

**Generation of Residue Data for Vegetable  
Minor-use Permit Applications - 2009 -  
AgriSolutions**

Andrew Keats  
AgriSolutions Australia Pty Ltd

Project Number: VG09135

## **VG09135**

This report is published by Horticulture Australia Ltd to pass on information concerning horticultural research and development undertaken for the vegetables industry.

The research contained in this report was funded by Horticulture Australia Ltd with the financial support of:  
Dow AgroSciences Australia Ltd  
the vegetables industry.

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ISBN 0 7341 2560 7

Published and distributed by:  
Horticulture Australia Ltd  
Level 7  
179 Elizabeth Street  
Sydney NSW 2000  
Telephone: (02) 8295 2300  
Fax: (02) 8295 2399

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# **Final Report**

for

## **Project VG09135**

Generation of Residue Data for Vegetable Minor-use Permit  
Applications - 2009 – AgriSolutions Australia Pty Ltd

Andrew Keats (December 2010)

Horticulture Australia Limited Project VG09135

December 2010

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This report provides an outline of trials that were undertaken to generate pesticide residue data in a range of vegetable crops to support minor-use permit applications to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

This project has been funded by HAL using the vegetable levy and a voluntary contribution from Dow AgroSciences Australia Ltd with matched funds from the Federal Government.

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## **Project Summary:**

Horticulture Australia Limited (HAL) and the Australian Vegetable and Potato Growers Federation (Ausveg) requires the generation of pesticide residue data in a range of vegetables crops to support minor-use permit applications to the Australian Pesticides and Veterinary Medicines Authority (APVMA). The key outcome of this project is the gaining of approval from the APVMA for use of the listed pesticide uses under APVMA minor use permits.

The project was conducted to determine the residue profile of methoxyfenozide and bentazone-sodium when applied to cucumber, capsicum, leafy lettuce, green peas, sugar snap peas and snow peas grown in either field conditions or protected structures. Eleven field trials were conducted during this project. Details of the field trials are discussed in the following pages.

The field and analytical components were conducted by AgriSolutions Australia Pty Ltd. The study was conducted under the OECD Principles of Good Laboratory Practice (GLP).

This project was funded by the Vegetable Industry levy with matched funding from the Australian Government.

## **Introduction:**

In Australia, before an agrochemical product can be sold or used, it must first be registered with the APVMA. In order for a manufacturer to gain registration approval for a product, they are required to submit a comprehensive data package to the APVMA. The costs for generating and collating such data can be substantial and unfortunately many horticultural crops are too small individually for agrochemical manufacturers to bear the cost of registering products for use. As a result, horticulturalists are often placed in situations where they risk severe crop losses from insects, weeds and diseases. On the other hand, they risk buyers rejecting their produce and other penalties if they are detected using products that are not registered. The need to gain minor-use permits and new registrations has come about due to loss of some agrochemicals and/or uses due to chemical reviews and product rationalisation. Horticultural produce must meet minimum standards relating to quality, safety and consumer expectation. Quality Assurance programs, dealing with the whole production process including agrochemical use, residues, and withholding periods, demand that growers only exercise Good Management Practices. The introduction of new and emerging crops, pesticide resistance, integrated pest management, the continual vigilance of horticultural industries for improved agrochemical choices and the disinclination of manufacturers to register for minor crops has led to the need for this project. The selective use of pesticides to control pests, weeds and diseases plays an important role in increasing production, improving the quality of Australia's horticultural crops and enabling growers to earn reasonable returns on their investments. At the same time, today's health conscious society is extremely sensitive to issues relating to chemical use and it is essential that consumers be protected by adequate regulations governing the use of agrochemicals. 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/data that verifies that the permitted use will be effective and will not have any harmful effects on humans, the crops or the environment. In early 2000, the vegetable industry undertook a national approach to permits by working with industry generated 'wishlists' for new pesticide uses, but this led to congestion in Australian Pesticide & Veterinary Management Authority (APVMA) system and dissatisfaction amongst growers and grower groups. This was in part due to widespread duplication of the requests made for permits in the absence of a truly co-ordinated system and concern over the priority assessment for each pesticide. This approach was also unable to give relevant priority to new pesticide technologies and available Integrated Pest Management (IPM) friendly pesticides that were outside the industry's experience. A new approach to address the current and future pesticide requirements for horticultural crops has been developed using the Strategic Agrichemical Review Process (SARP). This approach had the benefit of IPM compatibility, where possible, improved scope for resistance management, sound biological profile and residue and trade acceptance domestically and for export. This review process provides the vegetable industry with sound pesticide options for the future that the industry can pursue for registration with the manufacturer, or minor-use permits with APVMA for clearly identified crop protection needs, many of which will also assist the expansion of effective IPM strategies. All of the studies in this tender have been identified through the SARP.

**Study Number:**

AVG50

**Study Name:**

Determination of Bentazone-sodium residues in pea pods and fodder following one application of Basagran<sup>®</sup> 480 SL

**Study Objective:**

This study was conducted to generate residue data on Basagran<sup>®</sup> (480 g/L bentazone) {present as the sodium salt} in peas. Two trials were conducted in Tasmania to determine the levels of residues of bentazone in peas following one foliar application of the herbicide Basagran<sup>®</sup> 480 SL.

The purpose of the study was to generate data in support the renewal of a minor use permit to use Basagran<sup>®</sup> 480 SL in peas in Tasmania being made to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

**Study Justification:**

Basagran<sup>®</sup> 480 SL is used for the control of a range of weeds in pea crops. Residue data are needed to support a renewal application for a minor use permit to use Basagran<sup>®</sup> 480 SL in pea crops. The data from this study has been submitted to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

**Analytical Method Details:**

The method utilised for the analyses of the snow pea and sugar snap pea specimens obtained during Study No. HAL1637 was as follows:

BASF Method No. 438/2 (L0044/02): "*Method for the Determination of Bentazone, 6-0H-Bentazone and 8-0H-Bentazone in Plant and Animal Matrices*". December 2008

Residues of bentazone were extracted from crop samples with 70/30 methanol/water. The sample was centrifuged, and an aliquot of the extract evaporated to aqueous residue and diluted with water, cleaned up using C18 solid phase extraction (SPE) cartridges. The SPE cartridge was eluted with 20/80 acetone/hexane. After addition of ascorbic acid as an antioxidant, the eluate was evaporated to dryness and the residues reconstituted in methanol/water. The purified extract was then analysed by LC-MS/MS.



## **Field Trial Details:**

Two field trials were conducted during this study in processing pea crops.

**Trial one** was conducted at Ulverstone, North Western Tasmania on a clay loam soil. The cultivar was Small Sieve Freezer.

A single rate of Basagran<sup>®</sup> was applied once at the 6 – 7 node growth stage.

Pod specimens were collected on two occasions, just prior to commercial harvest and at commercial harvest. Fodder specimens were collected at commercial harvest. Untreated pod and fodder specimens were also collected and analysed.

Residue levels in pods were reported on a fresh weight basis. Residue levels in fodder were reported on both a fresh weight basis and a dry weight basis.

The levels of bentazone-sodium residues determined for both sampling intervals were at or below the temporary MRL of T0.05 as established by the APVMA for peas.

**Trial two** was conducted at Cressy in the Central North of Tasmania on a clay loam soil. The cultivar was Resal.

A single rate of Basagran<sup>®</sup> was applied once at the 6 – 7 node growth stage.

Pod specimens were collected on two occasions, just prior to commercial harvest and at commercial harvest. Fodder specimens were collected at commercial harvest. Untreated pod and fodder specimens were also collected and analysed.

Residue levels in pods were reported on a fresh weight basis. Residue levels in fodder were reported on both a fresh weight basis and a dry weight basis.

The levels of bentazone-sodium residues determined for both sampling intervals were at or below the temporary MRL of T0.05 as established by the APVMA for peas.

## **Submission:**

The final report and permit application for this study was submitted to APVMA in July 2010.

**Study Number:**

HAL1454

**Study Name:**

Determination of methoxyfenozide residues in cucumbers, capsicums and leafy lettuce following three applications of Prodigy™ 240 SC

**Study Objective:**

This study was conducted to generate residue data on Prodigy™ 240 SC (240 g/L methoxyfenozide) in cucumbers, capsicums and leafy lettuce. Six trials were conducted to determine the levels of residues of methoxyfenozide in cucumbers, capsicums and leafy lettuce following three foliar applications of the insecticide Prodigy™.

The purpose of the study was to generate data in support of a minor use permit application to use Prodigy™ in cucumbers, capsicums and leafy lettuce being made to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

**Study Justification:**

Prodigy™ 240 SC is to be used for the control of *Helicoverpa spp* in cucumber, capsicum and leafy lettuce crops. Residue data are needed to support an application for a minor use permit to use Prodigy™ 240 SC in cucumber, capsicum and leafy lettuce crops. The data from this study will be submitted to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

The method utilised for the analyses of the snow pea and sugar snap pea specimens obtained during Study No. HAL1454 was as follows:

**Analytical Method Details:**

Dow AgroSciences LLC Method GRM: 02.25 "*Determination of Residues of Methoxyfenozide in High Moisture Crops by Liquid Chromatography with Tandem Mass Spectrometry Detection*". Dow AgroSciences LLC, Indiana 46268-1054. Date: October 4, 2002.

Residues of methoxyfenozide were extracted from a high moisture crop sample with a 90% methanol/10% 0.1N hydrochloric acid solution. The sample was centrifuged, and an aliquot of the extract was diluted with water and purified using Phenomenex Strata solid phase extraction (SPE) cartridges. The SPE cartridge was washed with a 60% water/40% methanol/0.1% formic acid solution and eluted with acetonitrile. The eluate was evaporated to dryness and the residues reconstituted in mobile phase. The purified extract was then analysed by LC-MS/MS.

## Field Trial Details:

Six field trials were conducted during this study.

**Trial one** was conducted at Hampton, South East Queensland. The crop was cucumbers and the cultivar was Lebanese. The crop was grown hydroponically in a greenhouse. The growing medium was sawdust.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Cucumber specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated cucumbers were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period to be equivalent to the 0.01 mg/kg methoxyfenozide level of quantitation at the final sample timing.

**Trial two** was conducted at Virginia, South Australia. The crop was cucumbers and the cultivar was Myrthos. The crop was grown hydroponically in a greenhouse.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Cucumber specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated cucumbers were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period to be just above the 0.01 mg/kg methoxyfenozide level of quantitation at the final sample timing.

**Trial three** was conducted at Doonan, South East Queensland. The crop was capsicum and the cultivar was Warlock. The crop was grown hydroponically in a greenhouse. The growing medium was hydroponic bags.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Capsicum specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated cucumbers were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period so that by the final sample timing, residues were well below the MRL of 3, as established by the APVMA for fruiting vegetables (non-cucurbits).

**Trial four** was conducted at Virginia, South Australia. The crop was capsicum and the cultivar was Remy. The crop was grown hydroponically in a greenhouse.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Capsicum specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated capsicums were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period so that by the final sample timing, residues were well below the MRL of 3, as established by the APVMA for fruiting vegetables (non-cucurbits).

**Trial five** was conducted at Bli Bli, South East Queensland. The crop was leafy lettuce and the cultivar was Green Oak. The crop was grown hydroponically in a greenhouse. The growing medium was hydroponic bags.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Leafy lettuce specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated leafy lettuces were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period and were similar to the levels in the capsicums from trials three and four.

**Trial six** was conducted at Tranmere, South Australia. The crop was leafy lettuce and the cultivar was Blackbelt. The crop was grown hydroponically in a greenhouse.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Leafy lettuce specimens were collected on five occasions, from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated leafy lettuces were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period but were higher than the levels in the leafy lettuces from trial five.

### **Submission:**

The final report and permit application for this study was submitted to APVMA in August 2010.

**Study Number:**

HAL1637

**Study Name:**

Determination of methoxyfenozide residues in snow peas or sugar snap peas following three applications of Prodigy™ 240 SC.

**Study Objective:**

This study was conducted to generate residue data on Prodigy™ (240 g/L methoxyfenozide) in snow peas or sugar snap peas. Three trials were conducted to determine the levels of residues of methoxyfenozide in snow peas or sugar snap peas following three foliar applications of the insecticide Prodigy™ 240 SC.

The purpose of the study was to generate data in support of a minor use permit application to use Prodigy™ 240 SC in snow peas or sugar snap peas being made to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

**Study Justification:**

Prodigy™ 240 SC is to be used for the control of *Helicoverpa spp* in snow pea or sugar snap pea crops. Residue data are needed to support an application for a minor use permit to use Prodigy™ 240 SC in snow pea or sugar snap pea crops. The data from this study will be submitted to the Australian Pesticides and Veterinary Medicines Authority (APVMA).

The method utilised for the analyses of the snow pea and sugar snap pea specimens obtained during Study No. HAL1637 was as follows:

**Analytical Method Details:**

Dow AgroSciences LLC Method GRM: 02.25 "*Determination of Residues of Methoxyfenozide in High Moisture Crops by Liquid Chromatography with Tandem Mass Spectrometry Detection*". Dow AgroSciences LLC, Indiana 46268-1054. Date: October 4, 2002.

Residues of methoxyfenozide were extracted from a high moisture crop sample with a 90% methanol/10% 0.1N hydrochloric acid solution. The sample was centrifuged, and an aliquot of the extract was diluted with water and purified using Phenomenex Strata solid phase extraction (SPE) cartridges. The SPE cartridge was washed with a 60% water/40% methanol/0.1% formic acid solution and eluted with acetonitrile. The eluate was evaporated to dryness and the residues reconstituted in mobile phase. The purified extract was then analysed by LC-MS/MS.

### **Field Trial Details:**

Three field trials were conducted during this study.

**Trial one** was conducted at Doonan, South East Queensland. The crop was sugar snap peas and the cultivar was Sugar Bon. The crop was grown in soil in a greenhouse. The soil type was Brown Loam.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Sugar snap pea specimens were collected on five occasions from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated sugar snap peas were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period.

**Trial two** was conducted at Virginia, South Australia. The crop was snow peas and the cultivar was Yates Climbing. The crop was grown in soil in a greenhouse. The soil type was loam.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Snow pea specimens were collected on five occasions from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated snow peas were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period.

**Trial three** was conducted at Doonan, South East Queensland. The crop was snow peas and the cultivar was Oregon Giant. The crop was field grown in a brown loam soil.

A single rate of Prodigy™ was applied three times at seven day intervals prior to maturity.

Snow pea specimens were collected on five occasions from 0 through to 7 days after the last application, so as to provide data on the chemical residue decline. Untreated snow peas were also collected and analysed.

The levels of methoxyfenozide residues determined by the analysis declined over the sampling period.

### **Submission:**

The final report and permit application for this study was submitted to APVMA in December 2010.