kg/ha	Average injury figure on 14.8
Untreated	-	0
Copper oxychloride	7 - 10	0
Triphenyl tin acetate w.p. 20%	1.2 - 1.5	1.3
Triphenyl tin hydroxide w.p. 20%	1.2 - 1.5	0

Figures: 0 = no injury; 1 = slight injury; 2 = moderate injury;
3 = heavy injury

It had meanwhile been found that the degree of phytotoxicity of the tin compounds could be influenced to a considerable extent by the formulation. It became evident, for instance, that besides the influence the particle size of the active substance has on the biological behaviour, the addition of certain organic colloids to the formulation can reduce the phytotoxicity, pH also appeared to exert a considerable influence. The application of this knowledge to new formulations led to improvements in the field results; though favourable fungitoxic properties were maintained, the phytotoxicity was reduced.

Once again it appeared that on potatoes, the hydroxide was better than the acetate. Some injury was still caused by the new acetate formulation but the triphenyl tin hydroxide formulation gave hardly any injury. Table 6 illustrates the effect of triphenyl tin hydroxide in various dosages compared with, amongst others, triphenyl tin acetate. If triphenyl tin hydroxide and triphenyl tin acetate are compared at similar dosages, it will be seen that the seasonal average of the Phytophthora infestation for triphenyl tin hydroxide was 5.6 and for triphenyl tin acetate 5.3. For this evaluation a scale in which 0 = heavy infestation and 10 = no infestation, was used.

The scale for injury runs from 0 = no injury to 5 = serious injury. That caused by triphenyl tin hydroxide in this trial was 0.7 against 1.5 for the acetate. All the tin treatments gave higher yields of healthy tubers than the untreated.

Table 7 shows that triphenyl tin hydroxide controlled the leaf infestation better than triphenyl tin acetate, and that the net tuber yield and the percentage of diseased tubers was lower with triphenyl tin hydroxide than with triphenyl tin acetate.

Table 6. Boekesteyn (sand, variety Bintje)

	Spraying dates and dosages in kg prep. per ha. on:				Seasonal averages leaf infestation 1)	Transf. 2)	Average injury figures 3)	Average yield of 50 plants in tubers >30 mm.	
	15/6	11/7	27/7	4/8				Healthy in kg	% diseased
Untreated					3.2	34.8	1.0	32	12.8
Triphenyl tin acetate w.p.20%	1.5	1.6	1.7	1.8	5.3	46.1	1.5	34	13.8
Triphenyl tin hydroxide w.p.20%	1.5	1.6	1.7	1.8	5.6	49.4	0.7	34	13.0
Triphenyl tin hydroxide w.p.20%	1.8	2.0	2.2	2.4	5.8	50.1	0.8	38	13.2
Triphenyl tin chloride w.p.20%	1.5	1.6	1.7	1.8	5.4	47.6		34	15.9
P 0.05						6.3		N.S.	N.S.
P 0.01						9.5			

1) 10 = free from Phytophthora infestans; 0 = completely dead
2) Transformation: bg sin $\sqrt{10x}$
3) 0 = injury
1, 2, 3 = light, moderate and heavy injury, respectively.

Table 7. Boekesteyn (sand, variety Bintje)

	Spraying dates and dosages in kg prep. per ha on:				Seasonal averages leaf infestation 1)	Transf. 2)	Average injury figures 3)	Average yield of 50 plants in tubers >30 mm.	
	15/6	11/7	26/7	4/8				Healthy in kg	% diseased
Untreated					2.8	31.6	0.8	42	9.1
Triphenyl tin acetate w.p. 20%	1.5	1.6	1.7	1.8	4.7	43.6	1.3	36	15
Triphenyl tin hydroxide w.p. 20%	1.5	1.6	1.7	1.8	5.1	46.6	1.3	39	12.5
Triphenyl tin chloride w.p. 20%	1.5	1.6	1.7	1.8	4.9	44.5	1.5	41	11.3
P 0.05						1.7		N.S.	N.S.
P 0.01						2.6			

1) 10 = free from Phytophthora infestans; 0 = completely dead
2) Transformation bg sin $\sqrt{10x}$
3) 0 = no injury. 1, 2, 3 light, moderate and heavy injury, respectively.

Finally a few remarks on uses other than on potatoes. On celeriac, triphenyl tin hydroxide in 0.3% appeared to give a good control of Septoria apii. On beets, Cercospora can be controlled effectively with triphenyl tin hydroxide. The dosage in this application was taken at the same level as that applied on potatoes, but will be further investigated.

Encouraging results were obtained on tomatoes in the control of P. infestans and Cladosporium fulvum. Injury occasionally occurred with the old formulations, but the new formulations of triphenyl tin hydroxide were much better in this respect. On apples complete control of scab was obtained, even with a small number of sprayings. Although some phytotoxicity occurred, the triphenyl tin hydroxide formulation approved to be safer than triphenyl tin acetate. Table 8 provides some data on results with triphenyl tin compounds on apples.

Table 8. 's-Graveland, Golden Delicious

	Dosages	Fruit russeting	Yields in kg
Triphenyl tin acetate w. p. 20%	0.1 - 0.05%	6	1500
Triphenyl tin hydroxide w. p. 20%	0.1 - 0.05%	6.5	1500
captan w. p.	0.25 - 0.125%	8	1800
P 0.01			554
P 0.05			366

Evaluation figures for fruit russeting: 1 = complete russeting
10 = no fruit russeting at all.

The quality of the apples treated with triphenyl tin hydroxide and triphenyl tin acetate was below that of the fruit treated with captan.

In another trial on apples, triphenyl tin hydroxide produced a higher yield than triphenyl tin acetate and captan, but the russeting of the apples was considerable (Table 9).

Table 9. Sevenum, Golden Delicious

	Yield in kg. per 3 trees
Triphenyl tin acetate w. p.	436
Triphenyl tin hydroxide w. p.	552
captan w. p.	480

A further investigation with the triphenyl tin hydroxide formulation, which has meanwhile been marketed for potatoes, beets and celery, is being made on various crops and in regions with different climates.

Finally the following chemical, physical and toxicological data are given:

Triphenyl tin hydroxide

Gross formula: $C_{18}H_{16}0\ Sn$
Molecular weight: 366.7
Appearance: white crystalline substance; Melting point: 118 - 120° C.
(If the substance is slowly heated, water is evolved)
Solubility: in ether at 20° C. 28 gm/l; 1.2 dichlorethane: 74 gm/l; methylene chloride: 171 gm/l; benzene: 41 gm/l; ethanol: abt. 100 gm/l; practically insoluble in water.

Acute toxicity in mice in mg/kg with 95% confidence limits.

		oral	intraperitoneal
LD 50	♀	511 (243 - 1080)	10.8 (6.65 - 17.5)
		619 (342 - 1120)	17.1 (12.6 - 23.3)

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PRELIMINARY RESULTS WITH A NEW COMPOUND FOR THE CONTROL OF APPLE MILDEW AND RED SPIDER IN THE U.K.

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Summary

HOE 2784 (2-s-butyl-4, 6-dinitrophenyl 3, 3-dimethylacrylate) has been found in Germany to be active against both apple mildew and red spider. Work on red spider in progress for the past two years in the U. K. and on apple mildew for the past year, and the compound has been found to be extremely effective for the control of apple mildew. When used in a programme of sprays every 14 days for the control of mildew, the control of red spider has been good and it has not been found necessary to include any other acaricide in the spray programme. When used as an acaricide alone in a programme of only two sprays the control of Fruit Tree Red Spider mite has been poor under U. K. conditions, and although more are required to give adequate control of this pest, it may not require the six or seven applications of a mildew programme.

Results are presented to illustrate the activity of the compound against both red spider and mildew and details are given showing how the compound is expected to fit into commercial spray programmes in the U. K.

Introduction

HOE 2784 has been developed in Germany by Farbwerke Hoechst A. G. and is based on the active compound 2-s-butyl-4, 6-dinitrophenyl 3, 3-dimethylacrylate. The compound was first reported by Emmel and Czech (1960) to have good acaricidal properties and in particular to be effective for the control of mites which have become resistant to organo-phosphorus or chlorinated hydrocarbon acaricides. Recently Hartel (in print) reported that the compound was also very effective for the control of true powdery mildews and discussed the relationship of chemical configuration to red spider and powdery mildew activity. In this work he showed that of all the compounds tested, the dimethylacrylate ester of dinoseb had the best all-round activity to spider and mildew with minimum phytotoxicity. In the following paper the general properties of the compound will be briefly reviewed and results presented on the performance of the compound in trials in the U. K. during 1961.

General Properties

The technical material is a white solid with a melting point of 67 - 69°C and a faint aromatic odour. It is insoluble in water but soluble in all the usual organic solvents.

Residues in plant material can be determined by Pietzka's method. The ester is extracted from the plant material with n-heptane and hydrolysed with sodium ethylate. The dinoseb formed is steam-distilled to remove interfering plant material and measured colorimetrically in dilute NaOH at a wavelength of 420 mμ

The compound is formulated as a 25% dispersible powder and the method of formulation is extremely critical. Faulty formulation can lead to almost complete deactivation of the active material. It is compatible with all normal fungicides such as captan, zineb and thiram. All dispersible powder formulations of insecticides are compatible apart from those based on organo-phosphorus compounds, with which phytotoxicity has been reported from Europe.

Activity against Powdery Mildews

Exploratory work by Besemer (1961) has shown that there are differences in the susceptibility of individual mildew species to HOE 2784. The compound has poor activity against mildew on roses, oaks and wheat but is particularly effective against apple mildew, and work so far carried out in the U. K. has only been against this disease.

Field Trials

Five field trials on Cox's Orange Pippin were carried out in the U. K. during 1961, three sprayed with hand lances using fully replicated plots and two applied with automatic equipment in unreplicated strips.

The three replicated trials were identical in design and situated in Cambridgeshire and Nottinghamshire. They were based on duplicate tree plots with a minimum of four replicates per treatment. These were applied with hand lances at 100 lb/in. as a drenching spray commencing at Pink Bud and repeating every 14 days as far as the weather permitted, to give a total of seven sprays through the season. Primary infection was assessed by counting infected fruit spurs at Pink Bud and secondary infection by selecting 10 extension shoots at random on each tree. The number of infected and clean leaves are counted and then expressed as a percentage infection. Results are given in Tables 1a, 1b and 1c.

Statistical analysis was carried out by using angular transformation of percentages.

Table 1a. Combined Apple Mildew/Red Spider trial carried out in Cambridgeshire

				Mildew Assessment		
Treatment	Rate/ 100 gall	% Primary infection at Pink Bud	% infected leaves 14.6.61	% infected leaves 7.7.61	% infected leaves including leaves dropped 27.7.61	% leaf drop 27.7.61
HOE 2784	1 lb	21.3	19.2	32.0	35.3	6.5
HOE 2784	2 lb	19.2	12.7	27.5	21.3	5.5
Dinocap	1 lb	24.1	22.9	28.5	35.3	6.8
Untreated	-	17.9	48.7	53.2	52.2	9.3

HOE 2784 at 2 lb is significantly better than all other treatments on 1st and 3rd assessments (p = 0.01).
All treatments significantly better than untreated (p = 0.01).
Dates of application:- 18.4.61, 4.5.61, 17.5.61, 31.5.61, 15.6.61, 28.6.61 and 17.7.61 of 25% dispensible powders.

Table 1b. Trial carried out in Nottinghamshire, Site A

				Mildew Assessment		
Treatment	Rate/ 100 gall	% Primary infection at Pink Bud	% infected leaves 30.5.61	% infected leaves 4.7.61	% infected leaves including leaves dropped 17.8.61	% leaf drop 17.8.61
HOE 2784	1 lb	5.3	8.2	27.2	19.4	2.2
HOE 2784	2 lb	2.3	5.8	12.2	10.6	2.8
Dinocap	1 lb	3.1	14.1	22.2	15.6	2.7
Untreated	-	1.9	22.2	52.4	33.5	6.2

HOE 2784 at 2 lb significantly better than all other treatments (p = 0.05); all treatments significantly better than untreated (p = 0.01) except dinocap at first assessment (not significant).
Dates of application:- 20.4.61, 5.5.61, 18.5.61, 1.6.61, 16.6.61, 29.6.61.and 18.7.61.

Table 1c. Trial carried out in Nottinghamshire, Site B

Treatment	Rate/ 100 gall	Mildew Assessment % Primary infection at Pink Bud	% infected leaves 29.5.61	% infected leaves 6.7.61	% infected leaves including leaves dropped 10.8.61	% leaf drop 10.8.61
HOE 2784	1 lb	15.7	8.6	21.3	19.4	2.3
HOE 2784	2 lb	10.1	1.9	16.9	18.7	3.0
Dinocap	1 lb	15.2	8.9	26.5	19.8	2.8
Untreated	-	12.5	30.0	66.0	37.3	6.7

HOE 2784 at 2 lb significantly better than all other treatments on 1st assessment only ($p = 0.01$); all treatments significantly better than untreated ($p = 0.01$).
Dates of application:- 19.4.61, 3.5.61, 17.5.61, 1.6.61, 16.6.61, 30.6.61 and 12.7.61.

These results show that rate for rate, HOE 2784 is as good as dinocap in mildew control. The cost of HOE 2784 permits a higher rate to be used, however, and it is clear that the 2 lb rate is giving a better control. No spray damage have been noted from any of the treatments. The incidence of mildew in all trials has been the highest recorded for some years. Of the two sites in Nottinghamshire, Site A had the lower primary infection but secondary infection built up rapidly as the season progressed. Site B had a heavy primary infection which caused a rapid build-up of secondary infection by the 6th July. Heavy rainfall occurred after this date and the infection receded naturally which explains the lower counts on the third assessment. Primary infection counts from all plots will be made in the spring of 1962.

The two unreplicated trials using automatic equipment on Cox's Orange Pippin were carried out in Nottinghamshire and Essex. One was designed to test the compound at high and medium volumes using a seven spray programme and the other to give a field comparison in one of the worst mildew areas in Essex, where six sprays were applied. The results are given in Tables 2a and 2b.

Table 2a. Unreplicated plots in Nottinghamshire, Site A

			Mildew Assessment				
Treatment	Rate/ acre	Vol. spray/ acre	% Primary infection at Pink Bud	% infected leaves 30.5.61	% infected leaves 4.7.61	% infected leaves 17.8.61	% leaf drop 17.8.61
HOE 2784	4 lb	200 gall	2.5	2.1	5.9	14.5	2.6
HOE 2784	4 lb	50 gall	2.6	1.9	6.1	15.4	2.6
Dinocap	2 lb	200 gall	2.0	1.3	6.6	14.1	3.0
Untreated	-	-	1.8	15.3	33.8	44.4	3.3

Dates of application:- 21.4.61, 8.5.61, 19.5.61, 2.6.61, 16.6.61, 30.6.61 and 14.7.61.

Table 2b. Combined Apple Mildew/Red Spider trial in Essex

Treatment	Rate/ acre	Vol. spray/ acre	% Primary infection at Pink Bud	% infected leaves 13.6.61	% infected leaves 14.7.61	% infected leaves 9.8.61	% leaf drop 9.8.61
HOE 2784	4 lb	200 gall	50.8	19.6	44.0	33.8	9.4
Dinocap	2 lb	200 gall	39.4	29.6	54.5	49.8	21.5

Dates of application:- 20.4.61, 8.5.61, 25.5.61, 16.6.61, 3.7.61 and 19.7.61.

Taking into account the higher primary infection counts on the HOE 2784 treatments, the results confirm under "commercial conditions" the findings in the smaller hand-sprayed trials and indicate that HOE 2784 can be sprayed at either high or medium volume to give the same degree of control of apple mildew.

In the Essex trial (Table 2b) mildew has been a very serious problem for three years and the primary infector counts illustrate this point. The grower concerned was satisfied with the control given by HOE 2784 and the figures suggest that the 'build-up' of mildew has been checked.

Activity against Red Spider Mites

Emmel and Czech (1960) and Lhoste and Lambert (1961) reported that HOE 2784 was active against red spider mites, including the Glasshouse Red Spider Mite and the Fruit Tree Red Spider mite which are of economic importance in the U. K. Laboratory tests have confirmed this activity. However, as damage was seen in tests on tomatoes, the main acaricidal work has been on top fruit and in particular against the Fruit Tree Red Spider mite.

After preliminary work in 1960, five field trials were carried out in 1961. Two of the trials were combined with those for apple mildew so that both the efficiency of the compound in a two spray acaricidal programme and the effect of a six or seven spray mildew programme could be observed. In the acaricidal programme two sprays were applied, the first at Pink Bud when the mites were beginning to hatch from the winter eggs and the second in June with the first codlin spray. The timing of the first application at Pink Bud was based on experience on the Continent. A timing trial carried out in Nottinghamshire in 1961 showed that there was no significant difference ($p = 0.05$) in the control obtained between applications of the compound at Pink Bud or Petal Fall.

As with the Mildew work trials were of the two types, three being replicated plots sprayed with hand lances and the other two unreplicated strips sprayed with automatic machines.

The replicated trials on Cox's Orange Pippin were based on one or two tree plots with at least four replicates per treatment. Red spider was assessed by taking a minimum of 50 leaves per plot and brushing the mites on to cards using a mite brushing machine similar to that described by Henderson and McBurnie (1943). The mites were then counted into one of three stages: adults, nymphs and larvae, and eggs.

Results are given in Tables 3a, 3b and 3c.

Table 3c. Combined Red Spider/Mildew trial in Cambridgeshire

Treatment	Rate/ 100 gall	No. of sprays	Fruit Tree Red Spider assessment (No. of mites/leaf)								
			7th June, 1961			10th July, 1961			24th Aug. 1961		
			Adults	N & L	Eggs	Adults	N & L	Eggs	Adults	N & L	Eggs
1. HOE 2784	2 lb	7	0.00	0.04	0.32	0.04	0.12	0.56	0.04	0.60	2.00
2. HOE 2784	1 lb	7	0.08	0.12	1.36	0.00	0.00	0.76	0.08	1.56	5.08
3. Dinocap	1 lb	7	0.24	0.60	1.40	0.00	0.40	0.96	0.60	4.52	10.32
4. HOE 2784	2 lb	2*	0.00	0.00	0.52	0.00	0.16	0.80	0.28	2.00	4.64
5. HOE 2784	1 lb	2*	0.04	0.24	1.00	0.00	0.56	0.36	0.60	2.68	7.52
6. Phenkapton	16 fl. oz	2*	0.00	0.00	0.20	0.00	0.04	0.04	0.04	0.48	1.24
7. Untreated	-	0	0.32	0.28	6.80	0.48	2.92	18.84	-	-	-
Least difference required for significance		$p = .05$	-	-	2.09	-	-	2.96	-	-	4.41
		$p = .01$	-		2.80	-	-	3.97	-	-	5.91

Dates of application

7 spray treatments, 19.4.61, 3.5.61, 12.5.61, 1.6.61, 16.6.61, 30.6.61 and 12.7.61.
*2 spray treatments, 19.4.61 and 16.6.61.
*Dinocap was applied at 1 lb/100 gallons on treatments 4 and 5 on 3.5.61, 12.5.61, 30.6.61 and 12.7.61 and on all dates on treatment 6.

Table 3b. Trial carried out in Essex

Treatment	Rate/ 100 gall	Fruit Tree Red Spider assessment (No. of mites/leaf)								
		29th May 1961			6th July 1961			1st August 1961		
		Adults	N & L	Eggs	Adults	N & L	Eggs	Adults	N & L	Eggs
1. HOE 2784	1 lb	1.34	0.38	4.16	0.77	6.27	18.18	5.84	64.80	39.52
2. HOE 2784	2 lb	0.26	1.15	1.60	1.73	2.82	16.51	4.48	67.68	46.56
3. Phenkapton	16 fl. oz	0.06	0.45	0.38	0.13	0.70	4.10	0.64	5.44	5.60
4. Untreated	-	1.98	0.45	6.27	2.18	7.81	32.38	5.20	106.08	68.08
Least difference required for significance	p = .05		-	2.32	-	-	9.17	-	-	16.63
	p = .01		-	3.13	-	-	12.35	-	-	22.40

Dates of application

Treatments 1 and 2	Pink Bud - 17th April, 1961.
Treatments 3	Petal Fall - 9th May, 1961.
All treatments	1st Codlin Spray - 13th June, 1961.

Table 3c. Trial carried out in Nottinghamshire

Treatment	Rate/ 100 gall	Fruit Tree Red Spider assessment (No. of mites/leaf) 20th June, 1961 Adults	N & L	Eggs	26th July, 1961 Adults	N & L	Eggs
1. HOE 2784	2 lb	0.40	1.24	17.28	0.36	9.40	12.88
2. HOE 2784	1 lb	0.40	4.56	25.08	1.28	24.92	25.68
3. Phenkapton	16 fl. oz	0.04	0.32	1.72	0.12	1.36	2.16
4. Demeton-methyl	12 fl. oz	0.04	0.64	2.76	0.00	1.44	3.04
5. Untreated	-	0.52	5.72	33.44	2.28	41.80	27.72
Least difference required for significance	p = .05	-	-	11.76	-	-	9.84
	p = .01	-	-	15.73	-	-	13.13

Dates of application

Treatments 1 and 2 Late Pink Bud - 24th April, 1961.
Treatments 3 and 4 Petal Fall - 10th May, 1961.
All treatments 23rd June, 1961.

These results show that the compound, even at 2 lb/100 gallons, does not give a satisfactory control of Red Spider when applied only twice during the season. When, however, it is applied regularly in a mildew programme, the control is adequate and no additional acaricide should be needed.

Both of the automatic machine trials were in Essex; in one a two spray acaricidal programme using medium and high volume was compared to a six spray mildew programme at high volume. The other trial was to compare at high volume a two spray acaricidal programme with a grower's spray schedule.

As the plots were unreplicated, sampling of red spider was carried out using 10 marked trees per plot. 50 leaves were taken at random from each of the marked trees making a total of 500 per plot. Results are given in Tables 4a and 4b.

Table 4a. Combined Red Spider/Mildew trial in Essex, Site A

Treatment	Rate & volume/ acre	No. of sprays	Fruit Tree Red Spider assessment (No. of mites/leaf)								
			29th May, 1961			6th July, 1961			1st Aug. 1961		
			Adults	N & L	Eggs	Adults	N & L	Eggs	Adults	N & L	Eggs
1. HOE 2784	4 lb in 200 gall	6	0.03	0.48	1.44	0.00	0.19	1.38	0.06	1.06	3.39
2. HOE 2784	4 lb in 200 gall	2	0.16	0.19	0.70	0.10	1.73	4.48	0.96	20.06	17.25
3. HOE 2784	4 lb in 50 gall	2	0.29	0.35	1.06	0.26	2.27	5.70	0.51	14.43	14.82

Variety: Cox's Orange Pippin.
Dates of application: 20.4.61, 8.5.61, 25.5.61, 16.6.61, 3.7.61 and 19.7.61.

Table 4b. Trials carried out in Essex, Site B

Treatment	Rate/ 100 gall	Fruit Tree Red Spider assessment (No. of mites/leaf)								
		29th July, 1961			6th July, 1961			1st Aug. 1961		
		Adults	N & L	Eggs	Adults	N & L	Eggs	Adults	N & L	Eggs
1. HOE 2784	2 lb	0.58	19.36	1.28	4.29	22.01	31.36	0.22	17.34	48.10
2. Phosphamidon & malathion	16 fl.oz 30 fl.oz	0.13	23.84	0.10	0.35	5.09	3.55	0.16	2.24	2.56

Variety: Worcester Pearmain.
Dates of application: Treatment 1 23.4.61, 16.6.61, 11.7.61 and 25.7.61.
Treatment 2 Phosphamidon 12.5.61
Malathion 19.5.61, 16.6.61 and 6.7.61.

* Bronzing was apparent on the 6th July in the HOE 2784 plot.

These results confirm those of the hand-sprayed trials that satisfactory control of Red Spider cannot be obtained by using only two sprays of the compound but when applied in a Mildew programme it is excellent. It may not, however, be necessary to apply as many as 6 or 7 sprays to obtain a satisfactory control.

The reasons for the inadequate control of a two spray programme under U.K. conditions appear to be two-fold. First, the persistence of the compound is only about fifteen days and, unless the hatch of mites from the winter eggs is very rapid, its potency will have gone before the hatch has been completed. Secondly the compound is unable to diffuse across the leaf surface and as a complete spray cover cannot usually be achieved, a larger number of mites survive the treatment than occurs with materials able to diffuse. Spraying several times increases the cover and consequently gives adequate control. Temperature may also be a factor as the compound has given excellent control of spider in some European countries.

Safety on apple varieties

A wide range of apples have been sprayed including all the main commercial varieties such as Cox's Orange Pippin, Worcester Pearmain, Lord Lambourne, Laxton Superb, Bramley and James Grieve and no varietal susceptibility has been observed.

Discussion

Even though apple mildew is one of the most difficult problems that growers have to face, many are not yet prepared to apply a seven spray programme of a mildew-specific fungicide because of the cost. The results of using HOE 2784 in such a programme show the excellent control of mildew and red spider obtained, and, as no additional acaricide is needed, it will be economic. Difficulties in controlling red spider are arising where the same acaricides are used too frequently without any rotation. The activity of HOE 2784 against mites resistant to other acaricides offers an alternative programme and will therefore help to prevent the build-up of strains resistant to other materials.

No great difficulty is foreseen in fitting HOE 2784 into commercial spray programmes. Although it may not be possible to use organo-phosphorus insecticides, insect pests can be controlled satisfactorily without phytotoxicity using a programme based on BHC-DDT pre-blossom, BHC at Petal Fall for sawfly and DDT or Sevin for codlin moth and summer tortrix. As the compound does not control apple scab an additional fungicide must be used and captan has proved satisfactory. Mixtures with thiram and zineb have also been used successfully on the Continent.

Conclusions

The results of the trials described show that HOE 2784 gives excellent control of apple mildew and red spider in a six or seven spray programme and will be an important addition to the pesticides available to the grower.

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Discussion

Q. Dr. W.E.Ripper

Menazon is obviously a major advance in selective aphis control and the research workers of Plant Protection Ltd., are to be congratulated. What is the mode of insecticidal action and how is the systemic effect achieved? As the prolonged protective effect is based on spray residues, may we ask whether the spray deposit is reasonably rainfast? Does the seed treatment of beet make the beet plants toxic to aphids long enough to convey protection during the months when aphids appear?

A. Mr. J.F.Newman

In reply to the first part, the mode of action of menazon is indeed an interesting subject on which we have limited information at the moment. With locusts, it has been found that menazon is a rather poor cholinesterase inhibitor. I have done some experiments with cercal reflex preparations of cockroaches and menazon is rather poor as a ganglionic blocking agent. I have also tried with cockroach preparations with the perineural membrane removed and there again menazon has poor activity.

With regard to the rainfastness of the deposit of menazon on the surface of leaves, we have had a certain amount of trouble with a dispersible powder formulation which we used in the early part of the season this year, and which was unsatisfactory on some leaves, especially the waxy leaves of sugar beet. We now have some very much improved formulations which we propose to use on sugar beet next year.

A. Mr. E.C.Edgar also replied to this question

With reference to the part of the question on sugar beet seed treatment, the period of protection depends to some extent on the dosage applied. Laboratory tests using 2.0% menazon by weight on the seed gave complete protection against aphid infestation for 6 weeks from sowing the seed and partial control for a much longer period. Higher loadings are desirable although 4.0% is the highest that can be achieved without employing special techniques involving stickers or pelleting. In one field trial this year a 2.0% w/w menazon seed dressing increased the yield of roots by 16%. This was on a late drilled crop and aphid attack was early in 1961.

A. Mr. J.F.Newman then enlarged further on the Mode of action

There are two possibilities. Either some sort of metabolism takes place in which menazon is converted into a more active inhibitor or menazon is active on an enzyme specific to aphids. These are two possibilities and more work is needed to elucidate the matter.

Q. Dr. Hubert Martin

Is the P_{32} revealed in Mr. de Pietri-Tonelli's extremely good radio autographs present in toxic form?

A. Mr. P. de Pietri-Tonelli

The first and second radio autographs were one of each leaf in order of peach trees and, of course, everything was radioactive when photographed. The black spots mark P32 from the insecticide and from all the other metabolites having P_{32}.

Q. Dr. L. Broadbent

The 1960 replicated trials with menazon were well done, but unfortunately sites were near diseased potato crops from which virus was carried to plants in the trials. Mr. Newman should not conclude from these two trials that menazon will be of value only in moderately satisfactory or marginal seed producing areas. Our spraying trials over several years have shown that a good aphicide will limit virus spread in many ware-growing districts and I would expect that further work will show menazon to be effective in such areas?

A. Mr. J. F. Newman

I think Dr. Broadbent is undoubtedly right and one really has to look at those results particularly in relation to the results on aphid populations. Over the last 10 years Dr. Broadbent, Mr. Burt and Mr. Heathcote have done a large number of experiments on the control of aphid-borne viruses in potatoes and they have shown quite clearly that the spray programme with 3 sprays will give the desired control of virus transmission in many parts of England and Wales. We have been able to show in much more limited experiments that we can get control by the tuber treatment which looks quite as good so far as aphid control is concerned and we suppose that the eventual virus control will also be as good. Farmers have not used the spray treatments to any great extent, due I think to the trouble involved in going through the crop several times. The relatively simple tuber application might be more attractive.

Q. Dr. W. E. Ripper

Are thrips killed by menazon?

A. Mr. J. F Newman

Menazon has low activity against thrips and we are quite as baffled as Dr. Ripper is at the moment.

Q. Dr. L. Broadbent

Has menazon been tried as a seed treatment on cereals and carrots to control virus in these crops?

A. Mr. J. F. Newman

In collaboration with Dr. Morgan Jones of the N. A. A. S., Wales, I did do a few trials on the cereal virus down in Pembroke. Dr. Morgan Jones selected fields which had showed very severe virus symptoms in past years, and we got a good attack of aphids as well, but no virus turned up whatever even in the controls.

Q. <u>Mr. M.J. Way</u>

Has Mr. Newman any information on the toxicity and persistence of menazon applied at the same rate per acre as a tuber treatment and as a granular application. In my experiments the same rate per acre was initially more toxic to aphids and also much more persistent when applied to the seed than when applied to granules to the seed harrow?

A. <u>Mr. J.R. Newman</u>

In the potato trials which I described, the rates were the same - 2 lb per ton in the case of tuber treatment and 2 lb per acre in the case of the granule treatment and planting was at the rate of 1 ton of seed per acre. Inevitably with granule treatment it takes time to get sufficient root development for the plant to pick up an adequate amount of menazon to produce control. In the trials, the aphid population had faded out by mid-July so we cannot say much about the relative persistence of granular and tuber treatments. The tuber treatment gave some protection right through to the middle of July.

<u>Mr. Edgar</u> added

The seed treatment is a placement technique getting the material exactly where it is wanted to give protection as soon as the seed germinates or the plant breaks through the soil, whereas with a granular treatment roots have to grow out before the toxicant is absorbed and the protection of the young plant may be delayed.

<u>Mr. Newman</u>

Carrots are one of the very few crops so far where we have obtained serious phytotoxicity with seed dressing. Foliar sprays might be more satisfactory

<u>Mr. Edgar</u>

I think Mr. Newman exaggerates phytotoxicity on carrots. It has occurred but I think we may be able to overcome this.

Q. <u>Mr. Worthing</u>

Can Mr. Elings give us any information of structure activity relationships in compounds of the WP 155 series? For instance, do small changes in structure completely remove the activity, or does the activity slowly tail-off with homologous compounds? How critical is the given structure?

A. <u>Mr. H. Elings</u>

We found quite a similar type of activity in rather a great number of derivatives of WP 155. So there are gradual changes in activity among members of the series and WP 155 is not to be considered an exception in it. WP 155 has been chosen also on other grounds as its excellent fungicidal properties.

Q. <u>Mr. E. Robert</u>

What is the mammalian or human toxicity of tin compounds?

A. Ir. A. J. Pieters

There are several data on the toxicity of triphenyl tin compounds, some of which you will find in the paper. In practice the toxicity of triphenyl tinhydroxide will for several applications give no difficulties. For instance it has been established that in potatoes no residues can be found in the tubers. Some data on the subject of chronic toxicity of the triphenyl tin compounds are known from literature. A Belgian publication named "Répertoire Toxicologique des Pesticides" of Simone Dormal and Georges Thomas (1960) states that 5 p.p.m. of triphenyl tinacetate in the food during 105 days caused no adverse effects to rats.

We can assume on physiological reasons that data on triphenyl tinacetate are applicable to triphenyl tinhydroxide also.

Q. Mr. I. F. Storey

During the past three years, we have carried out trials in Lincolnshire using tri-phenyltin acetate for the control of potato blight and have obtained very effective control both of the disease on the foliage and in the tuber. There has been no indication of any adverse effect on yield in spite of slight injury to the leaf from time to time. In the trials, we have obtained on average a yield increase of $1\frac{1}{2}$ tons per acre whereas with copper oxychloride the increase was of the order of 8 - 10 cwt per acre.

The tin compound has been the only one used which seems to have a direct effect on tuber blight. Is there any indication from the work in Holland how this effect has been obtained? Has there been a direct effect by protection of the tuber in the soil or is this the result of a toxic effect on the spores whilst present on the leaf?

A. Ir. A. J. Pieters

I regret not to be able to explain the mode of action of triphenyltinhydroxide on tuber blight. As far as we know no explanation of this phenomenon is available up till now. As far as herbicidal effects are concerned I do not think that we have seen such a thing in our trials; perhaps Mr. Elings could add to this being the man that has done the field trials.

Mr. Elings added

We sometimes see a slight scorching effect on some weeds, but they are by no means killed.

Q. Professor A. E. Muskett

Has Mr. Elings compared the effectiveness of WP 155 for the control of rose mildew with that for washing soda (2 oz per gallon) and milk?

A. Mr. Elings

The answer is no.

Q. Mr. A Casella

Could Mr. Higgons give information about toxicity of HOE. 2784?

A. Mr. D. J. Higgons

HOE 2784 is only a moderately toxic compound with an oral LD 50 to rat of 130 mg. / Kg. It has however, an extremely low toxicity by dermal absorption, and should therefore, be a relatively safe compound to use in practice.

Q. Mr. Peter G. Burgess

Has Mr. Elings tried WP 155 against rose mildews in glasshouses, especially with the rose variety Baccarat?

A. Mr. Elings

Yes, we have. The activity against mildew in roses in glasshouses is as good as against mildew on outdoor roses. Baccarat was included in many tests; we did not observe any phytotoxicity on this variety.

Q. Dr. Ripper

Has Dr. Elings tried to control Erisyphe graminis with WP 155 and has it a curative effect in severe outbreaks? Is it phloem translocated? Is it compatible with the standard plant hormone weedkillers?

A. Mr. Elings

Quite a number of the laboratory experiments were done with powdery mildew on barley, which is one of our standard test plants for powdery mildew screening. We made on field-test against Erisyphe on barley and the effect of WP 155 was good. So it is perhaps possible you could use WP 155 on cereals if you wanted to, but I am not sure it would not be too expensive for these crops. We did not yet look further into this matter and we do therefore not know if the curative effect would be sufficient in severe outbreaks. Probably WP 155 formulations are compatible physically and chemically with 2, 4D and MCPA-formulations. We do not yet know, how exactly WP 155 translocation takes place.

Q. Mr. H. G. Haigh

1. Have any comparative field trials of the organic tin compounds against Bordeau Mixture been carried out, similar to those described for copper oxychloride?

2. How does the cost of treatment with tin compounds compare with that for copper compounds, in view of the much higher price of tin?

3. In the tables in the paper, 50% presumably refers to the metal content of copper oxychloride. Does 20% refer to the metal content of the tin compound or does it refer to the % active ingredients?

A. Ir. A. J. Pieters

We have not used Bordeaux Mixture in our trials. Regarding the cost of the triphenyltinhydroxide, you have to realise that the concentration of the metal is very low as compared with copper. 50% refers to the metallic copper but 20% refers to the active compound viz triphenyltinhydroxide, which contains about 30% metallic tin. Regarding the price I think that it is necessary and it certainly seems possible to sell triphenyltinhydroxide at prices that will only slightly be higher than the copper products and I think in practice these two products will be used.