

**EFFECIENCY OF TWO ORGANOPHOSPHOURUS  
INSECTICIDES ON THE CITRUS WHITEFLY,  
*DIALEURODES CITRI* WITH SPECIAL  
REFERENCE TO THEIR RESIDUAL  
BEHAVIOUR ON ORANGE FRUITS.**

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**Abstract**

Two experiments were carried out during the two successive years 1986, 1987 in Tant El-Gizierah, Qalubia governorate to evaluate the efficiency of two organophosphorus insecticides against the citrus whitefly, *Dialeurodes citri* on navel orange trees. Selecron (profenofos) at the rate of 0.075% and Tokuthion 500 (prothiophos) at the rate of 0.15% were applied on orange trees and an untreated area was left for comparison. Both insecticides gave satisfactory results and no phytotoxicity was observed after application.

Residues of either pesticide were mainly detected on and in the orange peel, while the amounts penetrated into the pulp were negligible.

Pre - harvest intervals of 21 days and zero day were determined for prothiophos and profenofos according to the maximum residue limits of 0.05 and 3.0 ppm, respectively.

Persistence of either chemical followed a similar pattern but the  $RL_{50}$  was 6.0 days for profenofos and 10.5 days for prothiophos.

As the amounts of residues determined in the orange peel were relatively high at the pre - harvest intervals and as it might be used in flavour and jam manufacture, further studies should be directed to investigate the effect of washing, heat and cooking processes on the disappearance of pesticides residues from the peel.

## INTRODUCTION

Profenofos (Selecron, Curacron) (0-4-bromo-2-chloro-phenyl 0-ethyl S-propyl phosphorothioate), and prothiophos (Tokuthion) (0-2, 4-dichlorophenyl) 0-ethyl S - propyl phosphorodithioate) are two organophosphorus insecticides recommended in Egypt for the control of mealybugs and scale insects infesting citrus, guava and grape trees. Efficacy of profenofos and prothiophos was previously mentioned by Koli and Makar (1981).

The citrus whitefly *Dialeurodes citri* Ashmead is a new citrus pest in Egypt where there is no insecticide recommended for its control. These two organophosphorus insecticides which are recommended in other countries are tested in Egypt to evaluate their efficiency against this pest under the Egyptian climatic conditions.

Studying the residual behaviour of pesticides applied on edible crops is a main objective to determine the pre-harvest intervals (PHI's) that should pass following application and before human consumption. PHI is an important element of good Agricultural practice (GAP) in order to get the maximum benefit of pesticides with the minimum health hazards by maintaining pesticide residues below the Maximum Residue Limits (MRL's).

In the present investigation, the behaviour and persistence of profenofos and prothiophos residues were also studied on and in orange peel and in orange pulp.

## MATERIALS AND METHODS

### Treatments and Sampling

Two experiments were carried out during 1986 and 1987 at Tant El-Gizierah, Qalyubia governorate to evaluate the efficiency of Profenofos and prothiophos against the common citrus whitefly *Dialeurodes citri* Ashmead.

Profenofos (72%EC) at the rate of 0.075 % and prothiophos (500EC) at the rate of 0.15% were applied in October 1986 on navel orange trees, Qalyubia governorate, as full coverage spray by means of high pressure reciprocating pump and manually operating guns.

Each treatment had four replicates at random, each replicate was composed of 16 trees (4x4) and the control plots were isolated outside the experimental boundaries.

Samples were taken from the middle four trees, fifty leaves from each replicate representing the four cardinal direction, i.e. 200 leaves per treatment. Samples were collected before and after spraying for one, two and three weeks. Living immature stages of whiteflies were counted and recorded. To evaluate the efficiency of the tested insecticides, percentage of pest reduction was determined according to Henderson and Tilton equation (1955).

### Residue analysis

Mature fruits were picked from the middle trees of each plot which constituted 16 trees. The samples were taken one hour following application and then occasionally until 21 days after treatment. Samples were kept at  $-20^{\circ}\text{C}$  in polyethylene bags until time of analysis.

Samples after getting to room temperature, were weighed, the peels were taken off and the pulp was weighed. Methanol was used for extraction of profenofos and prothiophos residues from peels and pulp orange samples. Partitioning between aqueous methanol / chloroform was carried out.

Deactivated florasil (6% water) column was used for clean-up of prothiophos residues using benzene as eluent for peel and pulp samples (Mollhoff 1975).

Column chromatography using 2% deactivated silica gel eluted with hexane/acetone (95/5) was adopted in the clean-up of peel and pulp samples containing profenofos residues according to Abou-zahw (1980).

A Pye - Unicam (104) gas chromatography equipped with flame photometric detector in the phosphorus mode (526 nm filter) was used for prothiophos detection under the following conditions: Column glass 5 ft. x 4mm i.d. packed with 4% SE-30 + 6% OV-210 on gas chrom Q 80 - 100 mesh. temperatures ( $^{\circ}\text{C}$ ): Column 230, injection port 250, detector 230 and gas flow rates ml/min  $\text{N}_2 = 100$ ,  $\text{O}_2 = 20$ ,  $\text{H}_2 = 90$ , air = 80. Chart speed: 0.7 cm/min. According to these conditions, the retention time of prothiophos (RT) was 2.4 minutes.

Profenofos was detected using a Pye-Unicam gas chromatography equipped with  $\text{Ni}^{63}$  electron capture detector under the following conditions: Column gas 7 ft.



x 4 mm. i.d. packed with 1.5 % OV - 17 1.95 % OV - 210 on gas chrom Q 80 - 100 mesh, temperatures ( $^{\circ}\text{C}$ ): Column 248, injection port 260 , detector 270. Nitrogen as the Carrier gas was used at flow rate of 75 ml/ min. Under these conditions, profenofos appeared after 4 minutes with a chart speed of 0.7 cm/min.

Recoveries of prothiophos and profenofos in peel samples were 82.38 % and 87.83 %, respectively and in pulp Samples were 80.54 % and 78.86%, respectively. Limits of detection were 0.01 and 0.005 ppm for prothiophos and profenofos in respect.

## RESULTS AND DISCUSSION

### Effect of insecticides on whitefly

Results shown in Table 1 indicated that Selecron provided over 98% reduction in the population of immature stages after three weeks from application. Tokuthion provided more than 94.15 % reduction during the same period. Statistical analysis of the results showed no significant differences in the potency of both chemicals.

### Residue analysis

Data presented in tables 2 and 3 demonstrate the amounts of prothiophos and profenofos residues determined on and in orange peel and in orange pulp. Magnitude of residues was calculated based on the whole fruit and percentages of persistence are also tabulated.

The initial deposits in the peel of prothiophos and profenofos that were 1.58 and 4.76 ppm, decreased to 1.25 and 3.2 and 3.24 ppm after 24 h and to 0.71 and 2.80 ppm after 3 days from application , respectively . However, at the end of the experimental period 0.41 and 1.84 ppm were detected for prothiophos and profenofos, respectively.

Prothiophos and profenofos residues detected in the orange pulp were negligible and never exceeded the limits of determination (0.01 and 0.005 ppm, respectively) . However, these figures indicate the limited penetration of the two pesticides as the residues were confined only on and in the peel.

Residues of prothiophos and profenofos determined on and in the peel and in the

Insecticides and rate of application	Reduction in <i>D. Citri</i> population at indicated periods %						
	1986			1987			Mean %
	7/10	14/10	21/10	2/10	7/10	14/10	
Selecron 720 EC ( at 0.075%)	98	99	99	96	98	100	98.35
Tokuthion 500 EC (at 0.15 %)	92	95	96	92	94	96	94.15

Table 2. Residues, persistence and half-life of prothiophos 500 on and in navel orange fruits at different intervals following treatment at the rate of 0.15%

Days after treatment	Amount of residues			Persistence on whole fruit basis %
	Determined		Calculated	
	on and in the peel ppm	on the pulp ppm	on and in the whole fruit ppm	
0	1.58	<0.01*	0.194	100.00
1	1.25	<0.01	0.179	92.27
3	0.71	<0.01	0.111	57.23
7	0.62	<0.01	0.105	54.12
14	0.50	<0.01	0.089	45.88
21	0.41	<0.01	0.051	26.92

Half life ( $RL_{50}$ ) = 10.5 days

\*Limit of detection = 0.01 ppm

Table 3. Residues, persistence and half-life of prothiophos 72% on and in navel orange fruits at different intervals following treatment at the rate of 0.075%

Days after treatment	Amount of residues			Persistence on whole fruit basis %
	Determined		Calculated	
	on and in the peel ppm	on the pulp ppm	on and in the whole fruit ppm	
0	4.76	<0.005*	1.279	100.00
1	3.24	<0.005	1.034	80.84
3	2.80	<0.005	0.815	63.72
7	2.30	<0.005	0.587	45.90
14	2.11	<0.005	0.519	40.58
21	1.84	<0.005	0.423	33.07

Half life ( $RL_{50}$ ) = 6.0 days

\*Limit of detection = 0.005 ppm



pulp of orange fruits were calculated on whole fruit basis in order to be compared with their MRL's for calculating the pre-harvest intervals. Maximum residue limits of prothiophos and profenofos in citrus fruits were 0.05 and zero days, respectively as profenofos amounts did not reach its MRL immediately after application.

with regard to the residues on and in the orange peel which might be used in jam and flavour manufacturing, further investigation is needed to study the effect of washing, heat and other cooking processes on the disappearance of prothiophos and profenofos from the orange peel as the amounts of residues detected were relatively high at the pre-harvest intervals.

Persistence of prothiophos and profenofos (Tables 2&3) decreased from 92.27 and 80.84 % after 24 h to 57.23 and 63.72% after 3 days from application, respectively. By the end of the experimental period 26.29% persisted from the initial deposit of prothiophos and 33.07 % from profenofos. The half period ( $RL_{50}$ ) determined from the persistence curves were 10.5 and 6.0 days indicating shorter persistence of profenofos than prothiophos, respectively.

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## تقييم كفاءة مبيدين فوسفوريين ضد حشرة ذبابة الموالح البيضاء ودراسة الاثر الباقي لهذين المبيدين علي ثمار البرتقال

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أجريت تجربتين خلال عامي ١٩٨٦، ١٩٨٧ في طنط الجزيرة بمحافظة القليوبية لتقييم فعالية مبيدين حشريين فوسفوريين علي أشجار البرتقال ابوسره المصابه بذبابة الموالح البيضاء. عومل مبيد السليكرون بمعدل ٠.٠٧٥ % ومبيد التوكثيون بمعدل ٠.١٥ % وقد كانت نتيجة اباداة الحشره مرضيه بالنسبه لكل من المبيدين ولم تلاحظ آثار جانبية ضاره علي النبات عقب المعاملة . كما قدرت الاثار المتبقية لكل من المبيدين في قشره البرتقال حيث تركزت فيها بينما كانت الكميات التي قدرت داخل الثمار من الممكن تجاهلها .

وحددت فترات الأمان أو فترات ما قبل الحصاد بالنسبه للتوكثيون علي أنها ٢١ يوم وللليكرون علي أنها صفر حيث وصلت كميه المتبقيات إلي أقل من الحدود القصوي المسموح بها وهي ٠.٣ ، ٠.٠٥ جزء في المليون علي التوالي وذلك بحساب كيمه المتبقيات علي أساس الثمره الكامله كما حددت فتره نصف العمر للسليكرون علي أنها ٦ أيام وللتوكثيون علي أنها عشره ونصف يوم .

ويلاحظ أنه بالنسبه لقشره البرتقال والتي تستخدم في عمل المربي ، ومكسبات الطعم فإنه يلزم دراسات مستفيضة لدراسة تأثير عمليات الغسيل والحرارة والتصنيع علي اختفاء المبيدات من القشرة.