Horticulture Innovation Australia

Final Report

Generation of residue data for pesticide minor-use permit applications in almond tree crops

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1. MEDIA SUMMARY

In Australia, before an agrochemical product can be sold or used, it first must be registered by the Australian Pesticides and Veterinary Medicines Authority (APVMA). In order for a manufacturer to register a product they are required to submit a comprehensive data package to the APVMA. The costs for generating and collating such data are high and unfortunately many crops are too small for agrochemical manufacturers to bear the high cost of registering products for use in those crops. As a result, almond growers are often placed in situations where they risk severe crop losses from insects, weeds and diseases because appropriate pesticides are not available. On the other hand, they risk buyers rejecting their produce and other penalties if they are detected using products that are not registered for that specific use.

The APVMA's National Permit System adds some flexibility to the lengthy registration process and legalises the availability of products for minor-use purposes, not specified on the product label. However, off-label permits issued by the APVMA still must be applied for, along with information and data submitted, that verifies that the permitted use will be effective and will not have any harmful effects on humans, the crops or the environment.

In this project, a single study was conducted on the miticide/insecticide active ingredient abamectin. This study was conducted at two different field sites in Victoria and South Australia on almond tree crops. Abamectin has been registered for many years as a broad spectrum miticide and insecticide. It has registrations in a wide range of horticultural crops including apples, pears, tomatoes and strawberries. It is also registered on a wide range of winter and summer field crops. There has been an APVMA minor use permit in place for the use of abamectin in almonds (PER5658).

This study involved one application of VERTIMEC MITICIDE/INSECTICIDE (18/L abamectin) in tank mix with VICOL SUMMER OIL (825 g/L petroleum oil) at 28 days before harvest and sampling the crops at normal commercial harvest time. The sampled plant parts were then analysed for residues of abamectin. A detailed study report on the field and analytical components of the project was prepared and this will be used as part of a minor-use permit application to the APVMA. The report could also be used in the future in applications to the APVMA for label extensions to existing registered products.

The major outcome of this project is that if the permit application is approved, an additional pesticide will be available for use by almond growers. This project has been part of a larger programme of research into the minor-use of pesticides in horticulture. Although the outcomes of this project have been met there is an ongoing need for growers to have access to newer and better pesticides and so similar projects should be planned and conducted in the future.

2. TECHNICAL SUMMARY

Abamectin has been registered for many years as a broad spectrum miticide and insecticide. It has registrations in a wide range of horticultural crops including apples, pears, tomatoes and strawberries. It is also registered on a wide range of winter and summer field crops. There has been an APVMA minor use permit in place for the use of abamectin in almonds (PER5658).

A single study was conducted on almonds in 2011 and 2012. The study was conducted at two different field sites in Victoria and South Australia on commercial almond orchards. The study co-ordination was conducted by Agrisearch Services Pty Ltd at Reservoir, Victoria and the analytical component was conducted at Agrisearch Analytical Pty Ltd at Rozelle, New South Wales. The study was conducted under the OECD Principles of Good Laboratory Practice (GLP). The test substances and their active ingredients were as follows:

VERTIMEC MITICIDE INSECTICIDE – an emulsifiable concentrate formulation containing 18 g/L abamectin as the active constituent. The sample was supplied by Syngenta Crop Protection Pty Ltd.

VICOL SUMMER OIL – an oil miscible liquid formulation containing 825 g/L petroleum oil as the active constituent. The sample was supplied by Victorian Chemical Company Pty Ltd.

Field sites were selected at locations where almonds were commonly grown. Specific site details and requirements were as per the approved Study Plan and the Standard Operating Procedures (SOPs) of Agrisearch Services Pty Ltd. The treatment was applied at 28 days before commercial harvest according to Good Agricultural Practice and locally accepted procedures.

Both trials within the study were established using an unrandomised and unreplicated large block design. The pesticide treatments were applied in a manner, which simulated best commercial practice for the application of insecticides and miticides to almond trees. The method used replicated how the co-operator farmer typically grows and sprays the crop.

Sampling was carried out according to documented Standard Operating Procedures relevant to crop and plant portions to be sampled and analysed. Kernel samples that were collected from each field site were sent frozen to the nominated analytical laboratory and the samples were analysed as per the Study Plan with the laboratory report sent to the Study Director for inclusion in a composite Study Report.

The data generated from this study are being used as part of a minor-use permit application to the Australian Pesticides and Veterinary Medicines Authority APVMA. The data could also be used for future permit applications, pesticide label extensions or for inclusion in complete pesticide registration applications.

3. INTRODUCTION

A single study was conducted to determinate the residue level of abamectin following a single foliar application to almond trees. This study was conducted at two different field sites in Victoria and South Australia. The study co-ordination was conducted by Agrisearch Services Pty Ltd at Reservoir, Victoria and the analytical component was conducted at Agrisearch Analytical Pty Ltd at Rozelle, New South Wales. The study was conducted under the OECD Principles of Good Laboratory Practice (GLP).

This report contains the experimental methods used and presents the results obtained.

The study was conducted under Horticulture Australia Limited project AL11013 and Agrisearch Services Project HAL/GLP/11/01. The results from the study make up part of a submission to the APVMA for a minor use permit for the use of abamectin in almonds as per the APVMA permit PER5658.

4. MATERIALS AND METHODS

4.1 Site Details

The field component of the study was conducted in two commercial orchards located at Wood Wood Victoria and Virginia South Australia.

4.1.1 Site 1: Wood Wood Victoria

Crop	Almonds grown under normal agronomic practices for the region
Variety	Carmel
Planting Configuration	Single tree
Distance Between Rows	7.5 m
Spacing	4.5 m
Site History	Almond orchard
Prohibited Pesticides	The entire plot areas, including borders and buffer areas were not sprayed with any prohibited agrochemicals for any weeds, pests or diseases during the trial period.
Irrigation	Drip irrigation was applied as necessary during the trial period.
Soil Type	The site was located on a red sandy loam soil, which was typical of the production area in Wood Wood region. The site was wholly located on the same soil type.
Crop Stage at Application	The first application of the treatments was made 4 weeks before the optimum harvest time.
Weather Conditions	Normal weather conditions were experienced during the trial period. There were 5 wet days for a total of 65 mm or rain. The minimum temperature ranged from 9.0-24.3°C and the maximum ranged from 18.5-37.7°C.

4.1.2 Site 2: Virginia South Australia

Crop	Almonds grown under normal agronomic practices for the region
Variety	Carmel
Planting Configuration	Free standing trees
Distance Between Rows	7.2 m
Spacing	4.89 m
Site History	Permanent almond orchard
Prohibited Pesticides	The entire plot areas, including borders and buffer areas were not sprayed with any prohibited agrochemicals for any weeds, pests or diseases during the trial period.
Irrigation	Sprinkler irrigation was applied as necessary during the trial period.
Soil Type	The site was located on a brown loam soil, which was typical of the production area in the Virginia region. The site was wholly located on the same soil type.
Crop Stage at Application	The application of the treatments was made 4 weeks before the optimum harvest time.
Weather Conditions	Normal weather conditions were experienced during the trial period. There were 6 wet days for a total of 39 mm or rain. The minimum temperature ranged from 9.2-27.3°C and the maximum ranged from 18.4-40.4°C.

4.2 Treatments

Treatment	Rate Applied	Application Times DBCH	Sampling Interval From Application
1. Untreated control			28 DAT
2. VERTIMEC + VICOL SUMMER OIL	750 mL/ha + 5 L/ha	28	28 DAT

DBCH – days before commercial harvest.
DAT – days after application of Treatment 2.

4.3 Formulation

VERTIMEC MITICIDE INSECTICIDE – an emulsifiable concentrate formulation containing 18 g/L abamectin as the active constituent. The sample was supplied by Syngenta Crop Protection Pty Ltd.

VICOL SUMMER OIL – an oil miscible liquid formulation containing 825 g/L petroleum oil as the active constituent. The sample was supplied by Victorian Chemical Company Pty Ltd.

4.4 Experimental Design

Each trial was established using an unrandomised and unreplicated large block design. The individual plot sizes were four trees with a minimum area of 135 m^2. Each plot size was sufficient to produce duplicate, kernel samples in sufficient quantity and number to satisfy Australian and international sampling requirements.

The untreated plots were situated as up-slope and as up-wind from each treated plot as practical, to prevent contamination of the untreated plot. Each plot was marked to completely and uniquely identify it by its geometry, trial number and treatment number. Test plots were considered as restricted access areas with measures taken to exclude unauthorised persons from the test area.

4.5 Application Method

Treatments were applied by motorised hand gun, with a single solid cone or single hollow cone nozzle, in sufficient water to ensure even and thorough coverage of all parts of each tree. The method used replicated how the co-operator farmer typically sprays the crop.

4.5.1 Site 1: Wood Wood Victoria

Application Date	14 February 2012
Treatments Applied	2
Actual Application Volume	1990 L/ha
Time (hours)	1100-1130
Temperature	29°C
Relative Humidity	43%
Cloud Cover	0%
Wind	2-5 km/hr, SE
Growth Stage	BBCH 85
Comments	Fine, sunny, warm

4.5.2 Site 2: Virginia South Australia

Application Date	9 February 2012
Treatments Applied	2
Actual Application Volume	2008 L/ha
Time (hours)	1542-1635
Temperature	23°C
Relative Humidity	49%
Cloud Cover	20%
Wind	2-5 km/hr, SSW
Growth Stage	BBCH 87
Comments	Fine and mild

4.6 Sampling Schedule

Residue sampling was undertaken according to the Study Plan and Agrisearch Services Pty Ltd Standard Operating Procedures.

At least 1 kg of kernels was sampled from 4 individual almond trees of each treatment for each sample. The samples were taken from all parts of the plot, however the ends of each plot were not sampled. Two samples were taken from each treatment on each sampling date with one being the Primary Sample and the other the Reserve Sample.

4.6.1 Site 1: Wood Wood Victoria

Sample Number	Rep.	Treatment	Application Rate	Substrate Type	Sampling Interval from Application No.	Sampling Date
111007-1	Primary	1. Untreated	Untreated	Kernel	28 DAT	13-Mar-12
111007-2	Primary	2. VERTIMEC	750 mL/ha	Kernel	28 DAT	13-Mar-12
111007-3	Reserve	1. Untreated	Untreated	Kernel	28 DAT	13-Mar-12
111007-4	Reserve	2. VERTIMEC	750 mL/ha	Kernel	28 DAT	13-Mar-12

DAT = days after treatment

4.6.2 Site 2: Virginia South Australia

Sample Number	Rep.	Treatment	Application Rate	Substrate Type	Sampling Interval from Application No.	Sampling Date
111008-1	Primary	1. Untreated	Untreated	Kernel	28 DAT	08-Mar-12
111008-2	Primary	2. VERTIMEC	750 mL/ha	Kernel	28 DAT	08-Mar-12
111008-3	Reserve	1. Untreated	Untreated	Kernel	28 DAT	08-Mar-12
111008-4	Reserve	2. VERTIMEC	750 mL/ha	Kernel	28 DAT	08-Mar-12

DAT = days after treatment

4.7 Sample Handling

Samples were handled, stored and transported according to Agrisearch Services Standard Operating Procedures.

Samples were packed into double heavy polythene bags and individually labelled in the field. Untreated and treated samples were placed in separate containers in the vehicle and transported to, and placed in, the sample storage rooms, immediately upon return from the field. The untreated and treated samples were physically separate in the same storage room. The temperature of the sample storage room was monitored regularly to ensure the samples were maintained below the desired temperature and to ensure the sample storage room was functioning normally.

Samples remained in freezer storage prior to dispatching to the analytical laboratory. A Sample Shipping Chain of Custody accompanied the samples. A temperature data logger was placed with the samples in transit to monitor the temperature. All samples arrived at the laboratory in a frozen state and in excellent condition as documented on the Sample Shipping Chain of Custody.

4.8 Residue Analysis

All samples were processed and analysed by Agrisearch Analytical Pty Ltd at Rozelle, NSW.

Abamectin residues were determined according to:

> *"Determination of Macrocyclic Lactone Residues in Animal Tissues and Milk", AATM-R-53, Revision 9, Agrisearch Analytical Pty Ltd, August 2011.*

A summary of the method is presented below:

> *Residues of abamectin were extracted from blended homogeneous almonds by maceration of the sample with acetonitrile. The extract was filtered to remove solids and was then partitioned with hexane. The acetonitrile fraction was evaporated to dryness and the residuum was dissolved in hexane.*
>
> *The samples were further cleaned up using NH2 solid phase extraction (SPE) cartridge. The SPE cartridge was washed with hexane, toluene and methylene chloride. Macrocyclic lactones were eluted from the cartridge with methylene chloride:acetone (1:2). The extract was evaporated to dryness.*
>
> *The residuum was dissolved in a mixture of 1-methylimidazole solution in acetonitrile (1:1). Trifluoroacetic anhydride in acetonitrile (1:2) was added to initiate derivatisation. Avermectin B1a and its 8,9-Z isomer form an identical fluorescent compound as does avermectin B1b and its 8,9-Z isomer. Final determination of the derivatised B1a and B1b analytes was via reversed phase HPLC with fluorescence detection against a series of standards similarly derivatised.*

5. RESULTS

Results are summarised in Tables 1-2 below.

Table 1 Summary of Results – Residues of abamectin in Almonds Following the Application of VERTIMEC MITICIDE INSECTICIDE, Trial 111007, Wood Wood, Victoria

Sample Number	Application Rate	Sampling Interval from Application Number	Sampling Date	Abamectin^ (mg/kg)
111007-1	UTC	28 DAT	13-Mar-12	<LOD
111007-2	VERTIMEC 750 mL/ha	28 DAT	13-Mar-12	<LOD

LOD = Limit of Detection = 0.001 mg/kg
LOQ = Limit of Quantitation = 0.005 mg/kg
UTC = Untreated control
DAT = days after treatment
^ Abamectin = Sum of avermectin B1a, avermectin B1b, (Z)-8,9 avermectin B1a and (Z)-8,9 avermectin B1b.

Table 2 Summary of Results – Residues of abamectin in Almonds Following the Application of VERTIMEC MITICIDE INSECTICIDE, Trial 111008, Virginia, South Australia

Sample Number	Application Rate	Sampling Interval from Application Number	Sampling Date	Abamectin^ (mg/kg)
111008-1	UTC	28 DAT	08-Mar-12	<LOD
111008-2	VERTIMEC 750 mL/ha	28 DAT	08-Mar-12	<LOD

LOD = Limit of Detection = 0.001 mg/kg
LOQ = Limit of Quantitation = 0.005 mg/kg
UTC = Untreated control
DAT = days after treatment
^ Abamectin = Sum of avermectin B1a, avermectin B1b, (Z)-8,9 avermectin B1a and (Z)-8,9 avermectin B1b.

No residues of abamectin were detected in the almond samples that had been collected 28 days after the application of abamectin to almonds.

A minor use permit application, similar to PER5658, was prepared and sent to the HAL Minor Use Co-ordinator for submission to the APVMA.

6. DISCUSSION

In two trials, a single application of VERTIMECT MITICIDE INSECTICIDE (18 g/L abamectin) was made at 750 mL/ha (13.5 g ai/ha) at 28 days before commercial harvest. Samples for residue analysis were taken 28 days after the last application.

Residues of abamectin in almond kernels were below the Level Of Detection at both trial sites.

A comprehensive confidential report was prepared. This report was deemed to be compliant to the OECD Principles of Good Laboratory Practice (GLP). The report was submitted to HAL on 4 December 2012.

7. TECHNOLOGY TRANSFER

The data generated from the study reported on here will be included in submissions to the Australian Pesticides and Veterinary Medicines Authority. A minor use permit application for the use of abamectin in almonds has been prepared and forwarded to the HAL Minor Use Coordinator. The data may also be used for additional submissions for permit applications, pesticide label extensions or for inclusion in complete pesticide registration applications. The results of the applications are disseminated on the APVMA website, the Government Gazette and by industry publications. There is also an ongoing rationalisation of pesticide permits and the transfer of permits to current pesticide labels.

8. RECOMMENDATIONS

It is recommended that the permit application that has been prepared, is submitted by the HAL Minor Use Coordinator to the APVMA for the permit renewal of PER5658.

It is also recommended that the confidential GLP report be offered to registrants of abamectin for them to use in label extensions of their products.