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Fumigation of kumara with aerosol formulations of synthetic and natural pyrethroids for control of tropical armyworm and opogona

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A report prepared for **Vegfed** 

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## 1 Executive summary

Tropical army worm (TAW) and opogona are common pests of stored kumara. In Northland, stored tubers became infested with TAW predominantly after harvest early in the year whilst opogona damage is more prevalent later in the year. Current practices for controlling these pests include repeated use of organophosphate (OP) insecticides that are hazardous to apply. Moreover, these pests are becoming more resistant to OP pesticides following the excessive use of these chemicals. Consequently, residues in kumara have become a concern to growers. This project investigated alternative pesticides and assessed the associated risk of residues from their use.

A small-scale trial was conducted at Crop & Food Research for Vegfed to assess the possibility of using existing aerosol formulations of natural (Pestigas®) and synthetic (Permigas®) pyrethroids for the control of these important pests of stored kumara. Kumara tubers infested with opogona larvae and TAW larvae were fumigated in a shipping container with Permigas® and Pestigas® applied at three rates (0.5, 1.0 and 2.0 g product/m³). Permigas® at the highest rate gave 100% kill TAW larvae, but all rates of Pestigas® were ineffective for the control of this insect. Opogona was not killed by either of the fumigants at the three rates tested. After treatment, the kumara were stored in a cool-store and samples were removed periodically for pesticide residue assessment on days 1, 3, 7, 14, 21 and 28. Permethrin and pyrethrin residues were found to be well within the permitted maximum residue limits (MRL) of 0.5 mg/kg and 1.0 mg/kg respectively.

A large-scale commercial fumigation was carried out at Dargaville in three sheds using Permigas® at 2 g/m³ for the control of TAW, a major concern to growers when this product was evaluated. A single application of Permigas® assisted by a circulation fan caused greater than 95% mortality of TAW under commercial storage conditions. The associated residue risk was assessed under two scenarios. Two application regimes, at 10-day intervals with up to 4 applications, and at 7-day intervals, a worst case scenario with, up to 3 applications were simulated. Kumara were sampled at 1, 3, 5, 7, 14, 21 and 28 days after the last fumigation in each regime. Samples were sent to a commercial laboratory to determine the amount of residues in the treated kumara. Permethrin residues found in the kumara samples when fumigated with Permigas® under the two regimes were also within the permitted levels.

Permigas<sup>®</sup> can be recommended for the control of TAW in stored kumara. The risk of exceeding the permitted MRL at the application rates and frequencies evaluated in this trial was low for permethrin. Further work, however, needs to be done to assess other pesticides and associated residue risks for the control of opogona in kumara.

## 2 Introduction

Opogona (*Opogona omoscopa*), also called the detritus moth in Australia (Naumann 1993), and the tropical armyworm (TAW, *Spodoptera litura*) are the major insect pest problems currently facing the New Zealand kumara industry. Kumara tubers are damaged in storage by these two insects. TAW larvae can also damage tubers in the field. They can enter the storage shed with the tubers, but mainly invade storage sheds directly. Adults may deposit eggs on tubers left in the field after harvesting or when they enter shed storage. An opogona population is always present around storage sheds on tubers left over from the previous year because of poor screening of storage facilities (van Epenhuijsen 2000) and the use of higher storage temperatures than advised. Currently, organophosphate pesticides like Actellic<sup>®</sup> smoke generators and Nuvos™ are used by New Zealand kumara producers to control insects. Both of these methods are inefficient and are hazardous pesticides to apply. Furthermore, Nuvan is can be a fire hazard if inappropriately applied (A Carpenter pers. comm.).

Two aerosol formulations, Permigas® and Pestigas® marketed by BOC Gases New Zealand Ltd, are currently used for horticultural application off-label. Preliminary investigations have indicated that these pesticides can be used against these pests under laboratory conditions. Permigas® mainly contains a synthetic pyrethroid, permethrin, and Pestigas® contains a natural pyrethroid, pyrethrum, formulated as an aerosol in liquid carbon dioxide under high pressure. When released these formulations produce a white fog formed by large numbers of very small droplets. For the sake of clarity, the word 'fumigant' is used for these products and 'fumigation' to describe the application of these formulations.

Pestigas<sup>®</sup> contains 4.0 g/kg pyrethrum while Permigas<sup>®</sup> contains 4.0 g/kg permethrin plus 1.0 g/kg pyrethrins. Permigas<sup>®</sup> also contain 5.0 g/kg piperonyl butoxide (PBO) whilst Pestigas<sup>®</sup> contains 20.0 g/kg PBO. Both products are currently registered for use in factories, warehouses and stores for the control of flies, moths, cockroaches and other flying insects. Preliminary trials suggest that the synthetic pyrethroid, Permigas<sup>®</sup>, can be a viable option (A Carpenter pers. comm.) for the control of TAW and opogona.

The objective of research described in this report was to assess the possibility of using existing aerosol formulations on natural (Pestigas<sup>®</sup>) and synthetic (Permigas<sup>®</sup>) pyrethroids for the control of these important pests of stored kumara.

## 3 Methods

Onion bulk bins were modified for these studies. Each bulk bin, inside measurement  $1.17 \times 1.38 \times 0.86$  m, was fitted on the inside with a wire "cage" with dimensions of  $1.2 \times 1.0 \times 0.56$  m ( $0.67 \text{ m}^3$  capacity). The space around

this cage was filled up with kumara tubers. The roof of the cage consisted of a wooden structure with a small opening to insert infested opogona kumara or TAW larvae. Bags containing two layers of kumara tubers were laid down on a lid that closed off the opening to ensure the cage was completely surrounded by kumara on all sides. This was done to simulate normal kumara storage conditions. The bins were placed in the middle of a 20-foot shipping container that was used as a fumigation chamber (Fig. 1).

Prior to the experiment opogona pupae were released onto old infested kumara tubers obtained from Dargaville. The tubers were selected for frass. In this way infested kumara tubers were obtained and used in the fumigation tests. A 20-litre plastic bucket was three-quarters filled with these infested tubers and placed inside each of the wire cages described above.

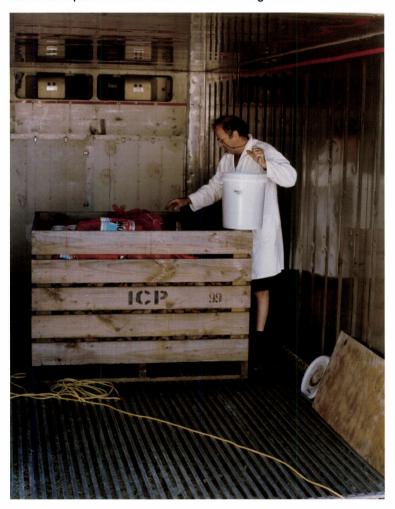


Figure 1: Bins with infested kumara placed in the cage. Containers with TAW on the left.

Ten TAW larvae, which were close to pupating in late January 2002, were placed with pieces of kumara in a small open plastic container that was also

placed inside the wire cages. Both the buckets and the plastic containers did not have lids.

Fumigation was carried out commencing 22 January in two shipping containers. Four replicates of each treatment were carried out over time. The fumigants were applied using a high pressure gun supplied by BOC Gases through an opening in the door of the shipping container. The fumigation was carried out at approximately 18°C with temperature rising during each day to 21-23°C.

The doors were kept closed for 1 hour after each treatment. A small fan placed on the container floor kept the air circulating within the chamber. After each treatment period, the doors of the container were opened and the main blower fans were switched on for 10 minutes to flush out any of the residual aerosol fog before the next fumigation treatment was carried out. Three rates of each aerosol formulation were used (Table 1).

In addition, a single overnight fumigation (16 hours exposure) in two shipping containers were carried out with the high rate of 2 g/m³ Pestigas® for TAW and 120 g/m³ EVQ601D (an experimental formulation of ethyl formate) for opogona.

Table 1: Pestigas<sup>®</sup> and Permigas<sup>®</sup> dose rates evaluated in the kumara fumigation trial.

Pestigas <sup>®</sup>	
Low	0.50 g/m³ (equivalent to 0.002 g/m³ pyrethrins)
Med	1.00 g/m <sup>3</sup> (equivalent to 0.004 g/m <sup>3</sup> pyrethrins)
High	2.00 g/m³ (equivalent to 0.008 g/m³ pyrethrins)
Permigas <sup>®</sup>	
Low	0.50 g/m³ (equivalent to 0.002 g/m³ permethrins + 0.0005 g/m³ pyrethrins)
Med	1.00 g/m³ (equivalent to 0.004 g/m³ permethrins + 0.001 g/m³ pyrethrins)
High	2.00 g/m³ (equivalent to 0.008 g/m³ permethrins + 0.002 g/m³ pyrethrins)

After the fumigation, the buckets and containers with insects were removed and covered with the meshed organza cloth. To monitor opogona control, the buckets were kept for approximately three months in a covered, sheltered ambient storage space and then, after 10 April at 22°C in a climate controlled storeroom to hasten the opogona life cycle.

The bins with opogona-infested tubers and other insects were inspected shortly after the treatments on 1 February. The next two assessments took place on 22 February and 6 May. All insects were recorded and removed or killed at the first and second assessments.

The containers with TAW were kept for 3 days at 23°C for the first assessment and then for another 3 weeks until pupation was completed. The numbers of adults were then counted.

### 4 Results

### 4.1 Small-scale efficacy trials

Apart from the opogona, the stored kumara tubers also contained earwigs, slaters, phoridae (larvae and other maggots living in rotten produce), whiteshouldered house moth (WSM), and spiders (Appendices I-III). As only very few insects were found at the first assessment data are not presented. Millipedes were not found again after the first assessment on 1 February. At that date only four adults and larvae of opogona were found in 11% of the 28 buckets containing kumara. The numbers of opogona increased steadily to infest 96% of the buckets at the last two assessments. The 3-4 mm light brown Desjardin's flat beetle (*Cryptamorpha desjardinsi*, Coleoptera: Silvanidae) was by far the most common beetle in the tubers. This beetle is common in Auckland and Northland as a fungal/mould feeder in mouldy stored foodstuffs and vegetable matter. Although not regarded as a primary pest, the numerous beetles on single tubers were a nuisance as well as a significant contaminant.

Table 2: Percentage of TAW larvae and emerged TAW adults in kumara tubers after fumigating with Permigas<sup>®</sup> and Pestigas<sup>®</sup> (means of four replicates).

Treatment	Killed larvae (%)	Emerged adults (%)
Untreated control	2.8	39.6
Permigas low	52.1	23.7
Permigas medium	80.6	5.6
Permigas high	100.0	0.0
Pestigas low	7.1	67.0
Pestigas medium	6.9	37.2
Pestigas high	2.8	42.9
LSD (P<0.05)	29.9	22.4

Permigas® at the high rate evaluated killed 100% of the TAW larvae (Table 2). The lower rates of Permigas® gave correspondingly lower degrees of effectiveness. Pestigas® was ineffective in killing the TAW, resulting in similar larval mortality as for the untreated control at all rates evaluated. Only Permigas® at the high and medium rates gave a significantly lower proportion of adults emerging than that from the untreated control. All Pestigas® rates as well as the Permigas® low rate resulted in as high, or higher, proportion of adults emerging than the experimental control. None of the fumigants had any effect on the total number of opogona adults and larvae (Table 3).

Significant damage to kumara tubers from opogona was seen where the tubers touched each other. However, the majority of the larvae were found in the mummified or completely soggy decaying tubers. Larvae of the WSM and opogona could not be distinguished from one another. Eight WSM adults (moths) were found at the last assessment compared to 12 in the first assessment. In order to be sure that the larvae found were mainly opogona, representative samples were reared in the laboratory from the last assessment. This showed that all of the larvae were opogona.

In the two single overnight treatments of Pestigas® and EVQ601D the doors of the shipping containers were kept closed overnight. These treatments were not replicated and hence caution must be exercised when interpreting mortality data. The buckets with kumara tubers contained hardly any insects (Appendices I and II). Maggots (Phoridae) and Desjardin's flat beetles were the only insects found in the rotten tubers The number of insects found in the EVQ601D treatment was low and some genera were absent. In contrast, no effect was observed on the TAW after the overnight treatment with the high dose rate of Pestigas®.

Table 3: Mean numbers of opogona (adults and larvae) and other insects found in buckets of kumara tubers after fumigation (means of four replicates).

Treatment	Opogona 2nd assessment	Opogona 3rd assessment	Opogona total	Other insects 2 <sup>nd</sup> + 3 <sup>rd</sup> assessment
Untreated control	5.5	38.2	43.7	119.7
Permigas low	4.0	69.2	73.2	148.8
Permigas medium	8.8	39.5	48.2	133.3
Permigas high	4.7	48.2	53.0	127.7
Pestigas low	17.5	38.2	43.7	119.7
Pestigas medium	6.0	29.3	35.2	131.5
Pestigas high	2.3	20.3	46.2	140.5
LSD (P<0.05)	13.03	45.9	52.1	46.3

### 4.2 Residue trial

At the efficacy trial, a large tray holding sufficient kumara tubers was placed at the top of the treatment bin to allow for maximum exposure of tubers to the spray deposits. Following each 1 hour treatment, the tray was removed and the kumara tubers were placed in a sack and stored in a coolroom at 18°C. Samples of kumara were removed periodically from each treatment on days 1, 3, 7, 14, 21 and 28 after fumigation. At each sampling occasion, two kumara tubers were removed from each treatment. Samples were bulked from the four replicates into one sample and placed immediately in a labelled plastic bag into a deep freeze. At the end of the sampling period the frozen

kumara samples were packed in polystyrene boxes and freighted frozen by Tranz Link Refrigerated to Hill Laboratories in Hamilton for residue analysis. Pesticide residue, determined in the kumara samples from the various fumigation treatments are presented in Tables 4 and 5. See Appendix IV for a laboratory report of the analysis.

Table 4: Pesticides residues found in kumara samples fumigated with Permigas<sup>®</sup> at three rates. DAF = days after one fumigation treatment.

Product	Rate	DAF	Pestio	Pesticide residues (mg/kg)				
	THE PARTY OF THE P	H <del>essel M. Qu</del> . Park 1, 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944 - 1944	Permethrins	Pyrethrins	Piperonyl butoxide			
	_	1	0.02	<0.03	0.02			
		3	0.03	<0.03	0.03			
	Ľow	7	0.03	<0.03	0.03			
	LOW	14	0.05	<0.03	0.05			
		21	0.02	<0.03	0.02			
		28	0.04	<0.03	0.03			
		1	0.03	<0.03	0.03			
		3	0.06	<0.03	0.06			
	Medium	7	0.07	<0.03	0.07			
Permigas <sup>®</sup>	Mediam	14	0.07	<0.03	0.06			
		21	0.12	0.03	0.12			
		28	0.12	<0.03	0.08			
		1	0.07	<0.03	0.08			
		3	0.10	<0.03	0.10			
	High	7	0.25	0.03	0.21			
	High	14	0.10	<0.03	0.10			
		21	0.06	0.06	0.31			
		28	0.14	<0.03	0.13			

Permethrin residues found in kumara samples after treatment with Permigas<sup>®</sup> ranged between 0.02 and 0.05, 0.03 and 0.12, and 0.06 and 0.25 mg/kg in the lower, medium and high treatments respectively. Piperonyl butoxide ranged between 0.02 and 0.31 mg/kg fruit.

Table 5: Pesticides residues found in kumara samples fumigated with Pestigas® at three rates. DAF=days after a single fumigation treatment.

Product	Rate	DAF	Pestic	Pesticide residues (mg/kg)				
		***************************************	Permethrins	Pyrethrins	Piperonyl butoxide			
	•	1	-	<0.03	0.15			
		3	-	<0.03	0.13			
	Low	7	-	<0.03	0.07			
	LOW	14	-	<0.03	0.08			
		21	-	<0.03	0.08			
		28	-	<0.03	0.12			
		1	-	0.07	0.36			
		3	-	0.05	0.30			
	Medium	7	-	0.06	0.34			
Pestigas <sup>®</sup>	Mediairi	14	-	0.04	0.38			
		21	-	<0.03	0.15			
		28	-	0.03	0.26			
	h pe . 1/11	1		0.15	0.71			
		3	. •	0.15	0.74			
	High	7	-	0.08	0.43			
	ingii	14	-	0.10	0.59			
		21	-	0.05	0.42			
		28		0.06	0.39			

In contrast, pyrethrins residues found in the kumara samples after treatment with Pestigas<sup>®</sup> ranged between 0.03 and 0.15 mg/kg fruit over all the rates applied. Piperonyl butoxide residues ranged between 0.07 and 0.74 mg/kg over the treatments.

## 5 On-site trials

### 5.1 Efficacy assessment

Based on results from small-scale trials at Crop & Food Research, a commercial scale evaluation was organised and carried out at two properties in Dargaville at the end of May when there was pressure from TAW in stored kumara sheds. This situation allowed a commercial evaluation of Permigas<sup>®</sup>. Two growers, Kerry Perreau and Andre de Bruin participated in this trial. Two sheds measuring about 125 m³ were used on Kerry's property and one at

Andre de Bruin's property. One hundred TAW collected from the surrounding storage sheds were used in the trial. Ten TAW of different sizes were placed in a plastic container. A meshed cloth was placed on the top of a cut out lid and secured to prevent the TAW from escaping. Ten containers were then placed at different locations in each of the two sheds, including the top, sides and between bins as well as on the floor in order to assess the extent of the pesticide coverage when applied as a fumigant. Two hundred and fifty grams of Permigas® (equivalent to 2 g Permigas/m³) was introduced into each shed using a high pressure handgun. Air circulating fans were left running for about 10 minutes and then switched off for the rest of the trial. The same treatment was repeated in Andre de Bruin's shed. The following day, the containers were assessed for the extent of TAW kill. Mortality of TAW in the treated sheds ranged between 95 and100% after one fumigation with Permigas®.

### 5.2 Residue assessment

At Kerry Perreau's storage shed a further 3 fumigations were carried out at 10-day intervals. Kumara samples were removed randomly from each shed on 1, 3, 5, 7, 14, 21 and 28 days after the last fumigation. At each sampling occasion, the kumara tubers were placed in a plastic bag and kept in deep a freeze. Samples for residue analysis were removed and frozen separately for each shed.

At Andre de Bruin's shed, a further 2 fumigations were carried out at 7-day intervals. Kumara samples were also removed from the shed 1, 3, 5, 7, 14, 21 and 28 days after the last fumigation. This frequent fumigation regime simulated a worst case scenario assuming TAW pressure was high. Results of the laboratory analysis are presented in Table 6.

At the completion of the trial, samples from the two properties were transported to Hill Laboratories in Hamilton for residue determination in the tubers. A laboratory report of residues determined in the tubers is attached in Appendix IV.

Table 6: Pesticide residues found in kumara sampled from three commercial sheds after several fumigation with Permigas<sup>®</sup>. DAF=days after last fumigation treatment.

Grower	Fumigation	DAF	<sup>1</sup> Pesticide residues F (mg/kg kumara tubers)				
	regime		Permethrins	Pyrethrins	Piperonyl butoxide		
Kerry	Four	1	0.22	0.04	0.21		
Perreau	fumigations	3	0.29	<0.03	0.26		
Shed 1	@ 10-day	5	0.46	0.07	0.39		
	intervals	7	0.20	0.03	0.17		
		14	0.29	< 0.03	0.25		
		21	0.45	0.04	0.37		
		28	0.16	< 0.03	0.14		
Kerry	Four	1	0.44	0.04	0.42		
Perreau	fumigations	3	0.61	0.10	0.58		
Shed 2	@10-day intervals	5	0.41	< 0.03	0.35		
	intervais	7	0.57	0.03	0.53		
		14	0.31	< 0.03	0.26		
		21	0.39	< 0.03	0.33		
		28	0.26	<0.03	0.20		
Andre	Three	1	0.20	<0.03	0.18		
de Bruin	fumigations	3	0.42	< 0.03	0.38		
Shed 1	@ 7-day	5	0.24	< 0.03	0.17		
	intervals	7	0.19	< 0.03	0.16		
		14	0.09	<0.03	0.08		
		21	<sup>2</sup> 0.77	<0.03	0.59		
		28	0.10	<0.03	0.05		

<sup>&</sup>lt;sup>1</sup>NZ (Maximum Residue Limits of Agricultural Compounds) Mandatory Food Standard 1999 (as ammended) list MRL's for permethrin = 0.5 mg/kg, pyrethrin = 1 mg/kg and piperonyl butoxide = 8 mg/kg.

A total of four fumigation treatments were carried out in two sheds at Kerry's property. Permethrin residues found in the tubers varied between 0.46 and 0.20 mg/kg in Shed 1, and 0.61 and 0.26 mg/kg in Shed 2 over the seven sampling occasions. At Andre's property, permethrin residues in the kumara tubers sampled ranged between 0.42 and 0.10 mg/kg. Unusually high permethrin residues (around 0.8 mg/kg) were found in tubers sampled three weeks after the last fumigation in Andre's shed. This could be due to batch variation as the last two samples were sent and analysed along with another lot of samples or an aberration in the result. This anomaly was, however, checked and confirmed to be the same.

<sup>&</sup>lt;sup>2</sup>This level was rechecked by the laboratory and confirmed. The high level determined could be because the samples were sent separately and analysed in a separate batch or it may be simply an unexplainable aberration in the result.

Pyrethrins residues at both properties were generally found to be lower than 0.04 mg/kg and piperonyl butoxide (PBO) ranged between 0.59 and 0.05 mg/kg. PBO was analysed because it is listed in the New Zealand Food Standards Code and has an MRL of 8 mg/kg.

## 6 Discussion

### 6.1 Efficacy of the fumigants

Permigas<sup>®</sup> is a non-water based permethrin formulation that is delivered as an aerosol fog. Droplet sizes range between 2 and 20 µm, which is ideal for use in enclosed spaces like kumara sheds. Penetration of Permigas<sup>®</sup> when used as a fumigant was variable; it was very efficacious for TAW control but not opogona control. TAW located in the containers and on kumara were readily exposed to the pesticide. In the initial trials, a single bin containing kumara and insects was placed in the middle of a shipping container and the fumigant introduced into the container. The aerosol fog droplets were sufficiently small to penetrate the containers holding the TAW.

In the commercial trial at Dargaville, the sheds were well stacked with bins full of kumara, leaving very little room for the natural movement of the fumigant throughout the produce. This was overcome by injecting 2 g/m³ Permigas® into the air-stream by pointing the gun in the direction of the air flow created by the circulating fan. The fumigant was carried throughout the shed for about 10 minutes using the air circulation fan. Permigas® caused more that 95% mortality in TAW after a single application. Remaining live larvae were killed with repeated applications. The mortality of TAW larvae in the containers placed at various locations in the shed did not show any significant variation, suggesting that the fumigant readily distributed throughout the stacked kumara bins in the shed. Moreover, a large number of TAW was also found dead on the floor and around bins suggesting that TAW, a nocturnal larva, must have came into contact with the pesticide from treated tubers and bins.

The poor control of opogona by Permigas<sup>®</sup> could be because the larvae bores deep into the kumara tubers where aerosol droplets may not be able to reach.

Pestigas<sup>®</sup> containing natural pyrethrins did not provide adequate control of the two pests evaluated.

Although damage to kumara tubers by opogona and TAW is of most concern for growers, it was interesting to see that at least eight other insect species were found in stored kumara. Many survived for more than three months in the plastic buckets. Decaying or mummified tubers in the bin provided a habitat for these insects. The presence of the white shouldered house moth was a surprise as its larvae may cause similar damage to that caused by opogona. There is a chance that the infestation occurred in Palmerston North as this insect has been found before in adjacent outside cultures.

## 6.2 Permigas® residues in kumara

The decay of permethrin, the active ingredient in Permigas®, over 28 days in both of the trials did not show any definite pattern, as would be expected from water-based application methods. This could be because the pesticide was applied as an aerosol fog and distributed evenly over the produce. Generally conditions in storage are less conducive to the breakdown of pesticides particularly in the case of the synthetic pyrethroid permethrin which has a long residual activity. However, when sprayed at the efficacious rate of 2 g/m³ Permigas®, permethrin residues from Permigas®, found in kumara were consistently lower than the permitted MRL's (0.5 mg/kg) over the period of the evaluation. As expected, in the commercial scale trial, repeated applications of Permigas® resulted in a slightly higher level of residues but remained within the MRL. Treating kumara with Permigas® at a more frequent interval (7-days compared with 10) did not result in any increase residues in kumara. Based on these findings, the kumara can be safely consumed in less than one week after fumigation at the dose rate evaluated.

## 7 Conclusion

Permigas<sup>®</sup> was found to be useful for the control of TAW in kumara tubers in storage. Permethrin residues resulting from spraying Permigas<sup>®</sup> were within the permitted MRL. Neither Pestigas<sup>®</sup> nor Permigas<sup>®</sup> at the dose rates evaluated can be recommended for the control of opogona infestations in kumara tubers. Further research will need to be carried out using higher dose rates of Permigas<sup>®</sup> or other pesticides.

## 8 Acknowledgements

New Zealand Kumara Distributors supplied infested tubers and Andre de Bruin (Delta Produce Ltd) supplied some additional opogona-infested tubers. Alby Marsh and Ken Somerfield gave technical assistance. We thank Kerry Perreau and Andre de Bruin who made their storage sheds available for the trial. BOC Gases is thanked for providing Permigas®, Pestigas®, high pressure handgun and assistance in the commercial trials.

## 9 References

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van Epenhuijsen, C.W. 2000: Damage to kumara tubers, sweet potato, (*Ipomoea batatas* L.) by opogona (*Opogona omoscopa*) and the tropical armyworm (*Spodoptera litura*). Crop & Food Research Confidential Report No. 261.

New Zealand Food Safety Authority. New Zealand (Maximum residue limits of agricultural compounds) Mandatory Food Standard 1999 (as amended).

# **Appendices**

Appendix I: Insect counts of tropical armyworm (TAM) in kumara tubers after different Pestigas<sup>®</sup> or Permigas<sup>®</sup> dose rate treatments. Counting of all insects was stopped once a total of 65 was reached.

Treatment	No. of larvae treated	No. of dead larvae	No. of dead pupae	No. of adults emerged
Pestigas low	.8	0	3	5
Permigas low	8	5	2	1
Pestigas med	6	1	3	2
Permigas mid	6	6	0	0
Pestigas high	9	1	3	5
Permigas high	10	10	0	0
Untreated control	8	0	6	4
Pestigas low	7	1	1	5
Permigas low	7	6	1	0
Pestigas med	9	0	7	2
Permigas med	10	10	0	0
Pestigas high	7	0	4	3
Permigas high	9	9	0	0
Untreated control	9	1	5	3
Pestigas low	7	1	1	5
Permigas low	10	6	2	2
Pestigas med	10	0	4	6
Permigas med	. 9	9	0	0
Pestigas high	9	0	6	3
Permigas high	10	10	0	0
Untreated control	8	0	6	2
Pestigas low	8	0	3	5
Permigas low	8	0	3	5
Pestigas med	9	1	5	3
Permigas med	9	2	5	2
Pestigas high	10	0	6	4
Permigas high	10	10	0	0
Untreated control	6	0	3	3

Appendix II: Numbers of Insects in kumara tubers for the second assessment after application of different Permigas<sup>®</sup> or Pestigas<sup>®</sup> dose rate treatments. Counting of all insects was stopped once a total of 65 was reached.

Treatment	Opogona adults	Opogona larvae	WHS adults	Phoridae adults + maggots	Beetles	Spiders	Others
Pestigas low	1	50	0	7	27	0	0
Permigas low	1	1	1	3	12	2	0
Pestigas med	0	6	1	28	46	. 6	3
Permigas med	3	18	2	16	19	1	0
Pestigas high	1	0	0	5	29	2	0
Permigas high	. 2	6	0	17	35	2	1
Untreated control	5	8	0	.3	19	4	2
Permigas low	3	5	1	41	46	6	0
Pestigas low	4	9	0	23	23	0	1
Permigas med	2	0	0	2	13	2	1
Pestigas med	3	3	0	7	22	5	3
Pestigas high	0	0	0	4	22	3	1
Permigas high	0	2	1	5	25	3	0
Untreated control	1	4	0	1	21	4	1
Permigas low	. 1	4	0	10	35	4	3
Pestigas low	1	4	0	4	65*	3	2
Pestigas med	2	5	0	3	65*	5	8

Treatment	Opogona adults	Opogona larvae	WHS adults	Phoridae adults + maggots	Beetles	Spiders	Others
Permigas med	0	6	0	10	12	4	0
Permigas high	1	4	0	4	6	0	0
Pestigas high	0	2	1	6	14	3	1
Untreated control	1	2	1	3	60	1	0
Pestigas low	0	1	0	0	65*	2	0
Permigas low	1	0	0	0	24	5	0
Permigas med	0	6	0	3	20	5	4
Pestigas med	1	4	3	1	45	2	0
Pestigas high	1	5	1	6	46	2	7
Permigas high	-1	3	0	6	16	1	0
Untreated control	1	0	0	1	25	3	0
EVQ601D	0	0	0	0	·1	0	0

Appendix III: Numbers of insects in kumara tubers for the third assessment after treatment with different Permigas  $^{\circledR}$  or Pestigas  $^{\circledR}$  dose rate treatments. Counting of all insects was stopped once a total of 65 was reached.

Treatment	Opogona adults	Opogona larvae	WSM adults	Phoridae	Beetles	Spiders	Slaters	Others
Pestigas low	11	29	0	0	65	10	37	1
Permigas low	5	65	0	2	65	10	5	65
Pestigas med	8	1	2	0	37	0	5	0
Permigas med	20	65	0	1	65	65	38	0
Pestigas high	3	0	1	28	65	9	0	0
Permigas high	3	39	0	21	65	2	24	, O 1
Untreated Control	0	45	0	9	25	28	1	0
Pestigas low	11	52	1	5	65	4	0	1
Permigas low	2	65	0	4	65	1	15	1
Pestigas med	2	40	0	0	65	15	6	2
Permigas med	2	24	0	5	65	5	0	3
Pestigas high	2	0	0	65	51	1	0	15
Permigas high	8	65	3	24	65	6	0	1
Untreated Control	28	53	0	13	65	8	35	1
Pestigas low	45	55	0	43	65	4	1	0
Permigas low	2	2	0	34	65	0	3	0
Pestigas med	31	26	0	0	65	3	4	0
Permigas med	11	12	0	1	51	3	5	0
Pestigas high	3	5	0	1	65	6	9,	2

Treatment	Opogona adults	Opogona larvae	WSM adults	Phoridae	Beetles	Spiders	Slaters	Others
Permigas high	0	0	0	22	65	2	10	2
Untreated Control	2	12	1	1	65	3	3	3
Pestigas low	.1	3	0	0	65	4	0	0
Permigas low	5	39	0	0	65	0	0	1
Pestigas med	6	2	0	0	65	2	35	0
Permigas med	5	20	0	10	65	0	4	0
Pestigas high	11	65	0	4	65	0	4	0
Permigas high	5	65	0	24	65	1	2	3
Untreated Control	1	12	0	1	65	1	2	0
EVQ601D	0	0	0	50	50	0	0	0

Appendix IV: Pesticide residues found in kumara samples sent to Hill Laboratories – A laboratory report.

# **Hill Laboratories**

R I Hill Laboratories Limited

Address: 1 Clyde Street, Private Bag 3205, Hamilton, New Zealand Telephone: +64 (7) 858-2000 Facsimile: +64 (7) 858-2001 Email:
mail@hill-labs.co.nz
Internet:
www.hill-labs.co.nz



Client: Crop & Food Research

Address: Private Bag 11600
Palmerston North

Contact: Hari Krishna

Laboratory No: 181430
Date Registered: 14/03/2002
Date Completed: 16/04/2002
Page Number: 1 of 3

Client's Reference: Crop and Food Research

The results for the analyses you requested are as follows:

Sample Type: Biological Materials, Kumara

GC Pesticides Table

Sample Name	#1		#2	#3	1	#4	#5
Lab No	181430/1	18	1430/2	181430/3	4	181430/4	181430/5
Units	(mg/kg as rcvd)	, ,	g/kg as cvd)	(mg/kg as rcvd)		(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.02		0.03	0.08		0.03	0.06
Permethrin	0.02	Activities and a commence of	0.03	0.07	i	0.03	0.06
Pyrethrin	< 0.03	. <	0.03	< 0.03		< 0.03	< 0.03

GC Pesticides Table

GC Pesticides Table					
Sample Name	#6	#7	#8	#9	#10
Lab No	181430/6	181430/7	181430/8	181430/9	181430/10
Units	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.10	0.03	0.07	0.21	0.05
Permethrin	0.10	0.03	0.07	0.25	0.05
Pyrethrin	< 0.03	< 0.03	< 0.03	0.03	< 0.03

GC Pesticides Table

Sample Name	#11	# 12	ì	#13	#14	#15
Lab No	181430/11	181430/12	dea'r maran	181430/13	181430/14	181430/15
Units	(mg/kg as rcvd)	(mg/kg as rcvd)		(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.06	0.10	İ	0.02	0.12	0.31
Permethrin	0.07	0.10		0.02	0.12	0.06
Pyrethrin	< 0.03	< 0.03		< 0.03	0.03	0.06



Sample Name	#16	#17	# 18
Lab No	181430/16	181430/17	181430/18
Units	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.03	0.08	0.13
Permethrin	0.04	0.12	0.14
Pyrethrin	< 0.03	< 0.03	< 0.03

### GC Pesticides Table

Sample Name	#19	#20	#21	#22	#23
Lab No	181430/19	181430/20	181430/21	181430/22	181430/23
Units	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.15	0.36	0.71	0.13	0.30
Pyrethrin	< 0.03	0.07	0.15	< 0.03	0.05

#### GC Pesticides Table

Sample Name	#24	#25	#26	#27	#28
Lab No	181430/24	181430/25	181430/26	181430/27	181430/28
	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.74	0.07	0.34	0.43	0.08
Pyrethrin	0.15	< 0.03	0.06	0.08	< 0.03

### GC Pesticides Table

Sample Name	#29	#30	#31	#32	#33
Lab No	181430/29	181430/30	181430/31	181430/32	181430/33
Units	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.38	0.59	0.08	0.15	0.42
Pyrethrin	0.04	0.10	< 0.03	< 0.03	0.05

#### GC Pesticides Table

Sample Name	#34	#35	#36
Lab No	181430/34	181430/35	181430/36
Units	(mg/kg as rcvd)	(mg/kg as rcvd)	(mg/kg as rcvd)
Piperonyl Butoxide	0.12	0.26	0.39
Pyrethrin	< 0.03	0.03	0.06

**Summary of Methods Used and Detection Limits**The following table(s) gives a brief description of the methods used to conduct the analyses for this job.

The detection limits given below are those attainable in a relatively clean matrix. Detection limits may be higher for individual samples should insufficient sample be available, or if the matrix requires that dilutions be performed during analysis.

Substance Type: Biological Materials

Parameter Parameter	Method Used	Detection Limit
Pesticide GC Multiresidue Method	Ethylacetate extraction, SPE cleanup, GC-MS (SIM) analysis	N/A
Piperonyl Butoxide	GC-MS (SIM)	0.01 mg/kg as rcvd
Permethrin	GC-MS (SIM)	0.01 mg/kg as rcvd
Pyrethrin	GC-MS (SIM)	0.03 mg/kg as rcvd

### **Analyst's Comments:**

These samples were collected by yourselves and analysed as received at the laboratory.

Samples are held at the laboratory for two months (where appropriate) after reporting of results. After this date they are discarded unless otherwise advised by the submitter.

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Colin Malcolm, BSc

Pesticides Client Manager

# **Hill Laboratories**

R 1 Hill Laboratories Limited

Address:
1 Clyde Street,
Private Bag 3205,
Hamilton, New Zealand

Telephone: +64 (7) 858-2000 Facsimile: +64 (7) 858-2001 Email:
mail@hill-labs.co.nz
Internet:
www.hill-labs.co.nz



Client: Crop & Food Research

Address: Private Bag 11600,

Palmerston North

Contact: Hari Krishna

Laboratory No: 188356

Date Registered: 28/06/2002

Date Completed: 5/07/2002

Page Number: 1 of 3

Client's Reference: Crop and Food Research

### Summary of Methods Used and Detection Limits

The following table(s) gives a brief description of the methods used to conduct the analyses for this job.

The detection limits given below are those attainable in a relatively clean matrix. Detection limits may be higher for individual samples should insufficient sample be available, or if the matrix requires that dilutions be performed during analysis.

Substance Type: Biological Materials (Kumara)

Parameter	Method Used	Detection Limit
Piperonyl Butoxide, Permethrin, Pyrethrin	See attached report	See report

#### Analyst's Comments:

These samples were collected by yourselves and analysed as received at the laboratory.

Samples are held at the laboratory for two months (where appropriate) after reporting of results. After this date they are discarded unless otherwise advised by the submitter.

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Colin Malcolm, BSc

Pesticides Client Manager



RESIDUE REPORT FOR: Crop & Food Research

Trial Ref: Insecticides in Kumara

Laboratory Details: Analysis of Piperonyl Butoxide, Permethrin and Pyrethrin in kumara samples.

Laboratory Details. Allarysis of I	
	Shaun Clay
	Hill Laboratories, 1 Clyde Street, Hamilton, New Zealand
Date & State of Samples	Received on 28/6/2002 in frozen state and issued Laboratory Number
received (temperature)	188356/1-5
Storage Conditions &	Frozen at -18°C until analysis on 1/7/2002
Duration	
Crop Part Analysed	Whole Sample
Summary of Method:	Each kumara was cut in half and homogenized to a pulp in a food processor. A 20g sub-sample was removed and extracted in ethyl acetate. The mixture was blended at
- Preparation	high speed to a homogenous pulp then filtered. An aliquot of this was removed and
- Extraction	cleaned up by Solid Phase Extraction (SPE). The analytes were determined by GC-
- Cleanup	MS in the selected ion mode (SIM), monitoring the 176, 177 and 193 ions for piperonyl butoxide. The ions 183, 163 and 165 were monitored for permethrin. The
- Analysis	ions 123, 150 and 93 were monitored for pyrethrin.
- Reference	Results expressed on an as received basis and not recovery corrected.
	Reference: Method adapted from A.H. Roos, et al, Analytica Chimica Acta,
a a	196 (1987) 95-102.
Metabolites Detected	N/A
Method Efficiency:	NIA
- Extraction efficiency	N/A
5	
- % Recovery	Permethrin: 105% at 0.5mg/kg level
	Piperonyl Butoxide: 93% at 0.5mg/kg level
	Pyrethin: 80% at 0.5mg/kg level
	Note: Spike recovery experiment was conducted on a sample (188356/5) which had
	significant residues found for permethrin and piperonyl butoxide.
- Storage Degradation	Not determined
Correction Factor	Not applied
Levels in Untreated Samples	N/A
Method Sensitivity (LOD)	Permethrin: 0.01mg/kg
	Piperonyl Butoxide: 0.01mg/kg
	Pyrethrin: 0.03mg/kg
Full Report of Procedures and	N/A
sample chromatograms	

### Residue Results

Sample Name	Lab No	Permethrin (mg/kg as rcvd)	Piperonyl Butoxide (mg/kg as rcvd)	Pyrethrin (mg/kg as rcvd)
ADB Shed 1 Day 1 13/6/02	188356/1	0.20	0.18	< 0.03
ADB Store 1 Day 3 15/6/02	188356/4	0.39, 0.45	0.35, 0.40	< 0.03. < 0.03
ADB Store 1 Day 5 17/6/02	188356/2	0.24	0.17	< 0.03
ADB Shed 1 Day 7 19/6/02	188356/3	0.19	0.16	< 0.03
ADB Shed 1 Day 14 26/6/02	188356/5	0.09	0.08	< 0.03

# **Hill Laboratories**

R I Hill Laboratories Limited

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Client: Crop & Food Research

Address: Tennent Drive,

PALMERSTON NORTH

Contact: Hari Krishna

Laboratory No: 190106

Date Registered: 29/07/2002 Date Completed: 12/08/2002

Page Number: 1 of 3

The results for the analyses you requested are as follows:

Summary of Methods Used and Detection Limits

The following table(s) gives a brief description of the methods used to conduct the analyses for this job.

The detection limits given below are those attainable in a relatively clean matrix. Detection limits may be higher for individual samples should insufficient sample be available, or if the matrix requires that dilutions be performed during analysis.

Substance Type: Biological Materials (Kumara)

Parameter	Method Used	Detection Limit
	See attached report	See report

#### **Analyst's Comments:**

These samples were collected by yourselves and analysed as received at the laboratory.

Samples are held at the laboratory for two months (where appropriate) after reporting of results. After this date they are discarded unless otherwise advised by the submitter.

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Colin Malcolm, BSc

Pesticides Client Manager



### RESIDUE REPORT FOR: Crop & Food Research

Trial Ref: Insecticides in Kumara

Laboratory Details: Analysis of Piperonyl Butoxide, Permethrin and Pyrethrin in kumara samples.

Analyst Name	Shaun Clay				
Laboratory	Hill Laboratories, 1 Clyde Street, Hamilton, New Zealand				
Date & State of Samples	Received on 29/07/2002 in frozen state and issued Laboratory Number				
received (temperature)	190106/1-16				
Storage Conditions &	Frozen at -18°C until analysis on 07/08/2002				
Duration					
Crop Part Analysed	Whole Sample				
Summary of Method: - Preparation - Extraction	Each kumara was cut in half and homogenized to a pulp in a food processor. A 20g sub-sample was removed and extracted in ethyl acetate. The mixture was blended at high speed to a homogenous pulp then filtered. An aliquot of this was removed and cleaned up by Solid Phase Extraction (SPE). The				
- Cleanup	analytes were determined by GC-MS in the selected ion mode (SIM),				
- Analysis	monitoring the 176, 177 and 193 ions for piperonyl butoxide. The ions 183, 163 and 165 were monitored for permethrin. The ions 123, 150 and 93 were				
- Reference	monitored for pyrethrin.				
	Results expressed on an as received basis and not recovery corrected.				
	Reference: Method adapted from A.H. Roos, et al, Analytica Chimica Acta,				
	196 (1987) 95-102.				
Metabolites Detected	N/A				
Method Efficiency:	ALIA				
- Extraction efficiency	N/A				
4					
- % Recovery	Permethrin: 73% at 0.5mg/kg level				
	Piperonyl Butoxide: 76% at 0.5mg/kg level				
	Pyrethin: 72% at 0.5mg/kg level				
	Note: Spike recovery experiment was conducted on a sample (190106/9)				
	which had residues found for permethrin and piperonyl butoxide.				
- Storage Degradation	Not determined				
Correction Factor	Not applied				
Levels in Untreated Samples	N/A				
Method Sensitivity (LOD)	Permethrin: 0.01mg/kg				
	Piperonyl Butoxide: 0.01mg/kg				
· ×	Pyrethrin: 0.03mg/kg				
Full Report of Procedures and	N/A				
sample chromatograms					

### Residue Results

Sample Name	Lab No	Permethrin (mg/kg as rcvd)	Piperonyl Butoxide (mg/kg as rcvd)	Pyrethrin (mg/kg as rcvd)
KP Shed 1 Day 1 29/6/02	190106/1	0.22	0.21	0.04
KP Shed 1 Day 3 1/7/02	190106/2	0.29	0.26	< 0.03
KP Shed 1 Day 5 3/7/02	190106/3	0.46	0.39	0.07
KP Shed 1 Day 7 5/7/02	190106/4	0.20	0.17	0.03
KP Shed 1 Day 14 12/7/02	190106/5	0.29	0.25	< 0.03
KP Shed 1 Day 22 22/7/02	190106/7	0.45	0.37	0.04
KP Shed 1 Day 28 25/7/02	190106/8	0.16	0.14	< 0.03
ADB Shed 1 Day 21 2/7/02	190106/6	0.77	0.59	< 0.03
ADB Shed 1 Day 29 11/7/02	190106/9	0.10, 0.09	0.05, 0.05	< 0.03, < 0.03
KP Shed 2 Day 1 29/6/02	190106/10	0.48, 0.40	0.45, 0.39	0.04, 0.04
KP Shed 2 Day 3 1/7/02	190106/11	0.61	0.58	0.10
KP Shed 2 Day 5 3/7/02	190106/12	0.41	0.35	< 0.03
KP Shed 2 Day 7 5/7/02	190106/13	0.57	0.53	0.03
KP Shed 2 Day 14 12/7/02	190106/14	0.31	0.26	< 0.03
KP Shed 2 Day 22 22/7/02	190106/15	0.39	0.33	< 0.03
KP Shed 2 Day 28 25/7/02	190106/16	0.26	0.20	< 0.03