

The reconsideration of approval of the active constituent Endosulfan, registrations of products containing Endosulfan and their associated labels.

FINAL REVIEW REPORT AND REGULATORY DECISION

VOLUME 2 TECHNICAL REPORT

June 2005

Australian Pesticides & Veterinary Medicines Authority

Canberra Australia © This work is copyright. Apart from any use permitted under the *Copyright Act 1968*, no part may be reproduced without permission from the Australian Pesticides and Veterinary Medicines Authority: 2004.

This review report for the active constituent Endosulfan and products containing Endosulfan is published by the Australian Pesticides and Veterinary Medicines Authority. For further information about this review or the Pesticides Review Program, contact:

Manager Pesticides Review Australian Pesticides and Veterinary Medicines Authority PO Box E240 KINGSTON ACT 2604 Australia

Telephone: 61 2 6272 3213 Facsimile: 61 2 6272 3218

Email: chemrev@apvma.gov.au APVMA web site: http://www.apvma.gov.au

#### **FOREWORD**

The APVMA is an independent statutory authority with responsibility for the regulation of agricultural and veterinary chemicals in Australia. Its statutory powers are provided in the *Agricultural and Veterinary Chemicals Code Act*, 1994 (Agvet Codes).

The APVMA can reconsider the approval of active constituents, the registration of chemical products or the approval of labels for containers of chemical products at any time. This is specified in Part 2, Division 4 of the Agvet Codes.

The basis for the reconsideration is whether the APVMA is satisfied that continued use of the active constituent endosulfan and products containing endosulfan in accordance with the instructions for their use:

- would not be an undue hazard to the safety of people exposed to it during its handling; and/or
- would not be likely to have an effect that is harmful to human beings; and /or
- would not be likely have an unintended effect that is harmful to animals, plants or things or to the environment; and/or
- would not unduly prejudice trade or commerce between Australia and places outside Australia.

A reconsideration may be initiated when new research or evidence has raised concerns about the use or safety of a particular chemical, a product or its label.

The process for reconsideration includes a call for information from a variety of sources, a review of that information and, following public consultation, a decision about the future use of the chemical or product.

In undertaking reviews, the APVMA works in close cooperation with advisory agencies including the Department of Health and Aging, the Department of Environment and Heritage, the National Occupational Health and Safety Commission, and State Departments of Agriculture as well as other expert advisors, as appropriate.

The APVMA has a policy of encouraging openness and transparency in its activities and community involvement in decision-making. The publication of review reports is a part of that process.

The APVMA also makes these reports available to the regulatory agencies of other countries under bilateral agreements. It is proposed that countries receiving these reports will not utilise them for registration purposes unless they are also provided with the raw data from the relevant applicant.

This document contains the draft Final Review Report – Technical Reports of 'The reconsideration of approval of the active constituent Endosulfan, registrations of products containing Endosulfan and their associated labels' and relates to all products containing endosulfan. The review's findings and recommendations are based on information collected from a variety of sources. The information and technical data required by the APVMA to review the safety of both new and existing chemical products must be derived according to accepted scientific principles, as must the methods of assessment undertaken.

The draft review report containing the APVMA preliminary assessments (The NRA Review of Endosulfan, Volume I, August 1998) and the technical reports from its advisory agencies (Volume II) for all registrations and approvals relating to endosulfan are available from the APVMA website: <a href="http://www.apvma.gov.au/chemrev/chemrev.shtml">http://www.apvma.gov.au/chemrev/chemrev.shtml</a>.

# TABLE OF CONTENTS

8 RESII	DUES ASSESSMENT TECHNICAL REPORT	7
8.1 N	METABOLISM STUDIES	7
8.1.1	Hen Studies	7
8.1.2	Lactating Cow Studies	9
8.1.3	Sheep	
8.2 R	RESIDUE DEFINITION	11
	NALYTICAL METHODOLOGY	
8.3.1	Crop matrices	11
8.3.2	Animal tissues and milk	
8.4 S	TORAGE STABILITY	
8.4.1	Storage stability in crop matrices.	
8.4.2	Stability of endosulfan in animal tissues and milk	
8.5 C	CROP RESIDUE STUDIES	
8.5.1	Citrus fruit (oranges, mandarins, lemons)	
8.5.2	Pome fruit (apples, pears)	
8.5.3	Stone fruit (peaches, nectarines, apricots)	
8.5.4	Grapes	
8.5.5	Tropic/sub-tropic fruit-inedib peel (avocado, custard apple, mango, pawpaw, persimmon)	29
8.5.6	Brassica vegetables (broccoli, cabbage, cauliflower, Brussels sprouts)	33
8.5.7	Cucurbits (cucumber, melons, zucchini)	
8.5.8	Fruiting vegetables (capsicum, eggplant, tomato, sweet corn)	
8.5.9	Leafy vegetables (chinese cabbage, silverbeet, lettuce)	
8.5.10	Legume vegetables (beans, peas)	
8.5.11	Pulse crops (navy beans, faba beans, cow peas, field peas, lupins, chickpeas)	
8.5.12	Root and tuber vegetables (beetroot, carrot, potato, sweet potato)	
8.5.13	Stalk and stem vegetables (celery, rhubarb)	
8.5.14	Cereal Crops (sorghum, wheat, barley)	
8.5.15	Tree Nuts (macadamia)	
8.5.16	Oilseeds (Cotton, Soyabeans, Sunflowers, Canola)	
	NIMAL TRANSFER STUDIES	
8.6.1	Cattle studies	
8.6.2	Pigs	
8.6.3	Sheep	
	ROCESSING STUDIES	
	RESIDUES REFERENCES	
	S APPENDIX 1	
	S APPENDIX 2	
RESIDUE	S APPENDIX 3	99
RESIDUE	S APPENDIX 4	101

#### GLOSSARY OF TERMS AND ABBREVIATIONS

AAAA Australian Aerial Agricultural Association

ACAHS Australian Centre for Agricultural Health & Safety

ADI Acceptable Daily Intake

a.i. Active Ingredient

ai/100L active ingredient per 100 Litres aPAD Acute Population Adjusted Dose

ATV All Terrain Vehicles
BCF Bioconcentration Factor

bw Body weight

CAS Chemical Abstracts Service
CNS Central Nervous System
CP Pressure control nozzles

cPAD Chronic Population Adjusted Dose C-PAS Centre for Pesticide Application Safety

CRDC Cotton Research & Development Corporation

CRP Chemical Review Program
CXL Codex Maximum Residue Level

d Days

DFR Dislodgeable Foliar Residue EC Emulsifiable concentrate

ECRP Existing Chemical Review Program (APVMA)

EPA US Environmental Protection Agency

ER Oestrogen Receptor

FIFRA Federal Insecticide, Fungicide, and Rodenticide Act

FFDCA Federal Food, Drug, and Cosmetic Act

FOB Functional Observation Battery FQPA Food Quality Protection Act

g Gram

g ai/ha grams of active ingredient per hectare

GAP Good Agricultural Practice
HPA Hypothalamic-pituitary-adrenal
HPG Hypothalamic-pituitary-gonadal
HPT Hypothalamic-pituitary-thyroid

HRs Highest Residues

IPM Integrated Pest Management

kg Kilogram L Litre

LOAEL Lowest Observed Adverse Effect Level

LOD Limit of Detection

LOEL Lowest Observable Effect Level

MFL Maximum Feed Level

mg Milligram

mg/kg milligrams per kilogram

mL Millilitre

M/L	Mixing/loading
M/L/A/C	Mixing/loading/application/cleaning
MOE	Margins of Exposure
MRL	Maximum Residue Limits
NEDI	National Estimated Dietary Intake
NESTI	National Estimated Short Term Intake
NOAEL	No Observed Adverse Effect Level
NOEC	No Observable Effect Concentration
NOEL	No Observable Effect Level
NOHSC	National Occupational Health & Safety commission
OCS	Office of Chemical Safety
OHS	Occupational Health and Safety
OP	Organophosphorus compound
OPP	EPA Office of Pesticide Programs
OPPTS	EPA Office of Prevention, Pesticides and Toxic Substances
PAD	Population Adjusted Dose
PADI	Provisional Acceptable Daily Intake
PF	Processing Factor
ppb	Parts Per Billion
PPE	Personal Protective Equipment
ppm	parts per million
PVC	Polyvinyl chloride
RBC	Red Blood Cell
RED	Reregistration Eligibility Decision
REI	Restricted Entry Interval
RfD	Reference Dose
RLEM	Red Legged Earth Mite
SHBG	Sex hormone–binding globulin
<b>STMRs</b>	Supervised Trial Median Residues
SUSDP	Standards for the Uniform Scheduling of Drugs and Poisons
TC	Transfer Coefficient
TGA	Therapeutic Goods Administration
TGAC	Technical Grade Active Constituent
ULV	Ultra-low Volume
US EPA	United States Environment Protection Authority
WHP	With Holding Period
	<del>-</del>

#### 8 RESIDUES ASSESSMENT TECHNICAL REPORT

#### 8.1 METABOLISM STUDIES

#### 8.1.1 Hen Studies

Distribution, elimination and the nature of the metabolite residues in the eggs and edible tissues of the laying hen. C.M.M. Reynolds, 26 March 1996. AgrEvo U.K. Limited, Study Number TOX/94306.

Six laying hens were dosed by capsule daily for 12 consecutive days, at a mean dose level of 0.7 mg/kg bodyweight (dose range 0.65-0.72 mg/kg bw/day). The dose was composed of  $^{14}$ C endosulfan  $^{1}$  ( $\alpha$  – endosulfan:  $\beta$  – endosulfan 68:32) absorbed onto ground grain contained in a gelatin capsule. This was equivalent to 11 ppm in the feed (range 9.9-12.5 ppm), based on mean individual bodyweights and feed intakes over the 12 day dosing period.

Excreta were collected at 24 hour intervals over the 12 day period and combined with cage washes for quantification of radioactivity. Eggs were collected twice a day over 24 hour intervals; yolks and whites were separated for quantification. At 24 hours after administration of the final dose, the hens were slaughtered and samples of skin, muscle (breast and thigh), liver, subcutaneous and abdominal fat and unlaid eggs were collected for analysis.

Initial radioactivity was measured using LSC; TLC and HPLC were used to separate and identify various metabolites.

Elimination of radioactivity via excreta (and cage washes), accounted for a mean recovery of up to 50% of the administered dose within 24 hours after administration. By day 4, the recovered radioactivity accounted for up to 93% of the administered dose. The mean daily recovery of the administered dose was 86% in excreta over all hens during the 12 day study period.

Total radioactive residues (TRR, mg/kg endosulfan equivalents) in tissues and eggs are shown in Table 2 below.

Table 2: TRR in hen tissues and undeveloped eggs following dosing of <sup>14</sup>C-endosulfan at 0.7 mg/kg bodyweight/day (Reynolds, 1996).

Tissue		TRR (mg/kg endosulfan equivalents)							
	Hen 1	Hen 2	Hen 3	Hen 4	Hen 5	Hen 6	Mean ± SD		
Liver	0.444	0.423	0.334	0.393	0.533	0.671	0.466 ±0.119		
Muscle	0.031	0.025	0.027	0.027	0.024	0.036	0.028 ±0.004		
Skin	0.600	0.593	0.519	0.786	0.808	0.825	0.689 ±0.133		
Fat (Subcutaneous)	0.695	0.819	0.935	0.799	0.918	1.083	$0.875 \pm 0.134$		
Fat (Abdominal)	0.906	0.876	1.103	0.952	0.970	1.035	0.974 ±0.084		
Undeveloped eggs	0.713	0.636	0.995	0.806	0.610	0.847	$0.768 \pm 0.145$		

<sup>&</sup>lt;sup>1</sup> Labelled in the methylene bridge and in the 6,7,8,9 carbon positions of the cyclopentane ring.

The data show that the highest levels of radioactivity are present in skin, subcutaneous and abdominal fats and undeveloped eggs.

Total radioactivity in whole eggs was determined by measuring levels in egg yolks and whites separately. TRR in egg yolks and whites reached a plateau by day 10 of dosing, with levels in yolks increasing rapidly within 48 hours of administration of dosing. There was a noticeable difference in the magnitude of radioactivity in egg yolks and whites, with levels in egg yolks being up to  $100 \times$  those found in egg whites. Maximum radioactive residues were 1.08 and 0.028 mg/kg endosulfan equivalents in yolks and whites, respectively.

Extraction of radioactivity in tissues and eggs is shown in Table 3.

Table 3: Characterisation of radioactivity in hen tissues and eggs following dosing of <sup>14</sup>C endosulfan at 0.7 mg/kg bodyweight/day (Reynolds, 1996).

		%TRR in Sample (mg/kg endosulfan equivalents)							
	Muscle	Fat (A)	Fat (S)	Skin	Liver	Egg Yolk			
E-SO <sub>4</sub>	35.83 (0.01)	65.45 (0.638)	61.10 (0.535)	51.32 (0.354)	45.62 (0.132)	46.38 (0.396)			
E-lactone	3.51 (<0.001)	4.99 (0.049)	5.03 (0.044)	4.50 (0.031)	6.27 (0.018)	1.82 (0.016)			
$\alpha - E$	6.52 (0.002)	16.77 (0.163)	16.15 (0.141)	11.66 (0.080)	0.99 (0.003)	4.74 (0.04)			
β – E	4.40 (0.001)	7.80 (0.076)	8.90 (0.078)	4.79 (0.033)	1.60 (0.005)	1.32 (0.011)			
E-diol	_	_	_	_	4.25 (0.012)	_			
Extractable	64.40	97.04	98.21	94.43	91.41	92.17			
Total Identified	54.97	95.01	95.89	89.82	58.73	80.47			
Polar	4.71	0.56	4.71	17.55	23.27	20.18			
Extracted activity	8.15	_	0.51	0.52		0.89			
Unextractable	35.60	2.96	1.79	5.57	8.58	7.83			
Losses	1.39	1.19	1.81	3.86	8.85	7.61			
Total	100	99.72	100	99.77	99.43	96.8			

Fat (A) = Abdominal fat; Fat (S) = Subcutaneous fat.

E- $SO_4$  = endosulfan sulfate; E-lactone = endosulfan lactone;  $\alpha - E$  = alpha-endosulfan;  $\beta - E$  = beta-endosulfan; E-diol = endosulfan diol.

The results show that endosulfan sulfate is the major metabolite present in hen tissues and eggs, following oral dosing with endosulfan for 12 consecutive days. Concentrations ranged from 35 to 65% of the total administered  $^{14}$ C, or 0.01 to 0.64 mg/kg equivalents. The highest concentrations of endosulfan sulfate,  $\alpha$ – and  $\beta$ – endosulfan are found in abdominal and subcutaneous fats and skin. This indicates that endosulfan and its major metabolite endosulfan sulfate are preferentially found in the fat of dosed hens, compared to muscle, liver and eggs.

Unchanged endosulfan is also present at lower proportions, with varying amounts of the  $\alpha$ - and  $\beta$ - stereoisomers. Endosulfan lactone is present in all samples, however endosulfan diol is only found in liver.

Extraction of excreta and cage washings indicated that the majority of the extractable radioactivity was polar in nature, with low proportions of  $\alpha$ – and  $\beta$ – endosulfan, endosulfan sulfate and endosulfan diol being present. Further characterisation of the radioactivity in egg whites was not undertaken due to the very low levels present in the samples.

## 8.1.2 Lactating Cow Studies

Distribution, elimination and the nature of the metabolite residues in the milk and edible tissues of a lactating cow. J.M. Leah and C.M.M. Reynolds, 21 May 1996. AgrEvo U.K. Limited, Study Numbers TOX/94308 and TOX/94308A.

A single fresian cow was dosed orally by capsule for 5 consecutive days at 0.64 mg/kg bodyweight. This dose is equivalent to 21.6 ppm in the feed (450 kg animal with a mean daily feed intake of 13.35 kg/day).

Urine and faeces were collected at 24 hour intervals during the period of the study. The animal was milked twice a day, and the separate milkings were analysed individually. At 22 hours after the final dose, the animal was slaughtered and samples of liver, kidney, heart, lungs, muscle (hindquarter and psoas), fat (renal, omental and subcutaneous), rumen, bile and abomasal fluid were taken for analysis.

Radioactivity was measured using LSC; isolation and characterisation of metabolites was achieved using TLC and HPLC following solvent extraction and enzyme hydrolysis.

Elimination of the dose was rapid, with 42% of the dose being excreted within the first 2 days of the study. Recovered radioactivity in urine and faeces reached a maximum of up to 50.4% of the administered dose on day 3 of the study, with a mean daily recovery of 43.6% in excreta.

Radioactivity in milk reached peak levels in the sample taken approximately 6 hours after administration, then declined until the next dosing. Maximum radioactivity (0.171 mg/kg endosulfan equivalents) was observed in milk at 102 hours after the first dose (6 hours after the day 4 dose).

Total radioactive residues in various tissues are shown in Table 4.

Table 4: Total <sup>14</sup>C in tissues following oral dosing at 0.64 mg/kg bodyweight/day for 5 days (Leah and Reynolds, 1996).

Sample	TRR (mg/kg endosulfan equivalents)
Liver	3.572
Kidney	0.785
Muscle (hindquarter)	0.031
Muscle (psoas)	0.052
Fat (omental)	1.278
Fat (renal)	0.840
Fat (subcutaneous)	0.305
Heart	0.161
Lungs	0.673
Bile	1.844
Rumen contents	6.328
Abomasal contents	0.199

The results show that TRR are highest in rumen contents, liver, bile and fat. Isolation and characterisation of the radioactivity in edible tissues and milk is shown in Table 5.

Table 5: Characterisation of metabolites in cattle tissue following oral dosing at 0.64 mg/kg bodyweight/day for 5 days (Leah and Reynolds, 1996).

		% <sup>14</sup> C in Tissue (mg/kg endosulfan equivalents)							
% <sup>14</sup> C	Liver	Kidney	Heart	Muscle (psoas)	Fat (omental)	Fat (renal)	Fat (sub- cutaneous)	Milk (54 hrs)	
Extracted	37.98	43.49	55.24	69.24	95.70	96.46	81.34	95.32	
Released	53.87	52.24	33.18	13.86					
Identified	87.80	86.03	49.22	65.87	83.64	83.83	67.79	88.57	
α – E	2.72		2.17	15.14					

β – E		3.08						
E-SO <sub>4</sub>	27.16	12.58	14.21	50.73	82.08	83.83	64.09	88.57
	(0.969)	(0.099)	(0.023)	(0.026)	(1.049)	(0.704)	(0.195)	(0.130)
E-lactone	9.16	6.86	29.53					
E-diol	6.48	4.96						
HE-ether		4.77						
E-ether	6.76	2.13						
Polar	35.49	51.65	3.32		1.56			
Below limits of	0	0	39.19	15.65	0.32	1.86	0	3.22
analysis								
TRR (mg/kg eq.)	3.572	0.785	0.161	0.052	1.278	0.840	0.305	0.147

Released by enzyme hydrolysis.

The data show that of the extractable radioactivity, the predominant metabolite present in all tissues and milk is endosulfan sulfate. The highest concentrations are present in fats (renal, omental and subcutaneous) and milk (54 hours).

Although the highest levels of total radioactivity in edible tissues were present in liver, further characterisation shows that a large proportion of that radioactivity is composed of polar compounds, which were not identified as major metabolites or transformation products. In addition, endosulfan lactone and endosulfan diol were present in both liver and kidney at levels <10% of the TRR.

# **8.1.3** Sheep

Metabolism of Endosulfan in Milk Sheep, S.G. Gorbach, O.E. Christ, H-M. Kellner, G. Kloss and E. Borner, *J. Agr. Food Chem.*, (1968), <u>16</u>, 950 – 953.

In this published study, two fresian milk sheep were treated with a single oral dose of <sup>14</sup>C endosulfan (labelled in the methylene bridge carbon) at dose levels of 0.26 and 0.30 mg/kg bodyweight. Urine and faeces were collected at 24 hours intervals for 22 days. Similarly, the

sheep were milked twice a day for 22 days and samples analysed for radioactivity at regular intervals after administration of the dose. Total radioactivity in the blood was determined at 2, 4, 6, 8, 12, 24 and 48 hours after dosing, then daily up to day 21. At 40 days after administration, one of the animals was slaughtered and tissues were collected for radioanalysis.

Elimination of radioactivity was rapid with maximum concentrations up to 34% of the administered dose recovered by day 2 or 3 after dosing. After 22 days, up to 91% and 93% of the administered dose had been excreted in sheep 1 and 2 respectively.

In milk, a maximum of 0.4% of the administered dose was eliminated in the first two days after administration. The highest concentration was 0.15 mg/kg endosulfan equivalents (sheep 2 at 24 hours after dosing). By 7 days, the total radioactivity had fallen to 0.015 mg/kg endosulfan equivalents. Radioanalysis of skim milk and cream showed that 88% of the total radioactivity was present in the cream. Results for whole milk are shown in Table 6.

Table 6: TTR in sheep milk, following a single oral dose of 0.26 or 0.30 mg/kg bodyweight (Gorbach et al, 1968).

Days after dosing	Sheep 1		She	ep 2
	Vol. Of sample, mls	% TRR	Vol. Of sample, mls	% TRR
1	560	0.35	430	0.46
2	460	0.33	500	0.45
3	290	0.13	460	0.44
4 – 7	200	0.037	200	0.35
8 – 12	490	0.019	900	0.088
13 – 17	200	0.002	100	0.036
Total		0.868		1.824

Characterisation of the radioactivity in cream by TLC and GC indicated that endosulfan sulfate accounted for almost all of the <sup>14</sup>C in that fraction. No further characterisation of the radioactivity in skim milk was conducted.

Very little information on the levels of radioactivity in tissues was reported.

#### 8.2 RESIDUE DEFINITION

In metabolism studies in hens, sheep and cattle, the majority of the radioactive residues in tissues, eggs and milk are composed primarily of endosulfan sulfate with minor amounts of  $\alpha$ -endosulfan and  $\beta$ -endosulfan and endosulfan lactone.

Plant metabolism studies in apples, tomatoes and cucumber were evaluated and reported in the 1998 NRA review of endosulfan. Therein it was reported that endosulfan sulfate was the predominant component of the total radioactivity in apple leaves and cucumbers, however  $\alpha$ - and  $\beta$ -endosulfan comprise the majority of the radioactivity in apples (fruit) and tomatoes. In additional studies reported in the 1989 JMPR evaluation of endosulfan, endosulfan sulfate was found to be the main metabolite in plants.

The studies confirm that the current residue definition of endosulfan (sum of  $\alpha$ - and  $\beta$ - endosulfan and endosulfan sulphate) is appropriate for the determination of residues in crops and animal tissues, milk and eggs. The definition should be expressed as 'sum of  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate, expressed as endosulfan'.

#### 8.3 ANALYTICAL METHODOLOGY

# 8.3.1 Crop matrices

A method for the determination for endosulfan residues in a number of crops and crop matrices, was provided by the State Chemistry Laboratory (Victoria) with each of the residue trials. Recoveries and limits of quantitation are reported with each trial. A brief description of the method is given below.

Homogenised sample is mixed with acetone:hexane 20:80 and shaken on a mechanical shaker for an hour and left to stand overnight. An aliquot of the supernatant is transferred to a Kuderna-Danish apparatus and evaporated over a water bath with inversion into hexane. The extract is made up to a volume of 5ml. An aliquot is then loaded onto a Florisil column and eluted with  $CH_3CN$ :hexane: $CH_2Cl_2$  (1.5:48.5:50 v/v/v) and collected into a Kuderna-Danish flask with a concentrator tube. The solvent is evaporated with inversion into hexane and taken down to a volume of 2 ml. The remaining residue is quantified using GC/ECD with an external standard. Typical recoveries ranged from 0.005 to 0.5 mg/kg, with most recoveries being conducted with fortification at 0.02 mg/kg.

## 8.3.2 Animal tissues and milk

Validation of the Analytical Method for the Determination of Endosulfan ( $\alpha$ ,  $\beta$  and sulfate) in Animal Tissues, Egg (White and Yolk) and dairy Matrices based upon FDA Pesticide Analytical Manual, Volume I Multi-Residue Methodology. AgrEvo Project ID BJ-95R-14, Laboratory Project ID 95-0061, 13 November 1997.

Samples that were free of endosulfan were purchased and fortified with endosulfan at levels of 0.025, 0.25 and 0.70ppm (( $\alpha$ ,  $\beta$  and sulfate). In addition, samples from <sup>14</sup>C metabolism studies in cattle and hens<sup>2</sup> were also included for method validation. These were received as homogenised samples.

Fortified samples were homogenised and blended thoroughly prior to subsampling for analysis. Labelled poultry and milk samples required additional processing prior to subsampling. <sup>14</sup>C milk required dilution with control milk and similarly, <sup>14</sup>C poultry liver required dilution with control matrix.

Tissue samples were blended three times with 75:25 petroleum ether:acetone,  $Na_2SO_4$  and celite then filtered. The filtrate was evaporated to dryness and dissolved in petroleum ether and partitioned with  $CH_3CN$  three times. The  $CH_3CN$  was combined with  $H_2O$  and saturated NaCl and partitioned with petroleum ether. The aqueous phase was partitioned again with petroleum ether and then discarded. The petroleum ether phases were combined, washed with  $H_2O$  and dried over  $Na_2SO_4$ . The ether was evaporated to dryness and dissolved in hexane and loaded onto a Florisil SPE column. The column was washed with hexane and eluted with 50:48.5:1.5  $CH_2Cl_2$ :hexane: $CH_3CN$ . The eluate was evaporated to dryness, re-dissolved in toluene and analysed using GC with ECD (gas chromatography with electron capture detector).

To milk, EtOH and potassium oxalate were added and the mixture was shaken for 30 seconds. Ether was added and the mixture shaken again for 30 seconds. Petroluem ether was added and the mixture shaken again. The sample was centrifuged and the top organic phase was removed and combined with saturated NaCl solution. Centrifugation and extraction was repeated twice and the combined organic phases were partitioned with saturated NaCl. The aqueous phase was discarded and the remaining organic phase was washed with H<sub>2</sub>O. The H<sub>2</sub>O was discarded and the remaining organic phase was dried over Na<sub>2</sub>SO<sub>4</sub>. The sample was evaporated to dryness and dissolved in petroleum ether and partitioned four times with CH<sub>3</sub>CN. The CH<sub>3</sub>CN was combined with saturated NaCl and partitioned with petroleum ether twice. The petroleum ether phases were combined and washed with H<sub>2</sub>O and dried over Na<sub>2</sub>SO<sub>4</sub>. The sample was evaporated to dryness, dissolved in hexane and loaded onto a Florisil SPE column. The remaining clean-up steps are similar to those for tissues. Residues in milk were detected using GC with ECD.

The extraction steps for egg whites were similar to those described for milk. With egg yolks, the petroleum ether filtrate was passed through alumina-N prior to partitioning with petroleum ether and CH<sub>3</sub>CN.

The <sup>14</sup>C samples were quantified using GC/MS with single ion monitoring.

The limits of quantitation were reported as 0.01 mg/kg in milk, muscle, liver and kidney and 0.05 mg/kg in fat (section 3.2.1 animal transfer study). Method validation results are given in Table 7.

Table 7: Validated method recoveries in animal tissues, eggs and milk

Matrix	Mean Recover	ies in Fortified San	nples (% ± SD)	Mean Recoverie	s in <sup>14</sup> C Labelled S	amples (% ± SD)
	α	β	$SO_4$	α	β	SO <sub>4</sub>
Beef muscle	83 ± 3	82 ± 3	87 ±3	77 ± 7	82 ± 6	81 ± 6
Beef liver	$87 \pm 8$	$85 \pm 8$	$88 \pm 6$	$76 \pm 6$	$83 \pm 2$	$89 \pm 4$

<sup>&</sup>lt;sup>2</sup> AgrEvo TOX94308 cow study and TOX94306 hen study.

-

					1	
Beef fat	$84 \pm 7$	$87 \pm 5$	$94 \pm 3$			
Milk	$84 \pm 10$	$86 \pm 11$	$85 \pm 11$	$77 \pm 2$	$81 \pm 3$	$84 \pm 5$
Egg Whites	$78 \pm 5$	$86 \pm 7$	$85 \pm 9$	$88 \pm 8$	$88 \pm 5$	$88 \pm 3$
Egg Yolks	$80 \pm 6$	$81 \pm 6$	$86 \pm 6$	$76 \pm 3$	$76 \pm 4$	$76 \pm 4$
Beef heart				$80 \pm 8$	$86 \pm 3$	$81 \pm 4$
Beef kidney				$86 \pm 7$	$86 \pm 8$	$87 \pm 6$
Renal fat				$63 \pm 23$	94 ± 7	$89 \pm 10$
Omental fat				$72 \pm 8$	$75 \pm 5$	$73 \pm 5$
Poultry liver				$98 \pm 18$	$96 \pm 14$	$100 \pm 11$
Poultry muscle				$94 \pm 4$	91 ± 2	$90 \pm 3$
Poultry fat				$52 \pm 26$	$66 \pm 19$	$80 \pm 9$

The results from the GC/ECD analysis of the <sup>14</sup>C samples were in agreement with the levels found in the metabolism studies, except for poultry liver where only an average of 37% of the expected result was found. In conclusion, the validated method detected all incurred residues of endosulfan in tissues, milk and eggs (white and yolk), with the exception of poultry liver.

#### 8.4 STORAGE STABILITY

## **8.4.1** Storage stability in crop matrices

Determination of Endosulfan Residue Stability in Lemons, Leafy Lettuce and Beetroot. State Chemistry Laboratory Report No. 0111227, 28 November 2001.

Samples of lemons, leafy lettuce and beetroot from trials sponsored by Nufarm were repeat analysed for endosulfan residues following storage for up to 10 months. Samples were stored at -20 °C until preparation, initial analysis and after analysis. The results are shown in Table 8.

Sample Storage Interval Endosulfan Residues (mg/kg) SO<sub>4</sub> Total Lemons Initial analysis 0.10 0.14 0.012 0.25 0.0490.10 0.008 0.16 0.052 0.12 0.007 0.17 5 months 0.15 0.010 0.27 0.11 0.059 0.13 0.006 0.20 0.052 0.12 0.007 0.18 Lettuce Initial analysis 1.6 1.5 0.31 3.4 0.34 0.33 0.33 1.0 0.15 0.14 0.19 0.48 12 months 1.5 1.2 0.28 3.0 1.2 0.42 0.39 0.36 0.12 0.19 0.45 0.14 Beetroot Initial analysis 0.18 0.11 0.10 0.39 0.32 0.10 0.11 0.11 0.062 0.063 0.075 0.20 10 months 0.53 0.98 0.30 0.15 0.084 0.082 0.086 0.25 0.062 0.085 0.20

Table 8: Storage stability of endosulfan residues in lemons, leafy lettuce and beetroot.

The data show that incurred endosulfan residues do not significantly degrade upon freezer storage. The residues found in the stored samples

ranged 78 - 125% of the levels found in the

initial analyses. One beetroot sample however gave an anomalous result and this was explained by the laboratory as being due to sample inhomogeneity during sub-sampling. Samples in the horticultural trials were stored up to 12 months in most cases. Therefore the above data demonstrate that the residues found at the time of analysis reflect the likely levels that were present at the time of harvest or sampling.

## 8.4.2 Stability of endosulfan in animal tissues and milk

Freezer Storage Stability of Endosulfan (alpha, beta and Sulfate) on Animal Tissue and Dairy Matrices, D.A. Winkler. Bayer CropScience Report No. BJ96R006, Laboratory Project ID 96-0046, 22 June 1998.

The storage stability of endosulfan in beef muscle, beef liver, egg whites, egg yolks and milk was investigated for 3, 6, 9 and 12 months of freezer storage. Endosulfan free samples were purchased and control samples were fortified at 0.25 ppm with endosulfan ( $\alpha$ -,  $\beta$ - and sulfate) and stored at < -10 °C for 12 months. Unfortified samples were also stored for 12 months under the same conditions and at each storage interval, one unfortified control and two freshly fortified controls were analysed concurrently with the stored fortified samples to determine method recoveries. The results are shown in Table 9.

Table 9: Storage stability of endosulfan residues in animal tissues, eggs and milk

Matrix	Storage Interval	Recoveries in Stored Samples (%)		%)	Recoveries in I	reshly Fortified	Samples (%)
		α	β	sulfate	α	β	sulfate
Beef muscle	0				77, 81, 82, 81	81, 84, 85, 85	86, 86, 86, 84
	3 months	86, 82	88, 82	89, 82	85, 83	88, 86	90, 88
	6 months	87, 82	86, 79	90, 82	84, 81	81, 78	86, 81
	9 months	77, 76	81, 81	84, 84	79, 74	82, 78	86, 83
	12 months	82, 95	87, 101	86, 101	90, 84	92, 86	90, 85
Beef liver	0				87, 88, 93, 87	89, 90, 96, 83	87, 87, 93, 82
	3 months	76, 75	75, 74	75, 73	73, 73	75, 73	75, 72
	6 months	70, 69	71, 73	69, 72	59*, 72	70, 72	66, 73
	9 months	59, 57	75, 74	83, 82	59*, 63	74, 78	80, 83
	12 months	82, 81	94, 90	118, 111	69, 75	73, 77	73, 91
Egg whites	0				78, 78, 80, 80	85, 85, 89, 90	87, 87, 88, 88
	3 months	76, 79	78, 81	83, 84	84, 83	88,. 88	87, 87
	6 months	66, 66	71, 70	84, 81	72, 76	77, 78	81, 85
	9 months	67, 68	74, 74	80, 82	72, 75	74, 75	80, 82
	12 months	61, 70	62, 70	78, 83	75, 69	64, 73	69, 78
Egg yolks	0	82, 74	84, 77	64, 74	72, 71, 71, 66	76, 81, 84, 81	81, 84, 87, 82
	3 months	78, 78	83, 84	83, 84	75, 81	78, 87	80, 78
	6 months	72, 83	73, 83	67, 75	78, 71	82, 78	83, 85
	9 months	72, 63	64, 50	76, 58	77, 84	76, 82	70, 71
	12 months	83, 73	83, 81	80, 74	100, 95	104, 93	54*, 52*
Milk	0				73, 85, 92, 94	84, 88, 96,	76, 81, 92, 90
						100	
	3 months	83, 73	83, 81	80, 74	79, 75	74, 75	74, 77
	6 months	86, 83	87, 86	86, 85	85, 85	88, 86	86, 86
	9 months	75, 74	77, 76	76, 75	73, 79	72, 80	75, 82
	12 months	98, 104	103, 114	104, 115	89, 97	92, 100	93, 104

<sup>\*</sup> Outliers

The data show that endosulfan residues in the matrices investigated are stable for up to 12 months of freezer storage. Some data points were found below 60%, however these were reported as outliers as values at similar time points were within acceptable limits of 70 to 100%.

#### 8.5 Crop Residue studies

The residue trials are reviewed in the crop sequence of the *Codex Alimentarius Classification of Foods* and *Animal Feeds*. Although the trials included control plots, residues in the untreated samples are only reported when they exceeded the limit of quantitation (LOQ). The LOQ is typically 0.02 mg/kg for each component of the residue definition in most crops, unless otherwise indicated at the bottom of each Table.

In the tabulation of the residue data, <LOQ is reported in numerical form, i.e. <0.02 mg/kg. The calculation of total endosulfan residues from the components of the residue definition is outlined below:

α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>	Total (mg/kg)
< 0.02	< 0.02	< 0.02	< 0.02
< 0.02	< 0.02	0.05	0.05
< 0.02	0.04	0.05	0.09
0.03	0.04	0.05	0.11

Where endosulfan sulfate residues are present at levels above LOQ, however both  $\alpha$ - and  $\beta$ -endosulfan are present at <LOQ, the total residue will comprise the levels of endosulfan sulfate only, as plant and animal metabolism studies indicate that endosulfan sulfate is the predominant component of the total residue. Where the residues of all individual components are <LOQ, then the total endosulfan residue will be equivalent to the LOQ of endosulfan sulfate.

# 8.5.1 Citrus fruit (oranges, mandarins, lemons)

Residues at Harvest in Oranges. European Union, Southern Zone 1998, E.H.-J. Klein. Hoechst Schering AgrEvo Germany. Report No. ER 98 ECS 740, 21 April 1999.

Four trials to determine endosulfan residues in oranges were conducted in Spain and Italy during 1998. Orange trees were treated with 2 applications of an endosulfan EC product at a rate of 1123 g ai/ha. Trees were sprayed when the fruit were about 50% of the final size and at the beginning of fruit colouring (BBCH 75 – 81), at an interval of 14 days. Sprays were applied using high pressure sprayers (Spain) or knapsack mistblowers (Italy). Plot sizes were  $160\text{m}^2$  and  $250\text{ m}^2$  in Spain; plot sizes were not recorded for the Italian trials. Samples of fruit were taken at 0, 21 and 23 days after treatment and stored frozen until analysis. Analyses were completed within 5 months of sample collection; residues were determined in pulp and peel and reported on a whole fruit basis. The results are given in Table 10.

Table 10: Endosulfan residues in oranges from trials conducted in Spain and Italy (1998).

Trial site, Year,	Application	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Sollana, Spain 1998	1123	2 (14)	0	Whole fruit	0.21	0.20	0.08	0.49
(Navelina)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.85	0.83	0.30	1.98
			23	Whole fruit	0.02	0.03	0.11	0.16
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.07	0.11	0.43	0.61
Quart de Poblet,	1123	2 (14)	0	Whole fruit	0.34	0.24	0.09	0.67
Spain 1998	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
(Navelina)				Peel	1.30	0.93	0.34	2.57
			23	Whole fruit	0.04	0.05	0.19	0.28
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.11	0.16	0.71	0.98
Pisticci, Italy 1998	1123	2 (14)	0	Whole fruit	0.21	0.18	0.08	0.47
(Washington Navel)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.85	0.72	0.31	1.88
			21	Whole fruit	0.03	0.04	0.06	0.13
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.07	0.13	0.19	0.39
Pisticci, Italy 1998	1123	2 (14)	0	Whole fruit	0.21	0.18	0.05	0.44
(Tarocco)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.78	0.66	0.18	1.62
			21	Whole fruit	0.03	0.06	0.08	0.17
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
				Peel	0.09	0.20	0.27	0.56

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in orange pulp and peel are shown in Table 11.

Table 11: Recoveries of endosulfan in fortified orange peel and pulp

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Orange peel	0.02	90	92	107			
	1	100, 104	116, 116	113, 118			
Orange pulp	0.02	84, 99	93, 93	97, 99			

Mean recoveries were 98, 108 and 113% for  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate, respectively in orange peel and 91, 93 and 98%, respectively in orange pulp.

Decline of Residues in Orange Fruits and Transfer of Residues into Juice. European Union, Southern Zone 1997, E.H.-J. Klein. Hoechst Schering AgrEvo Germany. Report No. ER 97 ECS 740, 15 March 1999.

At five trial sites in Spain, Greece and Italy, orange trees were treated with 2 applications of an endosulfan EC product at a rate of 1123 g ai/ha. In addition, a higher rate of 3368 g ai/ha was applied at 3 of the sites (Spain and Italy) for collection of fruit for processing. Trees were sprayed when the fruit were about 80% of the final size and at the beginning of fruit colouring (BBCH 78 – 81), at an interval of 14 days. Applications were made using high pressure sprayers (Spain and Greece) or motorised knapsack sprayers (Italy). Plot sizes were 100 and  $100\text{m}^2$  in Spain,  $121\text{m}^2$  in Greece and  $100\text{m}^2$  in Italy. Samples of fruit were taken at regular intervals after treatment (0, 7, 14, 21 and 28 days) stored frozen until analysis. Analyses were completed within 10-11 months of sample collection; residues were determined in pulp and peel and reported on a whole fruit basis. For processed samples residues in juice and pomace/peel were also determined. Results are given in Table 12.

Table 12: Endosulfan residues in oranges from trials conducted in Spain, Italy and Greece (1997 – 1998).

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	SO <sub>4</sub>	Total
Moncada, Spain	1123	2 (14)	0	Peel	1.1	0.82	0.58	2.5
1997 (Navel)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			7	Peel	0.42	0.53	0.83	1.78
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			14	Peel	0.22	0.3	0.64	1.16
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			21	Peel	0.22	0.32	0.69	1.23
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			28	Peel	0.12	0.19	0.63	0.94
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			0	Whole fruit	0.26	0.20	0.14	0.60
			7		0.08	0.10	0.15	0.33
			14		0.05	0.07	0.14	0.26
			21		0.05	0.07	0.14	0.26
			28		0.03	0.05	0.13	0.21
	3368	2 (14)	21	Whole fruit	0.09	0.14	0.25	0.48
	(112.3 g ai/hL)			Juice, raw	< 0.02	< 0.02	< 0.02	< 0.02
				Pomace/peel	0.14	0.19	0.51	0.84
Quart de Poblet,	1123	2 (14)	0	Peel	1.1	0.72	0.54	2.36
Spain 1997	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02
(Navelina)			7	Peel	0.32	0.45	0.75	1.52
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			13	Peel	0.27	0.36	0.72	1.35
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02
			21	Peel	0.17	0.24	0.57	0.98

Pulp
Record Page
Real Content
Record Nuovo   Lay 1998   Control of the control
Record   Part
S. Teodoro Nuovo   I123   S. Teodoro Nuovo   I124   S. Teodoro Nuovo   I125   S. Teodoro Nuovo   I125   S. Teodoro Nuovo   I126   S. Teodoro Nuovo   I127   S. Teodoro Nuovo   I128   S. Teodoro Nuovo   I129   S. Teodoro Nuovo   I120   S. Teodoro Nuovo
S. Teodoro Nuovo   1123   S. Teodoro Nuovo   Inly 1998   C. S. Teodoro Nuovo   Inly 1098   C. S. Teodoro Nuovo   Inly 1998   C. S. Teodoro Nuovo   C. Teodoro
S. Teodoro Nuovo   I123   (37.5 g ai/hL)   (37.5 g ai/h
S. Teodoro Nuovo   I123   (37.5 g ai/hL)   (37.5 g ai/h
Care
Teron, Greece 1997 (Santa Lutsiana)
Froon, Greece 1997 (Santa Lutsiana)   1123 (37.5 g ai/hL)   7   Peel
Santa Lutsiana  (37.5 g ai/hL)
Peel
Pulp
Pel
Name
Pecl
Pulp
S. Teodoro Nuovo   I123
Note
S. Teodoro Nuovo Italy 1998 (Naveline)
S. Teodoro Nuovo (Naveline)
S. Teodoro Nuovo   I123
S. Teodoro Nuovo   I123   Control
S. Teodoro Nuovo   1123
S. Teodoro Nuovo   1123
S. Teodoro Nuovo Italy 1998 (37.5 g ai/hL)   123 (37.5 g ai/hL)   124
Rady 1998 (Naveline)
(Naveline)  (Navel
Pulp
14
Pulp
19
Pulp
Peel
Pulp
Note that the property is a second of the property is a
Tendero Nuovo   Titaly 1997 (Washington Navel)   Titaly 1997 (Washington Navel)   Titaly 1997   Ti
14
S. Teodoro Nuovo   I123 (37.5 g ai/hL)   Yellow   Yello
S. Teodoro Nuovo   1123 (37.5 g ai/hL)   Yellow   2 (14)   19   Whole fruit   Unit
S. Teodoro Nuovo   S. Teodoro Nuovo   I123 (37.5 g ai/hL)   S. Teodoro Navel)   S. Teodoro Nuovo   I124 (2 (14)   19   Whole fruit   Juice, raw   Vol.02 (2 (0.02)   Vol.02
Comparison of the comparison
S. Teodoro Nuovo   S. Teodoro Nuovo   I123   (37.5 g ai/hL)
S. Teodoro Nuovo   1123   2 (14)   0   Peel   0.42   0.38   0.11   0.91     Italy 1997 (37.5 g ai/hL)   7   Peel   0.14   0.27   0.13   0.54     Pulp   <0.02   <0.02   <0.02   <0.02     Pulp   <0.02   <0.02   <0.02     Pulp   <0.02   <0.02   <0.02     Pulp   <0.02   <0.02   <0.02     Peel   0.11   0.23   0.14   0.48     Pulp   <0.02   <0.02   <0.02   <0.02     Peel   0.08   0.15   0.17   0.40     Pulp   <0.02   <0.02   <0.02   <0.02     Peel   0.08   0.15   0.17   0.40     Pulp   <0.02   <0.02   <0.02   <0.02     Peel   0.08   0.15   0.17   0.40     Pulp   <0.02   <0.02   <0.02   <0.02     Peel   0.08   0.15   0.17   0.40     Pulp   <0.05   0.08   0.11   0.24
S. Teodoro Nuovo Italy 1997 (Washington Navel)    Variable
Italy 1997 (Washington Navel)         (37.5 g ai/hL)         Pulp (30.02 (0.02
(Washington Navel)     7     Peel Pulp     0.14 0.27 0.02 0.02 0.02 0.02 0.02 0.02 0.02
Pulp         <0.02         <0.02         <0.02         <0.02         <0.02           14         Peel         0.11         0.23         0.14         0.48           Pulp         <0.02
14     Peel Pulp     0.11 0.23 0.14 0.48 0.02       21     Peel 0.08 0.15 0.17 0.40 0.02 0.02       21     Pulp 0.002 0.02 0.02 0.02 0.02       28     Peel 0.05 0.08 0.11 0.24
$\begin{array}{ c c c c c c } \hline & Pulp & <0.02 & <0.02 & <0.02 & <0.02 \\ \hline 21 & Peel & 0.08 & 0.15 & 0.17 & 0.40 \\ Pulp & <0.02 & <0.02 & <0.02 & <0.02 \\ \hline 28 & Peel & 0.05 & 0.08 & 0.11 & 0.24 \\ \hline \end{array}$
21         Peel Pulp         0.08          0.15          0.17          0.40            Pulp          <0.02
Pulp         <0.02         <0.02         <0.02         <0.02           28         Peel         0.05         0.08         0.11         0.24
28 Peel 0.05 0.08 0.11 0.24
Pulp <0.02 <0.02 <0.02 <0.02
0 Whole fruit 0.11 0.10 0.03 0.24
7 0.04 0.08 0.04 0.16
14 0.03 0.06 0.04 0.13
21 0.03 0.05 0.05 0.13 0.02 0.03 0.03 0.08

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in whole fruit, pulp, peel and juice are shown in Table 13.

Table 13: Recoveries of endosulfan in fortified oranges, pulp, peel and juice.

Sample	α	-endosulfan	β-еі	ndosulfan	enc	dosulfan SO <sub>4</sub>
	Fortification	% Recovery	Fortification	% Recovery	Fortification	% Recovery
	(mg/kg)		(mg/kg)		(mg/kg)	
Whole fruit	0.02	73, 78, 91	0.02	63, 68, 84	0.02	71, 78, 97
	0.5	98	0.5	96	0.5	98
	0.7	86	0.7	86	0.7	88
Peel	0.02	84, 85, 87, 88, 90, 100,	0.02	58, 71, 75, 76, 76,	0.019	60, 74, 74, 80
		106		78, 81		
	0.507	73, 93	0.507	67, 95	0.02	68, 80, 107
	1.0	83	1.0	83	0.485	65, 97
	1.5	83	1.5	81	1	83
	2.03	69, 82	2.03	74, 82	1.5	84
					1.94	72, 85
Juice	0.02	59, 72, 75, 75, 112	0.02	62, 64, 74, 76, 95	0.02	64, 67, 69, 88
Pulp	0.02	74, 74, 87, 111	0.02	68, 75, 79, 95	0.019	64, 76, 88, 110
	0.05	60	0.05	64	0.049	68
	0.101	66, 86	0.1	68, 74	0.097	70, 78
	0.203	108	0.2	107	0.194	111

Mean recoveries of  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate were 85, 79 and 86% respectively, in whole fruit, 86, 77 and 79% respectively in peel, 83, 79 and 83%, respectively in pulp and 78, 74 and 72% respectively in juice. Mean recoveries were within the acceptable limits of 70 – 110%.

Residues at Harvest in Mandarins. European Union, Southern Zone 1998, E.H.-J. Klein. Hoechst Schering AgrEvo Germany. Report No. ER 98 ECS 741, 22 April 1999.

Four trials were conducted in Spain and Italy during 1998 to determine endosulfan residues in mandarins. Mandarin trees were treated with 2 applications of an endosulfan EC product at a rate of 1123 g ai/ha. Trees were sprayed when the fruit were about 50% of the final size and at the beginning of fruit colouring (BBCH 75 – 81), at an interval of 14 days. Applications were made using motorised knapsack sprayers (Spain) or knapsack mistblowers (Italy). Plot sizes were 72 and  $100m^2$  in Spain and  $75m^2$  in Italy. Samples of fruit were taken at 0, 20 or 21 days after treatment and stored frozen until analysis. Analyses were completed within 4 – 5 months of sample collection; residues were determined in pulp and peel and reported on a whole fruit basis. The results are given in Table 14.

Table 14: Endosulfan residues in mandarins from trials conducted in Spain and Italy (1998).

Trial site, Year,	Application	on	WHP	Sample		Residues (mg/kg)			
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total	
Cantillana Spain	1123	2 (14)	0	Peel	1.4	1.4	0.48	3.28	
1998 (Clemenules)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
			20	Peel	0.06	0.21	0.52	0.79	
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
			0	Whole fruit	0.35	0.35	0.12	0.82	
			20		0.02	0.06	0.14	0.22	
Brenes, Spain	1123	2 (14)	0	Peel	1.10	0.96	0.24	2.3	
1998 (Ortanique)	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
			20	Peel	0.04	0.15	0.36	0.55	
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
			0	Whole fruit	0.28	0.24	0.07	0.59	
			20		0.02	0.04	0.10	0.16	
Pisticci, Italy 1998	1123	2 (14)	0	Peel	1.10	1.20	0.49	2.79	
(Clementino	(37.5 g ai/hL)			Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
Commune)			21	Peel	0.17	0.34	0.58	1.09	
				Pulp	< 0.02	< 0.02	< 0.02	< 0.02	
			0	Whole fruit	0.27	0.29	0.12	0.68	

			21		0.05	0.09	0.14	0.28
Pisticci, Italy 1998	1123	2 (14)	0	Pulp	1.10	1.10	0.42	2.62
(Oroval)	(37.5 g ai/hL)			Peel	0.03	0.03	< 0.02	< 0.08
			21	Pulp	0.13	0.29	0.58	1.0
				Peel	< 0.02	< 0.02	< 0.02	< 0.02
			0	Whole fruit	0.38	0.38	0.15	0.91
			21		0.05	0.10	0.19	0.34

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in peel and pulp are shown in Table 15.

Table 15: Recoveries of endosulfan in fortified mandarin peel and pulp.

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Mandarin peel	0.02	84, 91	89, 123	107, 122				
	1	97, 101	111, 112	112, 112				
Mandarin pulp	0.02	99, 103	80, 101	92, 99				

Mean recoveries of  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate were 93, 109 and 113%, respectively in peel and 101, 90 and 96%, respectively in pulp.

Trials to Determine the Level of Endosulfan in Oranges at Harvest Following Four Applications to the Crop. Report No. 1/10/544, Protocol No. ECR544, October 2001. Determination of Endosulfan Residues in Oranges, State Chemistry Laboratory Report No. 0102252 & 0103083.

In two trials conducted in South Australia and Victoria, an endosulfan EC formulation was applied to orange trees at spray concentrations of 10.5 or 21 g ai/hL ( $1\times$  or  $2\times$ ). Four sprays were applied at intervals of 14 days at Victoria and 13-15 days in South Australia; spray volumes of 2000L/ha were used; 210-420 g ai/ha. Treatments began at 42 days prior to the first harvest. Sprays were applied by hand lance and trial plots comprised 2 trees in South Australia and 4 trees in Victoria with a single replication. Fruit were sampled at 0, 1, 3 and 7 days after application. Samples were stored at  $-20^{\circ}$  C for 10 months prior to analysis. The results are shown in Table 16.

Table 16: Endosulfan residues in oranges from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Beverford, VIC,	10.5 g ai/hL	4	0	Oranges	0.035	0.052	0.015	0.10
2000 (Valencia)			1		0.020	0.044	0.019	0.083
			3		0.010	0.025	0.014	0.049
			7		0.007	0.024	0.023	0.054
	21 g ai/hL	4	0	Oranges	0.090	0.097	0.027	0.21
	_		3		0.058	0.12	0.046	0.22
Waikerie, SA, 2000	10.5 g ai/hL	4	0	Oranges	0.081	0.10	0.008	0.19
(Valencia)			1		0.032	0.077	0.013	0.12
			3		0.018	0.047	0.013	0.078
			7		0.007	0.017	0.009	0.033
	21 g ai/hL	4	0	Whole fruit	0.17	0.20	0.029	0.40
			3	Pulp	0.011	0.016	0.007	0.034
				Peel	0.10	0.21	0.067	0.38

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg.

Table 17: Recoveries of endosulfan in fortified oranges.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan β-endosulfan endosulfan					
Oranges	0.02	96	104	90			
	0.51	95	106	92			

Trials to Determine the Level of Endosulfan in Lemons at Harvest Following Four Applications to the Crop. Report No. 1/10/543, Protocol No. ECR543, October 2001. Determination of Endosulfan Residues in Lemons, State Chemistry Laboratory Report No. 0102253, 0103086 & 0105244.

In three trials conducted in South Australia, Queensland and Victoria, an endosulfan EC formulation was applied to lemon trees at spray concentrations of 10.5 or 21 g ai/hL ( $1 \times$  or  $2 \times$ ). Four sprays were applied at intervals of 14 days at two sites and 12 to 17 days at the third site; spray volumes of 1428, 2000 and 2000L/ha were used at Queensland, Victoria and South Australia, respectively; 150-420 g ai/ha. Treatments began at 42 days prior to the first harvest at various stages of fruiting. Sprays were applied by hand lance or motorised back-pack mister and trial plots comprised 2 trees in South Australia and 4 trees in Queensland and Victoria with a single replication at each site. Fruit were sampled at 0, 1, 3 and 7 days after application. Samples were stored at  $-20^{\circ}$  C for 11 months prior to analysis. The results are shown in Table 18.

Table 18: Endosulfan residues in lemons from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)	_	α	β	SO <sub>4</sub>	Total
Beverford, VIC,	10.5 g ai/hL	4	0	Lemons	0.10	0.14	0.012	0.25
2000 (Lisbon)			1		0.049	0.10	0.008	0.16
			3		0.052	0.12	0.007	0.17
			7		0.038	0.14	0.012	0.19
	21 g ai/hL	4	0	Lemons	0.36	0.35	0.013	0.72
			3		0.21	0.47	0.024	0.70
Koah, QLD, 2000	10.5 g ai/hL	4	0	Lemons	0.064	0.069	0.021	0.15
(Lisbon)			1		0.014	0.021	0.013	0.048
			3		0.006	0.014	0.013	0.033
			7		< 0.005	0.008	0.013	0.021
	21 g ai/hL	4	0	Whole fruit	0.068	0.072	0.021	0.16
			3	Pulp	< 0.005	< 0.005	< 0.005	< 0.005
				Peel	0.063	0.20	0.10	0.36
Montacute, SA,	10.5 g ai/hL	4	0	Lemons	0.082	0.095	0.016	0.19
2000 (Lisbon)			1		0.058	0.099	0.021	0.18
			3		0.045	0.090	0.029	0.16
			7		0.024	0.074	0.033	0.13
				Control	0.019	0.012	0.005	0.036
	21 g ai/hL	4	0	Lemons	0.18	0.23	0.028	0.44
			3		0.088	0.22	0.034	0.34

Table 19: Recoveries of endosulfan in fortified lemons.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Lemons	0.02	81	89	85			
	0.51	83	95	85			

Trials to Determine the Level of Endosulfan in Mandarins at Harvest Following Four Applications to the Crop. Report No. 1/10/542, Protocol No. ECR542, October 2001. Determination of Endosulfan Residues in Mandarins, State Chemistry Laboratory Report No. 0103082, 0105080 & 0105243.

In two trials conducted in South Australia and Queensland, an endosulfan EC formulation was applied to mandarins at spray concentrations of 10.5 or 21 g ai/hL ( $1\times$  or  $2\times$ ). Four sprays were applied at intervals of 14 days in Qld and 13 to 16 days in South Australia; spray volumes of 645 and 2000 L/ha were used at Queensland and South Australia, respectively, 68 or 210 g ai/ha. Treatments began at 42 days prior to the first harvest, from fruit fill to maturity. Sprays were applied by hand lance or motorised back-pack mister and trial plots comprised 4 trees in Queensland and 2 trees in South Australia, with a single replication at each site. Fruit were sampled at 0, 1, 3 and 7 days after application. Samples were stored at  $-20^{\circ}$  C for 4 months prior to analysis. The results are shown in Table 20.

Table 20: Endosulfan residues in mandarins from trials conducted in Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Emerald Creek, Qld	10.5 g ai/hL	4	0	Mandarins	0.034	0.046	0.012	0.092
2001 (Imperial)	-		1		0.031	0.076	0.040	0.15
			3		0.025	0.057	0.024	0.11
			7		0.011	0.039	0.033	0.083
	21 g ai/hL	4	0	Mandarins	0.037	0.099	0.10	0.24
			3		0.039	0.092	0.048	0.18
Renmark, SA 2001	10.5 g ai/hL	4	0	Mandarins	0.064	0.072	0.018	0.15
(Kara)	-		1		0.021	0.042	0.026	0.089
			3		0.013	0.033	0.025	0.071
			7		0.006	0.014	0.015	0.035
	21 g ai/hL	4	0	Mandarins	0.034	0.12	0.11	0.26
	-		3		0.030	0.067	0.044	0.14

Table 21: Recoveries of endosulfan in fortified mandarins.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Mandarins	0.02	92	98	96			
	0.5	88	110	89			

# 8.5.2 Pome fruit (apples, pears)

Several overseas studies for apples were provided in summary form, with little detail of field or analytical conditions. The data are shown in Table 22 below. Those studies in which the use patterns are comparable to Australian GAP are highlighted.

Table 22: Endosulfan residues in apples and processed fractions from trials conducted in Spain, France and Italy (1994).

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Alfamen Spain	565	2 (51)	0	Fruit	0.05	0.09	< 0.01	0.15
1994	(56.5 g ai/hL)		7		0.02	0.02	< 0.01	0.05
(Golden Smuthe)			12		< 0.01	0.02	< 0.01	0.03
			21		< 0.01	0.01	< 0.01	< 0.01
			28		< 0.01	< 0.01	< 0.01	< 0.01
	1130	2 (51)	0	Fruit	0.26	0.24	0.02	0.52
	(113 g ai/hL)		7		0.04	0.08	0.02	0.14
			12		0.02	0.04	0.02	0.08
			21		0.02	0.03	0.02	0.07
			28		0.01	0.02	0.01	0.04
La Almunia De	565	2 (51)	0	Fruit	0.07	0.09	< 0.01	0.17
Dona Godina Spain	(56.5 g ai/hL)		7		0.01	0.02	< 0.01	0.04
1994, (Starkingson)			12		0.02	0.02	< 0.01	0.05
			21		0.02	0.03	< 0.01	0.06
			28		< 0.01	< 0.01	< 0.01	< 0.01
	(113 g ai/hL)	2 (51)	0	Fruit	0.15	0.21	< 0.01	0.37
			7		0.04	0.06	0.02	0.12

		1	12		0.02	0.04	< 0.01	0.08
			21		0.02	0.03	< 0.01	0.06
			28		0.02	0.03	< 0.01	0.06
Meynes France	565	2 (76)	0	Fruit	< 0.01	< 0.01	< 0.01	< 0.01
1994 (Golden Spur)	(57 g ai/hL)	2 (70)	7	Truit	0.02	0.04	0.01	0.07
1)) (Golden Spur)	(37 g til/IIL)		13		< 0.01	< 0.01	< 0.01	< 0.01
			21		< 0.01	< 0.01	< 0.01	< 0.01
			28		< 0.01	< 0.01	< 0.01	< 0.01
	1130	2 (76)	0	Fruit	0.34	0.31	0.02	0.67
	(114 g ai/hL)	2 (70)	7	Truit	0.04	0.06	0.02	0.12
	(114 g al/IIL)		13		< 0.01	< 0.01	< 0.01	< 0.01
			21		0.04	0.04	0.05	0.13
			28		0.03	0.06	0.03	0.12
Gallo Italy, 1994	848	2 (52)	0	Fruit	0.33	0.34	< 0.01	0.68
(Golden Delicious)	(56.5 g ai/hL)	2 (32)	7	Truit	0.06	0.09	0.02	0.18
(Golden Benerous)	(30.3 g til/IIL)		14		0.08	0.12	0.03	0.23
			21		0.04	0.07	0.03	0.14
			28		0.03	0.06	0.02	0.11
	1696	2 (52)	0	Fruit	1	0.71	0.05	1.8
	(113 g ai/hL)	2 (32)	7	Fruit	0.14	0.23	0.04	0.41
	(113 g til/IIL)		14	Washed fruit	0.07	0.12	0.03	0.22
			1.	Unwashed fruit	0.1, 0.1	0.13, 0.13	0.03, 0.04	0.26, 0.27
				Cider	< 0.01	< 0.01	< 0.01	< 0.01
				Mash	0.02	0.03	0.01	0.06
				Pomace ①	0.24	0.36	0.09	0.69
				Wash water	< 0.01	< 0.01	< 0.01	< 0.01
			21	Fruit	0.16	0.26	0.05	0.47
			28	Fruit	0.08	0.13	0.03	0.25
S. Maria Codifiume	848	2 (82)	0	Fruit	0.35	0.16	< 0.01	0.52
Italy, 1994	(56.5 g ai/hL)	- ()	7		0.04	0.02	< 0.01	0.07
(Imperatore)	(0 010 8 111 111)		14		0.01	0.02	< 0.01	0.04
( I · · · · · · · · · · · · · · · · · ·			21		0.03	0.04	< 0.01	0.08
			28		< 0.01	0.02	< 0.01	0.03
	1696	2 (82)	0	Fruit	0.62	0.27	0.01	0.90
	(113 g ai/hL)	(- )	7	Fruit	0.11	0.12	0.02	0.25
	( - 8 )		14	Washed fruit	0.06	0.08	0.02	0.16
				Unwashed fruit	0.13, 0.05	0.13, 0.09	0.01, 0.05	0.27, 0.19
				Cider	< 0.01	< 0.01	< 0.01	< 0.01
				Mash	< 0.01	0.01	< 0.01	< 0.01
				Pomace ①	0.17	0.26	0.07	0.5
				Wash water	< 0.01	< 0.01	< 0.01	< 0.01
			21	Fruit	0.03	0.05	0.02	0.10
			28	Fruit	0.06	0.07	0.03	0.16

① Pomace was described as marc and pomace; no indication of wet weight/dry weight was given.

Table 23: Mean recoveries of endosulfan in fortified apples and processed commodities.

Sample	α-(	endosulfan	β-enc	losulfan	endosulfan SO <sub>4</sub>		
	Fortification (mg/kg)	% Recovery	Fortification (mg/kg)	% Recovery	Fortification (mg/kg)	% Recovery	
Apples	0.01 - 0.5	93	0.01 - 0.5	94	0.01 - 0.5	95	
Cider	0.01 - 0.02	90	0.01	95	0.01	97	
Mash	0.01	90	0.01	73	0.01	82	
Pomace	0.01	90	0.01	97	0.01	85	

The limit of quantitation in apple was 0.01 mg/kg for each component of the residue definition.

Trials to Determine the Level of Endosulfan in Apples at Harvest Following Six Applications to the Crop. Report No. 1/9/563, Protocol No. ECR563, October 2001. Determination of Endosulfan Residues in Apples, State Chemistry Laboratory Report No. 0103227, 0105044 & 0107122.

In two trials conducted in NSW and Qld, an endosulfan EC formulation was applied to apple trees at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ). Six sprays were applied at intervals of 7 to 21 days in NSW and at 14 days in Qld. Treatments began at 70 and 84 days prior to the first harvest at

NSW and Qld, respectively. Sprays were applied by hand lance and trial plots comprised 4 trees. Fruit were sampled at 0, 7, 14 and 21 days after application. Fruit sampled from the NSW trial on day 14 were processed into juice, wet pomace and dry pomace. Samples were stored at  $-20^{\circ}$  C for 7 months prior to analysis. The results are shown in Table 24.

<b>Table 24:</b>	Endosulfan	residues in	apples from	trials	conducted in	Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Bathurst NSW 2001	66.5 g ai/hL	6	0	Fruit	1.2	0.9	0.03	2.1
(Red Delicious)	_		7		0.25	0.48	0.04	0.77
			14		0.08	0.18	0.02	0.29
			21		0.09	0.16	0.02	0.27
	133 g ai/hL	6	0	Fruit	1.5	1.3	0.07	2.9
	_		7		0.35	0.64	0.05	1.0
			14		0.24	0.44	0.04	0.72
			21		0.20	0.34	0.04	0.58
	66.5 g ai/hL	6	14	Whole fruit	0.12	0.23	0.03	0.38
				Juice	0.005	0.017	< 0.005	0.02
				Wet pomace	0.25	0.46	0.05	0.76
				Dry pomace	0.72	1.4	0.12	2.2
Cottonvale, Qld	66.5 g ai/hL	6	14	Fruit	0.16	0.22	0.15	0.53
2001 (Royal Gala)								

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in whole fruit, apple juice, dry pomace and wet pomace are shown in Table 25.

Table 25: Recoveries of endosulfan in fortified apple and processed fractions

Sample	Fortification (mg/kg)		% Recovery	
		α-endosulfan	% Recovery β-endosulfan	endosulfan SO <sub>4</sub>
Juice	0.02	95	98	99
	0.5	92	102	100
Dry pomace	0.02	100	112	99
	0.5	85	96	97
Wet pomace	0.02	92	82	113
	0.5	102	111	110

Recoveries in whole fruit were not reported.

Trials to Determine the Level of Endosulfan in Pears at Harvest Following Six Applications to the Crop. Report No. 1/9/548, Protocol No. ECR548, October 2001. Determination of Endosulfan Residues in Pears, State Chemistry Laboratory Report No. 0103089 & 0103150.

In 2 trials conducted in Victoria and South Australia, an endosulfan EC formulation was applied to pear trees at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ). Six sprays were applied at intervals of 14 days; treatments began at approximately 70 days prior to the first harvest. Sprays were applied by hand gun or hand lance and trial plots comprised 8 trees in Victoria and 2 trees in South Australia. Fruit were sampled at 0, 7, 14 and 21 days after the final application. Samples were stored at  $-20^{\circ}$  C for 8 months prior to analysis. The results are shown in Table 26.

Table 26: Endosulfan residues in pears from trials conducted in Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Ardmona, VIC 2001	66.5 g ai/hL	6 (14)	0	Fruit	0.44	0.45	0.018	0.91
(Packham)			7		0.26	0.20	0.38	0.84
			14		0.22	0.37	0.20	0.79
			21		0.097	0.19	0.13	0.42
	133 g ai/hL	6 (14)	0	Fruit	0.86	0.74	0.23	1.8
			7		0.57	0.82	0.30	1.7
			14		0.48	0.77	0.33	1.6
			21		0.21	0.40	0.24	0.85
Paracombe, SA	66.5 g ai/hL	6 (14)	0	Fruit	0.74	0.67	0.32	1.7
2001 (Dutchess)			7		0.34	0.46	0.35	1.2
			14		0.062	0.14	0.24	0.44
			21		0.044	0.10	0.23	0.37

Table 27: Recoveries of endosulfan in fortified pears.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Pears	0.02	104	103	106			
	0.5	87	94	94			

## 8.5.3 Stone fruit (peaches, nectarines, apricots)

(Note, these uses are no longer registered. Submitted data not relevant to the reconsideration)

Residue trials in peaches were conducted in Australia, Italy, Greece and Spain. The studies are described in detail below.

Residue Trials on Peaches to Establish a Maximum Residue Level. Determination of Active Substance and Metabolite Decline Following 3 Applications. European Union (Southern Zone) 1997. E.H.-J. Klein Hoechst Schering AgrEvo Germany. Report No. ER 97 ECS 742, 3 December 1998.

In five trials in Italy and Spain, peach trees were treated with 3 applications of an endosulfan EC product at a rate of 800 g ai/ha. Application timings ranged from second fruit fall to ripe fruit (BBCH stages 73 – 89), at intervals of 13 or 14 days between each application. Sprays were applied using motorised knapsack sprayers (Spain) or knapsack mistblowers (Italy). Plot sizes were  $60m^2$  and  $81m^2$  in Italy and  $108m^2$  and  $252m^2$  in Spain. Samples of fruit were taken at 0, 7, 14 and 21 days after treatment and stored frozen until analysis. Analyses were completed within 7 months of sample collection; residues were determined in pulp and reported on a whole fruit basis. The results are given in Table 28.

Table 28: Endosulfan residues in peaches from trials conducted in Italy and Spain, 1997.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Sevilla, Spain 1997	855	3 (14)	0	Peach	0.43	0.27	< 0.02	0.72 (0.82)
(Baby Gold 6)	(57 g ai/hL)		7		0.30	0.34	0.03	0.67 (0.73)
			14		0.21	0.31	0.04	0.56 (0.60)
Sevilla, Spain 1997	855	3 (14)	0	Peach	1.05	0.76	0.04	1.85 (2.11)
(Sudanell)	(57 g ai/hL)		7		0.26	0.29	0.03	0.58 (0.63)
			14		0.14	0.22	0.04	0.40 (0.43)
			21		0.05	0.12	0.03	0.20 (0.21)
Romagna, Italy	855	3 (14)	0	Peach	0.37	0.28	< 0.02	0.67 (0.76)
1997	(57 g ai/hL)		7		0.13	0.11	< 0.02	0.26 (0.28)
(Star Red Gold)			14		0.11	0.15	< 0.02	0.28 (0.30)
			21		0.05	0.07	< 0.02	0.14 (0.15)
Romagna, Italy	855	3 (14)	0	Peach	0.38	0.22	< 0.02	0.62 (0.70)
1997 (Lafaiette)	(57 g ai/hL)		7		0.21	0.21	< 0.02	0.44 (0.46)
			14		0.04	0.05	< 0.02	0.11 (0.11)

#### Australian Pesticides and Veterinary Medicines Authority (APVMA)

			20		0.04	0.05	< 0.02	0.11 (0.11)
Puglia, Italy, 1997	855	3 (14)	0	Peach	0.75	0.19	0.02	0.96 (0.96)
(Maycrest)	(57 g ai/hL)		7		0.23	0.24	0.02	0.49 (0.54)
			14		0.04	0.09	0.03	0.16 (0.17)
			20		0.03	0.03	0.02	0.08 (0.08)

Values in parentheses are residues in pulp only.

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in peaches are shown in Table 29.

Table 29: Recoveries of endosulfan in fortified peaches

Sample	Fortification (mg/kg)		% Recovery	
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>
Peaches	0.02	73, 94, 125	92, 94, 102	82, 92, 98
	0.2	95, 113	97, 119	99, 103
	1	119	106	114

Mean recoveries were 103 and 102 and 98% for  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate, respectively.

Residues at Harvest in Peaches European Union, Southern Zone 1998, E.H.-J. Klein. Hoechst Schering AgrEvo Germany. Report No. ER 98 ECS 742, 8 April 1999.

In four trials in Italy, Greece and Spain, peach trees were treated with 3 applications of an endosulfan EC product at a rate of 800 g ai/ha. Application timings ranged from fruit fall after flowering to the beginning of fruit colour (BBCH stages 71-81), at intervals of 12 to 15 days between each application. Sprays were applied using high pressure sprayers (Greece and Spain) or knapsack mistblowers (Italy). Plot sizes were  $80m^2$  and  $101 m^2$  in Italy, 160 m2 in Greece and 150 and  $160m^2$  in Spain. Samples of fruit were taken at 0 and 21 days after treatment and stored frozen until analysis. Analyses were completed within 7 months of sample collection; residues were determined in pulp and reported on a whole fruit basis. The results are given in Table 30.

Table 30: Endosulfan residues in peaches from trials conducted in Italy, Greece and Spain, 1998.

Trial site, Year,	Application	on	WHP	Sample	Residues (mg/kg)			
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	SO <sub>4</sub>	Total
Bonifaio, Spain	855	3 (13,	0	Peach	1.20	0.75	0.12	2.07 (2.44)
1998 (Flor Down)	(57 g ai/hL)	15)	20		0.07	0.08	0.04	0.19 (0.21)
Sollana, Spain 1998	855	3 (14,	0	Peach	1.30	0.17	0.11	1.58 (1.14)
(Spind Graes)	(57 g ai/hL)	15)	19		0.08	0.05	0.04	0.17 (0.14)
Korifi, Greece 1998	855	3 (14)	0	Peach	0.63	0.36	0.03	1.02 (1.19)
(Louatel)	(57 g ai/hL)		21		0.07	0.07	0.02	0.16 (0.16)
Romagna, Italy	855	3 (12,	0	Peach	0.89	0.60	0.05	1.54 (1.73)
1998 (Maria Luisa)	(57 g ai/hL)	14)	20		0.07	0.10	0.05	0.22 (0.24)
Puglia, Italy 1998	855	3 (15,	0	Peach	0.48	0.33	0.02	0.83 (0.91)
(Spring Bell)	(57 g ai/hL)	13)	20		0.13	0.16	0.09	0.38 (0.40)

Residues values in parentheses are residues in pulp only.

The limits of quantitation for each component ( $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate) were 0.02 mg/kg. Recoveries for each compound in peaches are shown in Table 31.

Table 31: Recoveries of endosulfan in fortified peaches

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Peaches	0.02	82, 83	80, 82	70, 76			
	0.5	84. 89	90, 93	97. 98			

Mean recoveries were 84 and 86 and 85% for  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate, respectively.

Trials to Determine the Level of Endosulfan in Peaches at Harvest Following Three Applications to the Crop. Report No. 1/9/541, Protocol No. ECR541, October 2001. Determination of Endosulfan Residues in Peaches, State Chemistry Laboratory Report No. 0103151 & 0105233.

In two trials conducted in Victoria and Qld, an endosulfan EC formulation was applied to peach trees at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 13 – 14 days and spraying began at 28 days prior to the first harvest.

Sprays were applied by hand lance and trial plots comprised 4 trees in Victoria and 5 trees in Qld, with one replication. Fruit were sampled at 0, 14, 28 and 35 days after application. Samples were stored at – 20° C for up to 10 months prior to analysis. Residues in fruit pulp were determined and reported on a whole fruit basis. The results are shown in Table 32.

Table 32: Endosulfan residues in peaches from trials conducted in Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)	_	α	β	SO <sub>4</sub>	Total
Goulburn Valley,	66.5 g ai/hL	3 (14)	0	Peach	0.13	0.085	< 0.005	0.22
VIC, 2001		, ,	14		0.39	0.53	0.13	1.0
(Tatura 204)			28		0.08	0.15	0.07	0.30
			35		0.03	0.09	0.07	0.19
	133 g ai/hL	3 (14)	0	Peach	2.9	1.9	0.11	4.9
			14		0.29	0.33	0.05	0.73
			28		0.09	0.20	0.08	0.37
			35		0.03	0.11	0.07	0.21
Passchendale, QLD	66.5 g ai/hL	3 (14)	0	Peach	0.81	0.68	0.18	1.7
2000		, ,	14		0.26	0.35	0.14	0.75
(Crown Princess)			28		0.06	0.10	0.05	0.21
·			35		0.03	0.07	0.05	0.14
				Control	0.006	0.005	< 0.005	0.011

Table 33: Recoveries of endosulfan in fortified peaches.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Peaches	0.02	91	85	93		
	0.5	82	86	84		

Trials to Determine the Level of Endosulfan in Nectarines at Harvest Following Three Applications to the Crop. Report No. 1/9/536, Protocol No. ECR536, October 2001. Determination of Endosulfan Residues in Nectarines, State Chemistry Laboratory Report No. 0103085.

In a trial conducted in South Australia, an endosulfan EC formulation was applied to nectarine trees at spray concentrations of 66.5 g ai/hL (1×). Three sprays were applied at intervals of 14 days and spraying began at 28 days prior to the first harvest. Sprays were applied by hand lance and the trial plot comprised 6 trees with one replication. Fruit were sampled at 0, 14, 28 and 35 days after application.

Samples were stored at  $-20^{\circ}$  C for up to 10 months prior to analysis. Residues in fruit pulp were determined and reported on a whole fruit basis. The results are shown in Table 34.

Table 34: Endosulfan residues in nectarines from trials conducted in Australia 2001.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray Conc.	No.	(days)		α	β	SO <sub>4</sub>	Total
Montacute, SA 2000	66.5 g ai/hL	3 (14)	0	Nectarine	1.7	1.1	0.03	2.8
(Tasty Gold)	-		14		0.26	0.34	0.02	0.62
			28		0.04	0.11	0.03	0.18
			35		0.1	0.27	0.06	0.43

Table 35: Recoveries of endosulfan in fortified nectarines.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Nectarines	0.02	98	93	105			
	0.5	82	86	89			

Trials to Determine the Level of Endosulfan in Apricots at Harvest Following Three Applications to the Crop. Report No. 1/9/561, Protocol No. ECR561, October 2001. Determination of Endosulfan Residues in Apricots, State Chemistry Laboratory Report No. 0103084.

In a trial conducted in South Australia, an endosulfan EC formulation was applied to apricot trees at spray concentrations of 66.5 and 133 g ai/hL ( $1 \times$  and  $2 \times$ ). Three sprays were applied at intervals of 13 - 16 days and spraying began at 28 days prior to the first harvest. Sprays were applied by hand lance and the trial plot comprised 2 trees with one replication. Fruit were sampled at 0, 14, 28 and 35 days after application. Samples were stored at  $-20^{\circ}$  C for up to 11 months prior to analysis. Residues in fruit pulp were determined and reported on a whole fruit basis. The results are shown in Table 36.

Table 36: Endosulfan residues in apricots from trials conducted in Australia 2001.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Renmark, SA 2000	66.5 g ai/hL	3 (13	0	Apricots	1.7	1.5	0.52	3.7
(Storey)		16)	14		0.15	0.30	0.36	0.81
			28		0.11	0.29	0.63	1.0
			35		0.01	0.04	0.21	0.26
	133 g ai/hL	3 (13	0	Apricots	5.0	4.1	1.1	10
		16)	14		1.0	1.8	1.3	4.1
			28		0.33	0.74	1.1	2.2
			35		0.16	0.34	0.94	1.4

Table 37: Recoveries of endosulfan in fortified apricots.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Apricots	0.02	88	104	122			
	0.5	87	87	91			

#### **8.5.4** Grapes

Reports from several overseas studies for grapes were provided in summary form, with little detail of field or analytical conditions. The data are shown in Table 38 below.

Table 38: Endosulfan residues in grapes and processed fractions from trials conducted in Spain and Italy in 1994.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Rate (g ai/ha)	No.	(days)	Sample	α	В	SO <sub>4</sub>	Total
Requera, Spain	848 ①	3	0	Fruit	0.16	0.2	< 0.05	0.39
Requera, Spani	040 @	(14,14)	0	Truit	0.10	0.2	₹0.05	0.37
1994, (Cencibel)	(113 g ai/hL)	(1.,1.)	8	Fruit	< 0.05	0.08	< 0.05	< 0.13
, , , , , , , , , , , , , , , , , , , ,	,		15	Fruit	< 0.05	0.07	< 0.05	< 0.12
				Juice	< 0.05	< 0.05	< 0.05	< 0.05
				Pomace	0.07	0.2	0.06	0.33
				New wine	< 0.05	< 0.05	< 0.05	< 0.05
				Mature wine	< 0.05	< 0.05	< 0.05	< 0.05
			22	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
			29	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
Campo Arcis, Spain	848 ②	3 (14)	0	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
1994 (Bobal)	(113 g ai/hL)		8	Fruit	< 0.05	0.1	< 0.05	< 0.15
			15	Fruit	< 0.05	0.1	< 0.05	< 0.15
				Juice	< 0.05	< 0.05	< 0.05	< 0.05
				Pomace	< 0.05	0.17	0.07	0.24
				New wine	< 0.05	< 0.05	< 0.05	< 0.05
				Mature wine	< 0.05	< 0.05	< 0.05	< 0.05
			22	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
			29	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
Espartinas, Spain	339 ③	3 (14)	0	Fruit	0.13	0.12	< 0.05	0.25
1994, (Garrida)	(113 g ai/hL)		7	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
			13	Fruit	< 0.05	< 0.05	0.09	< 0.09
				Juice	< 0.05	< 0.05	< 0.05	< 0.05
				Pomace	< 0.05	< 0.05	< 0.05	< 0.05
				New wine	< 0.05	< 0.05	< 0.05	< 0.05
				Mature wine	< 0.05	< 0.05	< 0.05	< 0.05
			20	Fruit	< 0.05	< 0.05	< 0.05	< 0.05
Stornarella, Italy	1356 ⊕	3 (14)	0	Fruit	0.84	0.68	< 0.05	1.56
1994 (Sangiovese)	(113 g ai/hL)		7	Fruit	0.91	0.7	< 0.05	1.62
			13	Fruit	0.1	0.25	< 0.05	0.38
			14	Juice	< 0.05	< 0.05	< 0.05	< 0.05
				Pomace	0.13	0.48	0.07	0.68
				New wine	0.07	< 0.05	< 0.05	< 0.12
				Mature wine	< 0.05	< 0.05	< 0.05	< 0.05
			21	Fruit	< 0.05	0.12	< 0.05	0.17
			28	Fruit	0.05	0.13	< 0.05	0.23
Tebano, Italy 1994	1695 ©	3 (14)	0	Fruit	0.80	0.75	< 0.05	1.6
(Trebbiano)	(113 g ai/hL)		7	Fruit	0.26	0.37	< 0.05	0.66
			14	Fruit	0.20	0.31	< 0.05	0.53
				Juice	< 0.05	< 0.05	< 0.05	< 0.05
				Pomace	0.38	0.57	< 0.05	1
				New wine	< 0.05	< 0.05	< 0.05	< 0.05
			2.1	Mature wine	< 0.05	< 0.05	< 0.05	< 0.05
			21	Fruit	0.11	0.17	< 0.05	0.31
			28	Fruit	0.12	0.19	< 0.05	0.34

① Last spray at ripe for vintage; last sampling at end of mellowing (Eichhorn 35 – 41).

Table 39: Mean recoveries of endosulfan in fortified grapes and processed commodities.

Sample	α	-endosulfan	β-er	dosulfan	endosulfan SO <sub>4</sub>		
	Fortification	% Recovery	Fortification	% Recovery	Fortification	% Recovery	
	(mg/kg)		(mg/kg)		(mg/kg)		
Grapes	0.05 - 0.5	97	0.05 - 0.5	99	0.05 - 0.5	104	
Juice			0.05	88	0.05	88	
Pomace	0.05	84	0.05	106	0.05	110	
New wine	0.05	89	0.05	105	0.05	106	

② Last spray at beginning or maturity; last sampling at end of mellowing (Eichhorn 35 - 41).

③ Last spray at beginning or maturity; last sampling at ripe for vintage (Eichhorn 35 − 38).

① Last spray at beginning or maturity; last sampling at end of mellowing (Eichhorn 38).

⑤ Last spray at beginning or maturity; last sampling at end of mellowing (Eichhorn 35 − 41).

Reported recoveries in the trials were within acceptable limits of 70 - 110% for each component of the residue definition. Details of processing or descriptions of the various samples were not given, e.g. pomace, mature wine.

# 8.5.5 Tropic/sub-tropic fruit-inedib peel (avocado, custard apple, mango, pawpaw, persimmon)

Trials to Determine the Level of Endosulfan in Avocado at Harvest Following Six Applications to the Crop. Report No. 1/10/554, Protocol No. ECR554, October 2001. Determination of Endosulfan Residues in Avocadoes, State Chemistry Laboratory Report No. 0103228, 0104094 & 0107097.

In three trials conducted in Queensland, an endosulfan EC formulation was applied to avocado trees at spray concentrations of 70 or 140 g ai/hL ( $1 \times$  or  $2 \times$ ). Six sprays were applied at 13-15 day intervals, from fruit development to the maturing fruit stage. Sprays were applied by motorised backpack mister or hand held lance and treatment plots comprised 4 or 5 trees

with a single replication at each site. Mature fruit was collected at 0, 14, 21 and 28 days after application. Samples were stored at  $-20^{\circ}$  C for up to 10 months prior to analysis. Residues were determined in the flesh and calculated on a whole fruit basis; the results are shown in Table 40.

Table 40: Endosulfan residues in avocado from trials conducted in Australia 2000/2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Tolga, QLD, 2000	70 g ai/hL	6	0	Avocado	0.011	0.021	0.040	0.072
(Shepard)			14		< 0.005	< 0.005	0.010	0.010
			21		< 0.005	< 0.005	< 0.005	< 0.005
			28		< 0.005	< 0.005	< 0.005	< 0.005
Glasshouse Mtns,	70 g ai/hL	6	0	Avocado	0.32	0.29	0.049	0.66
QLD, 2001,			14		0.008	0.031	0.026	0.065
(Wurtz)			21		0.009	0.035	0.057	0.10
			28		0.015	0.048	0.044	0.11
	140 g ai/hL	6	0	Avocado	0.55	0.50	0.12	1.2
			14	Flesh	< 0.005	0.007	0.018	0.025
				Peel	0.19	0.70	0.35	1.2
			21	Avocado	0.016	0.080	0.066	0.16
			28	Flesh	< 0.005	0.012	0.035	0.047
				Peel	0.053	0.46	0.44	0.95
				Control	< 0.005	0.005	0.007	0.012
Tolga, QLD, 2000 (Hass)	70 g ai/hL	6	28	Avocado	< 0.005	< 0.005	< 0.005	< 0.005

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

The portion of the commodity to which the MRL applies is the whole commodity after removal of the stone, but calculated on a whole fruit basis.

Table 41: Recoveries of endosulfan in fortified avocado.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Avocado	0.02	71, 88	69, 84	75, 100			
	0.5	69, 70	69, 79	85, 84			

Trials to Determine the Level of Endosulfan on Custard Apples. Report No. 1/2/500, Protocol No. ECR500, February 2001. Supervised Residue Trial For Endosulfan On Custard Apples, R. Lee, Resource Sciences Laboratory, Indooroopilly, November 2000.

In two trials conducted in Queensland and New South Wales, an endosulfan EC formulation was applied to custard apple trees at spray concentrations of 70 or 140 g ai/hL ( $1 \times$  or  $2 \times$ ). Three sprays

were applied at 14 day intervals, with the final spray applied to fruit 12-20 cm in diameter. Sprays were applied by a power sprayer and treatment plots comprised 3 trees with a single replication at each site. Mature fruit was collected at 0, 7, 14 and 28 days after application. Samples were stored at  $-20^{\circ}$  C for up to 4 months prior to analysis. The residue results are shown in Table 42.

Table 42: Endosulfan residues in custard apples from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Nambour, QLD,	70 g ai/hL	3	0	Custard apple	0.71	0.39	0.22	1.32
2000 (African pride)			7		0.02	0.03	0.05	0.10
			14		0.01	0.01	0.07	0.09
			28		< 0.005	0.01	0.04	0.05
	140 g ai/hL	3	0	Custard apple	0.84	0.50	0.29	1.63
			7		0.03	0.08	0.16	0.27
			14		0.01	0.04	0.20	0.25
			28		< 0.005	0.01	0.06	0.07
Alstonville, NSW,	70 g ai/hL	3	0	Custard apple	0.62	0.35	0.17	1.14
2000 (Pinks			7		0.11	0.14	0.10	0.34
Mammoth)			14		0.05	0.07	0.06	0.18
			28		0.01	0.02	0.04	0.07
	140 g ai/hL	3	0	Custard apple	0.37	0.62	0.36	1.33
			7		0.30	0.49	0.17	0.95
			14		0.22	0.32	0.22	0.76
			28		0.03	0.08	0.12	0.23

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 43: Recoveries of endosulfan in fortified custard apple.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Custard apple	0.02	77, 41,48	52, 33, 38	53, 45, 53			
	0.2	96, 88	88, 78	109, 111			
	1	91, 97, 100	99, 99, 113	92, 100, 103			

Trials to Determine the Level of Endosulfan in Mangoes at Harvest Following Two Applications to the Crop. Report No. 1/10/537, Protocol No. ECR537, October 2001. Determination of Endosulfan Residues in Mangoes, State Chemistry Laboratory Report No. 0104096, 0105234 & 0105235.

In three trials conducted in Queensland and New South Wales, an endosulfan EC formulation was applied to mango trees at spray concentrations of 70 or 140 g ai/hL ( $1 \times$  or  $2 \times$ ). Two sprays were applied at an interval of 14 days, at stages ranging from immature fruit to maturing fruit. Sprays were applied by motorised backpack mister or hand lance and treatment plots comprised 2-5 trees with a single replication at each site. Mature fruit was collected at 0, 7, 14 and 28 days after application. Samples were stored at  $-20^{\circ}$  C for up to 10 months prior to analysis. Residues were determined in the skin and flesh and calculated on a whole fruit basis; the results are shown in Table 44.

Table 44: Endosulfan residues in mangoes from trials conducted in Australia 2001.

Trial site, Year,	Applicati	Application		Sample	Residues (mg/kg)			
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Dorroughby, NSW,	70 g ai/hL	2	0	Mango	0.24	0.17	0.007	0.42
2001 (Bowen)			7	_	0.071	0.067	0.060	0.20
			14		0.026	0.025	0.049	0.10
			28		0.012	0.015	0.063	0.090
Tolga, QLD, 2001,	70 g ai/hL	2	0	Mango	0.17	0.15	0.034	0.35
(Palmer)	_		7	_	0.035	0.035	0.10	0.17
			14		0.015	0.013	0.12	0.15

			28		0.009	0.006	0.15	0.16
	140 g ai/hL	2	0	Mango	0.26	0.18	0.048	0.49
			7		0.11	0.11	0.28	0.50
			14		0.051	0.055	0.34	0.45
			28		0.005	0.005	0.089	0.099
Wamuran, QLD,	70 g ai/hL		28	Mango	0.014	0.011	0.20	0.22
2001 (Kent)	-		28	Control	0.008	0.006	0.031	0.045

LOD = 0.005 m/kg; LOQ = 0.02 mg/kg.

Table 45: Recoveries of endosulfan in fortified mango.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Mango	0.02	96	78	97			
	0.5	94	98	94			

Trials to Determine the Level of Endosulfan in Pawpaws at Harvest Following Four Applications to the Crop. Report No. 1/10/539, Protocol No. ECR539, October 2001. Determination of Endosulfan Residues in Pawpaw, State Chemistry Laboratory Report No. 0012105 & 0012106.

In two trials conducted in Queensland, an endosulfan EC formulation was applied to pawpaws at spray concentrations of 70 or 140 g ai/hL ( $1 \times$  or  $2 \times$ ). Four sprays were applied at 14 day intervals to mature fruit. Sprays were applied by motorised backpack mister and plots were  $15 \times 2m$  at both sites with a single replication. Mature fruit was collected at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 11 months prior to analysis. Results are shown in Table 46.

Table 46: Endosulfan residues in pawpaw from trials conducted in Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)	_	α	β	$SO_4$	Total
Walkamin, QLD,	70 g ai/hL	4	0	Pawpaw	0.16	0.12	0.088	0.37
2000 (Ruby Red)			7		0.030	0.053	0.10	0.18
			14		0.013	0.024	0.094	0.13
			21		< 0.005	0.005	0.090	0.095
	140 g ai/hL	4	0	Pawpaw	0.37	0.33	0.18	0.88
			7		0.09	0.11	0.13	0.33
			14		0.025	0.074	0.13	0.23
			21		< 0.005	0.010	0.074	0.084
				Control	0.007	0.006	0.010	0.023
Mareeba, QLD,	70 g ai/hL	2	0	Pawpaw	0.11	0.076	0.051	0.24
2000 (Hybrid 1B)			7	_	0.005	0.011	0.079	0.095
			14		< 0.005	0.006	0.047	0.053
			21		< 0.005	< 0.005	0.045	0.045

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 47: Recoveries of endosulfan in fortified pawpaw.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Pawpaw	0.02	90	99	95			
	0.5	83	84	87			

Trials to Determine the Level of Endosulfan in Persimmons at Harvest Following Two Applications to the Crop. Report No. 1/10/545, Protocol No. ECR545, October 2001. Determination of Endosulfan Residues in Persimmon, State Chemistry Laboratory Report No. 0105225, 0105226 & 0105281.

In three trials conducted in Queensland and New South Wales, an endosulfan EC formulation was applied to persimmons at spray concentrations of 70 or 140 g ai/hL ( $1 \times$  or  $2 \times$ ). Two sprays were applied at 14 day intervals to immature and mature fruit. Sprays were applied by hand lance or hand

sprayer and treatment plots comprised 2-4 trees with a single replication. Mature fruit was collected at 0, 7, 14 and 28 days after application. Samples were stored at  $-20^{\circ}$  C for up to 6 months prior to analysis. Results are shown in Table 48.

Table 48: Endosulfan residues in persimmon from trials conducted in Australia 2001.

Trial site, Year,	Application	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	SO <sub>4</sub>	Total
Wollongbar, NSW,	70 g ai/hL	2	0	Persimmon	0.45	0.37	0.11	0.93
2001 (Fuji)			7		0.17	0.24	0.12	0.53
-			14		0.17	0.23	0.15	0.55
			28		0.53	0.58	0.65	1.8①
The Summitt, QLD,	70 g ai/hL	2	0	Persimmon	0.49	0.48	0.057	1.0
2001 (Fuyu)			7		0.38	0.42	0.086	0.89
			14		0.27	0.34	0.080	0.69
			28		0.14	0.21	0.15	0.50
	140 g ai/hL	2	0	Persimmon	1.4	1.2	0.080	2.7
			7		0.71	0.81	0.18	1.7
			14	Peel	2.6	2.8	0.87	6.3
				Flesh	0.13	0.086	0.028	0.24
			28	Peel	1.7	1.9	1.4	5.0
				Flesh	0.014	0.015	0.016	0.045
Glasshouse Mtns,	70 g ai/hL	2	28	Persimmon	0.14	0.13	0.45	0.72
QLD, 2001 (Fuji)				Control	0.17	0.16	0.46	0.79

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg. ① Samples size was 0.5 kg (3 units) instead of 2 kg.

Table 49: Recoveries of endosulfan in fortified persimmon

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Persimmon	0.02	101	110	106			
	0.5	78	84	86			

Determination Of Total Endosulfan Residues In Lychee Fruit Following Four Applications Of Endosulfan 350 EC Applied At Rates Of 52.5 g ai/100L and 105 g ai/100L. Mareeba Queensland 1999. J.C. Watson 9 May 2000.

In two trials conducted in Queensland, an endosulfan EC formulation was applied to lychee trees at spray concentrations of 52.5 or 105 g ai/hL (1× or 2×). Four sprays were applied at 10 day intervals from fruit fill to mature fruit. Sprays were applied using a backpack mister and treatment plots comprised single trees with 4 replications. Mature fruit was collected at 0, 3, 7, 14 days after application; samples from 2 replicates were combined prior to analysis, giving two samples for each interval. Samples were analysed within 4 months of collection. Results are shown in Table 50; combined replicates are reported.

Table 50: Endosulfan residues in lychees from trials conducted in Australia 1999.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Mareeba, QLD,	52.5 g ai/hL	4	0	Lychee	1.48	0.90	0.68	3.06
1999, (Bengal)					1.78	1.09	0.69	3.56
			3		0.36	0.30	0.42	1.08
					0.66	0.54	0.93	2.13
			7		0.43	0.37	0.82	1.62
					0.24	0.21	0.50	0.95
			14		0.11	0.084	0.24	0.43
					0.098	0.081	0.27	0.50
	105	4	0	Lychee	1.47	0.84	0.41	2.71
					3.83	1.72	1.10	6.65
			3		0.56	0.52	0.72	1.80
					0.64	0.56	0.82	2.02
			7		0.36	0.32	0.56	1.24

					0.38	0.32	0.72	1.42	
			14		0.35	0.28	0.62	1.25	
					0.26	0.19	0.53	0.98	
Bundaberg, QLD,	52.5 g ai/hL	4	7	Lychee	0.28	0.30	0.58	1.16	
1999 (Kwai Mai					0.19	0.17	0.48	0.84	
Pink)									

The limit of detection was 0.005 mg/kg and the limit of quantitation was 0.01 mg/kg. Recoveries were reported as the mean from six fortifications and are shown below in Table 51.

Table 51: Recoveries of endosulfan in fortified lychee.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	% Recovery β-endosulfan	endosulfan SO <sub>4</sub>			
Lychee	0.02	89	86	84			
	0.05	92	90	87			
	0.2	90	92	92			
	2	93	92	90			

## 8.5.6 Brassica vegetables (broccoli, cabbage, cauliflower, Brussels sprouts)

Trials to Determine the Level of Endosulfan in Broccoli at Harvest Following Three Applications to the Crop. Report No. 1/5/594, Protocol No. ECR594, May 2001. Determination of Endosulfan Residues in Cauliflower, State Chemistry Laboratory Report No. 0009232, 0009239, 0010031 & 0012135.

In two trials conducted in Victoria and Queensland, an endosulfan EC formulation was applied to broccoli at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ;  $0.6 - 2.1 \times$  application rate). Three sprays were applied at 10 - 11 day intervals, with the final spray applied at heading or early harvest. Sprays were applied by mini boom or backpack sprayer and plot sizes were  $2.8 \times 10$ m and  $3.6 \times 11$ m in Qld and Victoria, respectively, with a single replication at each site. Broccoli heads were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for 4 months prior to analysis. The results are shown in Table 52.

Table 52: Endosulfan residues in broccoli from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Stanthorpe, Qld,	66.5 g ai/hL	3	0	Broccoli	1.70	0.92	< 0.005	2.6
2000, (Babylon)	(533 – 768 g		3		0.51	0.36	0.034	0.90
	ai/ha)		7		0.12	0.14	0.025	0.29
			14		0.008	0.006	0.004	0.018
	133 g ai/hL	3	0	Broccoli	1.91	1.05	0.009	3.0
	(1066 1536 g		3		0.70	0.51	0.047	1.3
	ai/ha)		7		0.27	0.27	0.064	0.60
			14		0.016	0.024	0.020	0.060
Cranbourne, VIC,	66.5 g ai/hL	3	0	Broccoli	0.55	0.27	0.011	0.84
2000 (Greenbelt)	(465 g ai/ha)		3		0.39	0.25	0.06	0.70
			7		0.08	0.08	0.012	0.172
			14		< 0.005	< 0.005	< 0.005	< 0.005
	133 g ai/hL	3	0		1.25	0.71	< 0.005	2.0
	(931 g ai/ha)		3		0.36	0.27	0.077	0.71
			7		0.11	0.12	0.055	0.28
100 0005 1	100 005 4		14		0.008	0.007	0.010	0.025

LOD = 0.005 mg/kg; LOO = 0.05 mg/kg.

Table 53: Recoveries of endosulfan in fortified broccoli.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Broccoli	0.07	80, 106	138, 161	53, 84		

Trials to Determine the Level of Endosulfan in Cauliflower at Harvest Following Three Applications to the Crop. Report No. 1/5/550, Protocol No. ECR550, May 2001. Determination of Endosulfan Residues in Cauliflower, State Chemistry Laboratory Report No. 0101001 & 0011009

In two trials conducted in Victoria and Western Australia, an endosulfan EC formulation was applied to cauliflower at spray concentrations of 66.5 or 133 g ai/hL

 $(1 \times \text{ or } 2 \times)$ . Three sprays were applied at 8-10 day intervals, with the final spray applied at maturity. Sprays were applied by hand held boom or backpack sprayer

and plot sizes were  $2 \times 40$ m and  $1.25 \times 5$ m in WA and Victoria, respectively, with a single replication at each site. Cauliflower heads were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for 6 months prior to analysis. The results are shown in Table 54.

Table 54: Endosulfan residues in cauliflower from trials conducted in Australia 2000.

Trial site, Year,	Application	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	SO <sub>4</sub>	Total
Medina, WA, 2000	66.5 g ai/hL	3	0	Cauliflower <sup>®</sup>	0.094	0.066	0.007	0.17
(Galacia)	(166 – 219 g ai/		3		0.084	0.046	0.011	0.14
	ha)		7		0.05	0.038	0.016	0.10
			14		0.029	0.028	0.035	0.092
	133 g ai/hL	3	0	Cauliflower	0.54	0.35	0.014	0.90
	(332 – 439 g ai/		3		0.054	0.04	0.013	0.11
	ha)		7		0.052	0.038	0.019	0.11
			14		0.047	0.027	0.042	0.12
			7	Control	0.028	0.008	< 0.005	0.036
Werribee, VIC,	66.5 g ai/hL	3	0	Cauliflower@	0.042	0.025	0.020	0.087
2000 (Chaser)	(315 g ai/ha)		3		0.039	0.029	0.006	0.074
			7		0.010	0.006	< 0.005	0.016
			14		< 0.005	< 0.005	< 0.005	< 0.005
	133 g ai/hL	3	0	Cauliflower	0.23	0.13	0.005	0.36
	(630 g ai/ha)		3		0.11	0.074	0.012	0.20
			7		0.046	0.040	0.008	0.094
			14		0.006	< 0.005	< 0.005	0.011

 $<sup>\</sup>odot$  Florets removed from trimmed heads for bagging.  $\odot$  Cauliflower removed from stalk then cut into quarters before bagging. LOD = 0.005 mg/kg; LOQ = 0.05 mg/kg.

Table 55: Recoveries of endosulfan in fortified cauliflower.

Sample	Fortification (mg/kg)		% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Cauliflower	0.05	118	94	92				
	0.5	118	95	85				

Trials to Determine the Level of Endosulfan in Cabbage at Harvest Following Three Applications to the Crop. Report No. 1/5/564, Protocol No. ECR564, May 2001. Determination of Endosulfan Residues in Cabbage, State Chemistry Laboratory Report No. 0011010 & 0012094.

In two trials conducted in Victoria and Queensland, an endosulfan EC formulation was applied to cabbage at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ). Three sprays were applied at 10 - 13 day intervals, with the final spray applied at 7 days before harvest. Sprays were applied by hand held boom or mini boom and plot sizes were  $1.5 \times 13.3$ m and  $50 \times 1.25$ m in Qld and Victoria, respectively,

with a single replication at each site. Cabbage heads were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for 10 months prior to analysis. The results are shown in Table 56.

Table 56: Endosulfan residues in cabbage from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)	_	α	β	$SO_4$	Total
Darling Downs, Qld	66.5 g ai/hL	3	0	Cabbage	0.31	0.20	0.019	0.53
2000 (Neptune)	(665 g ai/ha)		3		0.13	0.15	0.035	0.32
	_		7		0.026	0.029	0.043	0.098
			14		0.007	0.007	0.038	0.052
	133 g ai/hL	3	0	Cabbage	0.84	0.49	0.041	1.4
	(1330 g ai/ha)		3		0.21	0.24	0.062	0.51
			7		0.092	0.10	0.11	0.30
			14		0.014	0.014	0.045	0.073
Werribee, VIC,	66.5 g ai/hL	3	0	Cabbage	0.035	0.023	0.006	0.064
2000 (Green	(332 g ai/ha)		3		0.008	0.010	0.008	0.026
coronet)	_		7		0.006	0.008	0.017	0.031
			14		< 0.005	< 0.005	< 0.005	< 0.005
	133 g ai/hL	3	0	Cabbage	0.033	0.019	0.006	0.058
	(665 g ai/ha)		3		0.012	0.011	0.007	0.030
			7		0.006	0.012	0.008	0.026
			14		< 0.005	< 0.005	< 0.005	< 0.005

LOD = 0.005 mg/kg; LOQ = 0.05 mg/kg.

Table 57: Recoveries of endosulfan in fortified cabbage.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Cabbage	0.05	95	100	98		
	0.5	88	99	95		

Trials to Determine the Level of Endosulfan in Brussels sprouts at Harvest Following Three Applications to the Crop. Report No. 1/9/553, Protocol No. ECR553, September 2001. Determination of Endosulfan Residues in Brussels sprouts, State Chemistry Laboratory Report No. 0008260, 0008275, 0009078 & 0103090.

In two trials conducted in Victoria and South Australia, an endosulfan EC formulation was applied to Brussels sprouts at spray concentrations of 66.5 or 133 g ai/hL ( $1\times$  or  $2\times$  spray conc.;  $0.8\times-3.6\times$  application rate). Three sprays were applied at 10 day intervals, with the final spray applied at 7 days before harvest. Sprays were applied by backpack sprayer or mini boom and plot sizes were  $3\times10$ m and  $3.6\times10$ m in SA and Victoria, respectively, with a single replication at each site. Sprouts (buttons) were sampled at 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for 9 months prior to analysis. The results are shown in Table 58.

Table 58: Endosulfan residues in Brussels sprouts from trials conducted in Australia 2000/2001.

Trial site, Year,	ial site, Year, Application		WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Gumeracha, SA,	66.5 g ai/hL	3	0	Brussels sprout	6.0	4.1	0.22	10
2001 (Ariston)	(1330 g ai/ha)		3		1.6	1.6	0.22	3.4
	_		7		0.81	0.92	0.20	1.9
			14		0.53	0.64	0.18	1.4
	133 g ai/hL	3	0	Brussels sprout	6.8	4.7	0.23	12
	(2660 g ai/ha)		3		1.9	2.0	0.19	4.1
			7		2.0	2.2	0.27	4.8
			14		0.98	1.3	0.22	2.5
Cranbourne, VIC,	66.5 g ai/hL	3	0	Brussels sprout	0.10	0.093	0.063	0.26
2000 (Roger)	(582 g ai/ha)		3		0.13	0.14	0.061	0.33
			7		0.034	0.055	0.048	0.14

			14		0.014	0.032	0.044	0.090
1	133 g ai/hL	3	0	Brussels sprout	0.30	0.28	0.094	0.67
	(1163 g ai/ha)		3	_	0.11	0.16	0.053	0.32
			7		0.070	0.12	0.072	0.26
			14		0.023	0.052	0.053	0.13

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

It should be noted that rates approximating  $2\times$  and  $4\times$  the label rates were employed in the SA trials.

Table 59: Recoveries of endosulfan in fortified Brussels sprouts.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Brussels sprouts	0.02	92	88	98			
	0.5	67	83	58			

# 8.5.7 Cucurbits (cucumber, melons, zucchini)

Reports from several overseas field trials in musk melons were provided in summary form, with little detail of field or analytical conditions. The data are shown in Table 60 below.

Table 60: Endosulfan residues in melons from trials conducted in Spain and Italy in 1994.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	-	α	β	SO <sub>4</sub>	Total
Sevilla Spain, 1994	565 ①	3 (7,7)	0	Whole fruit				< 0.05
(Rixan)	(141 – 188 g			Pulp	< 0.05	< 0.05	0.095	0.10
	ai/hL)			Peel	< 0.05	< 0.05	< 0.05	< 0.05
	ĺ		3	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.096	0.10
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
			7	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.095	0.10
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
			14	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.115	0.12
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
			21	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
			29	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
Carmona Spain,	565 ©	3 (7,7)	0	Whole fruit				0.19
1994 (Daimiel)	(141 g ai/hL)			Pulp	< 0.05	< 0.05	0.08	< 0.08
				Peel	0.1	0.1	0.09	0.29
			3	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	< 0.05	< 0.05	0.09	0.09
			7	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	< 0.05	0.06	0.08	0.17
La Rinconada Spain	565 ③	3 (7,7)	0	Whole fruit				< 0.05
1994 (Daimiel)	(141 g ai/hL)			Pulp	< 0.05	< 0.05	0.07	0.07
				Peel	< 0.05	< 0.05	0.07	0.07
			3	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	0.08	< 0.05	0.06	0.06
			7	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	0.09	0.09
				Peel	< 0.05	< 0.05	0.07	0.07
S. Pietro in Casale,	565 ⊕	3 (7,7)	0	Whole fruit				0.46
Italy, 1994	(56.5 g ai/hL)			Pulp	< 0.05	< 0.05	< 0.05	< 0.05
(Tamaris)				Peel	0.43	0.29	< 0.05	0.75
			3	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	< 0.05	< 0.05
	ĺ			Peel	0.11	0.1	< 0.05	0.22

			7	Whole fruit				< 0.05
				Pulp	< 0.05	< 0.05	< 0.05	< 0.05
				Peel	0.11	0.14	< 0.05	0.29
			14	Whole fruit				
				Pulp				
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
			21	Whole fruit				
				Pulp				
				Peel	< 0.05	< 0.05	< 0.05	< 0.05
Ravarino Italy, 1994	565 ©	3 (7,7)	0	Whole fruit				0.47
(Calipso)	(56.5 g ai/hL)			Pulp	< 0.05	< 0.05	< 0.05	< 0.05
				Peel	0.47	0.5	< 0.05	1.01
			3	Whole fruit				0.22
				Pulp	< 0.05	< 0.05	< 0.05	< 0.05
				Peel	0.2	0.26	< 0.05	0.48
			7	Whole fruit				0.19
				Pulp	< 0.05	< 0.05	< 0.05	< 0.05
				Peel	0.16	0.28	0.05	0.49

LOQ = 0.05 mg/kg.

- ① Last spray at fruit development; sampling at fruit development (BBCH 70)
- ② Last spray at fruit development; sampling at start of fruit ripening (BBCH 70 80)
- ③ Last spray at fruit development; sampling at start of fruit ripening (BBCH 70 − 80)
- 4 Last spray at beginning of harvest; sampling at start of drying off (BBCH 81-89).
- ⑤ Last spray at beginning of harvest; sampling at start of drying off (BBCH 80 − 82).

Table 61: Mean recoveries of endosulfan in fortified melons

Sample	Fortification (mg/kg)		% Recovery	
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>
Pulp	0.1 - 0.5	90	98	98
Peel	0.05 - 1	83	93	95

Trials to Determine the Level of Endosulfan in Rockmelons at Harvest Following Four Applications to the Crop. Report No. 1/10/551, Protocol No. ECR551, October 2001. Determination of Endosulfan Residues in Rockmelons, State Chemistry Laboratory Report No. 0103148 & 0107094.

In two trials conducted in Victoria and Queensland, an endosulfan EC formulation was applied to rockmelon plants at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$  spray conc.;  $0.3 \times -2.1 \times$  application rate). Four sprays were applied at intervals of 14 days, with the final spray applied to mature fruit, or 7 days before harvest. Sprays were applied by hand held boom and plot sizes were  $12 \times 2m$  and  $2m^2$  in VIC and QLD, respectively, with a single replication at each site. Melons were sampled at 0, 3, 5 and 7 days after application. Samples taken at 0, 3 and 5 days from the Qld site were not analysed as the fruit were underdeveloped. Samples were stored at  $-20^{\circ}$  C for up to 7 months prior to analysis. The results are shown in Table 62.

Table 62: Endosulfan residues in rockmelons from trials conducted in Australia 2001.

Trial site, Year,	ar, Application		WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Kialla West, VIC,	66.5 g ai/hL	4	0	Rockmelon	0.39	0.28	0.032	0.70
2001 (Hiline)	(778 g ai/ha)		3		0.25	0.27	0.034	0.55
			5		0.12	0.11	0.037	0.27
			7		0.089	0.097	0.045	0.23
	133 g ai/hL	4	0		0.89	0.90	0.040	1.8
	(1556 g ai/ha)		3		0.26	0.32	0.047	0.63
			5		0.34	0.40	0.059	0.80
			7		0.13	0.16	0.060	0.35
			7	Control	< 0.005	< 0.005	0.011	0.011
Fernvale, QLD,	66.5 g ai/hL	4	0	Rockmelon <sup>①</sup>	0.30	0.26	0.13	0.69
2001 (Planters	(222 g ai/ha)		3		0.42	0.35	0.21	0.98
Jumbo)			5		0.23	0.30	0.70	1.2
			7		0.35	0.36	0.29	1.0
	133 g ai/hL	4	7	Rockmelon <sup>®</sup>	0.10	0.21	0.74	1.0

	(505 g ai/ha)	7	Control	0.006	< 0.005	0.014	0.02	

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 63: Recoveries of endosulfan in fortified rockmelons.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Rockmelon	0.02	80	104	103			
	0.5	97	99	98			

Trials to Determine the Level of Endosulfan in Cucumbers at Harvest Following Four Applications to the Crop. Report No. 1/10/547, Protocol No. ECR547, October 2001. Determination of Endosulfan Residues in Cucumbers, State Chemistry Laboratory Report No. 0105222 & 0105236.

In two trials conducted in Queensland and New South Wales, an endosulfan EC formulation was applied to cucumber plants at spray concentrations of 66.5 or 133 g ai/hL ( $1\times$  or  $2\times$  spray conc.;  $0.3\times-0.76\times$  application rate). Four sprays were applied at 12-16 day intervals, with the final spray applied to maturing fruit, or 7 days before harvest. Sprays were applied by hand held boom and plot sizes were  $10\times2$ m and  $1\times20$ m in NSW and Qld, respectively, with a single replication at each site. Cucumbers were sampled at 0, 3, 5 and 7 days after application. Samples were stored at  $-20^{\circ}$  C for 8 months prior to analysis. The results are shown in Table 64.

Table 64: Endosulfan residues in cucumbers from trials conducted in Australia 2000/2001.

Trial site, Year,	Application	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Darlington Pt, NSW	66.5 g ai/hL	4	0	Cucumber	0.065	0.056	0.028	0.15
2001 (Coolah)	(168 – 280 g		3		0.047	0.027	0.035	0.11
	ai/ha)		5		0.030	0.017	0.029	0.076
			7		0.051	0.031	0.035	0.12
	133 g ai/hL	4	0	Cucumber	0.31	0.20	0.054	0.56
	(336 - 560  g)		3		0.12	0.11	0.049	0.28
	ai/ha)		5		0.059	0.037	0.034	0.13
			7		0.14	0.11	0.082	0.33
Lowood, Qld, 2000,	66.5 g ai/hL	4	0	Cucumber	0.058	0.037	0.042	0.14
(Warner)	(150 g ai/ha)		3		0.028	0.017	0.034	0.079
			5		0.029	0.017	0.036	0.082
			7		0.031	0.021	0.042	0.094
	133 g ai/hL	4	0	Cucumber	0.071	0.044	0.050	0.16
	(300 g ai/ha)		3		0.041	0.024	0.054	0.12
			5		0.039	0.024	0.050	0.11
			7		0.044	0.032	0.056	0.13
				Control	0.007	< 0.005	< 0.005	0.012

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 65: Recoveries of endosulfan in fortified cucumbers.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Cucumber	0.02	99	93	94			
	0.5	88	97	93			

Trials to Determine the Level of Endosulfan in Zucchini at Harvest Following Four Applications to the Crop. Report No. 1/10/556, Protocol No. ECR556, October 2001. Determination of Endosulfan Residues in Zucchini, State Chemistry Laboratory Report No. 0012108, 0012109, 0102271 & 0105001.

In four trials conducted in Queensland, New South Wales and Western Australia, an endosulfan EC formulation was applied to zucchini plants at spray concentrations of 66.5 or 133 g ai/hL (1× or 2×

① One unit (500 g) sample collected only.

spray conc.;  $0.2 \times -0.9 \times$  application rate). Four sprays were applied at 13-15 day intervals, with the final spray applied to maturing fruit, or 7 days before harvest. Sprays were applied by hand held boom and plot sizes were  $1 \times 20$ m and  $10 \times 2$ m in Qld,  $10 \times 2$ m in NSW and  $1 \times 20$ m in WA, with a single replication at each site. Zucchini were sampled at 0, 3, 5 and 7 days after application. Samples were stored at  $-20^{\circ}$  C for up to 11 months prior to analysis. The results are shown in Table 66.

Table 66: Endosulfan residues in zucchini from trials conducted in Australia 2000/2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	mg/kg)		
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total		
Koraleigh, NSW,	66.5 g ai/hL	4	0	Zucchini	0.078	0.036	0.046	0.16		
2001, (Regal black)	(149 g ai/ha)		3		0.030	0.021	0.039	0.090		
			5		0.023	0.012	0.032	0.067		
			7		0.042	0.014	0.032	0.088		
	133 g ai/hL	4	0	Zucchini	0.069	0.038	0.019	0.013		
	(298 g ai/ha)		3		0.026	0.011	0.025	0.062		
	_		5		0.018	0.009	0.023	0.050		
			7		0.026	0.005	0.033	0.064		
				Control	0.007	< 0.005	< 0.005	0.012		
Walkamin Qld,	66.5 g ai/hL	4	0	Zucchini	0.028	0.13	0.038	0.20		
2000, (Regal black)	(205 - 324 g)		3		0.011	0.005	0.039	0.055		
	ai/ha)		5		0.011	< 0.005	0.034	0.045		
			7		0.005	< 0.005	0.032	0.037		
	133 g ai/hL	4	0	Zucchini	0.11	0.083	0.046	0.24		
	(300 g ai/ha)		3		0.006	0.005	0.019	0.030		
	_		5		0.008	0.005	0.019	0.032		
			7		0.008	0.008	0.019	0.035		
			7	Control	0.018	0.005	0.095	0.12		
Malanda, QLD,	66.5 g ai/hL	4	0	Zucchini	0.085	0.035	0.12	0.24		
2000 (Gold finger)	(177 – 344 g		3		0.021	0.007	0.059	0.087		
	ai/ha)		5		0.013	< 0.005	0.046	0.059		
			7		0.012	< 0.005	0.068	0.080		
	133 g ai/hL	4	7	Zucchini	0.016	< 0.005	0.058	0.074		
	(353 – 689 g		7	Control	< 0.005	< 0.005	0.069	0.069		
	ai/ha)									
Wattleup, WA,	66.5 g ai/hL	4	0	Zucchini	0.17	0.10	0.015	0.28		
2000 (Regal black)	(173 g ai/ha)		3		0.028	0.009	0.012	0.049		
			5		0.021	0.005	0.012	0.038		
			7		0.015	< 0.005	0.010	0.025		
	133 g ai/hL	4	7	Zucchini	0.019	< 0.005	0.011	0.030		
	(346 g ai/ha)									

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 67: Recoveries of endosulfan in fortified zucchini.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Zucchini	0.02	76	97	125			
	0.5	92	101	58			

### 8.5.8 Fruiting vegetables (capsicum, eggplant, tomato, sweet corn)

Trials to Determine the Level of Endosulfan in Capsicums at Harvest Following Three Applications to the Crop. Report No. 1/10/559, Protocol No. ECR559, October 2001. Determination of Endosulfan Residues in Capsicums, State Chemistry Laboratory Report No. 0012104, 0103081, 0103149 & 0103229.

In four trials conducted in Queensland, Victoria and South Australia, an endosulfan EC formulation was applied to capsicums at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 14 days, with the final spray applied at 7 days before harvest to mature fruit. Sprays were applied by hand held boom or back pack mister and plot sizes were

 $30 \times 2m$ ,  $3 \times 20m$  in Qld,  $1 \times 15m$  in Victoria and 7.5 m<sup>2</sup> in South Australia, with a single replication at each site. Capsicums were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for almost 12 months prior to analysis. The results are shown in Table 68.

Table 68: Endosulfan residues in capsicums from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Emerald Creek, Qld,	735 g ai/ha	3	0	Capsicums	0.076	0.098	0.012	0.19
2000 (Merlin)			3		0.056	0.087	0.012	0.16
			7		0.058	0.091	0.018	0.17
			14		0.066	0.11	0.18	0.36
	1470 g ai/ha	3	0	Capsicums	0.21	0.29	0.018	0.52
			3		0.065	0.10	0.013	0.18
			7		0.18	0.27	0.048	0.50
			14		0.065	0.14	0.032	0.24
Gumlu, Qld, 2000	735 g ai/ha	3	7	Capsicums	0.02	0.053	0.016	0.089
(Airies)			14	_	0.019	0.039	0.016	0.074
Shepparton, VIC,	735 g ai/ha	3	7	Capsicums	0.009	0.018	0.010	0.037
2000 (Target)			14	_	0.006	0.014	0.007	0.027
Virginia, SA, 2000	735 g ai/ha	3	0	Capsicums	0.36	0.39	0.13	0.88
(Yaspo)			3	_	0.054	0.13	0.22	0.40
			7		0.013	0.023	0.039	0.075
			14		< 0.005	< 0.005	0.006	0.006
	1470 g ai/ha	3	0	Capsicums	0.32	0.30	0.061	0.68
	_		3	_	0.037	0.10	0.13	0.27
			7		< 0.005	0.013	0.05	0.063
			14		0.005	0.014	0.015	0.034

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 69: Recoveries of endosulfan in fortified capsicums.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Capsicums	0.02	64	66	57			
	0.5	96	118	103			

Trials to Determine the Level of Endosulfan in Tomatoes at Harvest Following Three Applications to the Crop. Report No. 1/10/552, Protocol No. ECR552, October 2001. Determination of Endosulfan Residues in Tomatoes, State Chemistry Laboratory Report No. 0012107, 0103146, 0103147 & 0105232.

In four trials conducted in Queensland, Victoria and New South Wales, an endosulfan EC formulation was applied to tomatoes at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 14 days, with the final spray applied at fruiting. Sprays were applied by hand held boom and plot sizes were  $2 \times 10$ m,  $6 \times 1$ m in Qld,  $12 \times 1$ m in Victoria and  $30 \times 1$ m in New South Wales, with a single replication at each site. Tomatoes were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for almost 12 months prior to analysis. The results are shown in Table 70.

Table 70: Endosulfan residues in tomatoes from trials conducted in Australia 2000.

Trial site, Year,	, Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Walkamin, Qld,	735 g ai/ha	3	0	Tomatoes	0.037	0.045	0.007	0.089
2000 (Zola)			3		0.013	0.037	0.006	0.056
			7		0.005	0.021	0.009	0.035
			14		< 0.005	0.008	0.007	0.015
	1470 g ai/ha	3	0	Tomatoes	0.054	0.064	0.006	0.12
			3		0.025	0.074	0.01	0.11
			7		0.007	0.028	0.01	0.045

			14		0.006	0.033	0.014	0.053
Caffey, Qld, 2000	735 g ai/ha	3	7	Tomatoes	0.044	0.032	0.007	0.083
(Thunder)			14		< 0.005	< 0.005	< 0.005	< 0.005
Goulburn Valley,	735 g ai/ha	3	0	Tomatoes	0.027	0.026	0.006	0.059
VIC, 2000			3		0.025	0.035	0.009	0.069
(Granades)			7		0.007	0.013	0.007	0.027
			14		0.009	0.016	0.013	0.038
	1470 g ai/ha	3	0	Tomatoes	0.072	0.061	0.012	0.14
			3		0.020	0.033	0.009	0.062
			7		0.016	0.032	0.018	0.066
			14		0.014	0.025	0.018	0.057
Whitton, NSW,	735 g ai/ha	3	0	Tomatoes	0.037	0.044	< 0.005	0.081
2000 (Early nema			3		0.032	0.053	0.009	0.094
pride)			7		0.030	0.052	0.008	0.090
			14		0.009	0.011	< 0.005	0.020

LOD = 0.005 mg/k; LOQ = 0.02 mg/kg.

Table 71: Recoveries of endosulfan in fortified tomatoes.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Tomatoes	0.02	91, 96	102, 118	93, 98		
	0.5	93, 99	103, 116	95, 99		

Trials to Determine the Level of Endosulfan in Eggplant at Harvest Following Three Applications to the Crop. Report No. 1/10/540, Protocol No. ECR540, October 2001. Determination of Endosulfan Residues in Eggplant, State Chemistry Laboratory Report No. 0103153, 0105002, 0105223 & 0109043.

In four trials conducted in Queensland, Victoria and New South Wales, an endosulfan EC formulation was applied to eggplant at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 13-14 days, with the final spray applied at maturity to early harvest. Sprays were applied by hand held boom and plot sizes were  $1 \times 10$ m,  $1 \times 30$ m and  $4 \times 16$ m in Qld,  $1 \times 15$ m in Victoria and  $10 \times 2$ m in NSW, with a single replication at each site. Eggplant were sampled at intervals of 0, 0, 00 and 014 days after application. Samples were stored at 020 C for 07 months prior to analysis. The results are shown in Table 02.

Table 72: Endosulfan residues in eggplant from trials conducted in Australia 2001.

Trial site, Year,	Applicati	ion	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Koraleigh, NSW,	735 g ai/ha	3	0	Eggplant	0.034	0.030	< 0.005	0.064
2001, (Grace)			3		< 0.005	0.007	< 0.005	0.012
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
Glasshouse Mtns,	735 g ai/ha	3	0	Eggplant	0.33	0.21	0.026	0.57
QLD, 2001 (Venus)			3		0.043	0.062	0.031	0.14
			7		0.014	0.029	0.012	0.055
			14		0.007	0.016	0.018	0.041
	1470 g ai/ha	3	7	Eggplant	0.058	0.081	0.046	0.18
			7	Control	0.008	0.009	< 0.005	0.017
Shepparton, VIC,	735 g ai/ha	3	0	Eggplant	0.015	0.011	0.006	0.032
2001, (Black pearl)			3		< 0.005	< 0.005	< 0.005	< 0.005
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	0	Eggplant	0.012	0.015	< 0.005	0.027
			3		< 0.005	0.007	< 0.005	0.007
			7		< 0.005	< 0.005	0.006	0.006
			14		< 0.005	< 0.005	< 0.005	< 0.005
Gumlu, QLD, 2001,	735 g ai/ha	3	0	Eggplant	0.014	0.014	< 0.005	0.028
(Black pearl)			3		< 0.005	< 0.005	< 0.005	< 0.005

		7	< 0.005	< 0.005	< 0.005	< 0.005	
		14	< 0.005	< 0.005	< 0.005	< 0.005	

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 73: Recoveries of endosulfan in fortified eggplant.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Eggplant	0.02	84	107	83			
	0.5	90	93	88			

Trials to Determine the Level of Endosulfan in Sweet Corn at Harvest Following Three Applications to the Crop. Report No. 1/10/560, Protocol No. ECR560, October 2001. Determination of Endosulfan Residues in Sweet Corn, State Chemistry Laboratory Report No. 0102084, 0104037 & 0105231.

In three trials conducted in Queensland, Victoria and New South Wales, an endosulfan EC formulation was applied to sweet corn at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 7 – 9 days, with the final spray applied to semi-mature to mature cobs. Sprays were applied by hand held boom and plot sizes were 7 × 4m, 2 × 25m and 2 × 10m in Qld, Victoria and NSW, respectively, with a single replication at each site. Corn cobs

were sampled at intervals of 0, 3, 7 and 14 days after application. Samples were stored at  $-20^{\circ}$  C for 10 months prior to analysis. The results are shown in Table 74.

Table 74: Endosulfan residues in sweet corn from trials conducted in Australia 2000/2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Mulgowie, QLD,	735 g ai/ha	3	0	Corn cobs <sup>①</sup>	< 0.005	< 0.005	< 0.005	< 0.005
2000 (Golden			3		< 0.005	< 0.005	< 0.005	< 0.005
sweet)			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	7	Corn cobs	< 0.005	< 0.005	< 0.005	< 0.005
Warragal, VIC,	735 g ai/ha	3	0	Corn cobs	< 0.005	< 0.005	< 0.005	< 0.005
2001 (Honeysweet)			3		< 0.005	< 0.005	< 0.005	< 0.005
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	7	Corn cobs	< 0.005	< 0.005	< 0.005	< 0.005
Koraleigh, NSW,	735 g ai/ha	3	0	Corn cobs	< 0.005	< 0.005	< 0.005	< 0.005
2001 (Golden			3		< 0.005	< 0.005	< 0.005	< 0.005
sweet)			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	0	Corn cobs	< 0.005	< 0.005	< 0.005	< 0.005
			3		< 0.005	< 0.005	< 0.005	< 0.005
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 75: Recoveries of endosulfan in fortified sweet corn.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Sweet corn	0.02	64	98	75			
	0.5	81	88	87			

① Outer covering leaves and base of stalk were removed prior to bagging for transport.

#### 8.5.9 Leafy vegetables (chinese cabbage, silverbeet, lettuce)

Trials to Determine the Level of Endosulfan in Chinese Cabbage at Harvest Following Three Applications to the Crop. Report No. 1/10/549, Protocol No. ECR549, October 2001. Determination of Endosulfan Residues in Chinese Cabbage, State Chemistry Laboratory Report No. 0009125, 0009230, 0009237, 0010029 & 0104095.

In two trials conducted in Victoria and Queensland, an endosulfan EC formulation was applied to chinese cabbage at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ; 120 - 465 g ai/ha). Three sprays were applied at intervals of 10 - 14 days, with the final spray applied to seedlings. Sprays were applied by hand held boom and plot sizes were  $3 \times 12$ m and  $1 \times 5 - 10$ m (seedling trays) in Victoria and Queensland, respectively, with a single replication at each site. Whole plants were sampled when the crops were at the seedling stage or 30 cm high (depending on variety) at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 12 months prior to analysis. The results are shown in Table 76.

Table 76: Endosulfan residues in chinese cabbage from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Heatherton, VIC,	66.5 g ai/hL	3	0	Chinese	1.8	1.1	0.48	3.4
2000 (Bok choi,	(465 g ai/ha)		7	cabbage	0.24	0.24	0.54	1.0
Joi choi)			14		0.033	0.047	0.26	0.34
			21		0.012	0.017	0.23	0.26
				Control	0.008	0.005	0.012	0.025
Atherton, Qld,2000	66.5 g ai/hL	3	0	Chinese	18	10	1.1	29
(Bok choi)	(120 g ai/ha)		7	cabbage	0.60	1.1	1.9	3.6
			14		0.057	0.051	0.12	0.23
			21		0.036	0.047	0.17	0.25
	133 g ai/hL	3	0	Chinese	32	19	1.4	52
			7	cabbage	1.5	2.7	3.1	7.3
			14		0.056	0.060	0.11	0.23
			21		0.027	0.031	0.097	0.16
				Control	0.011	0.012	0.006	0.029

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 77: Recoveries of endosulfan in fortified bok choi.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Bok choi	0.02	107	137	125			
	0.5	92	85	94			

Trials to Determine the Level of Endosulfan in Silverbeet at Harvest Following Three Applications to the Crop. Report No. 1/10/546, Protocol No. ECR546, October 2001. Determination of Endosulfan Residues in Silverbeet, State Chemistry Laboratory Report No. 0009126, 0009231, 0010030 & 0107095.

In two trials conducted in Queensland and Victoria, an endosulfan EC formulation was applied to silverbeet at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ; 222 - 931 g ai/ha). Three sprays were applied at intervals of 14 days, with the final spray applied to plants that were 30 cm high. Sprays were applied by hand held boom and plot sizes were  $3 \times 12$ m and  $1 \times 5 - 10$ m (seedling trays) in Victoria and Queensland, respectively, with a single replication at each site. Whole plants were sampled at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 12 months prior to analysis. The results are shown in Table 78.

Table 78: Endosulfan residues in silverbeet from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	SO <sub>4</sub>	Total
Heatherton, VIC,	66.5 g ai/hL	3	0	Silverbeet	3.4	2.4	0.27	6.1
2000 (Silverstar)	(465 g ai/ha)		7	(leaves)	0.46	0.64	0.45	1.6
			14		0.31	0.59	0.66	1.6
			21		0.069	0.17	0.32	0.56
	133 g ai/hL	3	0	Silverbeet	10	6.3	0.72	17
	(931 g ai/ha)		7	leaves	1.1	1.9	0.89	3.9
			14		0.25	0.52	0.65	1.4
			21		0.09	0.24	0.34	0.67
			0	Control	0.033	0.020	0.014	0.067
Fernvale, Qld,2001	66.5 g ai/hL	3	0	Silverbeet	9.0	6.6	2.2	18
(Fordhook giant)	(222 g ai/ha)		7	leaves	0.63	1.4	1.7	3.7
			14		0.052	0.31	1.0	1.4
			21		0.017	0.041	0.25	0.31
				Control	0.005	0.005	0.015	0.025

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 79: Recoveries of endosulfan in fortified silverbeet.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Silverbeet	0.02	90	133	166			
	0.5	84	91	79			

Trials to Determine the Level of Endosulfan in Leafy Lettuce at Harvest Following Three Applications to the Crop. Report No. 1/10/555, Protocol No. ECR555, October 2001. Determination of Endosulfan Residues in Leafy Lettuce, State Chemistry Laboratory Report No. 0009019, 0102207, 0105043 & 0105228.

In four trials conducted in Queensland, Victoria and New South Wales, an endosulfan EC formulation was applied to leafy lettuces at spray concentrations of 66.5 or 133 g ai/hL ( $1 \times$  or  $2 \times$ ; 222 - 931 g ai/ha). Three sprays were applied at intervals of 14 - 17 days, with the final spray applied to plants that were at the head forming stage or 20 - 25 cm in diameter. Sprays were applied by hand held boom and plot sizes were  $50 \times 1.25$ m in VIC,  $2 \times 20$ m and 80m<sup>2</sup> in NSW and  $4 \times 6$ m (2X) in Queensland, with a single replication at each site. Whole plants were sampled at 0, 7, 14 and 21/28 days after application. Samples were stored at  $-20^{\circ}$  C for up to 11 months prior to analysis. The results are shown in Table 80.

Table 80: Endosulfan residues in leafy lettuce from trials conducted in Australia 2000/2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray conc.	No.	(days)		α	β	$SO_4$	Total
Werribee, VIC,	66.5 g ai/hL	3	0	Lettuce	1.6	1.5	0.31	3.4
2000 (Green oak)	(255 g ai/ha)		7		0.34	0.33	0.33	1.0
			14		0.15	0.14	0.19	0.48
			28		0.04	0.04	0.09	0.17
	133 g ai/hL	3	0	Lettuce	8.6	6.3	0.80	16
	(514 g ai/ha)		7		1.4	1.8	0.69	3.9
			14		0.54	0.68	0.53	1.8
			28		0.12	0.15	0.23	0.50
			14	Control	0.01	0.018	0.024	0.052
Agnes Banks, NSW,	66.5 g ai/hL	3	0	Lettuce	9.5	5.3	0.97	16
2000 (Red oak)	(312 – 623 g		7		0.61	0.63	0.84	2.1
	ai/ha)		14		0.26	0.29	0.65	1.2
			21		0.046	0.032	0.090	0.17
	133 g ai/hL	3	0	Lettuce	21	13	2.1	36
	(624 – 1246 g		7		1.5	1.9	1.4	4.8
	ai/ha)		14		0.27	0.28	0.52	1.1
			21		0.11	0.084	0.15	0.34

The lagoon, NSW,	66.5 g ai/hL	3	0	Lettuce	3.7	2.7	0.091	6.5
(Frill ice)	(239 – 479 g		7		0.096	0.12	0.20	0.42
	ai/ha)		14		0.008	0.016	0.024	0.048
			21		< 0.005	0.008	0.016	0.024
			28		0.016	0.008	0.018	0.042
Toowoomba, Qld,	66.5 g ai/hL	3	0	Lettuce	0.86	0.57	0.033	1.5
2001 (Nero cos)	(221 332 g		7		0.013	0.017	0.039	0.069
	ai/ha		14		< 0.005	0.005	0.020	0.025
			0	Control	0.022	0.008	0.012	0.042

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 81: Recoveries of endosulfan in fortified lettuce.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Lettuce	0.02	89	92	110			
	0.5	87	100	97			

# 8.5.10 Legume vegetables (beans, peas)

Reports from several overseas field trials in garden peas (Pisum sativum) were provided in summary form, with little detail of field or analytical conditions. The data are shown in Table 82 below.

Table 82: Endosulfan residues in garden peas (pods, peas and hulls) from trials conducted in France and Italy in 1994.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Marcellus, France	749 ①	3 (7,7)	0	Pea shoots	4	2.9	1.2	8.1
1994 (Cajou)	(250 g ai/hL)		7	Peas shoots	0.23	0.51	0.91	1.65
•			13	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
				Hulls	< 0.05	< 0.05	< 0.05	< 0.05
				Peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canned peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canning liquid	< 0.05	< 0.05	< 0.05	< 0.05
			21	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
Fourgues, France	780 ①	3 (7,7)	0	Pea shoots	3.4	2.7	1.6	7.7
1994 (Mona)	(250 g ai/hL)		7	Peas shoots	0.2	0.46	1.5	2.2
	_		13	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
				Hulls	< 0.05	< 0.05	0.08	0.08
				Peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canned peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canning liquid	< 0.05	< 0.05	< 0.05	< 0.05
			21	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
Anita, Italy, 1994	780 ①	3 (7,7)	0	Pea shoots	13	7	0.56	20.6
(Abador)	(250 g ai/hL)		7	Peas shoots	0.12	0.54	0.58	1.24
			14	Whole pods	< 0.05	< 0.05	0.06	0.06
				Hulls	< 0.05	< 0.05	< 0.05	< 0.05
				Peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canned peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canning liquid	< 0.05	< 0.05	< 0.05	< 0.05
			20	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
Conselice, Italy,	750 ①	3 (7,7)	0	Pea shoots	8.2	6.1	2.5	16.8
(Buget)	(250 g ai/hL)		7	Peas shoots	0.17	0.4	2.1	2.67
			14	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05
				Hulls	< 0.05	< 0.05	< 0.05	< 0.05
				Peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canned peas	< 0.05	< 0.05	< 0.05	< 0.05
				Canning liquid	< 0.05	< 0.05	< 0.05	< 0.05
			21	Whole pods	< 0.05	< 0.05	< 0.05	< 0.05

LOQ for hulls and peas = 0.05 mg/kg; LOQ for pods = 0.1 mg/kg.

① Last spray at beginning of fruit development; last sampling at harvest time for vining peas.

Table 83: Mean recoveries of endosulfan in fortified peas, shoots, pods and hulls.

Sample	α	-endosulfan		ndosulfan	endosulfan SO <sub>4</sub>		
	Fortification (mg/kg)	% Recovery	Fortification (mg/kg)	% Recovery	Fortification (mg/kg)	% Recovery	
Shoots	0.5 - 2.5	69	0.5 - 2.5	72	0.5 - 2.5	76	
Pods	0.1	87	0.1	83	0.1	82	
Hulls	0.05	77	0.05	75	0.05	90	
Peas	0.05	77	0.05	83	0.05	86	

Trials to Determine the Level of Endosulfan in Beans at Harvest Following Three Applications to the Crop. Report No. 1/10/538, Protocol No. ECR538, October 2001. Determination of Endosulfan Residues in Beans, State Chemistry Laboratory Report No. 0012103, 0103152 & 0104082.

In three trials conducted in Queensland, Victoria and Tasmania, an endosulfan EC formulation was applied to beans at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 13 – 15 days, with the final spray applied at flowering and fruit set. Sprays were applied by hand held boom or packback mister and plot sizes were  $0.6 \times 25 \text{m}$ ,  $10 \times 2 \text{m}$  and  $40 \times 1 \text{m}$  in Queensland, Tasmania and Victoria, respectively with a single replication. Beans were sampled at 0, 3, 7, 10 and 14 days after application. In the Qld trials, samples taken at 0 and 3 days were too immature and were not analysed. Samples were stored at –  $20^{\circ}$  C for up to 12 months prior to analysis. Residues in green beans are shown in Table 84.

Table 84: Endosulfan residues in green beans from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)	_	α	β	SO <sub>4</sub>	Total
Glen Alyn, Qld	735 g ai/ha	3	7	Beans (pods)	0.032	0.006	0.11	0.15
2000 (Festina)			10		0.015	0.005	0.062	0.082
			14		0.014	0.008	0.028	0.050
	1470 g ai/ha	3	7	Beans (pods)	0.18	0.12	0.58	0.88
			10		0.035	0.019	0.19	0.24
			14		0.034	0.022	0.11	0.17
			7	Control	0.11	0.010	0.007	0.13
Devonport, TAS,	735 g ai/ha	3	0	Beans (pods)	0.30	0.23	0.055	0.58
2000 (Montano)			3		0.081	0.066	0.090	0.24
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	7	Beans (pods)	0.050	0.033	0.11	0.19
Goulburn Valley,	735 g ai/ha	3	0	Beans (pods)	0.15	0.10	0.039	0.29
VIC, 2000 (Dwarf)	_		3		0.055	0.048	0.035	0.14
			7		0.022	0.021	0.049	0.092
			14		0.006	0.006	0.025	0.037

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 85: Recoveries of endosulfan in fortified beans.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Beans	0.02	105	103	90			
	0.5	80	77	76			

Trials to Determine the Level of Endosulfan in Peas at Harvest Following Three Applications to the Crop. Report No. 1/10/557, Protocol No. ECR557, October 2001. Determination of Endosulfan Residues in Peas, State Chemistry Laboratory Report No. 0012102, 0102117 & 0103028.

In three trials conducted in Queensland, Victoria and Tasmania, an endosulfan EC formulation was applied to peas at rates of 735 or 1470 g ai/ha ( $1 \times$  or  $2 \times$ ). Three sprays were applied at intervals of 14

days, with the final spray applied at pod filling to mature pods. Sprays were applied by mini boom and plot sizes were  $3 \times 30$ m,  $11 \times 6$ m and 80m<sup>2</sup> in Queensland, Tasmania and Victoria, respectively with a single replication. Peas were sampled at 0, 3, 7 and 14 days after application and pea hay was sampled at 7 and 14 days in Qld and at 28 days in Victoria. In the Victorian trial, the final sampling interval was 28 days after application and not 14 days. Samples were stored at  $-20^{\circ}$  C for up to 12 months prior to analysis. Residues in green peas (whole pods) and pea hay are shown in Table 86.

Table 86: Endosulfan residues in green peas (pods) and hay from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray rate	No.	(days)		α	β	$SO_4$	Total
Lockyer Valley,	735 g ai/ha	3	0	Peas (pods)	0.42	0.48	0.095	1.0
Qld, 2000 (Epic)	_		3		0.062	0.15	0.10	0.31
			7		0.009	0.026	0.047	0.082
			14		0.005	0.010	0.022	0.037
			7	Hay	0.42	1.6	1.1	3.1
			14		0.30	0.81	1.1	2.2
	1470 g ai/ha	3	0	Peas (pods)	1.5	1.4	0.20	3.1
			3		0.21	0.45	0.28	0.94
			7		0.06	0.11	0.24	0.41
			14		0.013	0.037	0.089	0.14
			7	Hay❶	0.81	3.5	3.8	8.1
			14		0.33	1.1	2.4	3.8
			7	Control hay	0.006	0.005	< 0.005	0.011
Devonport, TAS,	735 g ai/ha	3	0	Peas (pods)	0.46	0.55	0.05	1.1
2000 (Small sieve)			3		0.068	0.12	0.17	0.36
			7		0.015	0.022	0.087	0.12
			14		0.006	0.006	0.018	0.030
	1470 g ai/ha	3	7	Peas (pods)	0.033	0.10	0.20	0.33
Werribee, VIC,	735 g ai/ha	3	0	Peas (pods)	1.0	0.81	0.15	2.0
2000 (Melbourne			3		0.17	0.20	0.33	0.70
Market)			7		0.067	0.099	0.20	0.37
			28		< 0.005	0.005	0.008	0.013
			28	Hay <b>2</b>	0.015	0.041	0.066	0.12

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg for peas and pea hay.

Table 87: Recoveries of endosulfan in fortified peas (pods) and hay.

Sample	Fortification (mg/kg)	% Recovery α-endosulfan β-endosulfan lendosulfan SO4					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Peas	0.02	97	92	103			
	0.5	86	92	86			
Pea hay	0.02	88	89	87			
	0.5	103	96	100			

## 8.5.11 Pulse crops (navy beans, faba beans, cow peas, field peas, lupins, chickpeas)

Determination of Thiodan EC Residues in Navy Bean, AgrEvo Trial No ID98AUSE04/QD17-98, 12 January 2000. Determination of Endosulfan Residues in Navy Beans, State Chemistry Laboratory Report No. 0001086.

At a site in Laidley Qld, navy beans were treated with two applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). The application timings ranged from the 0.35 m crop (just prior to flowering) to early pod set stages; the intervals between sprays were 6 to 8 days. Individual plots were  $10m \times 5$  rows, with 4 replicate plots. The sprays were applied by air pressurised overhead boom. Samples of trash were collected 13 days earlier than the seed samples, as the crop matured slowly. Seed samples were collected at 61, 54, 48 and 41 days after application. Samples of trash were taken at 48, 41, 35 and 28

<sup>•</sup> Whole plants dried in the open. No indication of drying at the laboratory.

<sup>2</sup> No indication given of status of drying of the crop.

days after application. The interval between sample collection and analysis was 306 days; samples were stored at -20 °C prior to analysis.

Samples from replicate plots were analysed separately. Results are shown in Table 88.

Table 88: Residues in navy beans and trash following application of Thiodan EC at 735 g ai/ha (ID98AUSE04/QD17-98)

Trial site, Year,	Application	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Laidley, Qld, 1999	735	2	41	Navy bean	< 0.005	< 0.005	0.036	0.036
(Spearfelt)					< 0.005	< 0.005	0.046	0.046
					< 0.005	< 0.005	0.053	0.053
					< 0.005	< 0.005	0.031	0.031
			48	Navy bean	< 0.005	< 0.005	0.009	0.009
					< 0.005	< 0.005	0.013	0.013
					< 0.005	< 0.005	0.022	0.022
					< 0.005	< 0.005	0.013	0.013
			54	Navy bean	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.006	0.006
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.006	0.006
			61	Navy bean	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
			28	Bean trash	0.26	0.14	2.0	2.4 (15.00)
					0.27	0.20	1.4	1.9 (12.67)
					0.29	0.15	2.5	2.9 (16.11)
					0.14	0.06	0.94	1.1 (6.10)
			35	Bean trash	0.17	0.06	2.5	2.7 (15.00)
					0.11	0.05	1.4	1.6 (8.00)
					0.16	0.04	1.5	1.7 (9.44)
					0.15	0.07	1.6	1.8 (11.25)
			41	Bean trash	0.08	0.04	1.4	1.5 (7.89)
					0.04	0.02	0.74	0.80 (4.70)
					0.03	0.01	0.58	0.62 (4.42)
					0.07	0.03	1.5	1.6 (7.27)
			48	Bean trash	0.05	0.02	0.75	0.82 (4.31)
					0.05	0.02	0.84	0.91 (4.55)
					0.03	0.02	0.79	0.84 (4.42)
					0.03	0.01	0.53	0.57 (2.71)
				Controls	< 0.005	< 0.005	0.03	0.03 (0.16)
					< 0.005	< 0.005	0.02	0.02 (0.11)
					< 0.005	< 0.005	0.02	0.02 (0.09)
100 000 4	100 01 // 6			l	< 0.005	< 0.005	0.04	0.04 (0.19)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for beans and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 78–86% in trash.

Finite residues were present in one control seed sample (0.007 mg/kg  $\alpha$ -endosulfan) and in all of the control trash samples, at levels ranging 0.02 to 0.04 mg/kg total endosulfan.

Table 89: Recoveries of endosulfan in fortified navy bean samples (98AUSE04/QD17-98).

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Navy beans	0.1	73, 78	70, 80	69, 79				
Trash	0.1	101, 103	104, 108	100, 107				

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Determination of Endosulfan Residues in Navybean. Aventis Trial ID QD27-99, 18 December 2001. Determination of Endosulfan Residues in Navy Beans, State Chemistry Laboratory Report No. 0108134.

At a trial site in Laidley Qld, navy beans were treated with either one or two applications of Thiodan EC at a rate of 735 g ai/ha ( $1\times$ ). The application timings ranged from early flowering to early pod set (50 to 26 days before harvest); the intervals between sprays were 8 or 16 days.

Individual plots were 10m × 4 rows, with 4 replicate plots. The sprays were applied by air pressurised overhead boom. Samples of seed and trash were collected at 33 or 26 days after the final (or single spray). The interval between sample collection and analysis was 18 months; samples were stored at –20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed. Results are shown in Table 90.

Table 90: Residues in navy beans and trash following application of Thiodan EC at 735 g ai/ha (QD27-99)

Trial site, Year,	Application	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Laidley, Qld, 1999	735	2	33	Navy bean	< 0.005	< 0.005	0.026	0.026
(Spearfelt)					< 0.005	< 0.005	0.051	0.051
		2	26	Navy bean	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.040	0.040
		1	26	Navy bean	< 0.005	< 0.005	0.012	0.012
					< 0.005	< 0.005	0.018	0.018
		2	33	Bean trash	0.045	0.069	0.33	0.45 (0.65)
					0.14	0.16	0.81	1.1 (1.36)
		2	26	Bean trash	0.12	0.22	0.45	0.79 (1.27)
					0.25	0.46	0.78	1.5 (1.78)
		1	26	Bean trash	0.39	0.41	0.93	1.7 (2.36)
					0.80	1.3	2.7	4.8 (6.31)
				Control	< 0.005	0.006	0.024	0.03 (0.04)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for beans and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 16–38%.

Finite residues or 0.04 mg/kg were found in the untreated bean trash sample.

Table 91: Recoveries of endosulfan in fortified navy bean samples (98AUSE04/QD27-99).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Navy beans	0.1	106	112	119			
Trash	0.1	82	82	108			

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Faba Beans. One Trial, Bellata, New South Wales, Australia, 1999. Aventis Trial Number 9209, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Faba Beans, State Chemistry Laboratory Report No. 0010004.

In a trial at Bellata NSW, faba beans were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at flowering or pod filling. The trial plot was  $4m \times 10m$  and included four replications. Sprays were applied using a back pack with hand-held boom. Samples of forage from all treatments were collected at 62 days after post-emergent application, when the crop was at the  $1^{st}$  flower stage. Grain and straw samples were collected at harvest; 60 days after two applications or 196 days after a single application. The interval between sample collection and analysis was approximately 28 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed. Results are shown in Table 92.

Table 92: Residues in faba beans, forage and trash following application of Thiodan EC (9202)

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Bellata, NSW, 1999	175	1	62	Forage	< 0.005	< 0.005	0.008	0.008 (0.082)
(Barkool)					< 0.005	< 0.005	0.008	0.008 (0.104)
	350	1	62	Forage	< 0.005	< 0.005	0.009	0.009 (0.115)
					< 0.005	< 0.005	0.011	0.011 (0.10)
					< 0.005	< 0.005	0.009	0.009 (0.104)
					< 0.005	< 0.005	0.010	0.010 (0.088)
	175	1	196	Faba bean	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	196	Faba beans	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	60	Faba beans	< 0.005	< 0.005	0.028	0.028
					< 0.005	< 0.005	0.028	0.028
					< 0.005	0.054	0.046	0.10
					< 0.005	< 0.005	0.027	0.027
	175	1	196	Trash	< 0.005	< 0.005	0.014	0.014 (0.017)
					0.007	0.010	0.013	0.030 (0.033)
	350	1	196	Trash	< 0.005	0.013	0.018	0.031 (0.035)
					< 0.005	< 0.005	0.030	0.030 (0.034)
	350 + 735	1 + 1	60	Trash	0.017	0.24	0.27	0.53 (0.728)
					0.027	0.35	0.35	0.73 (0.847)
					0.069	0.58	0.45	1.1 (1.308)
					< 0.005	0.027	0.048	0.075 (0.084)
				Controls	0.005	0.006	0.007	0.018 (0.02)
					0.007	0.009	0.009	0.025 (0.028)

LOD = 0.05 mg/kg; LOQ = 0.1 mg/kg for beans, forage and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 88-92% in forage and 10-27% in trash samples.

Finite residues of 0.018 and 0.025 mg/kg total endosulfan were found in untreated controls. Recoveries in faba beans, forage and trash were within acceptable ranges.

Table 93: Recoveries of endosulfan in fortified faba bean samples (99AUSA02/9209).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Bean forage	0.1	84	87	86			
Faba beans	0.1	87	88	80			
Trash	0.1	89	87	89			

Determination of Thiodan EC Residues in Cow Peas. AgrEvo Trial No ID98AUSE04/QD16, 13 October 1999. Determination of Endosulfan Residues in Cow Pea, State Chemistry Laboratory Report No. 9907102.

At a site in Norwin Qld, cow peas were treated with two applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). Sprays were applied at 7 day intervals and application timings ranged from 8 weeks to 4 weeks before harvest. Individual plots were 8 rows  $\times$  20m, with 4 replications per treatment. The sprays were applied by hand-held boom. Samples of peas and trash were taken at 49, 42, 35 and 28 days after the final spray. The interval between sample collection and analysis was four months; samples were stored at 2 – 4 °C prior to analysis. Samples from replicate plots were analysed separately. Results are shown in Table 94.

Table 94: Residues in cow peas and trash following application of Thiodan EC at 735 g ai/ha (98AUSE04/QT16-98).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Norwin, Qld, 1999	735	2	49	Cow peas	< 0.005	0.005	0.081	0.086
(Big buff)				_	< 0.005	< 0.005	0.070	0.070
_					< 0.005	< 0.005	0.018	0.018
					< 0.005	0.006	0.092	0.098
		2	42	Cow peas	< 0.005	0.008	0.11	0.12
					< 0.005	0.005	0.094	0.099
					< 0.005	0.010	0.12	0.13
					< 0.005	< 0.005	0.083	0.083
		2	35	Cow peas	< 0.005	0.019	0.14	0.16
					< 0.005	0.018	0.14	0.16
					0.014	0.046	0.20	0.26
					0.008	0.039	0.17	0.22
		2	28	Cow peas	0.012	0.059	0.24	0.31
					0.014	0.061	0.25	0.32
					0.016	0.069	0.21	0.30
					0.016	0.072	0.26	0.35
	735	2	49	Trash	1.0	3.6	7.7	12 (14.3)
					0.95	3.9	7.2	12 (16.9)
					0.29	2.0	3.7	6.0 (7.7)
					0.75	3.8	6.5	11 (14.1)
		2	42	Trash	1.1	4.7	11	17 (20.7)
					1.6	5.5	10	17 (20.2)
					2.6	8.3	11	22 (25.6)
					0.74	3.9	7.2	12 (17.4)
		2	35	Trash	2.6	8.2	15	26 (30.3)
					1.9	6.0	11	19 (30.6)
					4.2	12	19	35 (46.0)
					2.5	6.8	12	21 (25.0)
		2	28	Trash	1.8	6.6	10	18 (28.6)
					3.4	9.6	13	26 (37.1)
					2.7	8.7	13	24 (34.8)
					2.0	6.3	9.6	18 (22.8)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for peas, and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged 14 - 44%.. Duplicate analyses of samples.

The data show that total endosulfan residues in cow pea trash range from 7.7 to 46 mg/kg dry weight following treatment from 7 to 4 weeks before harvest. The highest residues are observed for treatments applied between 5 and 4 weeks before harvest. Values for individual replicate samples vary, for examples there is a difference of greater than 10 mg/kg in the day 28 samples.

Recoveries in fortified samples are shown in Table 95.

Table 95: Recoveries of endosulfan in fortified cow pea samples (98AUSE04/QT16-98).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Cow peas	0.1	77, 79, 81, 102	78, 79, 81, 97	81, 82, 84, 104			
Pea trash	0.1	78, 88, 116, 116	75, 90, 104, 104	79, 93, 102, 102			

Determination of Thiodan EC Residues in Cow Peas. AgrEvo Trial No ID98AUSE04/QT17, 14 October 1999. Determination of Endosulfan Residues in Cow Pea, State Chemistry Laboratory Report No. 9907103.

At a site in Kincora Qld, cow peas were treated with two applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). Sprays were applied at 7 day intervals and application timings ranged from 8 weeks to 4 weeks before harvest. Individual plots were 8 rows  $\times$  20m, with 4 replications per treatment. The sprays were applied by hand-held boom. Samples of peas and trash were taken at 49, 42, 35 and 28

days after the final spray. The interval between sample collection and analysis was 139 days; samples were stored at 2-4 °C prior to analysis. Samples from replicate plots were analysed separately. Results are shown in Table 96.

Table 96: Residues in cow peas and trash following application of Thiodan EC at 735 g ai/ha (98AUSE04/QT17-98).

Trial site, Year,	Application		WHP	Sample		Residues (mg/kg)		
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Kincora, Qld, 1999	735	2	49	Cow peas	< 0.005	0.005	0.094	0.099
(Big buff)				_	< 0.005	< 0.005	0.053	0.053
					< 0.005	< 0.005	0.062	0.062
					< 0.005	0.006	0.058	0.058
		2	42	Cow peas	< 0.005	0.005	0.077	0.082
				_	< 0.005	< 0.005	0.073	0.073
					< 0.005	0.006	0.090	0.096
					< 0.005	< 0.005	0.071	0.071
		2	35	Cow peas	< 0.005	0.018	0.12	0.14
				_	< 0.005	0.018	0.14	0.16
					< 0.005	0.018	0.12	0.14
					0.006	0.024	0.16	0.19
		2	28	Cow peas	0.012	0.046	0.21	0.27
					0.009	0.040	0.19	0.24
					0.014	0.060	0.28	0.35
					0.014	0.058	0.25	0.32
		2	49	Trash	1.3	3.9	7.0	12 (15.4)
					0.12	0.95	3.6	4.7 (6.5)
					0.75	3.3	8.0	12 (15.2)
					0.39	1.2	3.9	5.5 (7.5)
		2	42	Trash	0.88	3.4	6.9	11 (14.1)
					1.5	6.4	12	20 (23.8)
					0.90	3.3	6.1	10 (18.2)
					0.92	4.2	8.8	14 (25.0)
		2	35	Trash	3.1	9.3	15	27 (32.9)
					2.9	7.8	11	22 (26.5)
					2.3	7.2	13	22 (26.2)
					2.2	7.1	15	24 (32.0)
		2	28	Trash	4.1	7.0	12	23 (29.1)
					1.6	5.0	6.4	13 (20.0)
					4.2	11	14	29 (47.5)
1.00 0.005 1					2.1	6.8	9.9	19 (24.7)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for peas and pea trash.

 $Figures\ of\ total\ endosulfan\ in\ parentheses\ are\ corrected\ for\ moisture\ content;\ moisture\ ranged\ 16-45\%\ in\ trash.$ 

The data show that total endosulfan residues in cow pea trash range from 6.5 to 47.5 mg/kg dry weight following treatment from 7 to 4 weeks before harvest. The highest residues are observed for treatments applied between 5 and 4 weeks before harvest. Values for individual replicate samples vary, for examples there is a difference of greater than 10 mg/kg in the day 28 samples, as found in the previous cow pea trial.

Recoveries of endosulfan in cow peas and trash were within acceptable limits of 70 - 110 % (Table 95).

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Field Peas. One Trial, Freeling South Australia, Australia, 1999. Aventis Trial Number 9208, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Field Peas, State Chemistry Laboratory Report No. 0007070.

In a trial at Freeling S.A., field peas were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at the green pod stage. The trial plot was 2m

× 15m and included four replications. Sprays were applied using a gas pressured back pack with handheld boom. Samples of forage from all treatments were collected at 47 days after post-emergent application, when the crop was at the 12-node stage. Additional forage samples from the 350 g ai/ha treatment were collected at 75 days or the 15-node stage. Grain and straw samples were collected at harvest; 49 days after two applications or 157 days after a single application. The interval between sample collection and analysis was approximately 14 to 15 months; samples were stored at –20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed, except for the forage samples taken at 75 days and peas and straw taken at 49 days, where the replicates were analysed individually. Results are shown in Table 97.

Table 97: Residues in field peas, forage and straw following application of Thiodan EC (99AUSA02/9208))

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Freeling, SA, 1999	175	1	47	Forage	< 0.005	< 0.005	< 0.005	< 0.005
(Var Alma)					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	47	Forage	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
		1	75	Forage	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	175	1	157	Field peas	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	157	Field peas	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	49	Field peas	< 0.005	0.011	< 0.005	0.011
					< 0.005	0.006	< 0.005	0.006
					< 0.005	0.007	< 0.005	0.007
					< 0.005	0.006	< 0.005	0.006
	175	1	157	Straw	< 0.005	0.018	0.017	0.035 (0.037)
					< 0.005	0.026	0.021	0.047 (0.050)
	350	1	157	Straw	< 0.005	0.020	0.024	0.044 (0.047)
					< 0.005	0.016	0.017	0.033 (0.035)
	350 + 735	1 + 1	49	Straw	0.028	0.21	0.061	0.30 (0.32)
					0.028	0.22	0.097	0.34 (0.37)
					0.030	0.24	0.098	0.37 (0.40)
					0.033	0.25	0.16	0.44 (0.48)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for peas, forage and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 86 – 88% in forage and 6–14% in trash samples.

The data show that there is little variation in residue results from single replicate analyses, as compared to combined replicates, for both straw and grain samples.

Table 98: Recoveries of endosulfan in fortified field pea samples (99AUSA02/9208).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Field peas	0.1	119	116	120			
Pea forage	0.1	95	98	103			
Pea straw	0.1	83	84	76			

Evaluation Of Thiodan EC For Residue Analysis in Lupin, Aventis Trial No. ID99AUSA02, June 2000. Determination of Endosulfan Residues in Lupin, State Chemistry Laboratory Report No. 0004086 and 0004087.

In a trial conducted at York W.A., lupins were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was immediately after sowing and the second application was at flowering. The trial

plot was  $2m \times 10m$  and included four replications. Sprays were applied using an air operated hand boom. Samples of forage following the single sprays were collected at 61 days after seeding. Grain and trash samples were collected at harvest, 49 days after two applications or 203 days after a single application. The interval between sample collection and analysis was approximately 12 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed. Results are shown in Table 99.

Table 99: Residues in lupins, forage and straw following application of Thiodan EC. (99AUSE02/LM08).

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
York, W.A., 1999	175	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
(Merrit)					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	175	1	203	Lupins	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	203		< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	49		0.009	0.028	0.018	0.055
					< 0.005	0.013	0.010	0.023
	175	1	203	Straw	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	203	Straw	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1+1	49	Trash	0.025	0.091	0.055	0.17 (0.19)
					0.036	0.11	0.064	0.21 (0.24)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for lupins, forage and straw.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 86 - 88% in forage and 12 - 15% in trash samples.

Table 100: Recoveries of endosulfan in fortified lupins, forage and straw samples (99AUSE02/LM08).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Lupins	0.1	70	70	73			
Forage	0.1	123	127	131			
Straw	0.1	71	70	70			

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Chickpeas. One Trial, Bellata, New South Wales, Australia, 1999. Aventis Trial Number 9210, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Chickpeas, State Chemistry Laboratory Report No. 0010005.

In a trial at Bellata NSW, chickpeas were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at the flowering/pod fill stage. The trial plot was  $2.5 \, \text{m} \times 19 \, \text{m}$  and included four replications. Sprays were applied using a gas pressured back pack with hand-held boom. Samples of forage from all treatments were collected at 65 days after post-emergent application, when the crop was approximately 30 cm in height and had not commenced flowering. Grain and straw samples were collected at harvest; 38 days after two applications or 142 days after a single application. The interval between sample collection and analysis was approximately 26 months; samples were stored at  $-20 \, ^{\circ}\text{C}$  prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed for half of the forage samples; for grain and straw taken at 38 days, the replicates were analysed individually. Results are shown in Table 101.

Table 101: Residues in chickpeas, forage and straw following application of Thiodan EC. (99AUSE02/9210).

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Bellata NSW, 1999	175	1	38	Forage	< 0.005	0.010	0.015	0.025 (0.152)
(Amethyst)					< 0.005	0.016	0.024	0.040 (0.225)
	350	1	38	Forage	< 0.005	0.020	0.038	0.058 (0.290)
					< 0.005	0.021	0.032	0.053 (0.265)
		1	38	Forage	< 0.005	0.014	0.025	0.039 (0.219)
					< 0.005	0.015	0.021	0.036 (0.176)
					0.005	0.022	0.031	0.058 (0.294)
					< 0.005	0.020	0.033	0.053 (0.273)
				Controls	< 0.005	0.006	0.007	0.013 (0.061)
					< 0.005	0.007	0.006	0.013 (0.061)
	175	1	142	Chickpeas	< 0.005	< 0.005	0.015	0.015
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	142	Chickpeas	< 0.005	< 0.005	0.006	0.006
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	38	Chickpeas	< 0.005	0.012	0.19	0.20
					< 0.005	< 0.005	0.12	0.12
					< 0.005	0.009	0.21	0.21
					< 0.005	0.010	0.14	0.15
	175	1	157	Straw	< 0.005	< 0.005	0.031	0.031 (0.046)
					< 0.005	0.013	0.055	0.068 (0.128)
	350	1	157	Straw	< 0.005	0.014	0.079	0.093 (0.171)
					< 0.005	< 0.005	0.043	0.043 (0.061)
	350 + 735	1 + 1	49	Straw	0.088	0.46	3.2	3.7 (9.44)
					0.064	0.27	2.4	2.7 (4.48)
					0.031	0.20	1.6	1.8 (2.17)
					0.064	0.21	2.4	2.7 (4.31)

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg for chickpeas, 0.1 mg/kg for forage and straw.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 78 – 84% in forage and 17 – 61% in straw samples.

The data show that residues in individual replicate straw samples vary by a factor of 2-4.

Table 102: Recoveries of endosulfan in fortified chickpeas, forage and straw samples (99AUSA02/9210).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Chickpeas	0.02	72	62	65			
	0.1	64	77	70			
Forage	0.1	80	74	66			
Straw	0.1	83, 93	81, 92	80, 97			

## 8.5.12 Root and tuber vegetables (beetroot, carrot, potato, sweet potato)

Trials to Determine the Level of Endosulfan in Beetroot at Harvest Following Four Applications to the Crop. Report No. 1/8/534, Protocol No. ECR534, August 2001. Determination of Endosulfan Residues in Beetroot, State Chemistry Laboratory Report No. 0012095.

In a single trial in Queensland, an endosulfan EC formulation was applied to beetroot plants at the rate of 735 or 1470 g ai/ha, (1× and 2×). Four sprays were applied at intervals of 14 days with the last spray applied at 14 days before harvest. Sprays were applied by mini boom; the trial plot was  $2 \times 40$  m with one replication. Tubers were sampled at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 4 months prior to analysis. Residues in beetroot tubers were determined and are shown in Table 103.

Table 103: Endosulfan residues in beetroot from trials conducted in Australia 2000.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray Rate	No.	(days)		α	β	$SO_4$	Total
Lockyer Valley, Qld	735 g ai/ha	4	0	Beetroot	0.18	0.11	0.10	0.39
2000 (Detroit short	(73.5 g ai/hL)		7		0.10	0.11	0.11	0.32
top)			14		0.062	0.063	0.075	0.20
			21		0.080	0.080	0.090	0.25
	1470 g ai/ha	4	0	Beetroot	0.38	0.27	0.13	0.78
	(147 g ai/hL)		7		0.25	0.22	0.16	0.63
			14		0.20	0.20	0.20	0.60
			21		0.16	0.15	0.15	0.46

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 104: Recoveries of endosulfan in fortified beetroot.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Beetroot	0.07	88	121	101		
	0.1	103	103	106		

Trials to Determine the Level of Endosulfan in Carrots at Harvest Following Three Applications to the Crop. Report No. 1/8/565, Protocol No. ECR565, August 2001. Determination of Endosulfan Residues in Carrots, State Chemistry Laboratory Report No. 0009020, 0101003 & 0103087.

In four trials conducted in Western Australia, South Australia and Victoria, carrots were treated with an endosulfan EC formulation at the rate of 735 or 1470 g ai/ha, (1× and 2×). Three sprays were applied at intervals of 14 days with the last spray applied to mature carrots. Sprays were applied by hand held boom and the trial plots were  $14 \text{ m}^2$  in SA,  $80\text{m}^2$  in Victoria and  $40 \times 2\text{m}$  in WA with one replication. Carrots were sampled at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 15 months prior to analysis. Residues in carrots were determined and are shown in Table 105.

Table 105: Endosulfan residues in carrots from trials conducted in Australia 2000.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Rate	No.	(days)		α	β	$SO_4$	Total
Virginia, SA, 2000,	735 g ai/ha	3	0	Carrot	0.016	0.030	0.028	0.074
(Ricarto)			7		0.017	0.034	0.025	0.076
			14		0.020	0.021	0.019	0.060
			21		0.034	0.054	0.046	0.13
	1470 g ai/ha	3	0	Carrot	0.040	0.066	0.046	0.015
			7		0.066	0.12	0.062	0.25
			14		0.044	0.080	0.062	0.19
			21		0.066	0.13	0.082	0.28
Virginia, SA, 2000,	735 g ai/ha	3	14	Carrot	0.019	0.043	0.033	0.095
(Ricarto)			21		0.018	0.023	0.019	0.060
			14	Control	0.013	< 0.005	0.005	0.018
Silvan, VIC, 2000	257 g ai/ha	3	0	Carrots	< 0.005	< 0.005	< 0.005	< 0.005
(Flakie)	_		7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		< 0.005	< 0.005	< 0.005	< 0.005
	514 g ai/ha	3	0		< 0.005	< 0.005	< 0.005	< 0.005
	_		7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		0.005	< 0.005	0.006	0.011
Medina, WA, 2000	735	3	14	Carrots	0.013	0.012	0.012	0.037
(Ivar)			21		0.019	0.019	0.016	0.054

LOQ = 0.005 mg/kg.

In the trial in Victoria, the product was applied as spray concentrations of 190 and 380 ml/100L, which corresponded to application rates of 257 and 514 g ai/ha or  $0.35 \times$  and  $0.7 \times$ . Therefore the trials are not appropriate for the establishment of an MRL.

Table 106: Recoveries of endosulfan in fortified carrot.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Carrot	0.005	95	104	97			
	0.04	79	80	69			
	0.5	78	101	88			

Trials to Determine the Level of Endosulfan in Potatoes at Harvest Following Three Applications to the Crop. Report No. 1/7/562, Protocol No. ECR562, July 2001. Determination of Endosulfan Residues in Potatoes, State Chemistry Laboratory Report No. 0011192, 0012096, 0101002, & 0103088.

In four trials conducted in Western Australia, South Australia, Victoria and Queensland, potatoes were treated with an endosulfan EC formulation at the rate of 735 or 1470 g ai/ha, (1× and 2×). Three sprays were applied at intervals of 14 days with the last spray applied to mature tubers at 14 days before harvest. Sprays were applied by hand held boom or small plot sprayer;

trial plots were  $10 \times 3m$  in Victoria,  $2 \times 40m$  in Queensland,  $2 \times 40m$  in Western Australia and  $36m^2$  in SA with one replication. Tubers were sampled at 0, 7, 14 and 21 days after application and were washed prior to dispatch for analysis. Samples were stored at  $-20^{\circ}$  C for up to 8 months prior to analysis. Residues in potatoes were determined and are shown in Table 107.

Table 107: Endosulfan residues in potatoes from trials conducted in Australia 2000.

г			1		_			
Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Rate	No.	(days)		α	β	$SO_4$	Total
Torquay, VIC, 2000	735 g ai/ha	3	0	Potato	0.005	0.005	< 0.005	0.010
(Sequoia)			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		< 0.005	< 0.005	0.005	0.005
	1470 g ai/ha	3	0	Potato	< 0.005	< 0.005	< 0.005	< 0.005
			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		< 0.005	< 0.005	< 0.005	< 0.005
Lockyer Valley,	735 g ai/ha	3	0	Potato	< 0.005	< 0.005	0.008	0.008
QLD, 2000	_		7		< 0.005	< 0.005	0.007	0.007
(Sebago)			14		< 0.005	< 0.005	0.007	0.007
			21		< 0.005	< 0.005	0.006	0.006
	1470 g ai/ha	3	0	Potato	< 0.005	< 0.005	0.007	0.007
	_		7		< 0.005	< 0.005	0.006	0.006
			14		< 0.005	< 0.005	0.008	0.008
			21		< 0.005	< 0.005	0.008	0.008
Medina, WA, 2000,	735 g ai/ha	3	0	Potato	0.26	0.15	< 0.005	0.41
(Delaware)	_		7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		< 0.005	< 0.005	< 0.005	< 0.005
Virginia, SA, 2000, (Collaben)	735 g ai/ha	3	14	Potato	< 0.005	< 0.005	0.007	0.007

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 108: Recoveries of endosulfan in fortified potato.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Potato	0.02	93	98	97		
	0.5	85	99	89		

Trials to Determine the Level of Endosulfan in Sweet Potatoes at Harvest Following Three Applications to the Crop. Report No. 1/8/533, Protocol No. ECR533, August 2001. Determination of Endosulfan Residues in Sweet Potatoes, State Chemistry Laboratory Report No. 0104097.

In a single trial in Queensland, sweet potatoes were treated with an endosulfan EC formulation at the rate of 735 or 1470 g ai/ha, (1× and 2×). Three sprays were applied at intervals of 14 - 15 days with the last spray applied to mature tubers at 14 days before harvest. Sprays were applied by small plot sprayer and the trial plot was  $1.5 \times 5$ m with one replication. Tubers were sampled at 0, 7, 14 and 21 days after application. Samples were stored at  $-20^{\circ}$  C for up to 6 months prior to analysis. Residues in sweet potatoes were determined and are shown in Table 109.

Table 109: Endosulfan residues in sweet potatoes from trials conducted in Australia 2000.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray Rate	No.	(days)		α	β	$SO_4$	Total
Southedge, QLD,	735 g ai/ha	3	0	Sweet potato	< 0.005	< 0.005	< 0.005	< 0.005
2000 (Sentinal)			7		< 0.005	< 0.005	< 0.005	< 0.005
			14		< 0.005	< 0.005	< 0.005	< 0.005
			21		< 0.005	< 0.005	< 0.005	< 0.005
	1470 g ai/ha	3	0	Sweet potato	0.005	< 0.005	< 0.005	0.005
			7		0.007	0.008	< 0.005	0.015
			14		0.007	0.006	< 0.005	0.013
			21		< 0.005	< 0.005	0.006	0.006

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 110: Recoveries of endosulfan in fortified sweet potato.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan β-endosulfan endosulfan				
Sweet potato	0.02	93	100	93		
	0.5	85	100	90		

### 8.5.13 Stalk and stem vegetables (celery, rhubarb)

Australian trials for celery and rhubarb were provided.

Trials to Determine the Level of Endosulfan in Celery at Harvest Following Three Applications to the Crop. Report No. 1/10/535, Protocol No. ECR535, October 2001. Determination of Endosulfan Residues in Celery, State Chemistry Laboratory Report No. 0009079, 0009109 & 0105229.

In trials conducted in Victoria and Qld, an endosulfan EC formulation was applied to celery at spray concentrations of 66.5 g ai/hL or 133 g ai/hL (233 – 765 g ai/ha,  $1 \times$  and  $2 \times$ ). Three sprays were applied at intervals of 14 days and spraying began when the plants were 20cm high or 35 days prior to harvest. Sprays were applied by hand held boom or motorised backpack mister; trial plots were  $3 \times 4$  m or  $10 \times 3.5$  m with one replication. Celery stalks were sampled at 0, 3, 7 and 10 days after application. Samples were stored at  $-20^{\circ}$  C for up to 8 months prior to analysis. Residues in celery stalks were determined and the results are shown in Table 111.

Table 111: Endosulfan residues in celery from trials conducted in Australia 2000.

Trial site, Year,	Application		WHP	Sample	Residues (mg/kg)			
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Toowoomba, Qld,	66.5 g ai/hL	3 (14)	0	Celery stalk	2.5	1.5	0.22	4.2
2001 (American			3		0.58	0.39	0.25	1.2
Stringless)			7		0.31	0.16	0.12	0.59
			10		0.56	0.29	0.26	1.1
	133 g ai/hL	3 (14)	7	Celery stalk	1.1	0.63	0.48	2.2
			10		1.6	0.85	0.58	3.0
				Controls	0.054	0.024	0.045	0.12
					0.064	0.029	0.058	0.15
Cranbourne, VIC,	66.5 g ai/hL	3 (14)	0	Celery stalk	0.18	0.12	0.053	0.35

#### Australian Pesticides and Veterinary Medicines Authority (APVMA)

2000, (Summit)			3		0.16	0.13	0.068	0.36	
			7		0.12	0.076	0.062	0.26	
			10		0.14	0.090	0.062	0.29	
	133 g ai/hL	3 (14)	7	Celery stalk	0.71	0.43	0.15	1.3	1
			10	·	0.34	0.20	0.081	0.62	

LOD = 0.005 m/kg; LOQ = 0.02 mg/kg.

Table 112: Recoveries of endosulfan in fortified celery.

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Celery	0.02	104	91	110			
	0.5	82	86	91			

Trials to Determine the Level of Endosulfan in Rhubarb at Harvest Following Two Applications to the Crop. Report No. 1/10/558, Protocol No. ECR558, October 2001. Determination of Endosulfan Residues in Rhubarb, State Chemistry Laboratory Report No. 0105230 & 0107096.

Two trials in rhubarb were conducted in Qld. An endosulfan EC formulation was applied to rhubarb at spray concentrations of 70 g ai/hL or 140 g ai/hL  $(130 - 468 \text{ g ai/ha}, 1 \times \text{ and } 2 \times)$ .

Two sprays were applied at a 14 days interval and spraying ranged from when 50% of the stalks were mature to 85% mature stalks or 21 and 7 days before commercial harvest.

Sprays were applied by hand held boom or motorised backpack mister; trial plots were  $10 \times 4$  m or  $10 \times 1$  m with one replication. Rhubarb stalks were sampled at 0, 3, 7 and 10 days after application. Samples were stored at  $-20^{\circ}$  C for between 4 and 12 months prior to analysis. Residues in rhubarb stalks were determined and the results are shown in Table 113.

Table 113: Endosulfan residues in rhubarb from trials conducted in Australia 2000 – 2001.

Trial site, Year, Application		WHP	Sample		Residues	s (mg/kg)		
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Hampton, Qld, 2000	70 g ai/hL	2 (14)	0	Rhubarb	0.015	0.29	0.26	0.57
(Sydney crimson)			3		0.016	0.061	0.013	0.090
			7		0.012	0.036	0.011	0.059
			10		0.017	0.042	0.020	0.079
	140 g ai/hL	2 (14)	7	Rhubarb	0.026	0.10	0.039	0.16
			10		0.015	0.060	0.028	0.10
Nth Tambourine,	70 g ai/hL	2 (14)	0	Rhubarb	2.1	1.5	0.094	3.7
Qld, 2001 (Big red)			3		0.41	0.33	0.065	0.80
			7		0.098	0.17	0.072	0.34
			10		0.056	0.087	0.053	0.20
	140 g ai/hL	2 (14)	7	Rhubarb	0.19	0.34	0.20	0.73
	-		10		0.063	0.12	0.063	0.25

LOD = 0.005 mg/kg; LOQ = 0.02 mg/kg.

Table 114: Recoveries of endosulfan in fortified rhubarb.

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	endosulfan SO <sub>4</sub>			
Rhubarb	0.02	96	105	93		
	0.5	93	94	90		

## 8.5.14 Cereal Crops (sorghum, wheat, barley)

Determination of Thiodan EC Residues in Sorghum, AgrEvo Trial No ID98AUSE06/NN12, 27 October 1999. Determination of Endosulfan Residues in Sorghum, State Chemistry Laboratory Report No. 9904013.

At a site in Gurley NSW, sorghum crops were treated with two foliar applications of Thiodan EC at a rate of 735 g ai/ha (1×). The application timings ranged from flowering to grain ripening, approximately 8 weeks to 4 weeks before harvest; the intervals between sprays were 5 to 9 days. Individual plots were 4 rows × 8m, with 4 replicate plots. The sprays were applied by hand held boom. Samples of grain and trash were collected at 46, 40, 31 and 26 days after application. The interval between sample collection and analysis was 169 days; samples were stored at 2 - 4 °C prior to analysis. Samples from replicate plots were analysed individually. Results are shown in Table 115.

Table 115: Residues in sorghum and trash following application of Thiodan EC at 735 g ai/ha (ID98AUSE06/NN12)

Trial site, Year,	Applicati	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Gurley NSW, 1998	735	2	26	Sorghum	0.080	0.26	0.44	0.78
(Buster)					0.030	0.11	0.41	0.55
					0.10	0.33	0.45	0.88
					0.15	0.42	0.52	1.1
		2	31	Sorghum	0.024	0.088	0.49	0.60
					0.033	0.091	0.54	0.66
					0.060	0.20	0.68	0.94
					0.031	0.13	0.58	0.74
		2	40	Sorghum	0.006	0.019	0.63	0.66
					0.006	0.018	0.52	0.54
					< 0.005	0.019	0.62	0.64
					0.007	0.024	0.57	0.60
		2	46	Sorghum	< 0.005	0.006	0.24	0.25
					< 0.005	0.006	0.17	0.18
					< 0.005	0.014	0.39	0.40
					< 0.005	0.013	0.39	0.40
		2	26	Trash	0.34	4.0	13	17 (42.50)
					0.19	1.6	10	12 (30)
					0.52	3.1	26	30 (78.9)
					0.43	3.5	19	23 (54.8)
		2	31	Trash	0.11	0.41	9.7	10 (26.3)
					0.095	0.45	8.4	8.9 (22.3)
					0.17	0.72	14	15 (46.9)
					0.18	0.83	14	15 (38.5)
		2	40	Trash	0.082	0.26	16	16.3 (48.5)
					0.024	0.11	7.5	7.6 (10.8)
					0.074	0.45	14	15 (42.8)
					0.048	0.28	11	11 (33.3)
		2	46	Trash	0.009	0.084	4	4.1 (11.7)
					0.012	0.11	4.4	4.5 (12.5)
					0.016	0.22	5	5.2 (10.6)
					0.024	0.22	5	5.2 (13)
				Controls	< 0.005	< 0.005	0.009	0.009
					< 0.005	< 0.005	0.009	0.009

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in grain and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 51-70% in trash.

Finite residues of endosulfan were found in untreated control samples at levels just above LOQ. The data show that there is significant variation in the endosulfan residues found in sorghum trash sampled at 26 to 40 days after application. Residues range from 22 to 79 mg/kg at 26 and 31 days after application. The current label withholding period for sorghum is 28 days.

Table 116: Recoveries of endosulfan in fortified sorghum samples (98AUSE06/NN12).

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Sorghum	0.1	72, 88, 91, 109, 114,	72, 86, 90, 108, 108,	88, 90, 90, 94, 98,				
		115, 119	125, 129	105, 110				
Trash	0.1	65, 76, 76, 82, 85,	70, 79, 79, 82, 85,	62, 72, 82, 93, 93,				
		93, 93	92, 96	101, 105				

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Determination of Thiodan EC Residues in Sorghum, AgrEvo Trial No ID98AUSE06/NN11, 27 October 1999. Determination of Endosulfan Residues in Sorghum, State Chemistry Laboratory Report No. 9904012.

At a second site in Gurley NSW, sorghum crops were treated with two foliar applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). The application timings ranged from head emergence to grain fill, approximately 8 weeks to 4 weeks before harvest; the intervals between sprays were 5 to 9 days. Individual plots were 4 rows  $\times$  8m, with 4 replicate plots. The sprays were applied by hand held boom. Samples of grain and trash were collected at 51, 41, 35 and 29 days after application. The interval between sample collection and analysis was 155 days; samples were stored at 2 – 4 °C prior to analysis. Samples from replicate plots were analysed individually. Results are shown in Table 117.

Table 117: Residues in sorghum and trash following application of Thiodan EC at 735 g ai/ha (ID98AUSE06/NN11)

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	•	α	β	$\tilde{SO}_4$	Total
Gurley NSW, 1998	735	2	29	Sorghum	0.010	0.05	0.77	0.87
(Buster*)					0.009	0.045	0.37	0.42
					< 0.005	0.028	0.41	0.44
					0.008	0.052	0.50	0.56
		2	35	Sorghum	< 0.005	0.020	0.25	0.27
					< 0.005	0.021	0.29	0.31
					< 0.005	0.017	0.32	0.34
					< 0.005	0.019	0.29	0.31
		2	41	Sorghum	< 0.005	0.010	0.31	0.32
					< 0.005	0.008	0.23	0.24
					< 0.005	0.009	0.30	0.31
					< 0.005	0.007	0.23	0.24
		2	51	Sorghum	< 0.005	< 0.005	0.19	0.19
					< 0.005	< 0.005	0.14	0.14
					< 0.005	< 0.005	0.094	0.094
					< 0.005	< 0.005	0.13	0.13
		2	26	Trash	0.64	2.7	20	23 (30.7)
					0.20	1.2	11	12 (15.4)
					0.52	1.9	19	21 (23.1)
					0.15	0.61	5.5	6.3 (7.1)
		2	35	Trash	0.20	0.81	16	17 (21.3)
					0.092	0.33	9.3	9.7 (11.8)
					0.066	0.32	10	10.4 (13.1)
					0.038	0.46	9.7	10.2 (15.2)
		2	41	Trash	0.040	0.31	9.9	10.4 (17.6))
					0.034	0.25	0.25	12 (21.4)
					0.032	0.30	12	12.3 (17.1)
					0.19	0.60	15	16 (19.7)
		2	51	Trash	0.067	0.34	16	16.4 (19.5)
					0.017	0.14	11	11 (15.5)
					0.22	1.6	19	21 (37.5)
					0.019	0.26	10	10.3 (17.1)
				Controls	< 0.005	< 0.005	0.012	0.012
					< 0.005	< 0.005	0.010	0.010
					< 0.005	< 0.005	0.007	0.007
I OD 0 005 /I /I				1	< 0.005	< 0.005	0.006	0.006

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in grain and trash.

Finite residues were found in all of the untreated control trash samples. The data show that residues in sorghum trash vary significantly in replicate samples, ranging from 7 to 31 mg/kg at 26 days after application.

<sup>\*</sup> Dryland sorghum variety.

 $Figures\ of\ total\ endosulfan\ in\ parentheses\ are\ corrected\ for\ moisture\ content;\ moisture\ ranged\ from\ 9-44\%\ in\ trash.$ 

Recoveries in fortified grain and trash are given in Table 116.

Determination of Endosulfan Residues in Grain Sorghum, AgrEvo Trial No ID98AUSE06/QD12-98, 22 June 1999. Determination of Endosulfan Residues in Sorghum, State Chemistry Laboratory Report No. 9906003.

Sorghum crops at a site in Forest Hill Qld, were treated with two foliar applications of Thiodan EC at a rate of 735 g ai/ha (1×). The application timings ranged from early head emergence to flowering, approximately 8 weeks to 4 weeks before harvest; the intervals between sprays were 5 to 8 days. The plot size was  $20m \times 6$  rows, containing 4 replications. The sprays were applied by air pressurised overhead boom. Samples of grain and trash were collected at 48, 40, 32 and 27 days after application. The interval between sample collection and analysis was 136 days; samples were stored at 2-4 °C prior to analysis. Samples from replicate plots were analysed individually. Results are shown in Table 118.

Table 118: Residues in sorghum and trash following application of Thiodan EC at 735 g ai/ha (ID98AUSE06/QD12-98)

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$\tilde{SO_4}$	Total
Forest Hill Qld,	735	2	27	Sorghum	0.005	0.021	0.28	0.31
1999, (MR Buster)					< 0.005	0.015	0.42	0.44
, , ,					< 0.005	0.021	0.41	0.43
					0.008	0.038	0.32	0.37
		2	32	Sorghum	< 0.005	0.008	0.24	0.25
					< 0.005	0.009	0.33	0.34
					< 0.005	0.016	0.49	0.51
					< 0.005	0.013	0.38	0.39
		2	40	Sorghum	< 0.005	0.005	0.20	0.20
					< 0.005	< 0.005	0.14	0.14
					< 0.005	< 0.005	0.18	0.18
					< 0.005	< 0.005	0.20	0.20
		2	48	Sorghum	< 0.005	< 0.005	0.093	0.093
					< 0.005	< 0.005	0.039	0.039
					< 0.005	< 0.005	0.052	0.052
					< 0.005	< 0.005	0.056	0.056
		2	27	Trash	0.032	0.15	2.0	2.2 (6.7)
					0.024	0.13	1.6	1.8 (4.7)
					0.030	0.19	1.8	2.0 (5.7)
					0.044	0.27	1.8	2.1 (5.8)
		2	32	Trash	0.008	0.062	0.94	1.0 (3.0)
					0.010	0.058	0.99	1.1 (3.2)
					0.024	0.16	1.5	1.7 (4.5)
					0.014	0.086	0.86	0.96 (3.0)
		2	40	Trash	< 0.005	0.035	0.87	0.91 (2.4)
					0.008	0.048	1.6	1.6 (4.6)
					0.008	0.048	1.1	1.2 (3.2)
					0.009	0.048	1.2	1.3 (3.7)
		2	48	Trash	0.007	0.034	1.1	1.1 (2.9)
					0.010	0.045	1.3	1.4 (4.4)
					0.009	0.044	1.7	1.8 (4.7)
					0.007	0.028	0.94	0.98 (2.8)
				Controls	0.006	0.016	0.12	0.14 (0.41)
					0.006	0.010	0.038	0.054
								(0.15)
					< 0.005	< 0.005	0.014	0.014
								(0.042)
					< 0.005	0.008	0.028	0.036
								(0.11)

 $LOD=0.005\ mg/kg;\ LOQ=0.1\ mg/kg$  for grain and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 62-68% in trash.

Finite residues were present in all untreated trash samples; although the levels are low, correction of the data would not change the overall situation with respect to establishment of an appropriate MRL for sorghum fodder. The pattern of variability in individual replicate samples is not observed in the samples from this trial, unlike the results from the previous two trials. Recoveries in fortified grain and trash are given in Table 116.

Determination of Thiodan EC Residues in Sorghum, AgrEvo Trial No ID98AUSE06/NN10, 27 October 1999. Determination of Endosulfan Residues in Sorghum, State Chemistry Laboratory Report No. 9904011.

In a trial conducted at Bellata NSW, sorghum crops were treated with two foliar applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). The application timings ranged from head emergence to grain fill, approximately 8 weeks to 4 weeks before harvest; the intervals between sprays were 5 to 9 days. The plot size was 4 rows  $\times$  8m, containing 4 replications. The sprays were applied by hand held boom. Samples of grain and trash were collected at 48, 43, 34 and 27 days after application. The interval between sample collection and analysis was 157 days; samples were stored at 2 – 4 °C prior to analysis. Samples from replicate plots were analysed individually. Results are shown in Table 119.

Table 119: Residues in sorghum and trash following application of Thiodan EC at 735 g ai/ha (ID98AUSE06/NN10)

Trial site, Year,	Application	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	~ <b>F</b>	α	β	SO <sub>4</sub>	Total
Bellata NSW, 1998	735	2	27	Sorghum	0.039	0.27	1.8	2.1
(Legend*)					0.045	0.28	1.4	1.7
					0.035	0.22	1.4	1.6
					0.034	0.22	1.3	1.5
		2	34	Sorghum	0.007	0.049	0.84	0.90
					0.009	0.057	0.96	1.03
					0.007	0.045	0.83	0.88
					0.013	0.071	0.95	1.0
		2	43	Sorghum	0.009	0.019	0.53	0.56
					< 0.005	0.014	0.70	0.71
					0.006	0.019	0.80	0.82
					< 0.005	0.014	0.51	0.52
		2	48	Sorghum	0.007	0.019	0.38	0.41
					0.005	0.014	0.29	0.31
					0.005	0.015	0.24	0.26
					0.005	0.022	0.37	0.40
		2	27	Trash	0.16	2.7	20	23 (54.8)
					0.14	2.2	17	19 (48.7)
					0.12	2.6	24	27 (62.8)
					0.091	1.8	18	20 (46.5)
		2	34	Trash	0.040	0.87	5.5	6.4 (16.4)
					0.048	1.0	10	11 (28.2)
					0.069	0.83	15	16 (35.5)
					0.091	0.82	17	18 (42.8)
		2	43	Trash	0.020	0.24	7	7.3 (24.3)
					0.042	0.82	12	13 (39.4)
					0.026	0.24	7.4	7.7 (24.1)
					0.066	1	12	13 (33.3)
		2	48	Trash	0.039	0.25	11	11 (35.5)
					0.040	0.29	12	12 (33.3)
					0.030	0.23	8.1	8.4 (23.3)
					0.082	1.3	19	20 (45.5)
				Controls	< 0.005	< 0.005	0.038	0.038
								(0.076)
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.014	0.014
								(0.042)
					< 0.005	0.011	0.016	0.027
								(0.077)

LOD = 0.005 g/kg; LOQ = 0.1 mg/kg in grain and trash.

Finite residues of endosulfan were present in untreated trash control samples. Total endosulfan residues in individual replicates taken at day 34 ranged 16 - 42 mg/kg and showed greater variation than samples taken at day 27, which ranged 46 - 63 mg/kg.

Recoveries in fortified grain and trash are given in Table 116.

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Barley. One Trial, Nathalia Victoria, Australia, 1999. Aventis Trial Number 9209, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Barley, State Chemistry Laboratory Report No. 0007071.

In a trial at Nathalia Victoria, barley crops were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at milk development (128 days after planting). The trial plot was  $2m \times 15m$  and included four replications. Sprays were applied using a gas pressurised back pack with hand-held boom. Samples of forage from all treatments were collected at 70 days after post-emergent application, when the crop was at the 5-7 tiller stage. Grain and straw samples were collected at harvest; 42 days after two applications or 158 days after a single application. The interval between sample collection and analysis was approximately 12 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed for forage samples; grain and straw were analysed as individual replicates. Results are shown in Table 120.

Table 120: Residues in barley, forage and straw following application of Thiodan EC (99AUSA02/9209)

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Nathalia VIC,	175	1	70	Forage	< 0.005	< 0.005	0.012	0.012 (0.067)
1999 (Galaxy)				_	< 0.005	< 0.005	0.011	0.011 (0.058)
	350	1	70	Forage	< 0.005	< 0.005	0.015	0.015 (0.079)
					< 0.005	< 0.005	0.015	0.015 (0.083)
	175	1	158	Barley	< 0.005	< 0.005	0.007	0.007
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	158	Barley	< 0.005	< 0.005	0.010	0.010
					< 0.005	< 0.005	0.008	0.008
	350 + 735	1 + 1	42	Barley	0.079	0.18	0.75	1.0
					0.063	0.13	0.87	1.1
					0.041	0.088	0.53	0.66
					0.092	0.20	1.0	1.3
	175	1	158	Straw	0.014	0.066	0.15	0.23 (0.25)
					0.008	0.033	0.066	0.11 (0.12)
	350	1	158	Straw	0.011	0.058	0.13	0.20 (0.22)
					0.009	0.059	0.12	0.19 (0.21)
	350 + 735	1 + 1	42	Straw	0.38	2.5	2.4	5.3 (5.7)
					0.31	2.1	2.3	4.7 (5.1)
					0.25	1.8	2.0	4.0 (4.3)
					0.41	2.8	2.7	5.9 (6.4)
				Controls	0.006	0.026	0.055	0.087 (0.093)
					0.005	0.027	0.051	0.083 (0.090)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for grain and straw.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 81 82% in forage and 7–10% in trash samples.

Finite residues were present in untreated control samples of straw.

<sup>\*</sup> Dryland sorghum variety.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 50-70% in trash.

Table 121: Recoveries of endosulfan in fortified barley samples (99AUSA02/9207).

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Barley	0.1	103	105	120				
Straw	0.1	85	86	77				
Forage	0.1	100	101	100				

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Barley. One Trial, Bellata, New South Wales, Australia, 1999. Aventis Trial Number 9206, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Barley, State Chemistry Laboratory Report No. 0009211.

In a trial at Bellata NSW, barley crops were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at flowering (141 days after planting). The trial plot was  $4m \times 15m$  and included four replications. Sprays were applied using a gas pressurised back pack with hand-held boom. Samples of forage from all treatments were collected at 56 days after post-emergent application, when the crop was at the early jointing stage. Grain and straw samples were collected at harvest, 53 days after two applications or 189 days after a single application. The interval between sample collection and analysis was approximately 29 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed for all samples taken following a single application; samples taken following 2 applications were analysed as individual replicates. Results are shown in Table 122.

Table 122: Residues in barley, forage and straw following application of Thiodan EC (99AUSA02/9206)

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Bellata NSW, 1999	175	1	56	Forage	< 0.005	< 0.005	0.009	0.009 (0.066)
(Grimmett)					< 0.005	< 0.005	0.009	0.009 (0.071)
	350	1	56	Forage	< 0.005	< 0.005	0.013	0.013 (0.086)
					< 0.005	< 0.005	0.010	0.010 (0.068)
				Control	< 0.005	< 0.005	0.007	0.007 (0.062)
	175	1	189	Barley	0.005	0.009	0.057	0.071
					< 0.005	< 0.005	0.014	0.014
	350	1	189	Barley	< 0.005	< 0.005	0.009	0.009
					< 0.005	0.007	0.043	0.050
	350 + 735	1 + 1	53	Barley	0.032	0.077	0.51	0.62
					0.018	0.041	0.49	0.55
					0.017	0.044	0.40	0.46
					0.019	0.041	0.66	0.72
	175	1	158	Straw	0.014	0.24	0.20	0.45 (0.51)
					< 0.005	0.052	0.086	0.14 (0.16)
	350	1	158	Straw	< 0.005	0.029	0.060	0.089 (0.10)
					0.007	0.088	0.23	0.32 (0.36)
	350 + 735	1 + 1	42	Straw	0.035	0.80	1.3	2.1 (2.4)
					0.038	1.1	2.0	3.1 (4.0)
					0.018	0.45	0.97	1.4 (1.6)
					0.036	0.86	1.7	2.6 (2.9)
				Controls	< 0.005	0.025	0.010	0.035 (0.039)
					< 0.005	< 0.005	0.009	0.009 (0.01)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in grain, straw and forage.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 85-89% in forage samples and 10-14% in straw samples.

Finite residues were present in one control forage sample and both straw samples. Straw and forage data may be corrected for levels present in untreated controls, recognising that the levels found in the one forage control are comparable to levels found in the treated samples.

Table 123: Recoveries of endosulfan in fortified barley samples (99AUSA02/9206).

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Barley	0.1	90	88	85				
Straw	0.1	88	90	81				
Forage	0.1	74	72	82				

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Evaluation Of Thiodan EC For Residue Analysis in Wheat, Aventis Trial No. ID99AUSA01, June 2000. Determination of Endosulfan Residues in Wheat, State Chemistry Laboratory Report No. 0004088 and 0004089.

In a trial conducted at York W.A., wheat was treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was immediately after sowing and the second application was at flowering. The trial plot was  $10m \times 2m$  and included four replications. Sprays were applied using an air operated hand boom. Samples of forage following the single sprays were collected at 61 days after seeding. Grain and trash samples were collected at harvest, 49 days after two applications or 203 days after a single application. The interval between sample collection and analysis was approximately 12 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed. Results are shown in Table 124.

Table 124: Residues in wheat, forage and straw following application of Thiodan EC. (99AUSE01/LM07).

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
York, W.A., 1999	175	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
(Westonia)					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
				-	< 0.005	< 0.005	< 0.005	< 0.005
	175	1	203	Wheat	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	203	Wheat	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	49	Wheat	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	175	1	203	Straw	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	203	Straw	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1+1	49	Trash	0.052	0.13	0.041	0.22 (0.24)
					0.060	0.17	0.058	0.29 (0.32)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in grain, straw and forage.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 75 – 88% in forage and 6 – 19% in straw samples.

Table 125: Recoveries of endosulfan in fortified wheat samples (99AUSA02/9206).

Sample	Fortification (mg/kg)	% Recovery						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Wheat	0.1	90	88	85				
Straw	0.1	94	94	88				
Forage	0.1	109	125	121				

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Wheat. One Trial, Bellata, New South Wales, Australia, 1999. Aventis Trial Number 9205, Study Number ID99AUSA02, September 2000.

Determination of Endosulfan Residues in Wheat, State Chemistry Laboratory Report No. 0009204.

In a trial at Bellata NSW, wheat was treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at flowering (103 days after planting). The trial plot was 4m × 15m and included four replications. Sprays were applied using a gas pressurised back pack with hand-held boom. Samples of forage from all treatments were collected at 55 days after post-emergent application, when the crop was at the tillering (elongation) stage. Grain and straw samples were collected at harvest, 35 days after two applications or 130 days after a single application. The interval between sample collection and analysis was approximately 24 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed for all samples taken following a single application; samples taken following 2 applications were analysed as individual replicates. Results are shown in Table 126.

Table 126: Residues in wheat, forage and straw following application of Thiodan EC (99AUSA02/9205)

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Bellata NSW, 1999	175	1	55	Forage	< 0.005	< 0.005	0.018	0.018 (0.12)
(Cunningham)					< 0.005	0.008	0.020	0.028 (0.19)
	350	1	55	Forage	< 0.005	0.011	0.022	0.033 (0.22)
					< 0.005	0.012	0.026	0.038 (0.25)
				Control	< 0.005	< 0.005	0.006	0.006 (0.038)
					< 0.005	< 0.005	0.006	0.006 (0.036)
	175	1	130	Wheat	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	130	Wheat	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	35	Wheat	< 0.005	< 0.005	0.046	0.046
					< 0.005	< 0.005	0.048	0.048
					0.015	0.017	0.067	0.099
					< 0.005	< 0.005	< 0.005	< 0.005
	175	1	130	Straw	0.012	0.006	0.025	0.043 (0.05)
					< 0.005	< 0.005	0.013	0.013 (0.02)
	350	1	130	Straw	< 0.005	< 0.005	0.021	0.021 (0.03)
					< 0.005	0.010	0.019	0.029 (0.03)
	350 + 735	1 + 1	35	Straw	0.070	0.19	0.42	0.68 (0.81)
					0.090	0.39	0.81	1.3 (1.6)
					0.072	0.33	0.69	1.1 (1.4)
					0.026	0.086	0.18	0.29 (0.39)
				Controls	< 0.005	< 0.005	0.005	0.005 (0.006)
					< 0.005	< 0.005	0.006	0.006 (0.007)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for grain, straw and forage.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 83-85% in forage samples and 13-26% in straw samples.

Table 127: Recoveries of endosulfan in fortified wheat samples (99AUSA02/9205).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Wheat	0.1	110	109	110			
Straw	0.1	99	93	86			
Forage	0.1	85	103	125			

Where control samples used for recovery studies had detectable levels of endosulfan residues, the recovery results were corrected for control levels.

### 8.5.15 Tree Nuts (macadamia)

Trials to Determine the Level of Endosulfan in Macadamia Nuts at Harvest Following Three Applications to the Crop. Report No. 1/10/532, Protocol No. ECR532, October 2001. Determination of Endosulfan Residues in Macadamias, State Chemistry Laboratory Report No. 0105078, 0105224 & 0105227.

In trials conducted in NSW and Qld, an endosulfan EC formulation was applied to macadamias at spray concentrations of 52.5 and 105 g ai/hL (656-735 g ai/ha,  $1\times$  and  $2\times$ ). Three sprays were applied at intervals of 14 or 18 days and spraying began at 30 days prior to the first commercial harvest or maturing nut stages. Sprays were applied by hand held boom and trial plots comprised 2-4 trees with one replication. Nuts were sampled at 0, 1, 2 and 4 days after application. Samples were stored at  $-20^{\circ}$  C for up to 8 months prior to analysis. Residues in nut meat were determined and the results are shown in Table 128.

Table 128: Endosulfan residues in macadamia nuts from trials conducted in Australia 2001.

Trial site, Year,	Applicati	on	WHP	Sample		Residues	s (mg/kg)	
(Variety)	Spray Conc.	No.	(days)		α	β	$SO_4$	Total
Dorroughby, NSW,	52.5 g ai/hL	3 (14)	0	Macadamias	0.016	0.016	< 0.005	0.037
2001 (334)			1		0.006	0.009	< 0.005	0.020
			2		< 0.005	< 0.0005	< 0.005	< 0.005
			4		< 0.005	< 0.005	< 0.005	< 0.005
	105 g ai/hL	3 (14)	2	Macadamias	< 0.005	< 0.005	< 0.005	< 0.005
Tolga, Qld, 2001	52.5 g ai/hL	3 (18)	0	Macadamias	< 0.005	< 0.005	< 0.005	< 0.005
(344)			1		< 0.005	< 0.005	< 0.005	< 0.005
			2		< 0.005	< 0.005	< 0.005	< 0.005
			4		< 0.005	< 0.005	< 0.005	< 0.005
	105 g ai/hL	3 (14)	2	Macadamias	< 0.005	< 0.005	< 0.005	< 0.005
Glasshouse Mtns,	52.5 g ai/hL	3 (14)	0	Macadamias	0.005	0.005	< 0.005	0.015
Qld, 2001 (344/741)			1		< 0.005	< 0.005	< 0.005	< 0.005
			2		< 0.005	< 0.005	< 0.005	< 0.005
			4		< 0.005	< 0.005	< 0.005	< 0.005

LOD = 0.005 mg/kg; LOQ = 0.01 mg/kg.

Table 129: Recoveries of endosulfan in fortified macadamia nuts

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Macadamias	0.01	77	87	76		
	0.1	80	96	104		

#### 8.5.16 Oilseeds (Cotton, Soyabeans, Sunflowers, Canola)

Determination of Thiodan EC Residues in Cotton, AgrEvo Trial No ID98AUSE02/NN08 – 98, 13 October 1999. Determination of Endosulfan Residues in Cotton, State Chemistry Laboratory Report No. 9908114.

At Moree NSW, cotton plants were treated with eight foliar applications of Thiodan EC at a rate of 735 g ai/ha ( $1\times$ ). Applications were made at 5 to 13 day intervals at varying stages of crop growth, ranging from the 5-leaf stage to early flowering (5-leaf, 7-leaf, 6-node, 8-node, squaring,  $1^{st}$  flower, early flowering).

The trial plots comprised 4 replicates of a randomised block of  $8m \times 15m$ . Application was by handheld boom with low volume sprays of 60L/ha. Samples of seed, trash and lint were taken at 93 days after the final application. The samples were frozen on the day of harvest, ginned, then refrozen for dispatch to the laboratory. The interval between sample collection and analysis was 190 days; samples were stored between 2-4 °C prior to analysis. Samples from each replicate plot were analysed separately.

Residues results and recoveries are shown in Tables 130 and 131.

Table 130: Total endosulfan residues in cotton seed, lint and trash (98AUSE02/NN08, 1999).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Moree, NSW,	735	8	93	Cotton seed	< 0.005	< 0.005	< 0.005	< 0.005
1999, (V15 Ingard)					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
				Cotton trash	< 0.005	< 0.005	0.007	0.007 (0.02)
					0.007	0.013	0.053	0.073 (0.013)
					< 0.005	< 0.005	0.009	0.009 (0.02)
					< 0.005	0.013	0.027	0.040 (0.05)
				Cotton lint	< 0.005	< 0.005	0.005	0.005
					< 0.005	< 0.005	0.009	0.009
					< 0.005	< 0.005	0.005	0.005
					< 0.005	0.007	0.014	0.021

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in seed and trash.

Corrected for moisture content.

The moisture content in the trash samples ranged from 29 to 67%, mean 40% from both untreated and treated samples. The individual trash samples are reported on an 'as received' basis and are corrected for moisture content.

Table 131: Recoveries of endosulfan in fortified cotton samples (98AUSE02/NN08, 98AUSE03/NN09).

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Cotton seed	0.1	71, 80, 81, 89, 105	70, 77, 79, 91, 109	75, 78, 82, 86, 87,		
				113		
Trash	0.1	83, 89, 92, 98, 101	84, 87, 91, 97, 103	78, 84, 91, 92, 98		
Lint	0.1	80, 86, 86, 87, 97,	76, 80, 84, 87, 95,	79, 82, 82, 83, 101,		
		102, 102, 115	100, 109, 115	101, 110, 111		

Determination of Thiodan EC Residues in Cotton, AgrEvo Trial No ID98AUSE03/NN09 – 98, 13 October 1999. Determination of Endosulfan Residues in Cotton, State Chemistry Laboratory Report No. 9908115.

At Narrabri NSW, cotton plants were treated with a total of ten foliar applications of Thiodan EC at a rate of 735 g ai/ha ( $1\times$ ). Eight applications made at the early stage (3-nodes to flowering) at intervals of 5 to 13 days and two applications were made at 1 and 20% bolls open, respectively.

The trial plots comprised 4 replicates of  $6m \times 15m$ . Application was by hand-held boom with low volume sprays of 60L/ha. Samples of seed, trash and lint were taken at 80 days after spray 8, 41 days after spray 9 and 27 days after the final application. The samples were frozen on the day of harvest, ginned, then refrozen for dispatch to the laboratory. The interval between sample collection and analysis was 172 days; samples were stored between 2-4 °C prior to analysis. Samples from each replicate plot were analysed separately.

Table 132: Total endosulfan residues in cotton seed, lint and trash (98AUSE03/NN09).

Trial site, Year,	Application	on	WHP	Sample		Residues	(mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Narrabri, NSW,	735	8	80	Cotton seed	0.008	0.009	0.016	0.033
1999, (V2 Ingard)					< 0.005	< 0.005	0.008	0.008
					< 0.005	0.008	0.023	0.031
					< 0.005	0.005	0.012	0.017
		9	41	Cotton seed	< 0.005	< 0.005	0.009	0.009
					0.005	0.008	0.016	0.029
					0.006	0.012	0.024	0.042

1 '		1	1	0.005	0.005	0.000	0.00
				< 0.005	< 0.005	0.008	0.08
	10	27	Cotton seed	0.010	0.014	0.031	0.055
				0.006	0.012	0.019	0.037
				0.009	0.012	0.026	0.047
				< 0.005	< 0.005	0.007	0.007
	8	80	Cotton trash	0.030	0.057	0.12	0.21 (0.41)
				0.009	0.019	0.042	0.07 (0.13)
				0.14	0.23	1.1	1.5 (2.45)
				< 0.005	0.016	0.036	0.052 (0.13)
	9	41	Cotton trash	0.13	0.19	0.64	0.96 (2.13)
				0.14	0.41	1.1	1.6 (2.67)
				0.04	0.11	0.28	0.43 (0.93)
				0.15	0.35	0.71	1.2 (2.85)
	10	) 27	Cotton trash	0.33	0.56	2.5	3.4 (11.72)
				0.036	0.14	0.36	0.54 (0.93)
				0.56	1.1	1.2	2.9 (5.92)
				0.25	0.46	1.0	1.7 (2.93)
			Controls	0.11	0.20	0.32	0.63 (1.57)
				0.018	0.043	0.12	0.18 (0.44)
				0.038	0.067	0.16	0.26 (0.56)
				0.026	0.055	0.15	0.23 (0.55)
	8	80	Cotton lint	0.005	0.009	0.017	0.031
				< 0.005	0.010	0.016	0.026
				0.016	0.026	0.026	0.074
				< 0.005	0.005	0.012	0.017
	9	41	Cotton lint	0.007	0.024	0.053	0.084
				0.014	0.029	0.060	0.10
				0.008	0.018	0.040	0.066
				< 0.005	0.008	0.016	0.024
	8	27	Cotton lint	0.017	0.029	0.023	0.069
		-	Cotton mit	0.025	0.066	0.023	0.19
				0.023	0.000	0.098	0.13
				0.043	0.056	0.19	0.33
I OD 0 005 /l I	00 01/			0.024	0.030	0.037	0.14

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg. Corrected for moisture content.

Finite residues of endosulfan were present in untreated samples of cotton trash, with levels ranging 0.13 to 11.7 mg/kg. The moisture content in the trash samples (control and treated) ranged from 39 – 71%; mean 52%. The individual trash samples are reported on an as received basis and are corrected for moisture content (italicised figures). Recoveries in fortified seed, trash and lint are reported in Table 131.

Determination of Endosulfan Residues in Soybean. AgrEvo Trial No ID98AUSE05/QD13-98, 22 June 1999. Determination of Endosulfan Residues in Soybean, State Chemistry Laboratory Report No. 9906004.

Thiodan 350 EC was applied to soybean crops at a trial site in Gatton, Qld. The crops were treated with two applications of Thiodan at 735 g ai/ha (1 $\times$ ). The sprays were applied at 7 day intervals with application timings ranging from 8 to 4 weeks before harvest or from early flowering to late flowering. Individual plots were 15m  $\times$  6 rows, with 4 replications per treatment. The sprays were applied by air pressurised overhead boom. Samples of beans and trash were taken at 49, 41, 34 and 27 days after the final spray. The interval between sample collection and analysis was 144 days; samples were stored at 2-4 °C prior to analysis. Samples from replicate plots were analysed separately. Results are not shown as there was difficulty identifying the control samples from the treated samples.

Determination of Thiodan EC Residues in Soybeans. AgrEvo Trial No ID98AUSE05/QT19-98, 14 October 1999. Determination of Endosulfan Residues in Soybean, State Chemistry Laboratory Report No. 9907105.

At a site in Norwin Qld, soybeans were treated with two applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). Sprays were applied at 7 day intervals and application timings ranged from 8 weeks to 4 weeks before harvest. Individual plots were 8 rows  $\times$  20m, with 4 replications per treatment. The sprays were applied by hand-held boom. Samples of beans and trash were collected at 49, 42, 35 and 28 days after the final spray. The interval between sample collection and analysis was 121 days; samples were stored at 2 – 4 °C prior to analysis. Samples from replicate plots were analysed separately. Results are shown in Table 133.

Table 133: Residues in soybeans and trash following application of Thiodan EC at 735 g ai/ha (98AUSE05/QT19-98).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Norwin, Qld, 1999	735	2	49	Soya beans	< 0.005	< 0.005	0.007	0.007
(A55939)					< 0.005	< 0.005	0.007	0.007
					< 0.005	< 0.005	0.007	0.007
					< 0.005	< 0.005	0.007	0.007
		2	42	Soya beans	< 0.005	0.006	0.024	0.030
					< 0.005	0.009	0.024	0.033
					< 0.005	0.009	0.024	0.033
					< 0.005	< 0.005	0.014	0.014
		2	35	Soya beans	< 0.005	< 0.005	0.013	0.013
					< 0.005	< 0.005	0.015	0.015
					0.006	0.010	0.020	0.036
					< 0.005	< 0.005	0.020	0.020
		2	28	Soya beans	< 0.005	0.007	0.011	0.018
					< 0.005	0.007	0.013	0.020
					< 0.005	0.010	0.023	0.033
					0.005	0.013	0.024	0.042
		2	49	Trash	0.012	0.048	0.61	0.67 (1.76)
					0.007	0.024	0.18	0.21 (0.54)
					0.005	0.020	0.20	0.22 (0.61)
					0.006	0.036	0.46	0.50 (1.35)
		2	42	Trash	0.027	0.17	0.78	0.98 (2.72)
					0.033	0.17	0.82	1.0 (3.03)
					0.026	0.13	0.75	0.91 (2.33)
					0.031	0.19	0.80	1.0 (2.77)
		2	35	Trash	0.038	0.16	0.71	0.91 (2.16)
				0.097	0.41	1.1	1.6 (4.10)	
					0.040	0.26	1.2	1.5 (4.05)
				0.036	0.22	0.76	1.0 (2.56)	
		2	28	Trash	0.044	0.22	0.93	1.2(3.24)
					0.062	0.21	0.98	1.2 (3.0)
					0.085	0.42	1.3	1.8 (4.39)
					0.030	0.12	0.40	0.55 (1.45)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for beans and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 54 - 67% in trash samples.

Table 134: Recoveries of endosulfan in fortified soya bean and trash samples (98AUSE04/QT19-98).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Soya beans	0.1	77, 93, 106, 106	78, 86, 95, 101	71, 79, 109, 142			
Trash	0.1	82, 93, 116, 128	103, 106, 118, 136	102, 126, 135, 142			

Determination of Thiodan EC Residues in Sunflowers. AgrEvo Trial No ID98AUSE05/NN13, 27 October 1999. Determination of Endosulfan Residues in Sunflower, State Chemistry Laboratory Report No. 9904014.

At a site in Edgeroi NSW, sunflowers were treated with two applications of Thiodan EC at a rate of 735 g ai/ha (1 $\times$ ). Sprays were applied at 7 day intervals and application timings ranged from 8 weeks to 5 weeks before harvest or from early flowering to head ripening. Individual plots were 6 rows  $\times$  15m, with 4 replications per treatment. The sprays were applied by hand-held boom. Samples of seeds and trash were collected at 49, 43, 34 and 29 days after the final spray. The interval between sample collection and analysis was 230 days; samples were stored at 2 – 4  $^{\circ}$ C prior to analysis. Samples from replicate plots were analysed separately. Results are shown in Table 135.

Table 135: Residues in sunflower seeds and trash following application of Thiodan EC at 735 g ai/ha (98AUSE05/NN13).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)	_	α	β	$SO_4$	Total
Edgeroi, NSW,	735	2	49	Sunflower seed	0.18	0.22	0.061	0.46
1998, (Sunolic 3)					0.095	0.15	0.053	0.30
					0.076	0.11	0.039	0.22
					0.15	0.21	0.062	0.42
		2	43	Sunflower seed	0.10	0.13	0.037	0.27
					0.16	0.21	0.067	0.44
					0.083	0.12	0.032	0.24
					0.35	0.47	0.084	0.90
		2	34	Sunflower seed	0.052	0.092	0.038	0.18
					0.099	0.24	0.17	0.51
					0.092	0.12	0.019	0.23
					0.049	0.086	0.029	0.16
		2	29	Sunflower seed	0.086	0.14	0.059	0.28
					0.069	0.15	0.091	0.31
					0.054	0.10	0.052	0.21
					0.046	0.063	0.015	0.12
		2	49	Trash	5.3	5.0	2.4	13 (14.6)
					4.1	5.5	4.2	14 (29.8)
					6.2	6.6	5.6	18 (46.1)
					8.9	9.6	8.7	27 (42.8)
		2	43	Trash	4.2	3.2	2.2	9.6 (14.5)
					9.8	9.3	10	29 (82.9)
					4.6	4.7	4.3	14 (38.9)
					7.1	4.8	4.9	17 (34.7)
		2	34	Trash	6.9	6.2	4.6	18 (62.1)
				6.3	5.7	6.6	19 (82.6)	
				5.3	5.8	6.0	17 (58.6)	
				4.3	3.4	3.5	11 (39.3)	
	2	29	Trash	1.9	2.0	2.2	6.1 (14.9)	
					1.2	0.9	1.0	3.1 (12.4)
					1.5	1.4	0.9	3.9 (13.0)
					0.8	1.0	0.9	2.7 (9.0)

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for seed and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 10-77% in trash samples.

The data for both seed and trash show that there is considerable variation in the residues found at each sampling interval and in each replicate sample. This is particularly notable in the trash samples where residues range by a factor of 4 in the day 43 replicate samples. The highest residues in trash were 82.9 mg/kg in a sample taken 43 days after treatment.

Table 136: Recoveries of endosulfan in fortified sunflower seed and trash samples (98AUSE05/NN13).

Sample	Fortification (mg/kg)	% Recovery				
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>		
Sunflower seed	0.1	72 (3), 79, 84, 87,	67, 73, 75, 82, 86,	70, 76, 77, 83, 84, 87		
		102	88, 90	(2)		
Trash	0.1	99, 104, 106, 113	101, 114, 128, 138	76, 82, 88, 91		

Evaluation Of Thiodan EC For Residue Analysis In Canola, Aventis Trial No. ID99AUSA03, June 2000. Determination of Endosulfan Residues in Canola, State Chemistry Laboratory Report No. 0004081 and 0004084.

In a trial conducted at York W.A., canola plants were treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was immediately after seeding and the second application was at flowering. The trial plot was  $2m \times 10m$  and included four replications. Sprays were applied using an air operated hand boom. Samples of forage following the single sprays were collected at 61 days after seeding. Grain and trash samples were collected at harvest; 49 days after two applications or 203 days after a single application. The interval between sample collection and analysis was approximately 12 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed. Results are shown in Table 137.

Table 137: Residues in canola, forage and trash following application of Thiodan EC. (99AUSE03/LM09).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
York, W.A., 1999	175	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
(Karoo)	350	1	61	Forage	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	175	1	203	Canola	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	203	Canola	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	49	Canola	< 0.005	< 0.005	0.008	0.008
					< 0.005	< 0.005	0.008	0.008
					< 0.005	0.007	< 0.005	0.007
					< 0.005	0.006	< 0.005	0.006
	175	1	203	Trash	< 0.005	< 0.005	0.006	0.006
					< 0.005	< 0.005	0.006	0.006
	350	1	203	Trash	< 0.005	< 0.005	0.009	0.009 (0.01)
					< 0.005	< 0.005	0.006	0.006
	350 + 735	1+1	49	Trash	0.045	0.092	0.20	0.34 (0.37)
					0.032	0.070	0.10	0.20 (0.22)
				Controls	< 0.005	0.006	0.006	0.012 (0.013)
					< 0.005	< 0.005	0.007	0.007

 $LOD=0.005\ mg/kg;\ LOQ=0.1\ mg/kg$  in seed, forage and trash.

 $Figures\ of\ total\ endosulfan\ in\ parentheses\ are\ corrected\ for\ moisture\ content;\ moisture\ ranged\ from\ 87-89\%\ in\ forage\ and\ 8-11\%\ in\ trash\ samples.$ 

Finite residues were present in untreated control samples of canola trash, at levels comparable to those present in the treated samples taken at harvest.

Table 138: Recoveries of endosulfan in fortified canola, forage and trash samples (99AUSA03).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Canola	0.1	74	73	74			
Trash	0.1	106	121	112			
Forage	0.1	74	74	77			

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Mallala, South Australia, Australia, 1999. Aventis Trial Number 9213, Study Number ID99AUSA03, September 2000.

Determination of Endosulfan Residues in Canola, State Chemistry Laboratory Report No. 0007072.

In a trial at Mallala S.A., canola was treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first

application was pre-emergent and the later application was at mid-flowering. The trial plot was  $2m \times 15m$  and included four replications. Sprays were applied using a gas pressured back pack with handheld boom. Samples of forage from all treatments were collected at 47 days after post-emergent application, when the crop was at the 6-7 leaf stage. Grain and straw samples were collected at harvest; 121 days after two applications or 211 days after a single application. The interval between sample collection and analysis was approximately 15 months; samples were stored at -20 °C prior to analysis. Samples from 2 replicate plots were combined (2 analyses from 4 replicate plots) and analysed, except for the canola and straw

samples taken at 121 days following two applications, where the replicates were analysed individually. Results are shown in Table 139.

Table 139: Residues in canola, forage and trash following application of Thiodan EC. (99AUSA03/9213).

Trial site, Year,	Applicati	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Mallala, SA, 1999	175	1	47	Forage	< 0.005	< 0.005	< 0.005	< 0.005
(Monty)				_	< 0.005	0.011	0.011	0.022 (0.22)
	350	1	47	Forage	< 0.005	0.012	0.016	0.028 (0.23)
					< 0.005	0.011	0.014	0.025 (0.25)
	175	1	211	Canola	0.010	< 0.005	< 0.005	0.010
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	211	Canola	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1 + 1	121	Canola	0.008	< 0.005	0.018	0.026
					< 0.005	< 0.005	0.022	0.022
					< 0.005	< 0.005	0.014	0.014
					< 0.005	< 0.005	0.022	0.022
	175	1	211	Trash	0.008	< 0.005	< 0.005	0.008
					< 0.005	< 0.005	0.025	0.025 (0.027)
	350	1	211	Trash	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.008	0.008
	350 + 735	1+1	121	Trash	0.11	0.023	0.19	0.32 (0.35)
	350 1 755	1.1	121	11451	< 0.005	0.038	0.17	0.21 (0.23)
					< 0.005	0.012	0.094	0.11 (0.12)
					< 0.005	0.008	0.12	0.13 (0.14)
				Controls	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	0.008	0.008

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg in seed, forage and trash.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 88 – 90% in forage and 8 – 11% in trash samples.

Table 140: Recoveries of endosulfan in fortified canola, forage and trash samples (99AUSA03).

Sample	Fortification (mg/kg)	% Recovery α-endosulfan β-endosulfan endosulfan SO <sub>4</sub>						
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>				
Canola	0.1	105	101	102				
Trash	0.1	191	92	79				
Forage	0.1	81	85	87				

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Bathurst New South Wales, Australia, 1999. Aventis Trial Number 9211, Study Number ID99AUSA03, September 2000.

Determination of Endosulfan Residues in Canola, State Chemistry Laboratory Report No. 0008139.

In a trial at Bathurst NSW, canola was treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at flowering. The trial plot was  $2m \times 15m$  and included four replications. Sprays were applied using a gas pressured back pack with hand-held

boom. Samples of forage from all treatments were collected at 98 days after post-emergent application, when the crop was at the 6-8 leaf stage. No grain or straw samples were collected due to premature harvest of the trial site. The interval between sample collection and analysis was approximately 27 months; samples were stored at -20 °C prior to analysis. Samples from each replicate plots were analysed individually. Results are shown in Table 141.

Table 141: Residues in canola forage following application of Thiodan EC. (99AUSA03/9211).

Trial site, Year,	ar, Application			Sample	Residues (mg/kg)				
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total	
Bathurst, NSW,	175	1	98	Forage	< 0.005	< 0.005	< 0.005	< 0.005	
1999, (Pinnacle)					< 0.005	< 0.005	< 0.005	< 0.005	
					< 0.005	0.005	0.007	0.012 (0.095)	
					< 0.005	< 0.005	< 0.005	< 0.005	
	350	1	98	Forage	< 0.005	0.005	0.006	0.011 (0.09)	
					< 0.005	< 0.005	< 0.005	< 0.005	
					< 0.005	0.005	0.005	0.010 (0.11)	
					< 0.005	0.005	0.005	0.011 (0.10)	

LOD = 0.005 mg/kg; LOQ = 0.1 in forage.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 87 – 91% in forage samples.

Recoveries in forage with fortification at 0.1 mg/kg were 115, 116 and 112% for  $\alpha$ -,  $\beta$ - and endosulfan sulfate, respectively.

Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Dookie, Victoria, Australia, 1999. Aventis Trial Number 9212, Study Number ID99AUSA03, September 2000.

Determination of Endosulfan Residues in Canola, State Chemistry Laboratory Report No. 0007069.

In a trial at Dookie Victoria, canola was treated with either a single application of Thiodan 350 EC at a rate of 175 or 350 g ai/ha, or two applications of Thiodan 350 EC at 350 and 735 g ai/ha. The first application was pre-emergent and the later application was at 59% petal fall.

The trial plot was  $2.5 \mathrm{m} \times 15 \mathrm{m}$  and included four replications. Sprays were applied using a gas pressured back pack with hand-held boom. Samples of forage from all treatments were collected at 79 days after the post-emergent application, when the crop was at the 10 leaf stage. Grain and straw samples were collected at harvest, at 188 days after a single application or 54 days after two applications. The interval between sample collection and analysis was approximately 30 months; samples were stored at -20 °C prior to analysis. Samples from replicate plots were analysed individually for seed and trash samples; forage samples were analysed as composites of two replicates. Results are shown in Table 142.

Table 142: Residues in canola, forage and straw following application of Thiodan EC. (99AUSA03/9212).

Trial site, Year,	Application	on	WHP	Sample		Residu	es (mg/kg)	
(Variety)	Rate (g ai/ha)	No.	(days)		α	β	$SO_4$	Total
Dookie, VIC, 1999	175	1	79	Forage	< 0.005	< 0.005	0.005	0.005
(Pinnacle)					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	79	Forage	< 0.005	< 0.005	0.005	0.005 (0.045)
					< 0.005	< 0.005	0.005	0.005 (0.044)
	175	1	188	Canola	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350	1	188	Canola	< 0.005	< 0.005	< 0.005	< 0.005
					< 0.005	< 0.005	< 0.005	< 0.005
	350 + 735	1+1	54	Canola	0.017	0.014	0.12	0.15
					0.009	0.009	0.046	0.064
					0.023	0.016	0.15	0.19
					0.014	0.009	0.097	0.12

175	1	188	Straw	< 0.005	< 0.005	0.032	0.032 (0.035)	
				< 0.005	0.022	0.065	0.087 (0.096)	
350	1	188	Straw	< 0.005	< 0.005	0.043	0.043 (0.047)	
				< 0.005	< 0.005	0.048	0.048 (0.053)	
350 + 735	1+1	54	Straw	0.052	0.15	0.99	1.20 (1.34)	
				0.014	0.040	0.27	0.32 (0.35)	
				0.065	0.15	0.97	1.20 (1.34)	
				0.043	0.12	0.88	1.0(1.11)	

LOD = 0.005 mg/kg; LOQ = 0.1 mg/kg for seed and forage.

Figures of total endosulfan in parentheses are corrected for moisture content; moisture ranged from 87-89% in forage samples and 9-11% in straw samples.

Table 143: Recoveries of endosulfan in fortified canola and forage (99AUSA03/9212).

Sample	Fortification (mg/kg)	% Recovery					
		α-endosulfan	β-endosulfan	endosulfan SO <sub>4</sub>			
Canola	0.1	72	68	68			
Forage	0.1	96	93	98			

Recoveries in straw were not reported.

#### 8.6 Animal Transfer Studies

#### **8.6.1** Cattle studies

Residues of  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate in milk and edible cattle tissues following 28 days feeding to lactating cows Endosulfan technical product, M.H. Peatman, C.M. Reynolds, J.H.M. Bright and T.L. Godfrey. AgrEvo U.K. Report No. RESID/99/8, Study ID 205/05/001, 26 May 1999.

Lactating cows were dosed for 28 consecutive days at concentrations of 0, 4, 12 and 30 ppm of technical endosulfan in the feed. Groups comprised 3 animals per dose, with an additional 4 animals for a 21 day depuration phase at the end of the 28 day dosing period. Dosing was conducted by feeding a dairy concentrate ration at milking (twice daily). At each milking, 3 kg of feed ration was weighed and a 10ml aliquot of the dose was distributed over the surface of the feed. The dose was not mixed any further in case of losses around the surface of the feed container. The doses were equivalent to 0.123, 0.352 and 0.835 mg/kg bodyweight/day<sup>3</sup>.

All cows were milked twice daily and milk yields were recorded for each day. Milk from the am and pm samples was combined prior to mixing and sub-sampling and freezing. Analysis was completed within 3 days of collection to demonstrate that a residue plateau had been reached in milk within the 28 day dosing period. Cream and skimmed milk samples were taken by removing the appropriate layer in the thawed sample prior to shaking.

Animals involved in the feeding phase of the study were sacrificed 16 – 24 hours after the final dose. During the 21-day depuration phase, animals were sacrificed at 7, 14 and 21 days following cessation of dosing at 30 ppm. Samples of muscle (loin, flank and diaphragm), liver, kidney and composite fat (subcutaneous, perirenal and omental) were taken from each animal. Samples were analysed within 3 – 4 months of slaughter. Storage stability in animal tissues and milk is discussed in section 5.3.2.

-

<sup>&</sup>lt;sup>3</sup> Average bodyweights and mean intakes were recorded in the study; mean feed intakes were 19.1, 19.4, 17.7 and 15.9 kg/day for groups 1,2, 3 and 4 respectively.

The reported limits of quantitation were 0.01 mg/kg for milk, muscle, liver and kidney and 0.05 mg/kg for fat.

The residue data for milk and tissues are shown in Table 144 and recoveries in individual matrices are given in Table 148. The depuration of endosulfan residues in various tissues up to 21 days after cessation of feeding is shown in Table 147.

Table 144: Residues in tissues of dairy cows following feeding of endosulfan at 4, 12 and 30 ppm for 28 days (Peatman et al. 1999).

Sample	mple Dose (ppm feed) Animal Endosulfan Residues (mg/kg)					
•			α	β	SO <sub>4</sub>	Total
Muscle	4	4	ND	ND	0.03	0.03
		5	ND	< 0.01	0.07	0.07
		6	ND	ND	0.03	0.03
	12	7	< 0.01	< 0.01	0.14	0.14
		8	ND	ND	0.32, 0.45, 0.39	0.45
		9	< 0.01	< 0.01	0.11	0.11
	30	10	<0.01, ND	<0.01, ND	1.4 (1.7, 2.0)	2.0
		11	< 0.01	< 0.01	0.28	0.28
		12	< 0.01	< 0.01	0.31	0.31
	Controls	1	<0.001, ND	ND, ND	0.0021, 0.0118	0.0118
		2	< 0.001	ND	0.003	0.003
		3	ND, ND	ND	0.002, 0.005	0.005
Liver	4	4	< 0.01	ND	0.55	0.55
		5	< 0.01	< 0.01	0.98	0.98
		6	ND	ND	0.59	0.59
	12	7	0.01	< 0.01	2.0	2.01
		8	ND, ND	0.01, ND	3.1, 2.5, 2.8	3.1
		9	ND	ND	1.2	1.2
	30	10	ND, ND	ND, 0.02	4.6, 2.8	4.6
		11	ND	ND	3.0	3.0
		12	ND	ND	3.7	3.7
	Controls	1	ND	ND	0.006	0.006
		2	ND	ND	0.016	0.016
		3	ND, ND	ND, ND	0.011, 0.014	0.014
Kidnev	4	4	< 0.01	< 0.01	0.08	0.08
		5	< 0.01	< 0.01	0.08	0.08
		6	< 0.01	< 0.01	0.06	0.06
	12	7	< 0.01	< 0.01	0.28	0.28
		8	ND	< 0.01	0.40	0.40
		9	< 0.01	< 0.01	0.24	0.24
	30	10	ND	< 0.01	0.85	0.85
		11	ND	< 0.01	0.55	0.55
		12	ND	< 0.01	0.60	0.60
	Controls	1	ND, <0.001	ND, ND	0.004, 0.003	0.004
		2	< 0.001	ND	0.003	0.003
		3	< 0.001	ND	0.003	0.003
Fat	4	4	ND	< 0.05	1.2	1.2
(composite)		5	ND	< 0.05	1.7	1.7
		6	ND	< 0.05	1.4	1.4
	12	7	ND	0.07	4.8	4.9
		8	ND	0.05	6.7	6.8
		9	ND	< 0.05	2.7	2.7
	30	10	< 0.05	0.07	12	12.1
		11	< 0.05	0.08	9.9	10
		12	ND	< 0.05	7.9	7.9
	Controls	1	ND	ND	ND, ND, 0.0137,	0.0184
			0.000	0.002	0.0184	0.02
		2	0.002	0.002	0.018	0.02
		3	ND	ND	0.012	0.012

LOQ = 0.01 mg/kg in muscle, liver and kidney; LOQ = 0.05 mg/kg in fat.

Table 145: Endosulfan residues in whole milk following dosing at 4, 12 and 30 ppm in the feed for 28 days

Days			ndosulfan resi	dues in milk o	f individual ar	nimals (mg/kg	; Feeding Ph	ase	
4 ppm		4	00		5	l go		6	go.
1	α ND	β	SO <sub>4</sub>	α ND	β	SO <sub>4</sub>	α ND	β	SO <sub>4</sub>
-1 1	<0.01	ND ND	<0.01	ND ND	ND ND	<0.01	ND ND	ND ND	<0.01
4	<0.01	< 0.01	0.07	<0.01	< 0.01	0.05	ND ND	<0.01	0.08
7	< 0.01	< 0.01	0.06	< 0.01	< 0.01	0.06	ND	< 0.01	0.06
10	< 0.01	< 0.01	0.07	< 0.01	< 0.01	0.07	ND	< 0.01	0.06
13	< 0.01	< 0.01	0.07	< 0.01	< 0.01	0.08	ND	< 0.01	0.07
16	< 0.01	< 0.01	0.06	< 0.01	< 0.01	0.07	ND	< 0.01	0.08
19	< 0.01	< 0.01	0.06	<0.01	< 0.01	0.07	ND	< 0.01	0.07
22 25	<0.01 <0.01	<0.01 <0.01	0.06 0.07	<0.01 <0.01	<0.01 <0.01	0.06 0.07	ND ND	<0.01 <0.01	0.06 0.06
28	< 0.01	< 0.01	0.07	<0.01	<0.01	0.07	ND ND	< 0.01	0.06
12 ppm	₹0.01	7	0.03	₹0.01	8	0.00	ND	9	0.00
-1	ND	ND	ND	ND	ND	ND	ND	ND	ND
1	ND	< 0.01	0.02	ND	< 0.01	0.02	ND	ND	0.02
4	< 0.01	< 0.01	0.18	ND	< 0.01	0.28	ND	< 0.01	0.14
7	< 0.01	< 0.01	0.24	ND	< 0.01	0.28	ND	< 0.01	0.17
9	< 0.01	< 0.01	0.24	ND	< 0.01	0.30	ND	< 0.01	0.15
10	ND	< 0.01	0.26	ND	<0.01	0.35	ND	< 0.01	0.19
13 16	ND ND	<0.01 <0.01	0.23 0.20	ND ND	<0.01 <0.01	0.40 0.31	ND ND	<0.01 <0.01	0.19 0.22
19	<0.01	< 0.01	0.20	ND ND	<0.01	0.31	ND ND	< 0.01	0.22
22	ND	< 0.01	0.22	ND ND	< 0.01	0.34	ND ND	<0.01	0.19
25	ND	< 0.01	0.24	ND	< 0.01	0.48	ND	< 0.01	0.22
28	ND	< 0.01	0.24	ND	< 0.01	0.41	ND	< 0.01	0.18
30 ppm		10			11			12	
-1	ND	ND	ND	ND	ND	ND	ND	ND	ND
1	ND	ND	0.03	ND	ND	0.06	ND	ND	0.02
4	ND	< 0.01	0.60	ND	<0.01	0.61	ND	ND	0.38
7 10	ND ND	<0.01 <0.01	0.60 0.53	ND ND	ND <0.01	0.45 0.78	ND ND	ND ND	0.44 0.38
13	ND ND	< 0.01	0.53	ND ND	< 0.01	0.78	ND ND	<0.01	0.58
16	ND	< 0.01	0.60	ND	< 0.01	0.73	ND	< 0.01	0.54
19	ND	< 0.01	0.75	ND	< 0.01	0.70	ND	< 0.01	0.60
22	ND	< 0.01	0.70	ND	< 0.01	0.63	ND	< 0.01	0.59
25	ND	ND	0.63	ND	< 0.01	0.52	ND	< 0.01	0.54
28	ND	ND	0.74	ND	< 0.01	0.66	ND	< 0.01	0.59
20				D	epuration Pha	ise	ı		
<b>30 ppm</b> 26 (-2)	< 0.01	0.01	0.95	ND	<b>14</b> <0.01	0.70	< 0.01	15 <0.01	0.82
29 (1)	<0.01	< 0.01	0.93	ND ND	<0.01	0.70	<0.01	<0.01	0.60
32 (4)	ND	< 0.01	0.10	ND ND	<0.01	0.13	ND	ND	0.16
35 (7)	ND	ND	0.06	ND	ND	0.06	ND	ND	0.09
38 (10)				ND	ND	0.08	ND	ND	0.11
41 (13)				ND	ND	0.06	ND	ND	0.08
44 (16)							ND	ND	0.05
47 (19)							ND ND	ND ND	0.06
50 (22) <b>30 ppm</b>		16			<u> </u>	<u> </u>	ND	ND	0.04
26 (-2)	< 0.01	<0.01	1.3	1					
29 (1)	ND	< 0.01	1.1						
32 (4)	ND	< 0.01	0.31						
35 (7)	ND	ND	0.16						
38 (10)	ND	ND	0.25						
41 (13)	ND	ND	0.12						
44 (16)	ND	ND	0.04						
47 (19) 50 (22)	ND ND	ND ND	0.06 0.04						
	mo/ko in milk	ND	0.04	l			l		

LOQ = 0.01 mg/kg in milk.

Table 146: Endosulfan residues in cream and skim milk from animals dosed at 12 ppm for 9 days.

	Cream									
Animal	Dose	Day	α	β	$SO_4$	Total (mg/kg)				
1	0	9	ND	ND	0.02	0.02				
7	12	9	< 0.01	0.02	1.4	1.42				
8	12	9	ND	< 0.01	0.89	0.89				
9	12	9	ND	< 0.01	0.81	0.81				
			Skim Milk							
1	0	9	< 0.001	ND	0.006	0.006				
7	12	9	ND	< 0.01	0.12	0.12				
8	12	9	ND	< 0.01	0.26	0.26				
9	12	9	ND	ND	0.13	0.13				

Table 147: Depuration of endosulfan residues in cattle tissues following withdrawal from dosing at 30 ppm

Sample	Depuration	Animal		Endosulfan	Residues (mg/kg)	
_	Interval (days)		α	β	$SO_4$	Total
Muscle	0	10	ND, <0.01	ND, <0.01	2.0, 1.4, 1.7	2.0, 1.4, 1.7
	0	11	< 0.01	< 0.01	0.28	0.28
	0	12	< 0.01	< 0.01	0.31	0.31
	7	13	ND	ND	0.06	0.06
	14	14	ND	ND	0.04	0.04
	21	15	ND	ND	0.03	0.03
	21	16	ND	ND	0.02	0.02
Liver	0	10	ND, ND	ND, 0.02	1.0, 4.6, 2.8	1, 4.6, 3
	0	11	ND	ND	3.0	3.0
	0	12	ND	ND	3.7	3.7
	7	13	ND	ND	0.76	0.76
	14	14	ND	ND	0.54	0.54
	21	15	ND	ND	0.36	0.36
	21	16	ND	ND	0.36	0.36
Kidney	0	10	ND	< 0.01	0.85	0.85
	0	11	ND	< 0.01	0.55	0.55
	0	12	ND	< 0.01	0.60	0.60
	7	13	ND	< 0.01	0.10	0.10
	14	14	ND	ND	0.07	0.07
	21	15	< 0.01	ND	0.06	0.06
	21	16	ND	ND	0.05	0.05
Fat	0	10	< 0.05	0.07	12	12
(composite)	0	11	< 0.05	0.08	9.9	10
	0	12	ND	< 0.05	7.9	8
	7	13	ND, ND	ND, ND	4.8, 5.3	4.8, 5.3
	14	14	ND, ND	ND, ND	2.1, 2.1	2.1, 2.1
	21	15	ND, ND	ND, ND	1.3, 1.3	1.3, 1.3
	21	16	ND, ND	ND, ND	0.98, 1.0	1, 1

Table 148: Recoveries of  $\alpha$ -,  $\beta$ - endosulfan and endosulfan sulfate in fortified tissues and milk.

Sample	Fortification Level (mg/kg)	% Recovery					
		α	β	SO <sub>4</sub>			
Muscle	0.01	61, 62, 64, 70	58, 60, 63, 71	81, 92, 93, 94			
	0.05	63, 63, 70	64, 64, 75	73, 89, 91			
	0.4	85	85	84			
	1	80	75	76			
	2	84	85	79			
Kidney	0.01	61, 62, 66	66, 67, 69	67, 76, 99			
	0.05	61, 67	64, 72	73, 73			
	0.16	91	96	77			
	0.2	72	74	67			
	0.4	69	74	68			
Liver	0.01	63, 67, 68	74, 79, 84	88, 91, 93			
	0.05	67, 68	77, 81	66, 68			
	0.2	83	89	96			
	1.2	90	92	89			
	2	84	88	89			

Fat	0.05	76, 76	81, 85, 86	76, 80, 84
	0.25	75, 87	79, 85	73, 80
	1	82, 92	85, 102	82, 90
	2	91	102	101
	4	88	92	90
	8	98	108	113
Whole milk	0.01	75, 78, 79	91, 92, 93	91, 91, 97
	0.02	77	82	85
	0.05	71, 72	93, 93	78, 81
	0.1	66, 68, 78, 79, 80, 80,	86, 87, 89, 94, 97, 98,	73, 76, 78, 80, 81, 83,
		88, 95	100, 123	89, 99
	0.2	60, 62,62, 63, 65, 71,	70, 79, 80, 81, 87, 91,	67, 68, 73, 77, 79, 85,
		75, 87, 98, 104	94, 98, 117, 121	88, 92, 93, 102
	0.3	61, 72, 72, 84, 90	71,81, 91, 88, 106	72, 73, 79, 79, 89
	0.5	71, 74, 75, 77, 78, 86,	94, 95, 95, 99, 107,	66, 76, 80, 82, 93, 95,
		97	107, 126	106
	1	69, 69, 70	81, 86, 86	83, 87, 88
Cream	1	60	77	68

#### 8.6.2 Pigs

Maier – Bode, H. Investigations on the Persistence of the Insecticide Endosulfan in the Vegetable and Animal Organism. Pharmakologisches Institut der Rheinischen Friedrich Wilhelms-Universitat, Bonn. Document A4 047. July 1966

In a brief report, three sows were fed 2 ppm endosulfan by gelatine capsule for 27, 54 or 81 consecutive days. The animals were slaughtered 24 hours after the final dose and total endosulfan ( $\alpha$ -,  $\beta$ - and  $SO_4$ ) was measured in various tissues. Mean levels of 0.07, 0.09 and 0.04 mg/kg endosulfan were found in composite samples of extractable fat from the neck, belly, omentum and kidney. Levels in other tissues were <0.01 mg/kg  $\alpha$ - and  $\beta$ -endosulfan and <0.02 mg/kg endosulfan  $SO_4$ .

#### 8.6.3 Sheep

Maier – Bode, H. Properties, Effect, Residues and Analytics of the Insecticide Endosulfan. Residues Reviews 1967.

In a review of endosulfan, a description is given of a feeding study conducted in milk sheep in  $1965^4$ . The sheep were given a daily oral dose of 15 mg of endosulfan for 28 consecutive days. During the dosing period, approximately 20% of the administered dose was excreted as unchanged endosulfan (technical), with a small proportion in the urine as water soluble transformation products, such as endosulfan diol. In milk, levels of 0.01-0.02 mg/kg endosulfan sulfate were detected; no  $\alpha$ - or  $\beta$ -endosulfan was present. In animals slaughtered on day 20 of the study, a maximum of 0.3 mg/kg endosulfan sulfate was found in composite renal and intestinal fat samples; no  $\alpha$ - or  $\beta$ -endosulfan was found.

#### 8.7 PROCESSING STUDIES

Processing studies in addition to those reported in the residues trials were not provided.

\_

<sup>&</sup>lt;sup>4</sup> Gorbach, S.G. (*Farbwerke Hoechst AG*): Untersuchungen über Thiodan im Stoffwechsel von Milchschafen. Unpublished internal report 1965. Untersuchungen über Thiodan im Stoffwechsel von Milchschafen, Növényvédelmi Tudományos értekezlet 1966, Februar 22 – 25. 88/1 – 7. A. Magyar Agrartudomanyi Egvesület Es Az Agrotröszt Kiadványa, Budapeat 1996.

#### 8.8 RESIDUES REFERENCES

AgrEvo Project ID BJ-95R-14, Laboratory Project ID 95-0061, 13 November 1997. Validation of the Analytical Method for the Determination of Endosulfan ( $\alpha$ ,  $\beta$  and sulfate) in Animal Tissues, Egg (White and Yolk) and dairy Matrices based upon FDA Pesticide Analytical Manual, Volume I Multi-Residue Methodology.

AgrEvo Trial No ID98AUSE02/NN08 – 98, 13 October 1999. Determination of Thiodan EC Residues in Cotton. State Chemistry Laboratory Report No. 9908114. Determination of Endosulfan Residues in Cotton.

AgrEvo Trial No ID98AUSE02/NN08 – 98, Determination of Thiodan EC Residues in Cotton, 13 October 1999. Determination of Endosulfan Residues in Cotton, State Chemistry Laboratory Report No. 9908114.

AgrEvo Trial No ID98AUSE03/NN09 – 98, 13 October 1999. Determination of Thiodan EC Residues in Cotton. State Chemistry Laboratory Report No. 9908115. Determination of Endosulfan Residues in Cotton.

AgrEvo Trial No ID98AUSE03/NN09 – 98, Determination of Thiodan EC Residues in Cotton, 13 October 1999. Determination of Endosulfan Residues in Cotton, State Chemistry Laboratory Report No. 9908115.

AgrEvo Trial No ID98AUSE04/QD16, Determination of Thiodan EC Residues in Cow Peas. 13 October 1999. State Chemistry Laboratory Report No. 9907102. Determination of Endosulfan Residues in Cow Pea.

AgrEvo Trial No ID98AUSE04/QD17-98, Determination of Thiodan EC Residues in Navy Bean, 12 January 2000. Determination of Endosulfan Residues in Navy Beans, State Chemistry Laboratory Report No. 0001086.

AgrEvo Trial No ID98AUSE04/QT17, Determination of Thiodan EC Residues in Cow Peas. 14 October 1999. State Chemistry Laboratory Report No. 9907103. Determination of Endosulfan Residues in Cow Pea.

AgrEvo Trial No ID98AUSE05/NN13, 27 October 1999. Determination of Thiodan EC Residues in Sunflowers. State Chemistry Laboratory Report No. 9904014. Determination of Endosulfan Residues in Sunflower.

AgrEvo Trial No ID98AUSE05/QD13-98, 22 June 1999. Determination of Endosulfan Residues in Soybean. State Chemistry Laboratory Report No. 9906004. Determination of Endosulfan Residues in Soybean.

AgrEvo Trial No ID98AUSE05/QT19-98, 14 October 1999. Determination of Thiodan EC Residues in Soybeans. State Chemistry Laboratory Report No. 9907105. Determination of Endosulfan Residues in Soybean.

AgrEvo Trial No ID98AUSE06/NN10, 27 October 1999. Determination of Thiodan EC Residues in Sorghum. State Chemistry Laboratory Report No. 9904011. Determination of Endosulfan Residues in Sorghum.

AgrEvo Trial No ID98AUSE06/NN11, 27 October 1999. Determination of Thiodan EC Residues in Sorghum. State Chemistry Laboratory Report No. 9904012. Determination of Endosulfan Residues in Sorghum.

AgrEvo Trial No ID98AUSE06/NN12, 27 October 1999. Determination of Thiodan EC Residues in Sorghum. State Chemistry Laboratory Report No. 9904013. Determination of Endosulfan Residues in Sorghum.

AgrEvo Trial No ID98AUSE06/QD12-98, 22 June 1999. Determination of Endosulfan Residues in Grain Sorghum. State Chemistry Laboratory Report No. 9906003. Determination of Endosulfan Residues in Sorghum.

Aventis Trial ID QD27-99, Determination of Endosulfan Residues in Navybean, 18 December 2001. Determination of Endosulfan Residues in Navy Beans, State Chemistry Laboratory Report No. 0108134.

Aventis Trial No. ID99AUSA01, June 2000. Evaluation Of Thiodan EC For Residue Analysis in Wheat. State Chemistry Laboratory Report No. 0004088 and 0004089. Determination of Endosulfan Residues in Wheat.

Aventis Trial No. ID99AUSA02, Evaluation Of Thiodan EC For Residue Analysis in Lupin, June 2000. State Chemistry Laboratory Report No. 0004086 and 0004087. Determination of Endosulfan Residues in Lupin.

Aventis Trial No. ID99AUSA03, June 2000. Evaluation Of Thiodan EC For Residue Analysis In Canola. State Chemistry Laboratory Report No. 0004081 and 0004084. Determination of Endosulfan Residues in Canola.

Aventis Trial Number 9205, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Wheat. One Trial, Bellata, New South Wales, Australia, 1999. State Chemistry Laboratory Report No. 0009204. Determination of Endosulfan Residues in Wheat.

Aventis Trial Number 9206, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Barley. One Trial, Bellata, New South Wales, Australia, 1999. State Chemistry Laboratory Report No. 0009211. Determination of Endosulfan Residues in Barley.

Aventis Trial Number 9208, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Field Peas. One Trial, Freeling South Australia, Australia, 1999. State Chemistry Laboratory Report No. 0007070. Determination of Endosulfan Residues in Field Peas.

Aventis Trial Number 9209, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Faba Beans. One Trial, Bellata, New South Wales, Australia, 1999. Determination of Endosulfan Residues in Faba Beans, State Chemistry Laboratory Report No. 0010004.

Aventis Trial Number 9209, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Barley. One Trial, Nathalia Victoria, Australia, 1999. State Chemistry Laboratory Report No. 0007071. Determination of Endosulfan Residues in Barley.

Aventis Trial Number 9210, Study Number ID99AUSA02, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following Application of Thiodan 350 EC To Chickpeas. One Trial, Bellata, New South Wales, Australia, 1999. State Chemistry Laboratory Report No. 0010005. Determination of Endosulfan Residues in Chickpeas.

Aventis Trial Number 9211, Study Number ID99AUSA03, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Bathurst New South Wales, Australia, 1999. State Chemistry Laboratory Report No. 0008139. Determination of Endosulfan Residues in Canola.

Aventis Trial Number 9212, Study Number ID99AUSA03, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Dookie, Victoria, Australia, 1999. State Chemistry Laboratory Report No. 0007069. Determination of Endosulfan Residues in Canola.

Aventis Trial Number 9213, Study Number ID99AUSA03, September 2000. Collection of Forage, Grain and Straw Samples For Later Residue Analysis Following The Application of Thiodan 350 EC To Canola. One Trial, Mallala, South Australia, Australia, 1999. State Chemistry Laboratory Report No. 0007072. Determination of Endosulfan Residues in Canola.

Gorbach, S.G., Christ, O.E., Kellner, H-M., Kloss, G. and Borner, E. Metabolism of Endosulfan in Milk Sheep, *J. Agr. Food Chem.*, (1968), **16**, 950 – 953.

Klein, E.H.-J. Hoechst Schering AgrEvo Germany. Report No. ER 97 ECS 740, 15 March 1999. Decline of Residues in Orange Fruits and Transfer of Residues into Juice. European Union, Southern Zone 1997.

Klein, E.H.-J. Hoechst Schering AgrEvo Germany. Report No. ER 97 ECS 742, 3 December 1998. Residue Trials on Peaches to Establish a Maximum Residue Level. Determination of Active Substance and Metabolite Decline Following 3 Applications. European Union (Southern Zone) 1997.

Klein, E.H.-J. Hoechst Schering AgrEvo Germany. Report No. ER 98 ECS 740, 21 April 1999. Residues at Harvest in Oranges. European Union, Southern Zone 1998.

Klein, E.H.-J. Hoechst Schering AgrEvo Germany. Report No. ER 98 ECS 742, 8 April 1999. Residues at Harvest in Peaches European Union, Southern Zone 1998.

Klein, E.H.-J. Schering AgrEvo Germany. Report No. ER 98 ECS 741, 22 April 1999. Residues at Harvest in Mandarins. European Union, Southern Zone 1998.

Leah, J.M. and Reynolds, C.M.M., 21 May 1996. Distribution, elimination and the nature of the metabolite residues in the milk and the edible tissues of a lactating cow. AgrEvo U.K. Limited, Study Numbers TOX/94308 and TOX/94308A.

Maier – Bode, H. Properties, Effect, Residues and Analytics of the Insecticide Endosulfan. *Residues Reviews* (1967), 1-45.

Maier – Bode, H. Investigations on the Persistence of the Insecticide Endosulfan in the Vegetable and Animal Organism. Pharmakologisches Institut der Rheinischen Friedrich Wilhelms-Universitat, Bonn. Document A4 047. July 1966

Needham, D., Creedy, C.L. and Hemmings, P.A., 9 April 1998. *Toxicokinetics in the rat following repeated daily oral administration of 1 mg/kg bodyweight for up to 28 days*. AgrEvo U.K. Limited, Study Number TOX 97099.

Peatman, M.H., Reynolds, C.M., Bright, J.H.M. and Godfrey, T.L. AgrEvo U.K. Report No. RESID/99/8, Study ID 205/05/001, 26 May 1999. Residues of  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate in milk and edible cattle tissues following 28 days feeding to lactating cows Endosulfan technical product.

Report No. 1/10/532, Protocol No. ECR532, October 2001. Trials to Determine the Level of Endosulfan in Macadamia Nuts at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0105078, 0105224 & 0105227. Determination of Endosulfan Residues in Macadamias.

Report No. 1/10/535, Protocol No. ECR535, October 2001. Trials to Determine the Level of Endosulfan in Celery at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009079, 0009109 & 0105229. Determination of Endosulfan Residues in Celery.

Report No. 1/10/537, Protocol No. ECR537, October 2001. Trials to Determine the Level of Endosulfan in Mangoes at Harvest Following Two Applications to the Crop. State Chemistry Laboratory Report No. 0104096, 0105234 & 0105235. Determination of Endosulfan Residues in Mangoes.

Report No. 1/10/538, Protocol No. ECR538, October 2001. Trials to Determine the Level of Endosulfan in Beans at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0012103, 0103152 & 0104082. Determination of Endosulfan Residues in Beans.

Report No. 1/10/538, Protocol No. ECR538, October 2001. Trials to Determine the Level of Endosulfan in Beans at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0012103, 0103152 & 0104082. Determination of Endosulfan Residues in Beans.

Report No. 1/10/539, Protocol No. ECR539, October 2001. Trials to Determine the Level of Endosulfan in Pawpaws at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0012105 & 0012106. Determination of Endosulfan Residues in Pawpaw.

Report No. 1/10/540, Protocol No. ECR540, October 2001. Trials to Determine the Level of Endosulfan in Eggplant at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0103153, 0105002, 0105223 & 0109043. Determination of Endosulfan Residues in Eggplant.

Report No. 1/10/542, Protocol No. ECR542, October 2001. Trials to Determine the Level of Endosulfan in Mandarins at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0103082, 0105080 & 0105243. Determination of Endosulfan Residues in Mandarins.

Report No. 1/10/543, Protocol No. ECR543, October 2001. Trials to Determine the Level of Endosulfan in Lemons at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0102253, 0103086 & 0105244. Determination of Endosulfan Residues in Lemons.

Report No. 1/10/544, Protocol No. ECR544, October 2001. Trials to Determine the Level of Endosulfan in Oranges at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0102252 & 0103083. Determination of Endosulfan Residues in Oranges.

Report No. 1/10/545, Protocol No. ECR545, October 2001. Trials to Determine the Level of Endosulfan in Persimmons at Harvest Following Two Applications to the Crop. State Chemistry Laboratory Report No. 0105225, 0105226 & 0105281. Determination of Endosulfan Residues in Persimmon.

Report No. 1/10/546, Protocol No. ECR546, October 2001. Trials to Determine the Level of Endosulfan in Silverbeet at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009126, 0009231, 0010030 & 0107095. Determination of Endosulfan Residues in Silverbeet.

Report No. 1/10/547, Protocol No. ECR547, October 2001. Trials to Determine the Level of Endosulfan in Cucumbers at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0105222 & 0105236. Determination of Endosulfan Residues in Cucumbers.

Report No. 1/10/549, Protocol No. ECR549, October 2001. Trials to Determine the Level of Endosulfan in Chinese Cabbage at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009125, 0009230, 0009237, 0010029 & 0104095. Determination of Endosulfan Residues in Chinese Cabbage.

Report No. 1/10/551, Protocol No. ECR551, October 2001. Trials to Determine the Level of Endosulfan in Rockmelons at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0103148 & 0107094. Determination of Endosulfan Residues in Rockmelons.

Report No. 1/10/552, Protocol No. ECR552, October 2001. Trials to Determine the Level of Endosulfan in Tomatoes at Harvest Following Three Applications to the Crop. State Chemistry

Laboratory Report No. 0012107, 0103146, 0103147 & 0105232. Determination of Endosulfan Residues in Tomatoes.

Report No. 1/10/554, Protocol No. ECR554, October 2001. Trials to Determine the Level of Endosulfan in Avocado at Harvest Following Six Applications to the Crop. State Chemistry Laboratory Report No. 0103228, 0104094 & 0107097. Determination of Endosulfan Residues in Avocadoes.

Report No. 1/10/555, Protocol No. ECR555, October 2001. Trials to Determine the Level of Endosulfan in Leafy Lettuce at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009019, 0102207, 0105043 & 0105228. Determination of Endosulfan Residues in Leafy Lettuce.

Report No. 1/10/556, Protocol No. ECR556, October 2001. Trials to Determine the Level of Endosulfan in Zucchini at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0012108, 0012109, 0102271 & 0105001. Determination of Endosulfan Residues in Zucchini.

Report No. 1/10/558, Protocol No. ECR558, October 2001. Trials to Determine the Level of Endosulfan in Rhubarb at Harvest Following Two Applications to the Crop. State Chemistry Laboratory Report No. 0105230 & 0107096. Determination of Endosulfan Residues in Rhubarb.

Report No. 1/10/559, Protocol No. ECR559, October 2001. Trials to Determine the Level of Endosulfan in Capsicums at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0012104, 0103081, 0103149 & 0103229. Determination of Endosulfan Residues in Capsicums.

Report No. 1/10/560, Protocol No. ECR560, October 2001. Trials to Determine the Level of Endosulfan in Sweet Corn at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0102084, 0104037 & 0105231. Determination of Endosulfan Residues in Sweet Corn.

Report No. 1/2/500, Protocol No. ECR500, February 2001. Trials to Determine the Level of Endosulfan on Custard Apples. Lee, R. Resource Sciences Laboratory, Indooroopilly, November 2000. Supervised Residue Trial For Endosulfan On Custard Apples.

Report No. 1/5/550, Protocol No. ECR550, May 2001. Trials to Determine the Level of Endosulfan in Cauliflower at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0101001 & 0011009. Determination of Endosulfan Residues in Cauliflower.

Report No. 1/5/564, Protocol No. ECR564, May 2001. Trials to Determine the Level of Endosulfan in Cabbage at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0011010 & 0012094. Determination of Endosulfan Residues in Cabbage.

Report No. 1/5/594, Protocol No. ECR594, May 2001. Trials to Determine the Level of Endosulfan in Broccoli at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009232, 0009239, 0010031 & 0012135. Determination of Endosulfan Residues in Cauliflower.

Report No. 1/7/562, Protocol No. ECR562, July 2001. Trials to Determine the Level of Endosulfan in Potatoes at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0011192, 0012096, 0101002, & 0103088. Determination of Endosulfan Residues in Potatoes.

Report No. 1/8/533, Protocol No. ECR533, August 2001. Trials to Determine the Level of Endosulfan in Sweet Potatoes at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0104097. Determination of Endosulfan Residues in Sweet Potatoes.

Report No. 1/8/534, Protocol No. ECR534, August 2001. Trials to Determine the Level of Endosulfan in Beetroot at Harvest Following Four Applications to the Crop. State Chemistry Laboratory Report No. 0012095. Determination of Endosulfan Residues in Beetroot.

Report No. 1/8/565, Protocol No. ECR565, August 2001. Trials to Determine the Level of Endosulfan in Carrots at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0009020, 0101003 & 0103087. Determination of Endosulfan Residues in Carrots.

Report No. 1/9/536, Protocol No. ECR536, October 2001. Trials to Determine the Level of Endosulfan in Nectarines at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0103085. Determination of Endosulfan Residues in Nectarines.

Report No. 1/9/541, Protocol No. ECR541, October 2001. Trials to Determine the Level of Endosulfan in Peaches at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0103151 & 0105233. Determination of Endosulfan Residues in Peaches.

Report No. 1/9/548, Protocol No. ECR548, October 2001. Trials to Determine the Level of Endosulfan in Pears at Harvest Following Six Applications to the Crop. State Chemistry Laboratory Report No. 0103089 & 0103150. Determination of Endosulfan Residues in Pears.

Report No. 1/9/553, Protocol No. ECR553, September 2001. Trials to Determine the Level of Endosulfan in Brussels sprouts at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0008260, 0008275, 0009078 & 0103090. Determination of Endosulfan Residues in Brussels sprouts.

Report No. 1/9/561, Protocol No. ECR561, October 2001. Trials to Determine the Level of Endosulfan in Apricots at Harvest Following Three Applications to the Crop. State Chemistry Laboratory Report No. 0103084. Determination of Endosulfan Residues in Apricots.

Report No. 1/9/563, Protocol No. ECR563, October 2001. Trials to Determine the Level of Endosulfan in Apples at Harvest Following Six Applications to the Crop. State Chemistry Laboratory Report No. 0103227, 0105044 & 0107122. Determination of Endosulfan Residues in Apples.

Reynolds, C.M.M., 26 March 1996. Distribution, elimination and the nature of the metabolite residues in the eggs and edible tissues of the laying hen. AgrEvo U.K. Limited, Study Number TOX/94306.

State Chemistry Laboratory Report No. 0111227, 28 November 2001. Determination of Endosulfan Residue Stability in Lemons, Leafy Lettuce and Beetroot.

Watson, J.C. 9 May 2000. Determination Of Total Endosulfan Residues In Lychee Fruit Following Four Applications Of Endosulfan 350 EC Applied At Rates Of 52.5 g ai/100L and 105 g ai/100L. Mareeba Queensland 1999.

Winkler, D.A. Bayer CropScience Report No. BJ96R006, Laboratory Project ID 96-0046, 22 June 1998. Freezer Storage Stability of Endosulfan (alpha, beta and Sulfate) on Animal Tissue and Dairy Matrices.

RESIDUES APPENDIX 1

#### **NOTICE**

# Extended Suspension of Registration and Label Approvals of products containing endosulfan

The NRA has extended the period that the registrations and associated label approvals of all endosulfan products are suspended. The affected registrations and label approvals are:

<b>Product Number</b>	Name of Product	Label Number(s)
	Nufarm Endosulfan 350 EC Insecticide	32799/0899
32799	[Nufarm Australia Limited]	32799/0400
		32799/1000
		32799/0301
		32799/0801
45570	Thionex 350 EC Insecticide Spray	45570/0299
	[Makhteshim-Agan (Australia) Pty Limited]	45570/1099
45838	Endosan Emulsifiable Concentrate Insecticide	45838/0899
	[Crop Care Australasia Pty Ltd]	45838/0300
		45838/0800
50004	Thiodan EC Insecticide	50004/0899
	[Bayer Cropscience Pty Ltd]	50004/1099
		50004/0702
52163	Farmoz Endosulfan 350 EC Insecticide	52163/0899
	[Farmoz Pty Limited]	

The suspensions are now effective from 24 September 2002 until 31 December 2003. Reasons for the suspension were that using the products in accordance with some of the instructions on the labels might be an undue hazard to the safety of people using anything containing its residues and might unduly prejudice trade.

The following instructions are UNCHANGED from those previously issued (Gazette No. NRA 10, 1 October 2002), except by addition of "When using or otherwise handling the product, follow the instructions of the current label except as follows:", which has been added to clarify the APVMA's original intent.

#### INSTRUCTIONS FOR SUPPLY AND USE OF SUSPENDED PRODUCT

These instructions apply to the use of endosulfan products during the period of suspension.

Product may be supplied only if a copy of these instructions is securely affixed to the container.

**READ THESE INSTRUCTIONS** before using or otherwise handling the product.

When using or otherwise handling the product, follow these instructions.

When using or otherwise handling the product, follow the instructions of the current label except as follows:

The current label may include instructions for using this product on pears, Brussels sprouts and leafy vegetables. It also includes a grazing withholding period of 42 days in respect of feeding treated commodities to livestock and a number of other feeding restraints. DO NOT follow those instructions.

In all states and territories the following are new instructions for Brussels sprouts, leafy vegetables, pears and animal feed commodities:

#### **Prohibited Crop Uses**

DO NOT apply to Brussels sprouts.

DO NOT apply to leafy vegetables.

"Leafy vegetables" means: leafy brassicas (excluding broccoli, cauliflower and head cabbage), silver beet, Chinese cabbage, choi sum, all lettuce varieties, cress, Japanese greens (mizuna, Indian mustard), spinach, pak choi and bok choi.

#### **New Withholding Period**

Pear: DO NOT harvest for 28 days after application.

#### **New Feeding Restraints**

DO NOT feed treated apple pomace, citrus pulp and peel; grape marc/pomace to livestock.

DO NOT feed treated pea vines or bean trash to livestock.

DO NOT feed treated cow peas, field peas or pigeon peas to livestock.

DO NOT feed fodder, stubble or trash from treated adzuki beans, chickpeas, cow peas, faba beans, field peas, lupins, mung beans, navy beans and pigeon peas to livestock.

DO NOT feed treated cereal grains (including sorghum and maize) to livestock.

DO NOT feed straw, fodder or trash from treated cereal crops (including sorghum and maize) to livestock.

DO NOT cut for stockfeed or allow livestock to graze treated vetch, lucerne (seed crop), medics (seed crop), clover (seed crop), chou moellier, forage cereals and pastures.

DO NOT feed wrapper leaves of treated Brassica and cole crops (cabbage, cauliflower and broccoli) or treated sweet corn trash to livestock.

DO NOT feed treated sunflower seed, safflower seed or linseed to livestock.

DO NOT feed fodder, stubble or trash from treated canola (rapeseed), linseed, safflower, soya beans, sunflower, peanuts to livestock.

DO NOT feed treated cotton fodder (excludes seed and hulls), stubble or trash to livestock.

Note: these feeding restraints do not apply where bare earth treatment with endosulfan for mite control has been made and no subsequent foliar treatment with endosulfan has been made.

Advisory Statement regarding livestock

If livestock have been fed any of the above commodities that have been treated with endosulfan prior to 25 September 2002, those animals should be kept on untreated feed for 90 days before slaughter.

If any of the above commodities that have been treated with endosulfan have been bailed or used in silage prior to 25 September 2002, they should not be fed to livestock.

#### WARNING

A person must not deal with the suspended products except in accordance with the instructions contained in this notice. A failure to comply with them will result in an offence against the Agvet Codes, about which the NRA will take appropriate action.

For any queries or further information about this matter, please contact:

Graeme Barden Manager, Pesticides Review

Phone: 02 6271 6549 Fax: 02 6272 3218

# RESIDUES APPENDIX 2

Crop	Pest		Withholding period			
		Rate (g ai/ha)	Conc. (g ai/100L)	No.	Timing	period
Avocado	Fruit spotting bug; banana – spotting bug, yellow peach moth		52.5	1 – 2	2 – 3 week intervals	14 days
	Brown loopers, grey loopers, redbanded thrips, red shouldered leaf beetle, swarming leaf beetle	735	70		Apply as required	
	Red shouldered leaf beetle (NSW)		52.5		Repeat when necessary	
Bananas	Fruit spotting bug; banana fruit caterpillar		52.5	1 – 2	2 – 3 week intervals when insects are present	14 days
Beans – green	Heliothis, cutworm, aphids, looper, jassids, green vegetable bug	735			Apply when heliothis are 7 to 10 mm long or as soon as green vegetable bugs are seen	2 days
Beans — soybeans, navy beans, mung beans	Heliothis, green vegetable bug, web spinner caterpillar, soybean moth, cutworms, loopers	735			Apply when heliothis are 7 to 10 mm long or at first sign of pest.	4 weeks
	RLEM, blue oat mite	175 or 350			Apply prior to seedling emergence	
Beetroot	Beetroot webworm, aphids Leafminer	735	66.5		Repeat at 10 – 14 day intervals	2 days
	Heliothis	735	70		Apply as required	
Berry fruit – currants & related fruit	Currant bud mite		66.5	2	Apply at first flower and again 3 weeks later	14 days
Blueberries	Monolepta beetle, caterpillars, plague thrips		52.5			14 days
	Grey cluster bug, Rutherglen bug		70			
Cabbages, Cauliflower & other Cole crops & leaf vegetables	Caterpillars, cabbage white butterfly, looper and riddler caterpillars, aphid (except grey cabbage aphid), Rutherglen bug, green vegetable bug, thrips, heliothis, jassids, cutworm	735	66.5 (or 10.5 g ai/15L knapsack)		Apply at 10 – 14 days or according to pest incidence.	Cole crops 2 days
Canola (oilseed rape)	Heliothis, Rutherglen bug, aphids, cabbage	735			Apply when heliothis are 7 to 10 mm long;	4 weeks

	white butterfly, ashles			ropost amova mar-1	Т
	white butterfly, cabbage moth			repeat sprays may be	
	Pink cutworm	350		necessary	
	RLEM, blue oat mite	175 or		Spray prior to	
	RELIVI, blue oat linte	350		seedling emergence	
Capsicums, okra, cape gooseberry	Tomato grub, thrips, aphids, Rutherglen bug, jassids, cutworms,	735	66.5	Apply every 10 – 14 days or according to pest pressure	7 days
	tomato russet mite, white flies, loopers, green vegetable bugs				
Carrots	Aphids, leafhoppers		66.5	Repeat as required	7 days
Cashews	Fruit spotting bug		70	Apply as required during December/January	14 days
Celery	Caterpillars, Aphids, Leafhoppers, Thrips		66.5	Apply every 10 – 14 days or according to pest pressure	2 days
Cereals	Armyworms	525			Apply at first sign of
	Heliothis	735			pest
	Pasture cockchafer	350 – 735			
Chickpeas,	Loopers, aphids,	735		Apply when heliothis	4 weeks
cowpeas,	cutworms, heliothis,			are 7 to 10 mm long	
pigeon peas, adzuki beans, faba beans	green vegetable bug			or as soon as green vegetable bugs are seen; apply at first sign of cutworm or	
	RLEM, blue oat mite	175 – 350		looper	
Chou moellier	Aphids, cutworm	735		Apply as soon as infestation occurs	4 weeks
	Pink cutworm	350		Apply early when larvae are small	
Citrus	Spined citrus bug		19.95		14 days
	Banana fruit caterpillar, citrus katydid, citrus planthopper, leafhoppers		70	Apply as required	
	Heliothis	735	70	Apply once only in spring	
	Bronze orange bug	350	19.95	Apply once only in spring	
Clover and medic seed crops	Heliothis	735		Apply early to small larvae	4 weeks
	Pink cutworm	350			
	RLEM and blue oat mite	175 – 350		Spray prior to seedling emergence	
Cotton	Heliothis, rough bollworm, aphids, thrips, cotton looper, jassids, green vegetable bug, cutworms, cotton tip worm, webspinner	735		Repeat sprays at 5 to 10 days depending on infestation level, rate of growth of cotton and insect checks	4 weeks

	caterpillar					
Cucurbits	Aphids, thrips, jassids, green vegetable bug		66.5		Apply every 10 to 14 days or as required. Do not use in greenhouses, glass houses, tunnels etc.	2 days
	Cucumber moth, heliothis		70			
	Cucurbit shield bug, fruitspotting bug, Rutherglen bug	735			Apply as required. or as required. Do not use in confined spaces e.g. greenhouses, glass houses, tunnels etc.	
Custard apples	Fruitspotting bug, banana-spotting bug, yellow peach moth,		52.5	1 – 2	Apply 1 or 2 sprays at 2 – 3 week intervals	14 days
	Blue triangle butterfly, loopers		52.5			
	Blue triangle butterfly		70	1	Spray once in late summer/autumn	
Eggplant	Heliothis, thrips, aphids, Rutherglen bug, jassids, cutworms, tomato russet mite, white flies,	735	66.5		Apply every 10 14 days or according to pest pressure.	7 days
	loopers, green vegetable bug, egg fruit caterpillar, yellow peach moth, potato moth				Do not use in confined spaces e.g. green houses, glass houses, tunnels etc.	
Grapevines	Thrips		66.5		Apply pre-blossom or as required	14 days
Guavas, persimmons	Fruitspotting bugs, caterpillars, loopers		52.5		Apply cover sprays in the spring and autumn. A follow up spray may be necessary 3 weeks after the initial spray	14 days
	Fruitspotting bugs (Qld only)		70		Apply at monthly intervals from time of fruit set or more frequently in orchards under constant pest pressure	
Kiwifruit	Caterpillars, Rutherglen bug, fruitspotting bugs		52.5		Apply every 10 to 14 days when pests are present	14 days
	Fruitspotting bugs (Qld),		70		Apply every 2 to 4 weeks during fruiting period depending on orchard infestation	
Linseed	Passion vine hopper Heliothis, green	735			Apply as required Apply at first sign of	4 weeks
	vegetable bug, thrips RLEM, blue oat mite	175 – 350			pest Apply prior to seedling emergence	

Longons	Duthardan bugs theins		70		Apply as required	14 days
Longans	Rutherglen bugs, thrips		70		Apply as required, repeat applications	14 days
	Emitenation bear		70		may be necessary	
	Fruitspotting bugs		70		Apply every 2 to 4	
					weeks during fruiting	
					period, depending on	
					infestation	
Loquats	Fruitspotting bugs		70		Apply every 2 to 4	14 days
					weeks during fruiting	
					period, depending on	
					infestation	
Lucerne	Heliothis, green	735			Apply as soon as pest	4 weeks & 42
	vegetable bug, thrips,				infestation appears	days WFS
	web-spinner caterpillar					
	Pink cutworm	350				
	RLEM, blue oat mite	175 –			Apply prior to	
	,	350			seedling emergence	
Lucerne seed	Lucerne seed wasp	735			Apply only when	4 weeks & 42
crops	Eucerne seed wasp	733			pests are present	days WFS
Lupins	Heliothis, aphids, green	735			Apply at firsts sign of	4 weeks
Lupins	vegetable bug	133			pest	4 WCCKS
	RLEM, Blue oat mite	175			Apply prior to	
	RLEM, Blue oat fille	175 –				
		350			seedling emergence	44.1
Lychees	Flower eating caterpillar,		52.5	2	Apply spray when	14 days
					trees are flowering. A	
					repeat spray may be	
					necessary	
	Lychee stink bug,		52.5	2	Apply when bug	
	fruitspotting bugs				activity is seen on	
					trees. A repeat spray	
					may be necessary	
					one week later	
	Fruitspotting bugs		70		Apply from October	
					to harvest whenever	
					bug damage fruit is	
					found	
Macadamias	Fruitspotting bugs		52.5	2	Apply at 3 weekly	14 days
Macadamas	Transporting dags		32.3	-	intervals when	1 i days
					premature nut fall is	
					evident	
	Banana-spotting bug		52.5	2-3	Apply at 2 weeks	
	Banana-spotting bug		32.3	2-3	intervals after	
	El	1	52.5	2 2	flowering	
	Flower caterpillar		52.5	2 - 3	Apply in the	
	m : :::		50.5		flowering period	
	Twig girdler, aphids,		52.5		Apply as required	
	macadamia lace bug					
	Green vegetable bug		52.5		Apply when heavy	
					infestations occur	
	Redshouldered leaf		52.5		Apply as required	
	beetle (monolepta					
	occiie (monorepia		1	1		l
	beetle)					
			70		Apply as required	
	beetle) Black citrus aphids	525	70 52.5			
Maize	beetle)	525 735			Apply as required Apply as required Apply at tasselling	4 weeks

		1	T	1	Τ	_
					emergence when	
					larvae are small.	
					Repeat sprays may	
					be necessary. For	
					aphids apply before	
					build up.	
Mammey	Redbanded thrips		70		Apply as required.	14 days
					Repeat applications	
					may be necessary.	
Mangoes	Flatid planthoppers		70		Apply as required to	14 days
	(including mango				flowers, foliage and	
	planthopper), flower-				fruit during the	
	eating caterpillars,				flowering to early	
	fruitspotting bugs, large				fruiting season	
	mango tip-borer, small					
	mango tip-borer					
	Redshouldered leaf				Apply as a high	
	beetle (monolepta				volume cover spray	
	beetle), banana-spotting				when bugs are	
	bug, redbanded thrips				detected	
Onions	Thrips		66.5		Apply as required	7 days
					when pests are	
					present	
Passionfruit	Passionvine bug,		52.5		Apply as required	14 days
	Rutherglen bug				when pests are	
					present	
	Green vegetable bug, fruitspotting bug		70			
Pasture (new	RLEM, blue oat mite	175 –			Spray prior to	4 weeks & 42
sowings –		350			seedling emergence	days WFS
direct drill or						
bare earth)						
Pawpaw	Fruitspotting bugs,		52.5	1 - 2	Apply at 2 to 3 week	14 days
	banana-spotting bug,				intervals when	
	yellow peach moth				infestation is present	
Peas- field &	Looper, aphids,	735				Green peas 2
green	cutworms, green					days
	vegetable bug, heliothis					Field peas 4 wks
	Pea weevil	350			Apply from start of	
					flowering onwards. If	
					any pea weevil are	
					present spray before	
					first pods form.	
					Check after spraying	
					and respray if	
					necessary.	
	RLEM, blue oat mite	175 – 350			Spray prior to seedling emergence	
Peanuts	Heliothis, jassids, green vegetable bug	735			5 5	7 days
Pecans	Heliothis, green	980				14 days
	vegetable bug		50.5		A mm1xx a	
	Christmas beetles,		52.5		Apply cover sprays	
	monolepta beetles,				once damage is	
	fruitspotting bugs,				detected in spring	
	yellow peach moth				and summer. It may	
					be necessary to apply	

					a follow-up spray	
					one week after first.	
Pistachios	Aphids, caterpillars		70		Apply as required	14 days
Pome fruit	Woolly aphids (apples only), green & black peach aphids, thrips, Rutherglen bug, heliothis, cherry aphid, dimpling bugs, Redshouldered leaf		66.5 52.5		Apply as a full cover spray as required.	
	beetle		32.3			
Pomegranate	Yellow peach moth		70	1-2	Apply once when damage is evident on small fruit and again 21 days later if necessary.	14 days
Potatoes	Thrips, aphids, leaf miner, jassids, potato moth, Rutherglen bug, green vegetable bug	735			Apply every 10 – 14 days or according to pest incidence.	7 days
Rambutans	Fruitspotting bugs		70		Apply from October to harvest whenever bug damaged fruit is found	14 days
Raspberries	Grey cluster bug, Rutherglen bug		70		Apply when swarms infest terminal flowers and fruit. Reapply if re-infestation occurs.	14 days
Red Beet	Red beet worm, Aphids, Leaf Miner	735			Apply thoroughly every 10 – 14 days	Not stated
Safflower	Heliothis, aphids, Rutherglen bug, grey cluster bug	735				4 weeks
	RLEM, blue oat mite	175 – 350			Apply prior to seedling emergence	
Sapodillas	Yellow peach moth	330	70		Apply and repeat as required.	14 days
Shallots	Army worms, heliothis, loopers	735	70		Apply as required	7 days
	Cutworms		70		Apply to plant bases/butts and/or soil surface at first sign or pests.	
	Rutherglen bug	735			Apply as required	
Silverbeet	Beet webworm	735	66.5		Spray to thoroughly wet leaves	2 days
Spinach	Aphids		66.5		Apply thoroughly at intervals of 10–14 dy	7 days
Sorghum	Heliothis, aphids, sorghum midge, sorghum head caterpillar, yellow peach moth	735			Apply at first sign of pest, generally from head emergence onward. Repeat sprays as necessary.	4 weeks
Soybeans	Grass blue butterfly, soybean moth	735			Apply at first sign of pest. Repeat sprays	4 weeks

4 days
4 days
4 days
weeks
days
days
4 1
4 days
days
uays
days
uays
weeks
da da

#### RESIDUES APPENDIX 3

# **Published Residues Data for Leafy Vegetables**

Crop	Trial site, Year	Application		Residues (mg/kg)		Comments	
(Variety)		Rate (g ai/ha)	No.	0 days	7 days		
Spinach	USA, 1961	1120	1	21 ①	4.7 ①	Maier-Bode (1968)	
Chard	Washington, USA	1120	2	57 ①	6.8 ①		
Collards	Washington, USA	840	4	39 ①	10 ①		
Lettuce	Washington, USA	1120	2	69 ①	8 ①		
	California, USA, 1958	1120	1	59 ①	9.5 ①		
	California, USA, 1966	1120	1	3	1.5		
	California, USA, 1966	1120	1	11	2.3		
Water cress	Hawaii, USA, 1961	1120	2	9.2 ①			
	Hawaii, USA, 1960	560	1	16 ①	0.2 ①		
Chard (Paros)	Germany, 1984	210	2	3.5	0.71	JMPR 1989	
(Paros)		210	2	5.44	0.38		
(Glutter Silber)		210	2	3.26	0.51		
(Luculles)		210	2	5.46	0.65		
(Luculles)		210	2	4.83	0.30		
(Grüner Schnitt)		210	2	5.03, 6.36	0.99, 1.74	0 day mean = 4.84 mg/kg	
Spinach (Atlanta)	Germany, 1984	210	2	8.97	1.5	JMPR 1989	
(Atlanta)		210	2	5.1 (4.7 – 5.49)	1.99 (1.74 – 2.24)		
(Atlanta)		210	2	3.71	0.72		
(Matador)		210	2	9.49	1.2		
(Matador)		210	2	9.95	1.72		
(Mazurka)		210	2	1.04	0.33		
(Mazurka)		210	2	2.57	0.43		
(Paloma)		210	2	2.69	0.95	0 day mean = $5.44$ mg/kg	
Leaf lettuce (Gelber Krauser)	Germany, 1984	210	2	4.23	0.24	JMPR 1989	
(Gelber Krauser)		210	2	6.87	0.57		
(Gelber Krauser)		210	2	10.28	1.04		
(Gelber hohlbluttringer		210	2	4.46	0.7		
Butter)		210	2				
		210	2	6.25	1.50		
(Lolo rosa)		210	2	7.56	0.47		
(Hilds gleber ruinder)		210	2	0.24	0.32	0  day mean = 5.7 mg/kg	
Head lettuce (Soraja)	Germany, 1984	210	2	5.21	0.55	JMPR 1989	
(Soraja)		210	2	6.11	2.43		
(Soraja)		210	2	3.66	2.36		
(Soraja)		210	2	6.26	1.17		

# Australian Pesticides and Veterinary Medicines Authority (APVMA)

(Hilds Neckarnisesen)		210	2	3.71	0.99	
(Hilds Neckarnisesen)		210	2	3.59	0.36	
(Attraktion)		210	2	1.38	0.42	
(Suzan)		210	2	11.62	1.52	
(Reskia)		210	2	4.66	0.71	
(Capitan)		210	2	5.07	0.70	0 day mean = $5.12$
						mg/kg
Heal lettuce	Germany 1984					JMPR 1989
(crisphead)	,					
(Great Lakes)		210	2	3.34	0.28	
(Great Lakes)		210	2	1.69	0.23	
(Great Lakes)		210	2	1.52	0.23	
(Great Lakes)		210	2	3.52	0.48	
(Great Lakes)		210	2	3.86	0.39	
(Minetto)		210	2	2.56	0.29	
(Minetto)		210	1	5.84	0.18	
(Minetto)		210	2	5.36	0.36	
(Astral)		210	2	2.19	0.43	
(Calmar)		210	2	1.29	0.33	0  day mean = 3.09
(Curring)			_	1.29	0.00	mg/kg
Endive (Bubikoff)	Germany, 1984	210	2	1.61	0.37	JMPR 1989
(Frisee sanda)	String, 170.	210	2	4.93	0.99	01/11/17/07
(Hilds diva)		210	2	1.91	0.20	
(Hilds diva)		210	2	2.26	0.77	
(Elna)		210	2	2.85	0.76	
(Malan)		210	2	1.21	0.45	
(Duka)		210	2	4.05	0.23	
(Kasseler		210	2	4.88	0.62	
Strunckchen)		210		1.00	0.02	
(Escariol gruner)		210	2	3.92	1.28	0 day mean = 3.06
(Escurior gruner)		210	~	3.72	1.20	mg/kg
Cos lettuce (Romea)	Germnay, 1984	210	2	1.98	0.48	JMPR 1989
(Valmaine)	Germinay, 1704	210	2	2.37	0.46	JIII K 1707
(Romea)		210	2	1.27	0.39	
(Cartan)		210	2	1.94	0.39	
(Kasseler)		210	2	3.58	0.46	
(Kasseler)		210	2	3.06	0.59	
(Gelbe kassesler)		210	$\frac{2}{2}$	1.59	0.57	0  day mean = 2.25
(Gelue Rassesiei)		210		1.37	0.57	mg/kg
1	l	1		1	1	mg/kg

RESIDUES APPENDIX 4

### Endosulfan

National Estimated Short-Term Intake (2 + years)
Acute RfD: 0.02 mg/kg bw
Commodity

Code	Name	MRL mg/kg	STMR or STMR-P, mg/kg	Process factor	HR or HR-P, mg/kg	Large portion, g/kg bw	Body weight, kg	Large portion, kg	Unit weight, g	% edible portion	Unit weight, edible portion, kg	Variability factor	Case	NESTI, mg/kg bw/day	% acute RfD
FC 0004	Orange				0.078	22.543	67	1.510	205	76	0.156	3	Case 2a	0.0021	10.6
FC 0003	Mandarin				0.11	5.188	67	0.348	86	76	0.065	3	Case 2a	0.0008	3.9
FC 0002	Lemon				0.17	2.234	67	0.150	165	66	0.109	3	Case 2a	0.0009	4.7
FP 0226	Apple				0.11	6.328	67	0.424	111	92	0.102	3	Case 2a	0.0010	5.2
FP 0230	Pear				0.21	6.149	67	0.412	235	90	0.212	3	Case 2a	0.0026	13.1
FS 0247	Peach				0.3	8.029	67	0.538	153	96	0.147	3	Case 2a	0.0037	18.620
FS 0245	Nectarine				0.18	9.015	67	0.604	126	96	0.121	3	Case 2a	0.0023	11.363
FS 0240	Apricot				0.26	8.442	67	0.566	58	94	0.055	3	Case 2a	0.0026	13.090
FI 0326	Avocado				0.065	3.299	67	0.221	269	70	0.188	3	Case 2a	0.0006	2.9
FI 0345	Mango				0.2	12.667	67	0.849	361	63	0.227	3	Case 2a	0.0039	19.5
FO 0350	Pawpaw				0.18	8.388	67	0.562	752	70	0.526	3	Case 2a	0.0043	21.7
FO 0352	Persimmon				0.89	10.03	67	0.672	112	69	0.077	3	Case 2a	0.0110	54.9
FI 0343	Lychee				1.62	8.388	67	0.562	20	69	0.014	3	Case 2a	0.0143	71.3
FI 0332	Custard apple				0.34	9.761	67	0.654	500	72	0.360	3	Case 2a	0.0070	34.9
VC 0046	Melon				0.15	7.773	67	0.521	1584	100	1.584	3	Case 2b	0.0035	17.5

VC 0424	Cucumbers	0	.12	2.232	67	0.150	267	97	0.259	3	Case 2b	0.0008	4.0
VC 0431	Zucchini	0	.09	1.507	67	0.101	98	93	0.091	3	Case 2a	0.0004	1.9
VO 0051	Capsicum		0.4	1.364	67	0.091	249	71	0.177	3	Case 2b	0.0016	8.2
VO 0448	Tomato	0.	094	3.739	67	0.251	130	99	0.129	3	Case 2a	0.0007	3.6
VO 0440	Eggplant	0.	055	7.266	67	0.487	378	84	0.318	3	Case 2a	0.0009	4.6
VB 0400	Broccoli	0	.29	2.406	67	0.161	318	56	0.178	3	Case 2b	0.0021	10.5
VB 0041	Cabbage	0.	098	3.541	67	0.237	950	84	0.798	3	Case 2b	0.0010	5.2
VB 0404	Cauliflower	C	0.1	2.627	67	0.176	575	100	0.575	3	Case 2b	8000.0	3.9
VR 0574	Beetroot	(	0.2	1.478	67	0.099	315	43	0.135	3	Case 2b	0.0009	4.4
VR 0577	Carrot	0.	095	2.284	67	0.153	95	85	0.081	3	Case 2a	0.0004	2.2
VR 0589	Potato	0	.02	7.164	67	0.480	150	82	0.123	3	Case 2a	0.0002	1.1
VR 0508	Sweet potato	0	.02	3.438	67	0.230	600	91	0.546	3	Case 2b	0.0002	1.0
VS 0624	Celery	0	.59	1.422	67	0.095	1920	79	1.517	3	Case 2b	0.0025	12.6
VS 0627	Rhubarb	0	.34	8.672	67	0.581	95	61	0.058	3	Case 2a	0.0035	17.7
	Macadamia												
TN 0669	nuts	0	.01	1.381	67	0.093	2	100	0.002	3	Case 2a	0.0000	0.1
GC 0080	Cereals	C	0.1	4.854	67	0.325					Case 1	0.0005	2.4
SO 0088	Oilseeds		1	0.786	67	0.053					Case 1	0.0008	3.9
VD 0070	Pulses	C	0.1	2.43	67	0.163					Case 1	0.0002	1.2
	Meat												
MM 0095	(mammalian)[in the fat]			7.782									4.8
IVIIVI UUJU	Fat = meat×0.2	1	1.7	0.3113	67	0.021					Case 1	0.0005	2.6
	Muscle =	'	,	0.0110	01	0.021					Oase I	0.0000	2.0
	meat×0.8	0	.07	6.22	67	0.417					Case 1	0.0004	2.2
MO 0105	Edible offal (mammalian)	0	.98	3.104	67	0.208					Case 1	0.0030	15.2
ML 0106	` ,	0.06	.55	29.654	67	1.987					Case 3	0.0030	8.9
VII 0 100	TVIII (O	0.00	I	20.00∓	0,	1.507	I			l	Just	0.0010	0.5

PM 0110	Poultry meat [in the fat]		6.436						1.6
	Fat = $meat \times 0.1$	0.05	0.6436	67	0.043		Case 1	0.0000	0.2
	Muscle = meat×0.9 Poultry, edible	0.05	5.792	67	0.388		Case 1	0.0003	1.4
PO 0111	offal of (1)	0.01	3.299	67	0.221		Case 1	0.0000	0.2
PE 0112	Eggs	0.02	1.703	67	0.114		Case 1	0.0000	0.2

# Endosulfan National Estimated Short-Term Intake (2 to 6 years) Acute RfD 0.02 mg/kg bw Commodity

Code	Name	MRL mg/kg	STMR or STMR-P, mg/kg	Process factor	HR or HR-P, mg/kg	Large portion, g/kg bw	Body weight, kg	Large portion, kg	Unit weight, g	% edible portion	Unit weight, edible portion, kg	Variability factor	Case	NESTI, mg/kg bw/day	% acute RfD
FC 0004	Orange				0.078	56.874	19	1.081	205	76	0.156	3	Case 2a	0.0057	28.6
FC 0003	Mandarin				0.11	18.716	19	0.356	86	76	0.065	3	Case 2a	0.0028	14.1
FC 0002	Lemon				0.17	14.259	19	0.271	165	66	0.109	3	Case 2a	0.0044	21.9
FP 0226	Apple				0.11	17.474	19	0.332	111	92	0.102	3	Case 2a	0.0031	15.5
FP 0230	Pear				0.21	24.468	19	0.465	235	90	0.212	3	Case 2a	0.0098	49.1
FS 0247	Peach				0.3	8.029	19	0.153	153	96	0.147	3	Case 2a	0.0070	35.235
FS 0245	Nectarine				0.18	9.015	19	0.171	126	96	0.121	3	Case 2a	0.0039	19.573
FS 0240	Apricot				0.26	8.442	19	0.160	58	94	0.055	3	Case 2a	0.0037	18.435

FI 0326	Avocado	0.065	4.184	19	0.079	269	70	0.188	3	Case 2b	0.0008	4.1
FI 0345	Mango	0.2	10.8947	19	0.207	361	63	0.227	3	Case 2b	0.0065	32.7
FI 0350	Pawpaw	0.18	1.806	19	0.034	752	70	0.526	3	Case 2b	0.0010	4.9
FI 0352	Persimmon	0.89	1.806	19	0.034	112	69	0.077	3	Case 2b	0.0048	24.1
FI 0343	Lychee	1.62	1.806	19	0.034	20	69	0.014	3	Case 2a	0.0053	26.4
FI 0332	Custard apple	0.34	1.806	19	0.034	500	72	0.360	3	Case 2b	0.0018	9.2
/C 0046	Melon	0.15	21.737	19	0.413	1584	100	1.584	3	Case 2b	0.0098	48.9
/C 0424	Cucumber	0.12	6.82	19	0.130	267	97	0.259	3	Case 2b	0.0025	12.3
VC 0431	Zucchini	0.09	6.82	19	0.130	98	93	0.091	3	Case 2a	0.0015	7.4
/O 0051	Capsicum	0.4	3.158	19	0.060	249	71	0.177	3	Case 2b	0.0038	18.9
/O 0448	Tomato	0.094	8.789	19	0.167	130	99	0.129	3	Case 2a	0.0021	10.5
/O 0440	Eggplant	0.055	3.158	19	0.060	378	84	0.318	3	Case 2b	0.0005	2.6
/B 0400	Broccoli	0.29	8.798	19	0.167	318	56	0.178	3	Case 2b	0.0077	38.3
/B 0041	Cabbage	0.098	4.453	19	0.085	950	84	0.798	3	Case 2b	0.0013	6.5
/B 0404	Cauliflower	0.1	6.497	19	0.123	575	100	0.575	3	Case 2b	0.0019	9.7
<b>.</b>	_								_			
VR 0574	Beetroot	0.2	3.789	19	0.072	315	43	0.135	3	Case 2b	0.0023	11.4
/R 0577	Carrot	0.095	11.336	19	0.215	95	85	0.081	3	Case 2a	0.0019	9.4
/R 0589	Potato	0.02	16.316	19	0.310	150	82	0.123	3	Case 2a	0.0006	2.9
/R 0508	Sweet potato	0.02	2.776	19	0.053	600	91	0.546	3	Case 2b	0.0002	0.8
/S 0624	Celery	0.59	5.053	19	0.096	1920	79	1.517	3	Case 2b	0.0089	44.7
/S 0627	Rhubarb	0.34	4.426	19	0.084	95	61	0.058	3	Case 2a	0.0036	17.9
TNI 0000	Macadamia	0.04	0.750	40	0.044	0	400	0.000		00-	0.0000	0.4
TN 0669	nuts	0.01	0.752	19	0.014	2	100	0.002	3	Case 2a	0.0000	0.1
GC 0080		0.1	2.629	19	0.050					Case 1	0.0003	1.3
800 088		1	1.408	19	0.027					Case 1	0.0014	7.0
√D 0070	Pulses	0.1	8.033	19	0.153					Case 1	0.0008	4.0

	Meat									
MM 0095	(mammalian)[in the fat]			13.715						27.2
IVIIVI 0095	-		4 7		40	0.050			0.0047	
	Fat = meat×0.2 Muscle =		1.7	2.743	19	0.052		Case 1	0.0047	23.3
	meat×0.8		0.07	10.972	19	0.208		Case 1	8000.0	3.8
	Edible offal									
MO 0105	(mammalian)		0.98	0.8666	19	0.016		Case 1	0.0008	4.2
ML 0106	Milks	0.06		76.325	19	1.450		Case 3	0.0046	22.9
PM 0110	Poultry meat			11.776						2.9
	Fat = meat×0.1		0.05	1.1776	19	0.022		Case 1	0.0001	0.3
	Muscle =									
	meat×0.9		0.05	10.598	19	0.201		Case 1	0.0005	2.6
	Poultry, edible									
PO 0111	offal of (1)		0.01	1.474	19	0.028		Case 1	0.0000	0.1
PE 0112	Eggs		0.02	3.949	19	0.075		Case 1	0.0001	0.4