

Determination of cyprodinil and fludioxonil residues in leafy vegetables

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DETERMINATION OF CYPRODINIL + FLUDIOXONIL RESIDUES IN LEAFY VEGETABLES

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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 vegetable crops are too small individually for agrochemical manufacturers to bear the high cost of registering products for use in those crops. As a result, vegetable 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.

This project was established to generate residue data following the application of cyprodinil + fludioxonil on head lettuce, leafy lettuce grown in the field and also hydroponically in protected structures, spinach and Chinese cabbage, for registration purposes in Australia. This project is part of a collaborative project between the Australian and US (IR-4) minor-use programs for data generation leading to sharing and registration of uses in both countries. This study was conducted at 11 different field sites in New South Wales, Queensland, Victoria and South Australia.

The study involved two or four applications of the pesticides on the target crops, sampling the crops at or around the normal commercial harvest time, and then analysing the sampled plant parts for residues of the target pesticide. A detailed study report on the field and analytical components was prepared and this was used as part of the permit applications to the APVMA.

The major outcome of this project is that pesticides that could not be legally used by vegetable growers will now be available. This project has been part of a larger programme of research that has been conducted over the past few years. 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

A study was conducted with cyprodinil + fludioxonil on head lettuce, leafy lettuce grown in the field and also hydroponically in protected structures, spinach and Chinese cabbage, for registration purposes in Australia. The study was conducted at 11 different field sites in New South Wales, Queensland, Victoria and South Australia. The study co-ordination was conducted by Agrisearch Services Pty Ltd at Orange, New South Wales 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 substance and its active ingredient were as follows:

SWITCH FUNGICIDE – a water dispersible granule formulation containing 375 g/kg cyprodinil and 250 g/kg fludioxonil

Field sites were selected at locations where the nominated crop was 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. Treatment application timing and sampling was according to Good Agricultural Practice and locally accepted procedures.

Each trial within the study was 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 fungicides to the target crops. 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 the crop and plant portion to be sampled and analysed.

Plant samples that were collected from each field site were sent frozen to Agrisearch Analytical Pty Ltd 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 for each of the eleven studies.

The data generated from the studies have been included or will be included in submissions to the Australian Pesticides and Veterinary Medicines Authority. These submissions are for permit applications, pesticide label extensions or for inclusion in complete pesticide registration applications.

3. INTRODUCTION

A study was conducted with cyprodinil + fludioxonil on head lettuce, leafy lettuce grown in the field and also hydroponically in protected structures, spinach and Chinese cabbage, for registration purposes in Australia. The study was conducted at 11 different field sites in New South Wales, Queensland, Victoria and South Australia. The study co-ordination was conducted by Agrisearch Services Pty Ltd at Orange, New South Wales 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 trial was conducted under Horticulture Australia Limited project VG08173 Agrisearch Project HAL/GLP/09/01.

4. MATERIALS AND METHODS

4.1 Study Details

This study was conducted to determine the tissue residue profile of cyprodinil and fludioxonil when applied to head lettuce, leafy lettuce, spinach and Chinese cabbage, following foliar applications of SWITCH FUNGICIDE.

The study co-ordination was conducted by Agrisearch Services Pty Ltd at 4/16 Jusfrute Drive, West Gosford NSW. The analytical component was conducted at Agrisearch Analytical, 1/48 Victoria Rd, Rozelle, NSW. The study was conducted under the OECD Principles of Good Laboratory Practice (GLP). The study was conducted according to an approved Study Plan that had been prepared as per the OECD GLP Guidelines.

4.2 Trial Sites

Field sites were selected at locations where the nominated crop was commonly grown. Eleven vegetable sites were established; nine grown in the field and two leafy lettuce trials grown hydroponically in protected structures. The head lettuce sites were at Mangrove Mountain NSW, Werribee Victoria and Gatton Queensland. The leafy lettuce sites grown in protected structures were at Doyalson NSW and Two Wells South Australia. The field grown leafy lettuce sites were at Gatton Queensland and Bacchus Marsh Victoria. The spinach trials were located at St Kilda South Australia and Clyde Victoria and the Chinese cabbage trials were located at Gatton Queensland and Freemans Reach NSW.

4.3 Trial Design

Each trial was established using an unrandomised and unreplicated large block design. The individual plot sizes generally ranged between 3-12 m² in area. Larger plot sizes were used if it was deemed necessary to obtain the required sample sizes. Each plot size was sufficient to produce duplicate, fresh-weight samples of produce on multiple occasions after the last application of each treatment, in sufficient quantity and number to satisfy 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.

4.4 Formulation

The pesticide formulation used in the study was as follows:

SWITCH FUNGICIDE – a water dispersible granule formulation containing 375 g/kg cyprodinil and 250 g/kg fludioxonil as the active constituents. The sample was supplied by AgriSolutions Pty Ltd.

4.5 Treatment Method

The treatments were applied in a manner that simulated best commercial practice for the application of SWITCH FUNGICIDE in field grown head lettuce, field and protected structure grown leafy lettuce, field grown spinach and field grown Chinese cabbage. Treatments were applied by boom spray in sufficient water to ensure even and thorough coverage of all parts of each plant. Total volume ranged from 232 L/ha to 935 L/ha.

4.6 Sampling Procedures

For head lettuce and Chinese cabbage, samples were taken from a minimum of 12 plants with a sample size of at least one kilogram. Some individual plants were sub-sampled by cutting along the vertical axis. For the leafy lettuce and spinach samples, more than one kilogram was sampled from at least 12 plants. The end plants 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.

The Primary Samples were the samples that were sent to the laboratory for analysis. The Reserve Samples remain in the freezer for at least 12 months after the completion of each study after which time they are discarded.

4.7 Treatment and Sampling Times

The treatments and sampling times for Trial 090329, on field grown head lettuce at Mangrove Mountain, NSW, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			4 DAT2 Treatment 2
2.	SWITCH	980.7 (1027) g/ha	2 (at 7 days)	0, 4, 7 DAT2
3.	SWITCH	980.7 (1000) g/ha	2 (at 7 days)	12 DAT2
4.	SWITCH	980.7 (1009) g/ha	4 (at 7 then 14 then 7 days)	0, 4, 7 DAT4
5.	SWITCH	980.7 (990.3) g/ha	4 (at 7 then 14 then 7 days)	12 and 15 DAT4

*Averaged over all applications

4DAT2 – 4 days after the second application of the treatment

The treatments and sampling times for Trial 090330, on field grown head lettuce at Werribee South, Victoria, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (985.1) g/ha	2 (at 6 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (1015) g/ha	2 (at 7 days)	10 DAT2
4.	SWITCH	980.7 (1053) g/ha	4 (at 7 then 14 then 6 days)	7 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090331, field grown head lettuce at Gatton, Queensland, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (988.5) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (1004) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (998.8) g/ha	4 (at 7 then 14 then 7 days)	0, 3, 7 DAT4
5.	SWITCH	980.7 (1015) g/ha	4 (at 6 then 14 then 7 days)	11 and 15 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090332, leafy lettuce grown hydroponically in protected structures at Doyalson, NSW, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (1163) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (1204) g/ha	2 (at 6 days)	11 DAT2
4.	SWITCH	980.7 (1197) g/ha	4 (at 6 then 15 then 7 days)	7 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090333, leafy lettuce grown hydroponically at Two Wells, South Australia, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (984.1) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (999.8) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (994.2) g/ha	4 (at 8 then 13 then 7 days)	0, 3, 7 DAT4
5.	SWITCH	980.7 (998.0) g/ha	4 (at 7 then 14 then 7 days)	11 and 15 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090334, field grown leafy lettuce at Gatton, Queensland, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (988.8) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (991.5) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (984.6) g/ha	4 (at 7 then 14 then 7 days)	7 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090335, field grown leafy lettuce at Bacchus Marsh, Victoria, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (956.1) g/ha	2 (at 6 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (963.7) g/ha	2 (at 8 days)	10 DAT2
4.	SWITCH	980.7 (1002) g/ha	4 (at 7 then 14 then 6 days)	0, 3, 7 DAT4
5.	SWITCH	980.7 (1081) g/ha	4 (at 7 then 14 then 8 days)	10 and 15 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090336, field grown spinach at St Kilda, South Australia, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (997.4) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (1003) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (988.5) g/ha	4 (at 7 then 14 then 7 days)	7 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090337, field grown spinach at Clyde, Victoria, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (1009) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (999.8) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (1051) g/ha	4 (at 7 then 14 then 7 days)	0, 3, 7 DAT4
5.	SWITCH	980.7 (1031) g/ha	4 (at 7 then 14 then 7 days)	11 and 15 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

The treatments and sampling times for Trial 090338, field grown Chinese cabbage at Gatton, Queensland, are presented in the table below:

Number	Treatment	Rate Applied (Actual*)	Number of Applications (and intervals)	Sampling Interval From Application Number
1.	Untreated control			3 DAT2 Treatment 2
2.	SWITCH	980.7 (985.1) g/ha	2 (at 7 days)	0, 3, 7 DAT2
3.	SWITCH	980.7 (1007) g/ha	2 (at 7 days)	11 DAT2
4.	SWITCH	980.7 (1008) g/ha	4 (at 7 then 14 then 7 days)	7 DAT4

*Averaged over all applications

3DAT2 – 3 days after the second application of the treatment

4.8 Analysis of Samples

Plant samples that were collected from each field site were sent frozen to the nominated analytical laboratory, Agrisearch Analytical Pty Ltd, as per the Study Plan. 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.

Cyprodinil and fludioxonil residues were determined according to an analytical method developed by Agrisearch Analytical Pty Ltd: “Determination of Multi-Residues in Fruit and Vegetables using DSPE” AATM-S-60, Revision 8, Agrisearch Analytical Pty Ltd, September 2008.”

5. RESULTS AND DISCUSSION

These data are presented in the relevant detailed GLP report used in submissions to the APVMA and other regulatory authorities. Such data are considered to be confidential at this stage and therefore are not presented in this report.

6. TECHNOLOGY TRANSFER

The data generated from the studies reported on here have been included or will be included in submissions to the Australian Pesticides and Veterinary Medicines Authority. These submissions are 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.

7. RECOMMENDATIONS

The major outcome of this project is that pesticides that could not be legally used by vegetable growers will now be available, thus providing growers with a broader range of options in the control of diseases and insect pests from which their crops suffer.

This project has been part of a larger programme of research that has been conducted over the past few years. 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.